



Short communication

Ultrasound-assisted emulsification solidified floating organic drops microextraction of ultra trace amount of Te (IV) prior to graphite furnace atomic absorption spectrometry determination

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ABSTRACT

In the present study, a new, simple and efficient method for the preconcentration of ultra trace amounts of Te (IV) is developed using ultrasound-assisted emulsification solidified floating organic drops microextraction (USAE-SFODME) before graphite furnace atomic absorption spectrometry determination. In this method, tellurium is extracted into the fine droplets of 1-undecanol after chelate formation with the water soluble ligand, ammonium pyrrolidinedithiocarbamate (APDC). Several factors such as pH, chelating agent amount, type and volume of the extracting solvent, sonication and centrifuging time that influence the extraction and complex formation are optimized. Under the optimum conditions, the calibration graph is linear in the range of 0.01–0.24 ng mL⁻¹ of tellurium in the original solution, with limit of detection of 0.003 ng mL⁻¹. The relative standard deviation (RSD) for seven replicated determinations of tellurium ion at 0.08 ng mL⁻¹ concentration level is calculated as 3.4%. The proposed method was successfully applied to the determination of Te (IV) in a standard soil and several water samples.

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1. Introduction

Tellurium is a p-type semiconductor and primarily used in semiconductor technology. It is also used in the glass and ceramic industries, as well as in metallurgy where it is added to steel and copper to improve machinability [1]. Due to its acute and chronic toxicity [2], the determination of trace amounts of tellurium has received increasing attention in recent years [3–7]. Tellurium is usually determined in a wide range of matrices (biological, clinical, geological, semiconductor, and metallurgical samples) and several techniques such as hydride generation chemiluminescence (HG-CL) [8], hydride generation atomic absorption spectrometry (HG-AAS) [7,9,10], electrothermal atomic absorption spectrometry (ETAAS) [11–13], inductively coupled plasma optical emission spectrometry (ICP-OES) [14], inductively coupled plasma-mass spectrometry (ICP-MS) [15,16], electrothermal vaporization-inductively coupled plasma-mass spectrometry (ETV-ICP-MS) [17], differential pulse polarography

(DPP) [18], stripping voltammetry [19,20], hydride generation atomic fluorescence spectrometry (HG-AFS) [21–24] and gas chromatography–mass spectrometry (GC-MS) [25] have been proposed for its determination. Tellurium concentration in natural samples is normally at low levels (10⁻⁶%) [1] and often below the detection limit of many modern instrumental techniques, such as flame atomic absorption spectrometry (FAAS), graphite furnace atomic absorption spectrometry (GFAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES). So, preconcentration processes on environmental samples are needed [12]. Several methods for the separation and preconcentration of tellurium are reported. These include hollow fiber liquid phase microextraction (HF-LPME) [12], on-line solid phase extraction [26], magnetic solid-phase extraction [16], coprecipitation [27,28] and ion exchange [29].

In 2006, a new liquid–liquid microextraction method called dispersive liquid–liquid microextraction (DLLME) was invented by Rezaee et al. [30]. Some of the advantages of the DLLME method are simplicity of operation, high speed and low consumption of extraction solvent (at μ L level). This method has been successfully applied for the preconcentration of Te in environmental samples [11]. Despite many benefits of DLLME, the choice of the extraction solvent is its main drawback. The method of solidification

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Table 1
Optimum instrumental conditions for tellurium determination.

Wavelength (nm)	214.3
Spectral bandwidth (nm)	0.2
Lamp current (mA)	10
Lamp type	HCL
Measurement mode	Peak height

Table 2
Optimum temperature program of GFAAS for determination of tellurium.

