# Determination of tetrafluoroborate in wastewaters by ion chromatography after ion pair liquid-liquid dispersive microextraction

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**ABSTRACT**

The ion chromatographic method was developed to determine tetrafluoroborate ion ($BF_4^-$) in different types of water using ion pair liquid-liquid dispersive microextraction. Tetrafluoroborate was extracted into an organic phase (1,2-dichloroethane) as an ion pair with a tetrabutylammonium cation ($TBA^+$). The most complete formation of $[(TBA^+)(BF_4^-)]$ was observed at ion-pair reagent concentration of at least 5 mmol $L^{-1}$ ($C(BF_4^-) \leq 1$mg $L^{-1}$). Ultrasonic irradiation was used to disperse the extractant. The achieved concentration factor (K) was $29\pm3$, and the degree of extraction (R) was $50\pm5\%$. The limit of detection of tetrafluoroborate using the microextraction technique was $7\times10^{-3}$ mg $L^{-1}$. The method applies to the analysis of different water origins. The presence of the main contained anions does not interfere with the microextraction and chromatographic determination of tetrafluoroborate. The maximum molar ratio of $BF_4^-$ to diverse ions is $1:10^4$ for fluoride, chloride, bromide, nitrate ions, and $1:10^2$ for sulfate and perchlorate ions.

## 1. Introduction

Today, the concept of "green chemistry" remains one of the main trends in analytical chemistry [1-4]. Decreased volumes of toxic reagents, automation, and miniaturization of analytical methods are the critical points of this concept [3]. One of the leading trends in green chemistry is using ionic liquids containing tetrafluoroborate ions [5, 6]. The control of its concentration in various objects is necessary at the stages of the synthesis and use of ionic liquids. Industrial wastewater can also contain various amounts of tetrafluoroborate. It's known that $BF_4^-$ use in the composition of non-aqueous electrolytes for chemical reactions and electrochemical

processes [7], as well as in electroplating to improve the quality of the electroplated layer [8]. Another alternative application area for $BF_4^-$ is agriculture. It is used there as a "green fungicide" [9] and an herbicide [10]. The wide use of $BF_4^-$ means a substantial difference in the composition of the analyzed samples. This necessitates the development of a universal method for analyte determination.

Spectrophotometry [11, 12, 13] and ion chromatography [14, 15] are the most common methods for determining tetrafluoroborate in aqueous solutions. The works [11, 12] are based on the conversion of all boron forms in the sample to the $BF_4^-$, reaction with an organic dye, and photometric determination of the product. However, the reaction stability depends on the temperature, the sample

pH, and the dye concentration. Also, the high consumption of toxic reagents is the disadvantage of this work. In addition, these methods make it possible to determine the concentration of total boron in the sample. The ion pair preconcentration of $BF_4^-$ and spectrophotometric determination are described in [13]. But large amounts of sample (20 ml) and toxic extractant (10 ml) were used in this work.

The determination of $BF_4^-$ in water samples by ion chromatography is often not sensitive enough (0.2 mg $L^{-1}$ - 1.4 mg $L^{-1}$) [14,15]. In this work, a procedure for ion pair liquid-liquid microextraction of $BF_4^-$ is proposed to increase the determination's selectivity and lower the detection limit. This method involves forming a neutral ion pair between the anion of the analyte and the cation of the ion-pair reagent due to electrostatic interaction [16]. It is known that cations of quaternary ammonium bases (QABs) can form ion pairs with inorganic anions from aqueous solutions [17]. The solubility of such compounds in organic solvents is higher than in water [18]. In addition to the lower detection limits, microextraction also makes it possible to reduce the load on the chromatographic system at high concentrations of diverse ions. It is important for the analysis of various samples, including industrial wastewater.
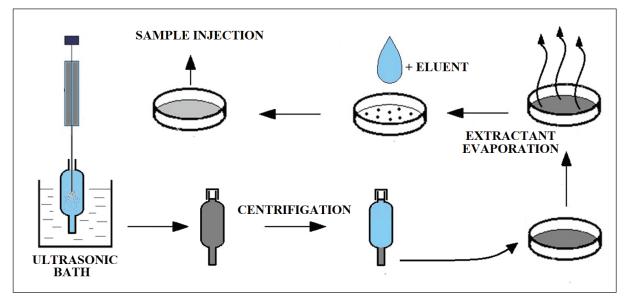
## 2. Materials and methods

### 2.1. Microextraction technique

A specially designed centrifuge tube (4.5 mL volume) was used for microextraction. 3.0 ml of analyzed water was placed in a tube, and a solution of the ion-pair reagent was added. Then the tube was placed in an ultrasonic bath, and 500 μL of the extractant was added. The resulting emulsion was separated by centrifugation. Further, the replacement of the organic matrix with an aqueous one was held. To do this, the extract (400 μL) was transferred in a Teflon vial, and the extractant was evaporated by using an infrared lamp. Then 50 μL of eluent was added to the dry residue. This solution was injected into the chromatograph for the determination of tetrafluoroborate ion ($BF_4^-$) in water samples (Schema 1).

