Cloud Point Extraction Using NDTT Reagent for Preconcentration and Determination of Copper in Some Environmental Water Samples by Flame Atomic Absorption Spectroscopy

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Abstract

A micellar-mediated phase separation using Triton X-114 was applied for preconcentration of copper, by formation of a complex with 6-(2-naphthyl)-2,3-dihydro-as-triazine-3-thione (NDTT) reagent, and its analysis by flame atomic absorption spectrometry (FAAS).The effect of experimental conditions such as pH, concentration of reagent and surfactant, equilibration temperature and time on cloud point extraction for maximum recovery were investigated. The complex of copper with NDTT was quantitatively extracted by Triton-X114 at 40 °C, then acidified methanol was added to surfactant-rich phase to decrease the viscosity for measurement of copper. The optimum conditions were pH = 8.5 using borate buffer, 0.05% (v/v) Triton X-114 and 3×10^{-6} mol l^{-1} of NDTT. Calibration graph of copper under optimum conditions was linear in the range of 0.22-100 ng ml^{-1} with a correlation coefficient of 0.998 and limit of detection of 0.22 ng ml^{-1}. The relative standard deviation (R.S.D.) for 5 replicate measurements at 50 ng ml^{-1} Cu level was 2.1% and the enrichment factor for this determination was 22.4. Finally, the proposed method was successfully applied for copper determination in some environmental water samples.

Keyword: Cloud point extraction, copper, triton X-114, NDTT, flame atomic absorption spectrometry

1. Introduction

The tendency and effort have been devoted on the studies for copper determination in aqueous and biological matrices since the concentration of copper is a good tool for environmental and toxicological monitoring. The removal of copper from aqueous medium such as effluents is of great interest for environmental and human health purposes.[1] In general, a daily copper intake of 1.5–2.0 mg is essential and copper at nearly 40 ng ml^{-1} is required for normal metabolism of many living organisms. But, in higher levels is toxic and severe oral intoxication will affect mainly the blood and kidneys. At these levels, copper can bind to the cell membrane and hinder the transport process through the cell wall.[1,2] Because of these and other copper effects on the taste of waters and corrosion, the trace copper content in water and food must be controlled on a daily basis and the European Commission has fixed the limit of 2 μg ml^{-1} for Cu in drinking water and the allowed limit of copper is set to 1.3 μg ml^{-1} in

the USA similar to that in Canada (1.0 μg ml^{-1}).[3,4]

The determination of copper is usually carried out by flame[5–7] and graphite[8,9] atomic absorption spectrometry (AAS), as well as spectrophotometry,[3,10] Chemiluminescence[11] and electrochemical methods.[4,12] However, due to presence of copper in low levels in environmental samples and the matrix effects, different separation and preconcentration techniques such as liquid-liquid[13] or solid phase,[1,5,7,14–16] precipitation,[17] ion-exchange[18] or flotation[19] are necessary. Disadvantages, such as significant chemical additives, solvent losses, complex equipments, large secondary wastes, prefiltration problems and time consuming, limit the application of these techniques.[6]

Micelles and other organized amphiphilic assembles are increasingly utilized in analytical chemistry. Their unique microheterogeneous structures capable of selective interaction with different solute molecules can strongly modify solubility, chemical equilibrium, kinetics and the spectroscopic properties of analytes and reagents. Separation procedures based on the peculiar properties of aque-

ous non-ionic and zwitterionic surfactant solution have also been proposed as an alternative to the use of traditional organic solvents. The first use of the cloud point extraction (CPE) technique was pioneered by Watanabe et al.[20] The analytical potential of the CPE has been discussed by several authors.[21–26]