Steps	Temperature (°C)	Time (s)	Argon flow rate (L min ⁻¹)
Drying	150	30	3.0
Ashing	900	15	3.0
Atomization	2600	5	0.0
Cleaning	2650	2	3.0

of floating organic drop microextraction (SFODME) was introduced by Khalili Zanjani et al. in 2007 [31] as a high-performance, powerful, rapid and inexpensive microextraction method. In this method, the solvent should have a melting point in the range of 10–30 °C and must be immiscible with water. Another dispersive liquid–liquid microextraction method based on the solidification of floating organic drop (DLLME–SFO) was introduced by Leong and Huang [32]. DLLME alone needs a greater amount of extraction solvents and hence is more toxic than DLLME–SFO. Recently, Mohamadi and Mostafavi [33] have developed a new procedure of solidified floating organic drop microextraction, which is based on the dispersion of a micro volume of water-immiscible extraction solvent in an aqueous sample solution using ultrasound energy without any dispersive solvent. A further separation of both liquid phases is carried out by centrifugation. The application of ultrasonic radiation facilitates the emulsification phenomenon and accelerates the mass-transfer process between two immiscible phases, which together with the large surface of contact between the phases leads to an increment in the extraction efficiency in a minimum amount of time [34,35]. The application of a miniaturized approach to the ultrasound assisted emulsification using a micro volume of extraction solvent provides the advantages of both ultrasonic radiation and SFODME. This technique is called ultrasound-assisted emulsification solidified floating organic drop microextraction (USAE–SFODME) [36].

The aim of this work is to combine USAE–SFODME with GFAAS for separation, preconcentration and determination ultra trace amounts of Te (IV) in water samples. In order to obtain a high enrichment factor, the influence of some experimental parameters affecting the complex formation and extraction conditions, are studied and optimized.

2. Experimental

2.1. Apparatus

The tellurium measurement was performed with a Varian Spectra AA 220 atomic absorption spectrometer (Australia, <http://www.varianinc.com>) with a deuterium lamp background correction, equipped with graphite furnace (GTA-110 series). The optimum operating parameters for GFAAS are given in Tables 1 and 2.

A metrohm 827 pH meter (model 827, Switzerland, <http://www.metrohm.com>) with a combined glass electrode for adjusting the pH of solutions were also used.

Fine droplets of organic solvent were obtained using a Sonorex RK255 ultrasonic water bath (Germany, <http://www.sonorex.com>) and an IEC-model HN-S centrifuge (USA,

<http://www.gsrtech.com/c/hns-ii.html>) was used to accelerate phase separation.

2.2. Reagents and solutions

The standard solution of Te (IV) (1000.0 µg mL⁻¹) was purchased from Merck (Darmstadt, Germany, <http://www.merck.com>) and stock solutions of tellurium (IV) were prepared daily by appropriate dilution. The chelating agent, ammonium pyrrolidinedithiocarbamate (0.001%, w/v) solution, was prepared daily by dissolving the appropriate amount of APDC (99%, Merck) in double distilled water. Other chemicals, such as HCl (37%), NaOH with the purity higher than 99% and 1-undecanol, 2-undecanol, *n*-hexadecane for use as an extraction solvent were purchased from Merck. A 500 µg mL⁻¹ palladium solution, the chemical modifier, was prepared by dissolving palladium (II) nitrate (Aldrich, Milwaukee, WI, USA, www.sigmaaldrich.com) in 1 mol L⁻¹ HCl solution. The vessels used for trace analysis were kept in 10% nitric acid for 24 h and subsequently washed three times with double distilled water.

2.3. Treatment of L'vov platform with palladium modifier

In order to prevent the loss of tellurium volatile compound in the GFAAS measurement, palladium as a permanent chemical modifier was electrically deposited on the L'vov platform surface. In the presence of a palladium modifier, tellurium has heat stability up to 900 °C [37]. The advantage of electro deposition is that even with one deposit of palladium, it is possible to replicate the experiment up to 500 times without losing tellurium. The electro deposition of Pd was performed using a Pt electrode as the anode and the platform of graphite tube as a cathode. Before electro deposition, the L'vov platform was heated 5 times to 2650 °C with a cooling step after each heating step. After electro deposition, the platform was washed with distilled water, dried at room temperature and again heated to 2000 °C for 4 s [37].

