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# Temperature-induced surfactant-mediated pre-concentration of uranium assisted by complexation

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**Abstract:** •Cloud-point extraction (CPE) was used with lipophilic chelating agent to extract uranium(VI) from aqueous solutions. The methodology used is based on the formation of metal complexes soluble in a micellar phase of a non-ionic surfactant, Triton X-114. The metal ions complexes are then extracted into the surfactant-rich phase at a temperature above the cloud-point temperature. The influence of surfactant concentration on extraction efficiency was studied and the advantage of adding 8-hydroxyquinoline (8HQ) as lipophilic chelating agent was evidenced. High extraction efficiency was observed, indicating the feasibility of extracting U(VI) using CPE. This study describes a four-step process – (1) extraction, (2) thermo-induced phase splitting, (3) back-extraction and (4) second phase splitting – for the recovery of uranium from water. During steps 1 and 2, uranium can be efficiently extracted at pH > 4 into the surfactant-rich phase formed by the splitting of the initial aqueous solution modified with a 1% Triton X-114. Thus, by using an 8HQ/U(VI) molar ratio of 10, the extraction yield is quantitative and the concentration factor obtained is superior to 100. Uranium can be then stripped with a diluted nitric acid solution (pH < 1) and the system can be recycled through a new four-step cycle.

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**Keywords:** cloud-point extraction; uranium; 8-hydroxyquinoline; Triton X-114

## INTRODUCTION

Assessment of the risk of impact from most radionuclides is based on the total radiological dose rate to the organism of concern. However, for uranium of natural isotopic composition there is a greater risk from chemical toxicity than radiological toxicity.<sup>1,2</sup>

Typical environmental problems associated with mill tailings and waste dumps are radon emanation and leaching of contaminants, including radionuclides and heavy metals, into surface water and groundwater.<sup>3</sup> Pyrite is a sensitive mineral in the geological environment, and its oxidation by iron-oxidizing bacteria produces sulfuric acid, which acidifies the pore water of abandoned metal tailings. Perturbations caused by aggressive mining/milling operations combined with highly acidic environments enhance metal mobilization, which is considered a direct pathway for groundwater contamination. Thus, because of the enrichment of uranium in ores of other metals and phosphate deposits, uranium is found in mine drainage from hard rock base- and precious metal mines, as well as industrial mineral mines.<sup>4</sup> Uranium is also detected in the copper waste dump leaching solutions of many large-scale porphyry copper operations.<sup>5</sup>

In view of uranium toxicity, precise determination in environmental materials is of utmost importance prior to pollution control measures. However, the direct

analysis of geological and environmental samples by atomic absorption spectrometry (AAS), inductively coupled plasma atomic emission spectrometry (ICP-AES) or mass spectrometry (ICP-MS) is still difficult because of the very low concentrations of uranium and also the presence of complex matrix. Thus, elaborate sample preparation involving analyte isolation and pre-concentration is generally necessary before the final analysis. Commonly used methods for extraction and pre-concentration of uranium have been recently reviewed.<sup>6</sup> Liquid–liquid extraction, ion exchange and solid phase extraction are commonly used methods. There is a trend to replace the solvent extraction procedure with ion exchange and solid phase extraction in order to minimize sample manipulation, analyte losses and use of toxic solvents. However, these methods are time-consuming and the tolerance of adsorbed matrix components is low. Thus alternative methods such as co-precipitation,<sup>7</sup> nanofiltration<sup>8</sup> and micellar systems have been developed.<sup>9</sup>

Separations and pre-concentration based on cloud-point extraction (CPE) are becoming a convenient alternative to conventional pre-concentration schemes.<sup>10</sup> The solubility of non-ionic surfactants in aqueous solution is depressed above a well-defined temperature known as the cloud-point temperature (CPT). By setting the solution at a temperature

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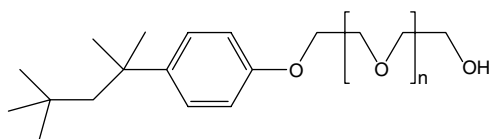


Figure 1. Structure of Triton X-114 ( $n = 6-8$ ).