Aqueous solutions of almost all non-ionic surfactants become turbid when heated to a temperature known as the cloud point. Above this temperature, the isotropic micellar solution separates into two transparent liquid phases: a surfactant-rich phase of very small volume composed mostly of the surfactant plus a small amount of water, and an aqueous phase, in equilibrium with the former, which contains a surfactant concentration close to its critical micellar concentration. The small volume of the surfactant-rich phase obtained with this methodology permits the design of extraction schemes that are simple, cheap, highly efficient, and of lower toxicity to the environment than those extractions that use organic solvents. The CPE phenomenon has been used for the extraction and preconcentration of organic compounds[27,28] and metal cations after the formation of sparingly water-soluble complexes. CPE has been shown to be an effective sample preconcentration technique for improving sensitivity and selectivity prior to atomic spectrometry,[29,30] high-performance liquid chromatography (HPLC),[31–33] flow injection analysis (FIA)[34] and capillary electrophoresis (CE).[35, 36]

The main purpose of this work was to apply CPE as a preconcentration step for FAAS determination of trace copper ion. In the developed system, 6-(2-naphthyl)-2,3-dihydro-as-triazine-3-thione (NDTT) was used as the ligand and Triton X-114 as surfactant. The proposed method is also applied to the determination of copper in some environmental water samples.

2. Experimental

2. 1. Apparatus

A Perkin-Elmer Model 2380 flame atomic absorption spectrometer equipped with Deuterium background correction, and air-acetylene flame was used for copper measurements. A copper hollow cathode lamp was used as the radiation source. The operating conditions of copper hollow-cathode were those recommended by the manufacturer. The wavelength used for copper determination was 324.8 nm. The acetylene flow rates and the burner height were adjusted in order to obtain the maximum absorbance signal by aspirating a solution containing the analyte with 5% (v/v) methanol in 0.1 mol l^{-1} nitric acid aqueous solution. A thermostated water bath maintained at the desired temperatures (Haake) was used for heating solutions and a centrifuge (Hettich) was used to accelerate the phase separation process. The pH of the solutions was controlled with a Genway pH-meter (model 3030).

2. 2. Reagents and Solutions

All reagents were of analytical grade and all solutions were prepared in deionized doubly distilled water. The NDTT was synthesized according to the procedure given in the literature.[37] The nonionic surfactant Triton X-114 (Sigma-Aldrich, Milwaukee, USA) was used without further purification. Stock solutions of copper(II) at a concentration of 1000 μg ml^{-1} was prepared from copper(II) nitrate (Fluka, Buches, Switzerland) in deionized doubly distilled water and working standard solutions were prepared by appropriate dilution of this solution. A stock buffer solution 0.1 mol l^{-1}, was prepared by dissolving appropriate amounts of $Na_2B_4O_7 \cdot 10H_2O$ (Fluka, Buches, Switzerland) in water. Nitric acid solution was prepared by direct dilution with deionized doubly distilled water from the concentrated solution (Merck, Darmstadt, Germany). Acetone, acetonitrile, ethanol and methanol (Fluka, Buches, switzerland) were used to decrease the viscosity of surfactant-rich phase.

2. 3. Procedure

For the cloud point extraction, aliquots of the cold solution containing the analytes, Triton X-114 and NDTT, buffered at a suitable pH, were placed into water bath. The temperature of the solutions was controlled by the thermometers dived both in the tubes and in the bath. The heating was performed with 0.5 °C min^{-1} rate and were kept for 15 min at 40 °C. Separation of the two phases was accomplished by centrifugation for 10 min at 4000 rpm. The phase was cooled down in an ice bath in order to increase the viscosity of the surfactant-rich phase. After the cloud point extraction, the aqueous phase was decanted by inverting the tube. To decrease the viscosity of the surfactant-rich phase and facilitate introduction in flame atomic absorption nebulizer, 200 μl of methanol solution containing 0.1 mol l^{-1} HNO$_3$ was added. The resultant solution was directly introduced into the FAAS by conventional aspiration.