2.4. Preparing graphite furnace for injection

Since, organic solvents used as extraction solvent are very volatile, heating causes solvent dispersion and the exiting of the analyte from the graphite furnace. To prevent this, the furnace space was saturated with organic solvent (1-undecanol). Before each analyte injection, 1-undecanol was injected to GFAAS and was heated to drying temperature.

2.5. General procedure

For USAE–SFODME, a 14.0 mL sample solution containing 1.2 ng of Te (IV) and 1.8 µL of APDC 0.001% was adjusted to pH 1.5 by a phosphate/phosphoric acid buffer and adjusted to a 15.0 mL in a screw cap glass test tube with conic bottom. 40.0 µL of 1-undecanol were added to the sample solution and sonicated for 4 min. A cloudy solution was formed in the test tube and the complex of Te–APDC was extracted into the fine droplets of 1-undecanol. The solution was now centrifuged at 2500 rpm for 8 min and the extraction solvent floated on the surface of aqueous solution. The glass tube was then transferred into a beaker containing crushed ice; the organic solvent solidified in 5 min and was transferred into a conical vial and melted quickly at room temperature. Now 20.0 µL of the organic phase was injected into the GFAAS for analysis.

The enhancement factor (EF) and percent extraction recovery (ER%) as analytical responses were calculated using the following equations:

$$EF = \frac{m_1}{m_2} \quad (1)$$

$$ER\% = \frac{C_{org}V_{org}}{C_{aq}V_{aq}} \times 100 \quad (2)$$

where m_1 and m_2 are slopes of the calibration curve after and prior preconcentration, V_{org} and C_{org} are the organic phase volume and Te (IV) concentration in the organic phase and V_{aq} and C_{aq} are the aqueous phase volume and Te (IV) concentration in the aqueous phase.

2.6. Sample preparation

2.6.1. Water samples

In order to test the applicability and reliability, the proposed method was applied to preconcentration of Te in different water samples. Tap water (Kerman drinking water, Iran), well water (Shahid Bahonar University of Kerman, Iran), river water (Shoor, Shahdad, Iran) and Oman sea water (Iran) were analyzed by USAE-SFODME combined with GFAAS for determination of tellurium. At first, the pre-reduction of tellurate to tellurite was done by gentle boiling in 6 mol L⁻¹ HCl medium for 45 min [38], and by adjusting pH to 1.5 with sodium hydroxide and filtering to remove any suspended material.

2.6.2. Soil sample

The accuracy and applicability of the proposed method was applied to the determination of tellurium in the Canadian Certified Reference Material (MA-1b) Project. A 2.0 mg sample was taken and dissolved completely by heating in a mixture of HNO₃, HCl and HF (2:6:2 volume ratios). The solution was cooled, diluted and filtered and the volume of the filtrate was raised to 100.0 mL with deionized distilled water in a volumetric flask.

3. Results and discussion

3.1. Optimization of the USAE-SFODME method

In this study, a combination of USAE-SFODME with GFAAS was developed for the determination of trace amounts of Te (IV). In order to obtain a high enhancement factor, the effect of different parameters affecting the complex formation and extraction conditions such as type and volume of extraction solvent, pH, chelating agent amount, centrifuging and sonication time were optimized. The proposed method was also employed for the determination of Te (IV) in soil sample and several water samples.

3.1.1. Effect of pH

In the microextraction method, the pH of the sample solution is a very important factor in the separation of trace metal ions and affects the formation of a complex with necessary hydrophobicity as well as the subsequent extraction. The effect of pH on complex formation was investigated in the pH range of 1–9. The results are shown in Fig. 1. As can be seen, the extraction recovery depends on the pH of sample solution and the highest extraction recovery was achieved at pH 1.5. When pH < 1, hydronium might compete with Te (IV) for complex formation with APDC reagent. On the other hand, the transformation of Te (IV) to Te (VI) may be responsible for a decrease in the extraction recovery of Te (IV) at pH values greater than 2. Therefore, pH of solutions was adjusted to 1.5 by a phosphate/phosphoric acid buffer.

