

## Simulation of physical-chemical Uranium speciation and Molybdenum for evaluation of environmental mitigation processes and radioprotection

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### Abstract

The understanding of the biogeochemical processes in which trace metals participate in mine regions requires the determination of the different physico-chemical species of the metals present. In the case of uranium extraction mines, the concern is focused on the efficiency of separating other elements present with similar chemical behavior. Added to this are the issues of radioprotection associated with the leaching process and mine acidity. Molybdenum is a trace element present in some uranium-rich regions. The challenge of separating the two elements is presented, reflected in the uranium enrichment process as a subsequent stage in the nuclear fuel cycle. Some separation techniques used have an environmental impact, making these elements available in the environment. In this sense, for this work, simulations were carried out using the PHREEQC program, based on thermodynamic variables and equilibrium constants. The experimental input data were obtained by extracting water in contact with soil from a region of a deactivated uranium mine, followed by analysis by ICP-MS. The simulations considered different acidity conditions and the presence or absence of organic matter in groundwater. It was possible to distinguish conditions in which the chemical availability of uranium and molybdenum differ, enabling their selective extraction. The simulation showed that the best condition for selective extraction of molybdenum from uranium mine is at acidic pH, since molybdenum forms molybdate ( $\text{MoO}_4^{2-}$ ) while uranium forms cationic complexes. Under neutral or basic conditions they form similar complexes, confirming the chemical similarity of the elements.

**Keywords:** Uranium mine; molybdenum; biogeochemical processes; PHREEQC.

## 1.- INTRODUCTION

Uranium ore concentrates (UOCs) can have a significant amount of molybdenum that both originates from variations in ore compositions and can also be attributed to processing: leaching, solvent extraction, resin extraction and precipitation. Because molybdenum and uranium share many common biogeochemical properties, it can form a variety of minerals hosted in uranium ores. Therefore, in the nuclear fuel cycle, molybdenum is an impurity difficult to be separated during the uranium extraction and purification processes. Its concentration must be below some limits specified by the International Atomic Energy Agency (IAEA), because: (i) volatile Mo fluorides would form during conversion to  $UF_6$  and would prevent the enrichment of  $^{235}U$  adequately and (ii) Mo would absorb neutrons and decrease the yield of nuclear fission. Thus, the more efficient selective separation of Mo in uranium processing is a relevant technical need [Migeon *et al.* 2021].

On the other hand, as they present similar geochemical behaviors, the study of molybdenum speciation in regions of uranium mines and, through these studies, directing attention to the mitigation of environmental contamination caused by the mine, favors the proposition of remediation methods for non-contaminated environments, only by molybdenum as from uranium itself [Frascoli and Hudson-Edwards 2018]. In several scenarios in nuclear engineering, molybdenum appears as an element that demands attention and, although there are many, a singular issue is revealed as a technical-scientific point of interest: its selective extraction compared to several other conjugates that present similar chemical behavior [Raiguel *et al.* 2019; Rizk *et al.* 2018].

For this work we propose a simulation study using the PHREEQC program, for the evaluation of groundwater [Shaw *et al.* 2011]. It aims to identify the similarities and differences in the biogeochemical behavior of uranium and molybdenum under different environmental conditions, in a region of a deactivated uranium mine. The experimental input data for the simulation were obtained by ICP-MS from water placed in contact with

the mine's soil [Lozano *et al.* 2020]. The simulation results are discussed regarding the selective extraction of molybdenum from uranium extracts.

## 2.- MATERIALS AND METHODS

Lozano *et al.*, [2020] proposed a method for recovering soil solution extracts from disturbed natural soils. The water retention curve (WRC), which is related to the hydraulic properties of soil, was determined as a way to define the range of suctions applied for the purpose of soil water extraction. We used this method to define the waters used in this work as representative of groundwater in contact with the soil of a deactivated uranium mine.

### 2.1.- Study area

Soil samples were collected from an area located in the west of Spain, in the province of Salamanca (40°43'N; 6°42'E). Each sample comprised 20-cm-deep layer of soil collected using an EIJKELKAMP split-tube sampler. Soil particles larger than 2 mm were removed after the soil samples were air-dried and the large aggregates broken into pieces. The remaining part of the samples was then thoroughly homogenized and chemically and physically characterized [Lozano *et al.* 2020]. The Esperanza mine is located in this region, which was explored through wells and galleries during the 1960s (Figure 1).