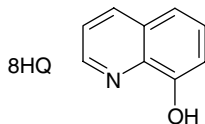


Figure 2. Structure of 8-hydroxyquinoline.

above CPT, the solution separates into a concentrated phase containing most of the surfactant, the coacervate phase, and a dilute aqueous phase. In this method, any component originally present in the solution that interacts with the surfactant aggregate, i.e. the micelles, either directly or after chelation by hydrophobic ligand can thus be extracted and concentrated in the small volume of the surfactant-rich phase. CPE arises from the partitioning of a solute between the two water-based phases depending on its affinity to the surfactant. CPE can be used for the extractive pre-concentration, separation and/or purification of metal ions, metal chelates, biomaterials and organic compounds,<sup>9,10</sup> and pilot-scale experiments for removal of wastewater contaminants have been developed.<sup>11</sup>

The purpose of this study was to evaluate the possibilities of CPE for the pre-concentration of uranium from water. In particular, the influence of surfactant concentration (Triton X-114; Fig. 1) on uranium extraction efficiency was studied and the advantage of adding 8-hydroxyquinoline (8HQ) (Fig. 2) was investigated. The use of 8HQ as a chelating agent for the concentration of trace elements has a long tradition.<sup>12</sup> In that context, 8HQ or immobilized 8HQ derivatives have already been used for the extraction,<sup>13</sup> separation and pre-concentration of uranium.<sup>14,15</sup>

## MATERIALS AND METHODS

All experiments were done in triplicate and data from representative batches are shown.

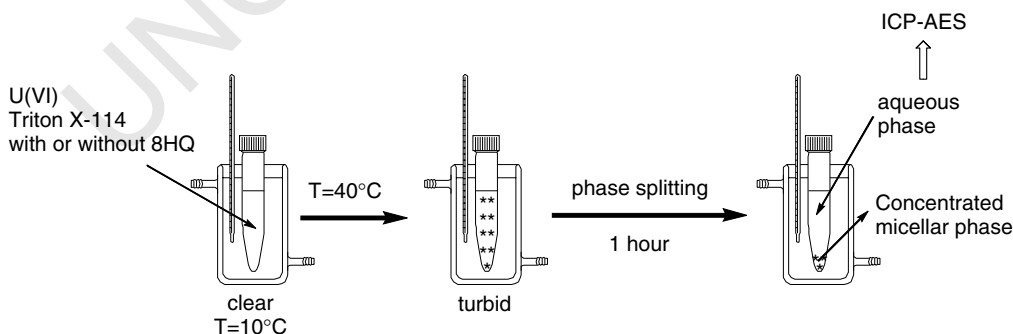


Figure 3. Cloud-point extraction experimental procedure.

## Chemicals and reagents

All reagents used were of analytical grade. Stock solutions of uranium nitrate (●Prolabo) were prepared by dissolving the appropriate amount of its salt in ultra-pure water (●Millipore Alpha-Q). Triton X-114 (Fig. 1) provided by ●Aldrich was used without further purification. Concentrated nitric acid (●Acros Organics) was used to prepare back-extraction solutions.

## Analytical procedure

### ICP-AES measurements

Uranium concentration was determined by ICP-AES using La as internal standard. The apparatus used in this work was a Liberty II from ●Varian.

### Temperature-induced phase separation

A thermostated bath (Haak CH from ●Fison) maintained at the desired temperature was used for CPT experiments.

## Cloud-point procedure

### CPE without 8HQ

This was performed by adding an aliquot of 50 mL of a solution containing U(VI) ( $0.1 \text{ mmol L}^{-1}$ ) to increasing amounts of pure Triton X-114. All surfactant concentrations were calculated as a percentage (w/w).

### CPE with 8HQ

This was performed by dissolving an appropriate amount of 8HQ (Fig. 2) in pure Triton X-114. An aliquot of 50 mL of a solution containing U(VI) ( $0.1 \text{ mmol L}^{-1}$ ) was then added to the 8-HQ/Triton X-114 solution.

In both cases, after homogenization of the mixture at  $10^\circ\text{C}$ , the solution was left to stand at  $40^\circ\text{C}$  for 1 h to obtain phase separation of the micellar phase into a surfactant-rich phase and an aqueous solution depleted in surfactant. The upper aqueous phase (i.e., the surfactant-poor phase) was then removed and its U(VI) concentration was determined by ICP-AES. The experimental procedure is illustrated in Fig. 3.