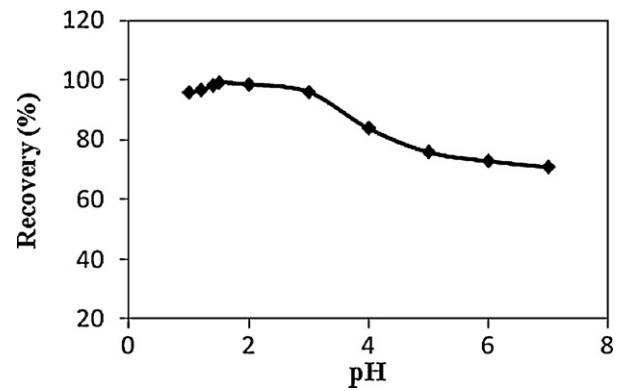


Fig. 1. Effect of pH on the extraction of 0.08 ng mL⁻¹ of Te (IV). Extraction conditions: aqueous sample volume, 15.0 mL; APDC 0.001% (w/v), 1.8 μL; extracting solvent volume, 40.0 μL; salt volume, 0.33%; sonication time, 4 min; centrifugation time, 8 min.

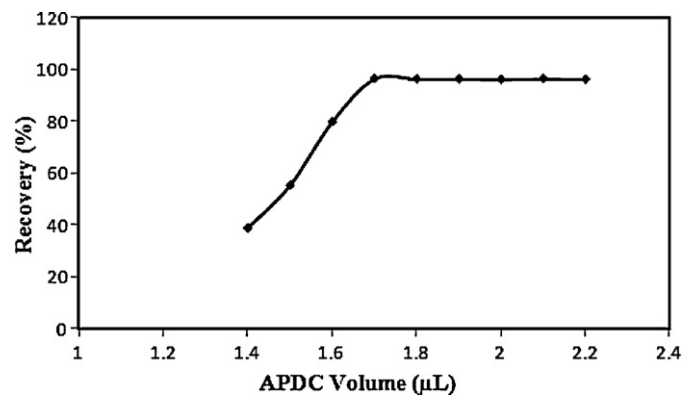


Fig. 2. Effect of APDC amount on the extraction of 0.08 ng mL⁻¹ of Te (IV). Extraction conditions: aqueous sample volume, 15.0 mL; pH 1.5; extracting solvent volume, 40.0 μL; salt volume, 0.33%; sonication time, 4 min; centrifugation time, 8 min.

3.1.2. Effect of APDC amount

The effect of ammonium pyrrolidinedithiocarbamate amount on the extraction efficiency was evaluated. Different volumes of 0.001% (w/v) APDC in the range of 1.4–2.2 μL were added to a 15.0 mL sample solution. As shown in Fig. 2, the recovery of Te (IV) increases with an increase in APDC volume up to 1.7 μL and is then constant. For subsequent experiments a volume of 1.8 μL of APDC was chosen.

3.1.3. Effect of type and volume of the extraction solvent

The extracting solvent for USAE-SFODME must have low volatility, low water solubility, melting point near room temperature, and must not interfere in the analytical techniques used for determination of analytes [39]. Accordingly, several extracting solvents, including 1-undecanol, 2-undecanone, and *n*-hexadecane were investigated. Among the tested extracting solvents, 1-undecanol showed the best extraction efficiency. Thus, 1-undecanol was chosen as the extracting solvent. In order to examine the effect of the extraction solvent volume, different volumes of 1-undecanol (25.0–70.0 μL) were used as the extraction solvent for the same USAE-SFODME procedure. It was observed that the extraction efficiency increases with an increase of the volume of 1-undecanol up to 35.0 μL and then remains constant. Hence a volume of 40.0 μL was used for further experiments.