### 2.2. Chromatographic conditions

All experiments were carried out on an LC-20A ion chromatograph (Shimadzu, Japan), which consisted of a Model LP-20ADsp liquid delivery pump and a conductivity detector Model CDD-10Avp. The separating column was OKA (50x4 mm i.d., Vagos, Tallinn, Estonia), and the suppressor column was KU-2x8 (100x4 mm i.d., Ekos-1, Moscow, Russia). Both columns and the conductivity detection cell



**Schema 1.** Microextraction technique for determination of tetrafluoroborate ions ($BF_4^-$) in water and wastewater samples based on ion chromatography

were placed inside the CTO-20AC column oven for temperature control (32 ºC). The eluent was obtained by a mixture of 1.0 mmol $L^{-1}$ $Na_2CO_3$ with 4.0 mmol $L^{-1}$ $NaHCO_3$, and the flow rate was set at 3 ml $min^{-1}$. This eluent has the optimum eluting power for the selective and fast determination of tetrafluoroborate. The volume of the injection loop was 50 μL.

## 2.3. Reagents

Ammonium tetrafluoroborate was used to prepare a standard solution supplied by JSC Vekton, Russia. The solution was kept in a polyethylene container for no more than a month. Sodium carbonate and sodium bicarbonate were used to prepare an eluent solution (JSC Vekton, Russia). All solutions were prepared using deionized water. 1,2-dichloroethane, chloroform, dichloromethane, and carbon tetrachloride were used as extractants (JSC Vekton, Russia). All reagents were analytical grade or better. Ion-pair reagents were tetrabutylammonium hydroxide TBAOH (40% aqueous solution, Sigma-Aldrich, Switzerland, CAS Number: 2052-49-5) and tetraethylammonium hydroxide TEAOH (25% aqueous solution, Acros Organics, India).
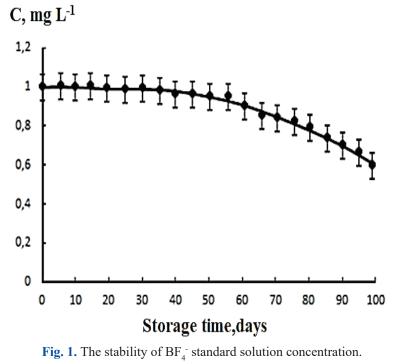
## 2.4. Other Equipment

The extractant was dispersed using a PSB-Gals 1335-05 ultrasonic bath with a radiation generator power of 50 W and a frequency of 35 kHz. SM-6M centrifuge with a rotor speed of 1200 rpm was used for separating the organic and aqueous phases. During replacing the matrix, the extractant was evaporated using a 300 W mirror infrared lamp. The Anion 4100 conductometer was used to control of purity of deionized water. Laboratory scales AUX 320 Shimadzu, micro doses HTL 20-200 μL, and Dragon LAB TopPette Pipette 2-20 μL were also used in work.

## 3. Results and discussions
### 3.1. Stability of tetrafluoroborate ion ($BF_4^-$)
Hydrolysis of $BF_4^-$ ions occurs in aqueous solutions at pH>1. Also, the rate of this process depends on the temperature [19]. About 0.7% of $BF_4^-$ decomposes in alkaline solutions within a day at 20°C [20]. So, we have studied the stability of a standard solution containing 1 mg $L^{-1}$ of $BF_4^-$. The results (Fig. 1) showed that the standard solution is stable for a month at a temperature of $3 \pm 1$ºC in a polyethylene container.