CPE of U(VI) from micellar solutions was evaluated in terms of extraction efficiency  $E$  and concentration

1 factor  $CF_{U(VI)}$ , defined as follows:

$$E_{U(VI)} = \frac{C_i V_i - C_f V_f}{C_i V_i} \times 100$$

2  
3  
4  
5 Total mass of U(VI) in the  
6 surfactant rich phase  
7  $CF_{U(VI)} = \frac{C_i V_i - C_f V_f}{C_f V_f} = \frac{\text{Total mass of U(VI) in the}}{\text{Total mass of U(VI) in the}} = \frac{C_i V_i - C_f V_f}{C_f V_f}$   
8 aqueous phase  
9

10 where  $C_i$  is the initial concentration of metal ion in  
11 the micellar solution,  $C_f$  is the concentration of metal  
12 ion in aqueous phase after CPE,  $V_i$  is the volume of  
13 the micellar solution and  $V_f$  is the volume of aqueous  
14 phase after CPE.

## 17 RESULTS AND DISCUSSION

### 18 Metal uptake by Triton X-114

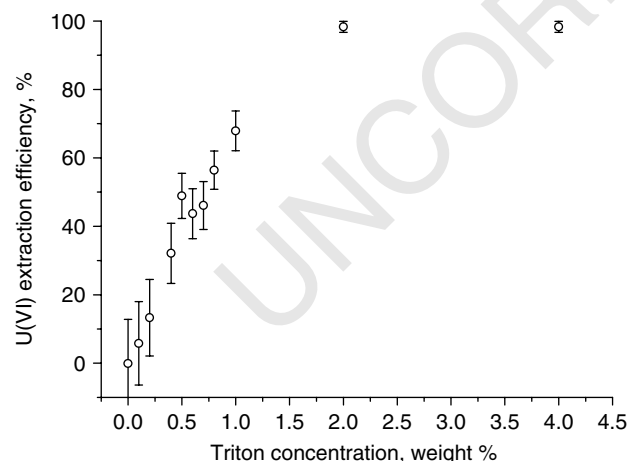
19 In Fig. 4 the variation of U(VI) extraction percentage  
20 as a function of Triton X-114 is presented. Triton  
21 X-114 is effective for U(VI) extraction, which is  
22 markedly increased by a Triton X-114 increase  
23 to reach a maximum of 98% for a triton X-114  
24 concentration of 2%.

25 Extraction of metal ions by CPE has already been  
26 demonstrated in the absence of chelating agent,<sup>12,13</sup>  
27 but metal ion separation could be improved by the  
28 formation of hydrophobic complexes. Since Watanabe  
29 and Tanaka's pioneering work<sup>14</sup> on nickel and zinc  
30 cation extraction, many chelating agents have been  
31 used to extract metal ions.<sup>15,16</sup>

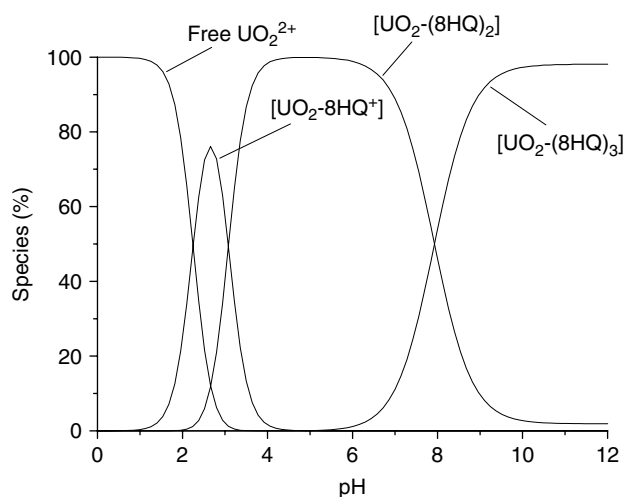
32 In order to minimize the surfactant concentration  
33 and increase the concentration factor we used the  
34 non-ionic surfactant Triton X-114 as the extractant in  
35 association with 8HQ as a chelating agent designed to  
36 form a U(VI) complex of altered solubility.

### 38 Metal uptake by Triton X-114 assisted by 39 complexation

40 Uranium complexes with 8HQ are characterized by  
41 high thermodynamic stability ( $\log \beta_3 = 23.8$ ,  $T =$   
42



58 **Figure 4.** U(VI) extraction efficiency (%) as a function of Triton X-114  
59 concentration (weight %).  $[U] = 0.1 \text{ mmol L}^{-1}$ ,  $\text{pH} = 4$ ,  $T = 40^\circ\text{C}$ ,  
60 equilibration time = 1 h.