3. Results and Discussion

3. 1. Effect of pH

Cloud point extraction of copper was performed in different pH buffer solutions. The separation of metal ions by the cloud point method involves prior formation of a complex with sufficient hydrophobicity to be extracted into the small volume of surfactant-rich phase, thus obtaining the desired preconcentration. pH plays a unique role on metal–chelate formation and subsequent extraction. Extraction yield depends on the pH at which complex formation is carried out. Fig. 1 shows the effect of pH on the extraction of copper complex. The effect of the sample pH on recovery was investigated within the range of 6.0–13.0 using borate buffers. As it is shown in Fig. 1 maximum recovery was obtained at pH range of 8.2–13.0 and pH 8.5 was chosen for extraction.
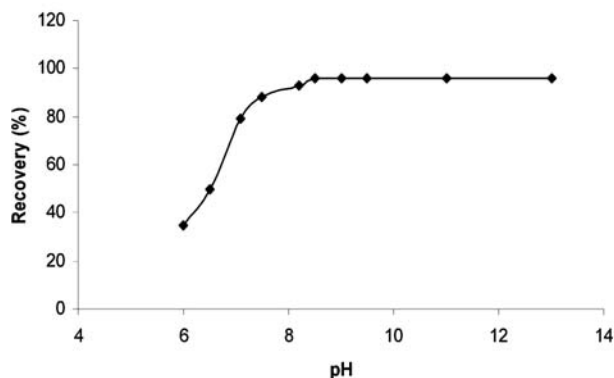
Figure 1. Effect of pH on the cloud point extraction using NDTT reagent of copper Conditions: Cu^{+2}: 50 ng ml^{-1}, Triton X-114 : 0.05%, NDTT: 3×10^{-6} mol l^{-1}

3. 2. Effect of NDTT Concentration

The extraction recovery as a function of the NDTT concentration is shown in Fig. 2. For this study, 10 ml of a solution containing 50.0 ng ml^{-1} copper in 0.05% (w/v) Triton X-114 with various amounts of NDTT was subjected to the cloud point preconcentration process. At this stated concentration of copper, ~100% extraction was achieved for a NDTT concentration of 6.3×10^{-7} mol l^{-1}. Due to consumption of the NDTT with other metals, an excess amount of reagent was used. Thus, a 3×10^{-6} mol l^{-1} NDTT solution was used in subsequent studies.
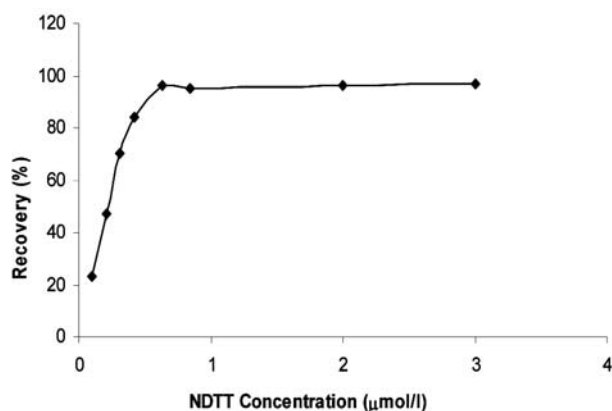

Figure 2. Effect of NDTT concentration on the cloud point extraction of copper Conditions : pH : 8.5, Cu^{+2}: 50 ng ml^{-1}, TritonX-114: 0.05%

3. 3. Effect of Triton X-114 Concentration

A successful cloud point extraction should maximize the extraction efficiency by minimizing the phase volume ratio ($V_{org}/V_{aqueous}$), thus improving its enrichment factor. Reports showed that Triton X-114 and PONPE-7.5