**Fig. 1.** The stability of $BF_4^-$ standard solution concentration.
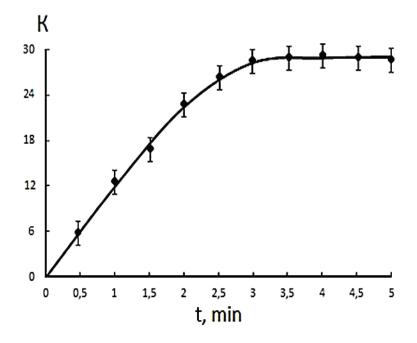Conditions: T = 3±1ºC, polyethylene container

**Fig. 2.** Effect of ultrasonic irradiation time. Extractant: 1,2-dichloroethane;
ion-pair reagent: TBAOH, concentration 5 mM (n=3, P=0.95)

### 3.2. Effect of ultrasonic irradiation time

The maximum concentration factor of $BF_4^-$ is reached at 3 min of ultrasonic irradiation and does not change further (Fig. 2). This dependence is valid for all chosen extractants and ion-pair reagents.

### 3.3. Effect of different extractants and ion pair reagents

Using tetrabutylammonium hydroxide as an ion-pair reagent made it possible to achieve better microextraction parameters (Table 1). Obviously, this is explained by the longer alkyl chain of tetrabutylammonium compared to tetraethylammonium cation. So, the extraction of ion pairs from the aqueous phase increases [21]. Parameters of microextraction depend on the polarity of the extractant solvent. Both the permittivity of solvent $\varepsilon_r$ and its dipole moment $\mu$ are important complementary characteristics, so the polarity of the extractant was estimated using the electrostatic coefficient EF [22], defined as the product of $\varepsilon_r$ and $\mu$. Thus, the simultaneous influence of both parameters is taken into account. The dependence of the concentrating factor of tetrafluoroborate on the electrostatic coefficient of the used extractant is shown in Figure 3. The higher the EF value of the solvent, the higher the efficiency of $[(TBA^+) (BF_4^-)]$ extraction.

### 3.4. Effect of ion pair reagent concentration

The minimum concentration of QAB was 5 mmol L$^{-1}$ for analyte concentrations range of 0.01–1

**Table 1.** The concentrating factor (K) and the degree of $BF_4^-$ extraction (R) (n=3, P=0.95).

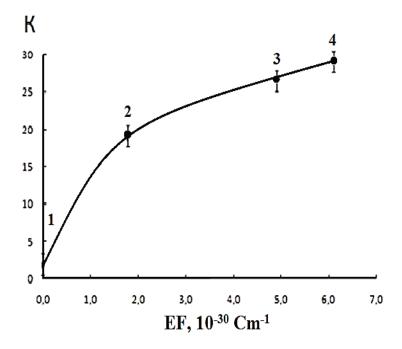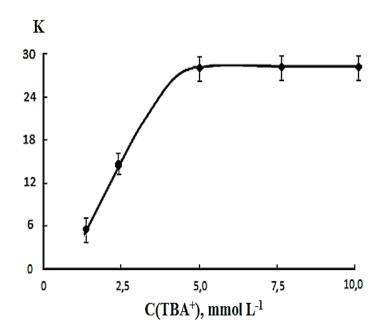| QAB | (% ,K (R | | | |
|---|---|---|---|---|
| | chloroform | 1,2-dichloroethane | carbon tetrachloride | dichloromethane |
| Tetraethyl-ammonium hydroxide | 0.1 ± 1.3 | 0.3 ± 3.1 | 0.1 ± 0.8 | 0.2 ± 2.4 |
| | (0.2 ± 2.2) | (0.5 ± 5.3) | (0.1 ± 1.4) | (0.3 ± 4.1) |
| Tetrabutyl-ammonium hydroxide | 2 ± 19 | 3 ± 29 | 0.2 ± 1.8 | 3 ± 26 |
| | (3 ± 27) | (5 ± 50) | (0.3 ± 3.0) | (4 ± 44) |

**Fig. 3.** Effect of extractant EF. Ion-pair reagent TBAOH, concentration 5 mM.
1 – carbon tetrachloride, 2 – chloroform, 3 – dichloromethane,
4 – 1,2-dichloroethane. (n=3, P=0.95) EF, $10^{-30}$ Cm$^{-1}$

mg L$^{-1}$ (Fig. 4). Abundance of ion pair reagents in the sample does not make the concentration results worse. It is necessary to shift the chemical equilibria to the formation of ion pairs.

Excess reagent was contained in the extract [23]. So, tetrabutylammonium hydroxide and tetraethylammonium hydroxide were chosen as ion-pair reagents. The fact was that hydroxide ions were neutralized by hydrogen ions in a suppression column and entered the detector as water molecules. It helped to decrease the background signal.