3.1.4. Effect of salt additions

Salting-out is a process of addition of electrolytes to an aqueous phase in order to increase the distribution ratio of a particular

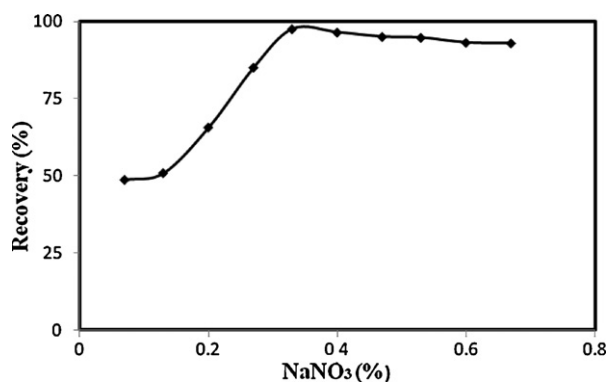


Fig. 3. Effect of salt addition on the extraction of 0.08 ng mL^{-1} of Te (IV). Extraction conditions: aqueous sample volume, 15.0 mL ; pH 1.5; APDC 0.001% (w/v), $1.8 \mu\text{L}$; extracting solvent volume, $40.0 \mu\text{L}$; sonication time, 4 min; centrifugation time, 8 min.

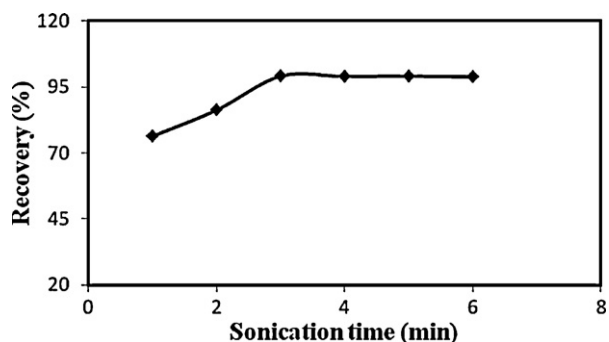


Fig. 4. Effect of sonication time on the extraction of 0.08 ng mL^{-1} of Te (IV). Extraction conditions: aqueous sample volume, 15.0 mL ; pH 1.5; APDC 0.001% (w/v), $1.8 \mu\text{L}$; extracting solvent volume, $40.0 \mu\text{L}$; salt volume, 0.33% ; centrifugation time, 8 min.

solute. The term also suggests reduction in the mutual miscibility of two liquids by addition of electrolytes. Weak intermolecular forces, e.g., hydrogen bonds, between organic molecules or non-electrolytes and water are easily disrupted by the hydration of electrolytes [40]. For investigating the influence of salt type on the Te (IV) USAE-SFODME performance, several experiments were performed by adding NaCl, KCl and NaNO_3 to the solution. Among the tested salts, NaNO_3 showed the best extraction efficiency. Now, the effect of the NaNO_3 was evaluated in the range $0.07\text{--}0.67\%$ (w/v). The resulting data (Fig. 3) showed that the maximum extraction efficiency was obtained in the presence of 0.33% of NaNO_3 . Thus 0.33% was chosen for subsequent studies.

3.1.5. Effect of sonication time

The time of sonication plays an important role in the dispersion, which affects the extraction efficiency of the analytes. Dispersion is the key step in determining whether extraction is successfully carried out or not. Sonication produces fine droplets of organic solvent into the aqueous bulk and results in the generation of a high contact area between the aqueous phase and the extraction solvent. Sonication time was examined in the range of 1–10 min under constant experimental conditions. As shown in Fig. 4, the extraction recovery increased with increasing sonication time up to 3 min, remaining constant for longer times. Thus, a time of 4 min was selected for the dispersive procedure in the subsequent experiments.

3.1.6. Effect of centrifuging time

Centrifugation was necessary to obtain two distinguishable phases in the extraction tubes. The effect of centrifuging time on the extraction efficiency was evaluated in the range of 2–10 min

Table 3
Effect of interfering ions.