(CPT near room temperature) were proper surfactants to perform CPE for trace elements.[38–39] Triton X-114 was chosen as a non-ionic surfactant because of its commercial availability in a high purified homogeneous form, low toxicological properties and cost. Also, low cloud point temperature (23–26 °C) and high density of the surfactant-rich phase facilitates phase separation by centrifugation. The effect of surfactant concentration was studied within the Triton X-114 concentration range from 0.01 to 0.25% (v/v). Fig. 3 shows the effect of the surfactant concentration on the intensity. Quantitative extraction obtained at its maximum when triton X-114 concentration was 0.05% (v/v). With increase of Triton X-114 concentration above 0.05%, the analytical sensitivity decreased, probably, due to dilution of the sample by additional surfactant solution. Accordingly, a concentration of 0.05% (v/v) Triton X-114 was chosen.
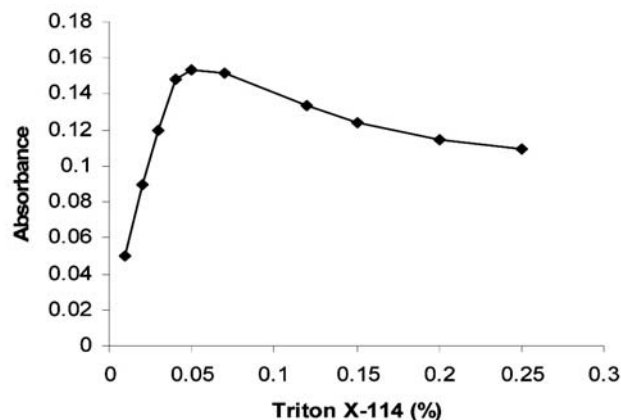

Figure 3. Effect of Triton X-114 concentration on the cloud point extraction of copper Conditions: pH: 8.5, Cu^{+2}: 50 ng ml^{-1}, NDTT: 3×10^{-6} mol l^{-1}

3. 4. Effect of the Equilibration Temperature and Time

Optimal incubation time and equilibration temperature are necessary to complete reaction, and to achieve easy phase separation and pre-concentration as efficient as possible. It was desirable to employ the shortest equilibration time and the lowest possible equilibration temperature, which compromise completion of reaction and efficient separation of phases. The dependence of extraction efficiency upon equilibration temperature was studied for a temperature span of 25–60 °C. Fig. 4 shows the effect of temperature on the recovery. It was found that an equilibration temperature of 40 °C is adequate to obtain quantitative extraction. The effect of equilibration and centrifugation time upon extraction efficiency was also studied in the range of 5–20 min. An equilibration time of 15 min and a centrifugation time of 10 min at

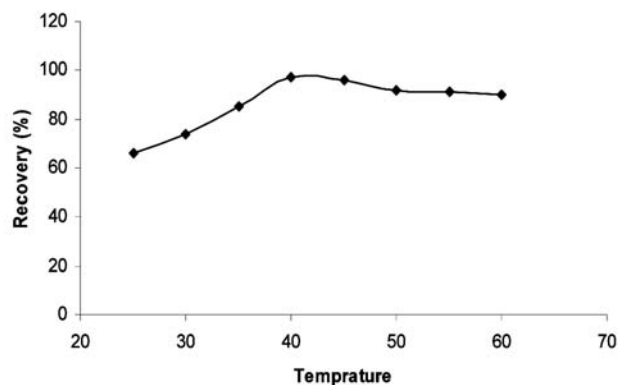4000 rpm were selected as the best to obtain quantitative extraction.

Figure 4. The effect of equilibration temprature on cloud point extraction of copper Conditions: pH: 8.5, Cu^{+2}: 50 ng ml^{-1}, TritonX-114: 0.05%, NDTT: 3×10^{-6} mol l^{-1}

3. 5. Effect of Viscosity

The surfactant- rich phase obtained after cloud point preconcentration is very viscose (~20 cP) owing to the high concentration of Trion X-114. Accordingly, after phase separation it is necessary to decrease the viscosity of the surfactant-rich phase to facilitate its handling and introduction into the atomizer. Several organic solvent and their acid mixtures were investigated, to increase the analytical signal of the FAAS. Solvents tested include acetone, acetonitrile, ethanol and methanol and the best results were obtained using methanol. A methanol solution containing 0.1 mol l^{-1} nitric acid was added to surfactant-rich phase after the separation of two phases. Results showed that the optimum volume of methanol was 200 µL.