77 **Figure 5.** Calculated speciation for U(VI) complexes with  
78 8-hydroxyquinoline (8HQ) evaluated as a function of pH for molar  
79 ratio of  $[8\text{HQ}]/[U] = 10$ ,  $[U] = 0.01 \text{ mmol L}^{-1}$ .

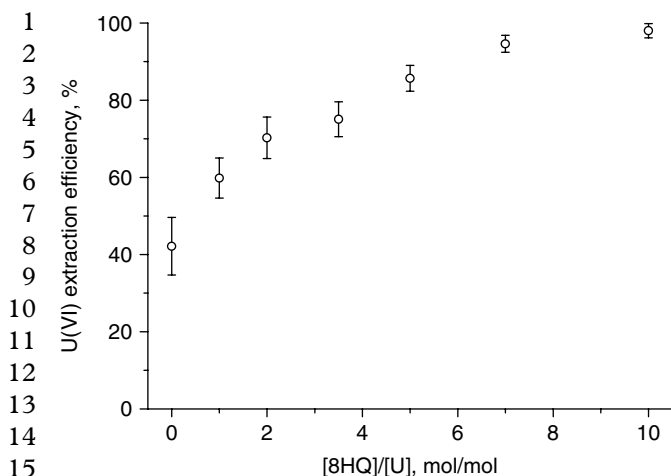
81  $25^\circ\text{C}$ ,  $\text{IS} = 0.1\text{M}$ .)<sup>17</sup> CPT of pure 1% weight Triton  
82 X-114 is close to  $23\text{--}25^\circ\text{C}$ . We have previously shown  
83 that 8HQ depresses the CPT of Triton X-114.<sup>20</sup> In  
84 practice, overheating by more than  $10^\circ\text{C}$  is essential  
85 to obtain phase splitting of the micellar phase into  
86 a surfactant-rich phase and an aqueous solution  
87 depleted in surfactant. In this study, when using  
88 8HQ, the temperature to obtain phase splitting is  
89  $40^\circ\text{C}$ , offering advantages in terms of experimental  
90 procedure. Furthermore, the surfactant-rich phase is  
91 the heaviest, allowing an easier work-up, since the  
92 upper aqueous phase can be easily removed by suction.

93 The pH is a critical parameter for CPE of  
94 U(VI) by 8HQ. Figure 5 shows the distribution  
95 of U(VI) complexes as a function of pH with  
96 a tenfold excess of 8HQ built using the HySS  
97 computer program.<sup>19</sup> U(VI) forms several complexes  
98 with 8HQ, i.e.,  $[\text{UO}_2-(8\text{HQ})^+]^+$ ,  $[\text{UO}_2-(8\text{HQ})_2]$  and  
99  $[\text{UO}_2-(8\text{HQ})_3]^-$ , and the complexation of U(VI) by  
100 8HQ or 8HQ derivatives is dominated by proton  
101 competition, which limits the metal ion binding.

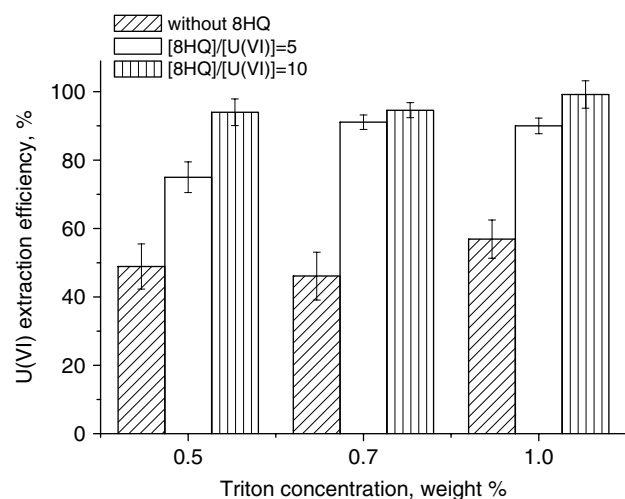
102 Figure 5 suggests that U(VI) is totally complexed by  
103 8HQ as the neutral species  $[\text{UO}_2-(8\text{HQ})_2]$  between  
104 about pH 4 and pH 6. We then investigated the CPE  
105 of U(VI) with increasing 8HQ/U(VI) molar ratios, at  
106 pH 4 and 0.7% weight Triton X-114 (Fig. 6).

107 Figure 6 shows that an 8HQ/U(VI) molar ratio of  
108 5 allows the recovery of more than 85% of uranium,  
109 whereas in the absence of 8HQ the yield of extraction  
110 was close to 40%. With an 8HQ/U(VI) molar ratio of  
111 10, more than 95% of uranium is extracted.