**Fig. 4.** Effect of ion pair reagent concentration. Extractant: 1,2-dichloroethane.
Ion-pair reagent: TBAOH, concentration 5 mM. C(BF$_4^-$) = 1 mg L$^{-1}$ (n=3, P=0.95)
C(TBA$^+$), mmol L$^{-1}$

**Table 2.** Summary of method parameters (Extractant : 1,2-dichloroethane. Ion-pair reagent:TBAOH, concentration 5 mM; n=3, P=0.95)

| Parameter name | Value |
|---|---|
| Linear Regression Equation | $S = 4011C \pm 180$, where $S$ – value of $BF_4^-$ peak area, $mV \times min$, $C$ – $BF_4^-$ concentration, mg $L^{-1}$ |
| Linearity Range of the calibration curve, mg $L^{-1}$ | 100 - 0.5 |
| % ,R | $50 \pm 5$ |
| K | $29 \pm 3$ |
| $C_{min}$, mg $L^{-1}$ | $10^{-1} \times 2$ |
| $C_{min, ex}$, mg $L^{-1}$ | $10^{-3} \times 7$ |

### 3.5. Detection limits

The limit of chromatographic detection $C_{min}$ was defined as $3.3\sigma$ ($\sigma$: the standard deviation of measurements of blank samples) [24]. The detection limit using microextraction $C_{min, ex}$ was calculated by dividing $C_{min}/K$ (Table 2).

### 3.6. Synthetic wastewaters analysis and effect of diverse anions

The microextraction efficiency and chromatographic determination are also affected by the sample composition. Many anions can compete with tetrafluoroborate in forming ion pairs with QABs.

Synthetic wastewater samples were prepared and analyzed to assess the interfering effect of diverse anions. The results are shown in Table 3.

Sulfate and perchlorate ions can have the greatest interfering effect. These anions also form stable ion pairs with tetrabutylammonium cation. When its concentration is over than shown in Table 3, the co-eluting occurs with $BF_4^-$. The peak geometry is violated, and the calculation of its area is difficult. However, under the chosen conditions, the separation of all components is not difficult. It is illustrated by the synthetic wastewater chromatograms (Fig. 5a, 5b).

**Table 3.** Effect of diverse anions
(Extractant − 1,2-dichloroethane. Ion-pair reagent − TBAOH, concentration 5 mM; n=3, P=0.95)

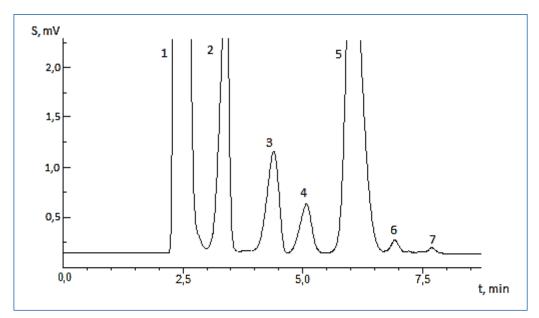| $BF_4^-$ concentration in synthetic samples, mgL$^{-1}$ | Interfering ions (maximum molar ratio) | Results of $BF_4^-$ determination, mgL$^{-1}$ |
|---|---|---|
| 1.0 | $F^-, Cl^-, Br^-, NO_3^-$ (1:10$^4$) | $1.00 \pm 0.17$ |
| 0.2 | | $0.20 \pm 0.04$ |
| 0.05 | $SO_4^{2-}, ClO_4^-$ (1:10$^2$) | $0.05 \pm 0.01$ |

**Fig. 5a.** Chromatograms of synthetic wastewater samples with direct analysis of samples,
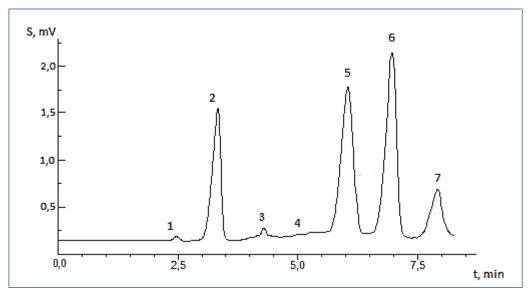$1 - F^-$, $2 - Cl^-$, $3 - Br^-$, $4 - NO_3^-$, $5 - SO_4^{2-}$, $6 - BF_4^-$, $7 - ClO_4^-$



**Fig. 5b.** Chromatograms of synthetic wastewater samples after microextraction.
$1 - F^-$, $2 - Cl^-$, $3 - Br^-$, $4 - NO_3^-$, $5 - SO_4^{2-}$, $6 - BF_4^-$, $7 - ClO_4^-$

### 3.7. Example of waste waters analysis and recoveries of $BF_4^-$

Using the proposed method, 3 samples of wastewater were analyzed to determine $BF_4^-$. Samples 1 and 2 were taken from the drain of the university electrochemical laboratory, and sample 3 was wastewater from the electroplating industry. All samples were filtered twice: firstly, with an ash-free paper filter with a pore size of 5-8 µm (Melior XXI LLC, Russia), then with a syringe filter with a pore size of 0.22 µm (Hawach Scientific Co., Ltd, China). $BF_4^-$ concentration was calculated using the calibration curve method. The results (Table 4) show that the proposed method is suitable for analyzing various origins samples. Recoveries of $BF_4^-$ are shown in Table 5.