3. 6. Analytical Figures of Merit

Table 1 summarizes the analytical characteristics of the optimized method, including limit of detection (LOD), linear range, reproducibility, enrichment factor and consumptive index (CI). The limit of detection, defined as $LOD = 3S_b/m$ (where S_b, and m are, standard deviation of the blank and slope of the calibration graph after preconcentration, respectively), was 0.22 ng ml^{-1}. The calibration curve for copper was linear from 0.22 to 100 ng ml^{-1} with a correlation coefficient of 0.9984. The relative standard deviation (RSD) for 5 replicate measurements of 50.0 ng ml^{-1} copper was 2.1%. The enrichment factor, E_f, defined as the following:

$$E_f = C_s/C_0$$

Where C_s is copper concentration (ng ml^{-1}) in the surfactant-rich phase after phase separation and C_0 is the initial concentration of copper (ng ml^{-1}) in blank aqueous phase. The enrichment factor obtained was 22.4 (*n = 5*).

The consumptive index, *CI*, is defined as following:

$$CI = Vs \, (mL) / EF$$

Where *Vs* and *EF* are sample volume (ml) and enrichment factor, respectively. There are some publications on cloud point extraction of copper using several reagent and different method. Comparison of the limit of detection, enrichment factors and consumptive index of theirs and the proposed method were given in Table 2.

Table 1. Analytical performance of the cloud point extraction method for copper determination

Sample volume (ml)	10
Limit of detection (ng ml^{-1})	0.22
Concentration range (ng ml^{-1})	0.22–100
Correlation coefficient (ml)	0.9984
Precision (n = 5) (%) R.S.D	2.1
Enrichment factor (n = 5)	22.4
Consumptive index (CI, ml)	0.44

Table 2. Comparison of the proposed method with other preconcentration procedures for Cu using several complexing reagents and TritonX-114

Regent	LOD (ng ml^{-1})	EF	CI (ml)	Detection	Ref
NDTT	0.22	22.4	0.44	FAAS	This work
TAN	0.27	64.3[a]	0.78	FAAS	[41]
O,O-DDTP	0.94	33.3[a]	0.30	FAAS	[42]
PAN	0.26	16.3	0.61	CE	[43]
A-O,O-DDTP	0.03	17	2.35	ICP-MS	[44]
Me-BTABr	1.08	17	0.50	FAAS	[45]
Thiamine	0.29	~10	2.50	FS	[46]

LOD: limit of detection; EF: enrichment factor; CI: consumptive index
TAN: 1-(2-thiazolylazo)-2-naphthol;
PAN: 1-(2-pyridylazo)-2-naphthol;
O,O-DDTP: *O,O*-diethyldithiophosphate;
A-O,O-DDTP: ammonium *O,O*-diethyl-dithiophosphate;
Me-BTABr: 2-[2´-(6-methylbenzothiazolylazo)]-4-bromophenol;
ICP-MS: Inductively Coupled Plasma Mass Spectrometry;
CE: Capillary Electrophoresis;
FS: Fluorescence Spectroscopy;
a: Enhancement Factor.

3. 7. Interference Studies

The effect of foreign ions on the recovery of copper was tested. Different amounts of common cations were added to the test solution containing 50.0 ng ml^{-1} of copper and the developed procedure was applied. An ion was considered as interferent, when it caused a variation in the absorbance of the sample greater than ±5%. The tolerance limits of various foreign ions are given in Table 3. These results demonstrate that large excess amounts of common cations and anions do not interfere on the determination of trace quantities of copper.