Coexisting	Tolerance limit ($C_{\text{ion}}/C_{\text{Te}}$) ^a	Recovery (%)
Mg^{2+}	5000	103.9
Cu^{2+}	1000	98.4
Ba^{2+}	1000	101.0
Zn^{2+}	500	102.3
Mn^{2+}	3000	99.6
Ni^{2+}	5000	96.5
Cd^{2+}	1500	100.1
Ca^{2+}	6000	103.3
Se^{4+}	300	96.5
Pb^{2+}	1000	101.4
Fe^{3+} ^b	1150	98.8
Sb^{3+}	200	97.8
Cl^-	9000	98.2
CH_3COO^-	7000	98.3
F^-	1000	102.8
SO_4^{2-}	10,000	104.1

^a C_{ion} : concentration of interfering ion; C_{Te} : concentration of tellurium.

^b Tolerance limit of Fe^{3+} after masking with fluoride ion.

Conditions: aqueous sample volume, 15.0 mL ; APDC 0.001% (w/v), $1.8 \mu\text{L}$; extracting solvent volume, $40.0 \mu\text{L}$; sonication time, 4 min; centrifugation time, 8 min.

at 2500 rpm. The extraction performance reached its peak when the solution was centrifuged at 2500 rpm for 8 min. When the centrifuging time was longer than 8 min, the recovery remained constant; a centrifuging time of 8 min was chosen.

3.1.7. Effects of co-existing ions

The effect of various interference ions in preconcentration of Te (IV) was studied under the optimized conditions. Solutions containing 0.010 ng mL^{-1} of Te (IV) and various amounts of several ions were prepared and were subjected to the USAE-SFODME procedure. The criterion for interference of each ion was set at $\pm 5.0\%$ in the recovery obtained for a solution containing Te (IV), without any interfering. Table 3 shows the substances studied and their maximum amounts tolerable.

3.2. Method validation

3.2.1. Analytical performance

Important parameters such as the linear range, precision, detection limit, and enhancement factor were determined to evaluate the method performance. The calibration curve was obtained after the standard series were subjected to the proposed procedure and then determined by GFAAS. Linearity was obtained with Te (IV) concentration in the range of $0.01\text{--}0.24 \text{ ng mL}^{-1}$ in the initial solution with a correlation coefficient of 0.9985 ($A = 3.6248C + 0.0270$). The limit of detection and quantification determined as $3S_b/m$ and $10S_b/m$ (where S_b is the standard deviation of the blank and m is the slope of the calibration curve after extraction) were 0.003 and 0.009 ng mL^{-1} , respectively. The relative standard deviation (RSD) for seven replicates of 0.08 ng mL^{-1} of Te (IV) was $\pm 3.4\%$ and the enhancement factor was 342.

3.2.2. Analysis of standard soil sample

To verify the accuracy of the method, the USAE-SFODME method was applied to the determination of tellurium in standard soil sample. The analytical results are given in Table 4. As can be seen, the obtained results are in good agreement with the reference value.

3.2.3. Analysis of standard water samples

The accuracy of the proposed method was evaluated by analyzing a standard reference material (SRM) 1643e from NIST (trace elements in water); with certified tellurium content of $1.09 \pm 0.11 \text{ ng mL}^{-1}$. The result is given in Table 5 and is in good agreement with reference value.

Table 4
Determination of tellurium in a standard soil sample.

Composition (% or $\mu\text{g g}^{-1}$)	Found ^a ($\mu\text{g g}^{-1}$)	Recovery (%)
Si; 24.5, Al; 6.11, Fe; 4.62, Ca; 4.60, K; 4.45, Mg; 2.56, C; 2.44, Na; 1.49, S; 1.17, Ti; 0.38, Ba; 0.18, P; 0.16, Mn; 0.09%, Cr; 200.0, Pb; 200.0, Rb; 160.0, Zr; 140.0, Cu; 100.0, Zn; 100.0, Bi; 100.0, Ni; 90.0, Mo; 80.0, Te; 40.0, Co; 30.0, Y; 20.0, W; 15.0, Sc; 13.0, As; 8.0, Ag, 3.9, Sb; 3.0, Au; 17.0 $\mu\text{g g}^{-1}$	38.7 \pm 1.2	96.7

^a Mean \pm standard deviation ($n = 4$).**Table 5**
Determination of Te (IV) in the water samples.