**Table 4.** The concentration of some components of different wastewater (n=3, P=0.95)

| Component | Concentration, mg L$^{-1}$ | | |
|---|---|---|---|
| | Sample № 1 | Sample № 2 | Sample № 3 |
| *Fe | $10^{-1} \cdot (2.0 \pm 0.4)$ | $10^{-1} \times (1.5 \pm 0.3)$ | $0.4 \pm 1.9$ |
| *Ni | $10^{-2} \times 1 >$ | $10^{-2} \times 1 >$ | $10^{-2} \times (2.0 \pm 0.4)$ |
| *Cu | $10^{-2} \times 1 >$ | $10^{-2} \times 1 >$ | $10^{-2} \times (8.0 \pm 1.5)$ |
| *Cr | $10^{-2} \times 1 >$ | $10^{-2} \times 1 >$ | $10^{-2} \times (3.0 \pm 0.8)$ |
| $^-$F | $1.1 \pm 0.1$ | $10^{-1} \times (9.3 \pm 0.9)$ | $11 \pm 2$ |
| $^-$Cl | $25 \pm 3$ | $32 \pm 4$ | $68 \pm 7$ |
| $SO_4^{2-}$ | $5.0 \pm 0.6$ | $8.1 \pm 0.9$ | $36 \pm 5$ |
| $BF_4^-$ | $10^{-2} \times (7.2 \pm 1.4)$ | $10^{-2} \times (2.2 \pm 0.4)$ | $10^{-1} \times (4.0 \pm 0.4)$ |

\* determined by atomic absorption analysis

**Table 5.** Recoveries of $BF_4^-$ (n=3, P=0.95)

| Original, mg L$^{-1}$ | Added, mg L$^{-1}$ | Found, mg L$^{-1}$ | Recovery, % |
|---|---|---|---|
| $10^{-1} \times (4.0 \pm 0.4)$ | $10^{-1} \times 1.0$ | $10^{-1} \times (5.1 \pm 0.6)$ | 102.5 |
| | $10^{-1} \times 2.5$ | $10^{-1} \times (6.4 \pm 0.7)$ | 97.5 |
| | $10^{-1} \times 5.0$ | $10^{-1} \times (9.0 \pm 0.9)$ | 100.0 |
| $10^{-2} \times (2.2 \pm 0.2)$ | $10^{-2} \times 1.0$ | $10^{-2} \times (3.3 \pm 0.9)$ | 104.5 |
| | $10^{-2} \times 2.5$ | $10^{-2} \times (4.9 \pm 1.1)$ | 109.1 |
| | $10^{-2} \times 5.0$ | $10^{-2} \times (7.1 \pm 1.3)$ | 95.5 |
| $10^{-2} \times (7.2 \pm 1.4)$ | $10^{-2} \times 1.0$ | $10^{-2} \times (8.0 \pm 1.6)$ | 97.2 |
| | $10^{-2} \times 2.5$ | $10^{-2} \times (9.6 \pm 2.0)$ | 98.6 |
| | $10^{-2} \times 5.0$ | $10^{-1} \times (1.3 \pm 0.2)$ | 111.1 |

## 4. Conclusion

A simple ion chromatographic method was developed to determine $BF_4^-$ using ion-pair liquid-liquid dispersive microextraction. The parameters of microextraction (concentration factor and degree of extraction) were calculated using ion-pair reagents with different hydrophobicity and extractants of various polarities. The higher the ion-pair reagent hydrophobicity and the electrostatic coefficient of the extractant, the higher the extraction efficiency. The best combination is using 1,2-dichloroethane and tetrabutylammonium hydroxide. The maximum molar ratios of analyte to diverse anions were established using synthetic wastewater. Analysis of real samples of industrial wastewater showed that other contained components don't interfere with the determination of $BF_4^-$. The proposed method can generally be applied to analyze water samples of various origins.

## 5. Acknowledgment

## 6. References

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