Table 3. Tolerance limits of interfering species in the determination of 50 ng ml^{-1} of copper (II)

Interferent-to-anlyte mole ratio	Interferent specieI
10000:1	Li$^+$, Na$^+$, K$^+$
2000:1	Ca^{2+},Mg^{2+}, Mn^{2+}, Bi^{3+}, Br$^-$, I$^-$, CH$_3$COO$^-$
1000:1	CO$_3^{2-}$, SO$_4^{2-}$, NO$_3^-$, Fe$_2^+$, Ba^{2+}, Cd^{2+}, Co^{2+}, Sr^{2+}, Zn^{2+}
100:1	Al^{3+}, Pb^{2+}
5:1	Hg^{2+}, Ni^2

3. 8. Determination of Copper in Some Environmental Water Sample

In order to examine the reliability of the proposed method for assaying of copper, the method was applied to determine the concentration of copper in some environmental water sample. Results in Table 4 show that, the added copper ions can be quantitatively recovered from the water samples by the proposed procedure. Recoveries (R) of spike additions (5.0 or 10.0 ng ml^{-1}) to water samples were quantitative. Recovery, *R*, was calculated as follows:

$$R (\%) = \{(C_m - C_o)/m\} \times 100$$

Where C_m, C_o and *m* are a value of copper measured in spiked sample, value of copper in unspiked sample and amount of copper added, respectively. These results demonstrate the applicability of the procedure for copper determination in water samples. For the validation of this method, water samples were analyzed for copper with proposed method and standard method [40] and the results are shown in Table 5. A comparison using *t*-test at 95%

confidence interval demonstrates that there is not significant difference among the achieved results using the proposed method and standard method.

Table 5. validation of proposed method

Sample	copper[a] (ng ml^{-1})	
	Proposed method	**ASTM method**
Distilled water	1.27 ± 0.10	n.d.[b]
Tap water	18.40 ± 0.29	18.32 ± 0.23
Rain water	2.23 ± 0.18	n.d
River water	5.60 ± 0.16	5.27 ± 0.14
Sea water	4.16 ± 0.08	3.90 ± 0.10
Drain water	35.75 ± 0.37	35.42 ± 0.31

[a] Mean S.D. (n = 3),   [b] Not detected

4. Conclusion

In this study the use of CPE method for copper determination using NDTT as new complex reagent was successfully developed. The proposed method permits analysis and quantification of copper (II) at ng ml^{-1} levels. Separation was efficient, resulting in a good enrichment factor and a low LOD. Finally, the results obtained in this work showed that the proposed method is applicable to a variety of copper containing samples. Therefore, this method will be applicable to the monitoring of small amounts of copper in environmental water sample.

Table 4. Results obtained for copper determination in environmental water samples[a]

Sample	concentration ofcopper (ng ml^{-1})		Recovery
	Added	**measured**	**(%)**
Distilled water	0.0	1.27 ± 0.10	–
	5.0	6.12 ± 0.12	96.8
	10.0	11.38 ± 0.18	101
Tap water	0.0	18.40 ± 0.29	–
	5.0	23.64 ± 0.40	104.8
	10.0	28.46 ± 0.34	100.6
Rain water	0.0	2.23 ± 0.18	–
	5.0	7.51 ± 0.16	105.6
	10.0	12.04 ± 0.18	98.1
River water	0.0	5.60 ± 0.16	–
	5.0	10.47 ± 0.20	97.4
	10.0	15.76 ± 0.28	101.6
Sea water	0.0	4.16 ± 0.08	–
	5.0	9.30 ± 0.17	102.8
	10.0	14.58 ± 0.22	104.2
Drain warer	0.0	35.75 ± 0.37	–
	5.0	40.70 ± 0.51	99
	10.0	45.82 ± 0.54	100.7

[a]Mean ± S. D. (n = 3)