Sample	Spiked (ng mL^{-1})	Found ^a (ng mL^{-1})	Recovery (%)
Tap water (Kerman)	0.00	N.D. ^b	–
	0.08	0.079 \pm 0.003	98.7
Well water (Kerman University)	0.00	B.L.R. ^c	–
	0.08	0.082 \pm 0.002	102.5
River water (Shoor, Shahdad)	0.00	N.D.	–
	0.08	0.078 \pm 0.003	97.5
Sea water (Oman)	0.00	B.L.R.	–
	0.08	0.084 \pm 0.002	105.0
SRM, 1643e (NIST)	0.00	1.06 \pm 0.04	97.2
	Certificated value (1.09 \pm 0.11)		

^a Mean \pm standard deviation ($n = 4$).^b Not detected.^c Below linear range.**Table 6**
Comparison of the published preconcentration methods for Te (IV) with the proposed method.

Preconcentration method	Determination technique	Enhancement factor	Detection limit (ng mL^{-1})	RSD (%)	Linear range (ng mL^{-1})	Ref.
–	CL ^a	–	2	± 3.0	10–200	[8]
HG-IAT ^b	FAAS ^c	222	0.9	± 7.0	–	[9]
DLLME	GFAAS	125	0.004	± 3.6	0.015–1	[11]
MSPE ^d	ICP-MS ^e	320	7.9×10^{-4}	± 7.0	–	[16]
On line-SPE	GFAAS	42	0.007	± 5.8	–	[26]
W-trap HG	AAS	28	0.08	± 5.8	0.5–20	[41]
Adsorption	UV-Vis-NIR spectrophotometer	25	13	± 2.0	–	[42]
Stripping voltammetry	CSV ^f	–	0.2	–	1–80	[43]
USAE-SFODME	GFAAS	342	0.003	± 3.4	0.01–0.24	This work

^a Chemiluminescence.^b Hydride generation integrated atom trapping.^c Flame atomic absorption spectrometry.^d Magnetic solid phase extraction.^e Inductively coupled plasma-mass spectrometry.^f Cathodic stripping voltammetry.

3.2.4. Analysis of water samples

The proposed method was also employed for the determination of Te in several water samples. The analytical results are summarized in Table 5 and indicate that the recoveries were reasonable for spiked samples in a range of 97.5–105.0%.

3.3. Comparison of USAE-SFODME with other methods

A comparison of USAE-DLLME combined with GFAAS and other techniques for separation and determination of Te (IV) is given in Table 6. We see that our method has low detection limit (0.003 ng mL^{-1}), high enhancement factor (342) and good linear range (0.01–0.24 ng mL^{-1}) for Te (IV). This also has a higher enhancement factor than all other method [8,9,11,16,26,41–43]. Moreover, the detection limit is lower than for all methods [8,9,11,26,41–43], except inductively coupled plasma-mass spectrometry [16]. On the other hand, the relative standard deviation (3.4%) is better than that obtained by inductively coupled plasma-mass spectrometry [16] and other methods with atomic absorption spectrometry detector [9,11,26,41]. All these results indicate that USAE-SFODME is a reproducible, sensitive and simple technique that can be used for preconcentration of tellurium.

4. Conclusion

In this study, ultrasound-assisted emulsification solidified floating organic drops microextraction prior to graphite furnace atomic absorption spectrometry determination, has been developed for separation and sensitive determination of Te (IV) in aqueous samples. The application of ultrasonic radiation is a powerful aid for improving the extraction efficiency of the extraction procedure in the lowest possible time. USAE-SFODME is a modified solvent microextraction method. The sample preparation time and the consumption of volatile organic solvents are minimized by this technique, which, unlike conventional methods has no need of a dispersive solvent. It also has several other advantages such as high recovery, simplicity of operation, low cost and high enrichment factor. Therefore, the method is of interest, especially for routine analytical work.

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