112 It was then found necessary to evaluate the influence  
113 of surfactant concentration on uranium extraction  
114 efficiency by varying the surfactant concentration for a  
115 constant 8HQ/U(VI) molar ratio. Figure 7 shows the  
116 results obtained for the CPE of U(VI) with increasing  
117 concentrations of Triton X-114, in the absence of  
118 8HQ and with 8HQ/U(VI) molar ratios of 5 and 10,  
119 after 1 h at the settling temperature.  
120



**Figure 6.** Effect of 8-hydroxyquinoline/uranium(VI) molar ratio on the extraction efficiency of U, 0.7 wt% Triton X-114, pH 4, [U] = 0.01 mmol L<sup>-1</sup>, equilibration temperature 40 °C, equilibration time 24 h.



**Figure 7.** U(VI) extraction efficiency for increasing concentrations of Triton X-114 in the absence and the presence of 8HQ, [U] = 0.01 mmol L<sup>-1</sup>, pH 4, equilibration temperature 40 °C, equilibration time 1 h, V<sub>i</sub> = 45 mL, V<sub>f</sub> = 44 mL.

In the absence of 8HQ and for increasing concentrations of surfactant from 0.5% to 1% weight, U(VI) extraction slightly increased with an efficiency of 49–60%. For the same surfactant concentrations, with a 8HQ/U(VI) molar ratio of 5, U(VI) extraction efficiency increased from 75% to 90%, whereas with an 8HQ/U(VI) molar ratio of 10 it increased from 94% to 99%.

Previous results showed that when increasing the 8HQ/U(VI) molar ratio from 5 to 10 an increase in Triton X-114 concentration from 0.5% to 1% exhibited a low influence on uranium extraction efficiency. Nevertheless, this low extraction efficiency dependence was correlated to a high CF increase varying from 9, when working with a 0.7% Triton X-114 concentration and an 8HQ/U(VI) molar ratio of 5, to 122 when working with a 1% Triton X-114 concentration and an 8HQ/U(VI) molar ratio of 10 (Table 1).

**Table 1.** Uranium concentration factor as a function of Triton X-114 concentration in the absence of 8HQ and for 8HQ/U(VI) molar ratios of 5 and 10. [U] = 0.01 mmol L<sup>-1</sup>, V<sub>i</sub> = 45 mL, V<sub>f</sub> = 44 mL, pH = 4, equilibration time = 1 h, equilibration temperature = 40 °C

Triton X-114 (% wt)	0.5			0.7			1.0		
[8HQ]/[U]	0	5	10	0	5	10	0	5	10
CF	<1	<1	<2	3	10	9	15	17	122

### Metal stripping

Figure 5 suggests that nitric acid can be used to strip U(VI) from the loaded organic solutions. Indeed, complexes formed with 8HQ can be back-extracted by bringing the surfactant-rich phase into contact with an aqueous solution at pH < 1. Thus, the surfactant-rich phase was brought into contact with a nitric acid solution. To obtain a homogeneous solution, the temperature of the mixture was lowered to 10 °C and then heated to 40 °C for 1 h to produce phase splitting. Using this procedure (1 mol L<sup>-1</sup> HNO<sub>3</sub>, phase ratio) the yield of stripping of U(VI) was found to be quantitative, within experimental error. Thus, experimental results obtained suggest that the recovery of more than 98% of U(VI) can be obtained in one cycle (extraction/back-extraction) starting from a feed solution containing 0.1 mmol L<sup>-1</sup> of U(VI).

### CONCLUSION

U(VI) can be extracted without using organic solvents by CPE using Triton X-114 as surfactant. Addition of 8HQ as chelating agent to the system leads to a decrease in Triton X-114 concentration needed and an increase in the concentration factor varying from 9 to 100, depending on the Triton X-114 concentration and the 8HQ/U(VI) molar ratio. The process consists of four steps: (1) extraction of U(VI) into the micelles of a non-ionic surfactant as a function of its association constants with a chelating extractant, 8HQ; (2) thermo-induced phase splitting; (3) back-extraction of U(VI) from the surfactant-rich phase using acidic solution; and (4) a second phase splitting. The separation can be achieved using an 8HQ/U(VI) molar ratio of 10 at pH 4 and U(VI) can be stripped with a dilute nitric acid solution (pH < 1).

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