5. References

1. E. Kendüzler, A. Rehber Türker, *Anal. Chim. Acta* **2003**, *480*, 259–266.
2. A. R. Ghiasvand, R. Ghaderi, A. Kakanejadifard, *Talanta* **2004**,62, 287–292.
3. P. Rumori, V. Cerdà, *Anal. Chim. Acta* **2003**, *486*, 227–235.
4. Z. Szigeti, I. Bitter, K. Toth, C. Latkoczy, D.J. Fliegel, D. Gunther, E. Pretsch, *Anal. Chim. Acta* **2005,** *532*, 129–136.
5. P. Hashemi, S. Bagheri, M. R. Fat'hi, *Talanta* **2005**, *68*, 72–78.
6. P. Ashtari, K. Wang, X. Yang, S. Huang, Y, Yamini, *Anal. Chim. Acta* **2005**, *550*, 18–23.
7. R. J. Cassella, O. I. B. Magalhães, M. T. Couto, E. L. S. Lima, M. A. F. S. Neves, F. M. B. Coutinho, *Talanta* **2005**, *67*, 121–128.

8. O. Acar, *Talanta* **2005**, *65*, 672–677.

9. J. Y. Cabon, *Spectrochim. Acta B* **2002**, *57*, 939–950.

10. J. J. Pinto, C. Moreno, M. García-Vergas, *Talanta* **2004**, *64*, 562–565.

11. S. Meseguer-Lloret, P. Campínas-Falcó, S. Cárdenas, M. Gallego and M. Valcárcel, *Talanta* **2004**, *64*, 1035–1035.

12. M. P. Hurst, K.W. Bruland, *Anal. Chim. Acta* **2005**, *546*, 68–78.

13. P. Bermejo-Barrera, A. Moreda-Piñeiro, R. González-Iglesias, A. Bermejo-Barrera, *Spectrochim. Acta B* **2002**, *57*, 1951–1966.

14. V. A. Lemos, P. X. Baliza, *Talanta* **2005**, *67*, 564–570.

15. N. Tokman, S. Akman, C. Ozeroglu, *Talanta* **2004**, *63*, 699–703.

16. E. L. da Silva, E.M. Ganzarolli, E. Carasek, *Talanta* **2004**, *62*, 727–733.

17. M. Soylak, S. Saracoglu, U. Divrikli, L. Elci, *Talanta* **2005**, *66*, 1098–1102.

18. S. Scaccia, G. Zappa, N. Basili, *J. Chromatogra. A* **2001**, *915*, 167–175.

19. K. Čundeva, T. Stafilov, G. Pavlovska, *Microchem. J.* **2000**, *65*, 165–175.

20. Z.K. Shihabi, *J. Chromatgr. A* **2000**, *902*, 107– 117.

21. A. Eiguren Fernandez, Z. Sosa Ferrera, J. J. Santana Rodriguez, *Quim. Anal.* **1997**, *16*, 283–289.

22. A. Eiguren Fern´andez, Z. Sosa Ferrera, J. J. Santana Rodriguez, *Anal. Chim. Acta* **1998**, *358*, 145–155.

23. W. L. Hinze, *Ann. Chim. (Rome)* **1987**, *77*, 167–208.

24. N. D. Gullickson, J. F. Scamehom and J. H. Harwell, *in Surfactant-Based Separation Processes,* eds. J. F. Schamehorn and J. H. Harwell, Marcel Dekker, New York, 1989, pp. 139–151.

25. H. Hoshino, T. Saitoh, H. Taketoni, T. Yotsuyanagi, H. Watanabe, K. Tachikawa, *Anal. Chim. Acta* **1983**, *147*, 339–345.

26. T. Saitoh, W. L. Hinze, *Talanta* **1995**, *42*, 119–127.

27. S. R. Sirimanne, J. R. Barr, D. G. Patterson, *Anal. Chem.* **1996**, *68*, 1556–1560.

28. T. Saitoh, W.L. Hinze, *Anal. Chem.* **1991**, *63*, 2520–2525.

29. J. L. Manzoori, A. Bavili-Tabrizi, *Anal. Chim. Acta* **2002**, *470*, 215–221.

30. J. R. Chen, K. C. Teo, *Anal. Chim. Acta* **2001**, *450*, 215–222.

31. C. García Pinto, J. L. Perez Pavon, B. Moreno Cordero, *Anal. Chem.* **1995**, *67*, 2606–2612.

32. R. Carabias Martinez, E. Rodriguez Gonzalo, M. G. Garcia Jimenez, C. Garcia Pinto, J. L. Perez Pavon, J. Hernandez Mendez, *J. Chromatogr. A* **1996**, *754*, 85–96.

33. A.-N. Tang, D.-Q. Jiang, Y. Jiang, S.-W. Wang, X.-P. Yan, *J. Chromatogr.A* **2004**, *1036*, 183–188.

34. Q. Fang, M. Du, C.W. Huie, *Anal. Chem.* **2001**, *73*, 3502–3505.

35. R. Carabias-Martinez, E. Rodriguez-Gonzalo, J. Dominguez-Alvarez, J. Hernandez-Mendez, *Anal. Chem.* **1999**, *71*, 2468–2474.

36. A.-N. Tang, D.-Q. Jiang, X.-P. Yan, *Anal. Chim. Acta* **2004**, *507*, 203–208.

37. I. Lalezari, H. Golgolab, *J. Heterocyclic Chem.* **1970**, *7*, 689–691.

38. E. K. Paleologos, C. D. Stalikas, S. M. Tzouwara-Karayanni, G. A. Pilidis, M. I. Karayannis, *J. Anal. Atom Spectrom.* **2000**, *15*, 287–291.

39. E. K. Paleologos, C. D. Stalikas, M. I. Karayannis, *Analyst.* **2001**, *126*, 389–393.

40. Annual Book of ASTM Standards, Water and Environmental Technology, vol. 1, sec. 11, D 1688–84, Philadelphia, 1986.

41. J. Chen, K. C. Teo, *Anal. Chim. Acta* **2001**, *450*, 215–222.

42. J. L. Manzoori, A. Bavili-Tabrizi, *Microchem. J.* **2002**, *72*, 1–7.

43. A. Tang, D. Jiang, X. Yan, *Anal. Chim. Acta* **2004**, *507*, 199–204.

44. M. A. M. Silva, V. L. A. Frescura, A. J.Curtius, *Spectrochim. Acta, Part B* **2000**, *55*, 803–813.

45. V. A. Lemos, J. S. Santos and P. X. Baliza, *J. Braz. Chem. Soc.* 2006, 17, 30–35.

46. A. Bavili-Tabrizi, *J. Hazard. Mater.* **2007**, *139*, 260–264.

Povzetek

Predkoncentracijo bakra smo izvedli s tvorbo kompleksa s 6-(2-naftil)-2,3-dihidro-triazin-3-tionom (NDTT) ter s fazno ločitvijo s pomočjo micel z uporabo Tritona X-114. Določitev je potekla z atomsko absorpcijsko spektrometrijo z atomizacijo v plamenu. Preučili smo učinek pH, koncentracij reagentov, temperature in časa na ekstrakcijo s tvorbo koloida. Kompleks bakra z NDTT smo kvantitativno ekstrahirali s Tritonom X-114 pri 40 °C, nakar smo dodali nakisan metanol za tmanjšanje viskoznosti. Optimalni pogoji so bili: boratni puffer pH 8,5, 0,05 % (v/v) Triton X-114 in $3 \cdot 10^{-6}$ mol l^{-1} NDTT. Umeritvena krivulja je bila linearna v območju 0,22–100 ng ml^{-1} (R = 0,998), meja zaznavnosti: 0,22 ng ml^{-1}. Relativni standardni odmik pri 5 ponovitvah določitve bakra koncentracije 50 ng ml^{-1} je bil 2,1 %, medtem ko je bil koeficient obogatitve 22.4. Postopek smo uspešno uporabili za določitev bakra v okoljskih vzorcih vode.