According to Santos-Francés *et al.* [2018], the region are located 700–800 m above sea level, and have annual precipitation of 500–600 mm, an average temperature of ~12 °C and annual evapotranspiration of 700–800 mm. The climate is sub-humid, temperate, and dry in the summer. The moisture and temperature regime of the soils is xeric and mesic, respectively. The climatophilic vegetation of this area corresponds to supra-Mediterranean silicic oak.



Figure 1.- Study region where the mine is located, from which soil was taken for the present work [Santos-Francés et al. 2018].

## 2.2.- Soil water extraction

Soil samples were separated according to texture and moistened with different amounts of deionized water to study the influence of rainfall volume (different degrees of moisture) [Rodríguez et al. 2018]. Soil water samples were collected by centrifugation to simulate the root suction pressure of the plants. Centrifugation to remove water was performed at different times after soil humidification to simulate periods of drought (time of contact of the soil with water) [Lozano *et al.* 2020]. For a combination of these variables, that is, soil texture, moisture content and water contact time, in addition to the extraction of the solution at three pressure levels, a total of 81 samples were prepared, based on a 3k factorial design [Medeiros *et al.* 2014].

The extracted solutions, named with the coding GE01 to GE81, were analyzed and the concentrations of the elements in the samples were determined by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) [Rodríguez *et al.* 2018]. The pH and conductivity were obtained directly by mixing an aliquot of soil with distilled water (1:1) and directly measuring it with a Hanna pH meter. The determination of the concentration of soil

nutrients and the amount of organic matter were carried out in partnership with the Soil Sciences Group of the Universidad de Salamanca, Faculty of Agricultural and Environmental Sciences. Through the results, 2 of the 81 analyzes were selected as a basis for the simulation by PHREEQC program presented in this work (GE21 and GE42). To choose these ICP-MS results for use in the simulation, the highest proportion of uranium and molybdenum verified was used as a criterion, since they are the target elements of the study.

Analysis by ICP-MS gives the total concentrations of elements in solution. Adopting an aqueous model, the necessary input data for the simulations are the concentrations of the elements present, the pH of the solution and the temperature, the latter being chosen as the standard temperature of 25°C. As the samples had organic matter, it was decided to simulate including the degradation reaction [Spirakis, 1996]. The database used in the simulations was from Lawrence Livermore National Laboratory (LLNL), due to the availability of information regarding the elements present in the samples in this database [Zavarin et al. 2018]. The simulation result shows speciation and activity of the aqueous species.

### **3.- RESULTS**

As mentioned above, the samples taken for this study come from a test based on a 3k factorial design, where  $k=4$  according to the chosen parameters. The two water samples have in common the same soil particle size with which they came into contact (clay, lower level) and moisture content (100%, higher level), differing in incubation time than for the sample G21 was 30 days (highest level) and for sample G42 it was 7 days (intermediate level), as well as regarding the suction pressure of the water to be analyzed, 153-193kPa for sample G21, and 32-41KPa for sample GE42. In table 1 we present some physicochemical variables of samples G21 and G42.

In Table 1 we present a characterization of the samples based on the analysis of elements present by ICP-MS. The data are complemented by other physicochemical parameters important for the simulation study (Table 2). It is observed that the ratio between the concentrations of uranium by molybdenum is much higher for the G21 sample, where molybdenum appears in a much lower concentration than uranium. Other differences between the samples are the high levels of manganese (Mn), iron (Fe), copper (Cu) and zinc (Zn), for sample G21 and mercury (Hg) and selenium (Se) for sample G42. In addition, sample G21 has a higher content of sulfates and G42 of nitrates. In fact, with regard to the ions present, the samples can be classified distinctly as mixed-mixed (G21) and bicarbonate-mixed (G42) [Reis *et al.* 2021].

Table 1.- Elements presents by ICP-MS.

	<b>G21</b>	<b>G42</b>
Li (µg/L)	0,07	1,02
Na (µg/L)	3097,9	5432,6
Mg (µg/L)	7592,5	5918,6
K (µg/L)	1591,4	2609,2
Ca(µg/L)	12928,3	11080,4
Co (µg/L)	8,24	14,98
Rb (µg/L)	1,91	2,36
Sr (µg/L)	124,76	114,75
Cd (µg/L)	Undetectable	Undetectable
Sn (µg/L)	0,97	1,73
Ba (µg/L)	37,30	41,67
Hg (µg/L)	Undetectable	2,73
Pb (µg/L)	0,66	<0.5
U (µg/L)	29,24	18,70
Al (µg/L)	3,98	Undetectable
Ti (µg/L)	0,70	<0.5
Cr (µg/L)	5,74	<0.5
Mn (µg/L)	11927,3	7374,4

Fe ( $\mu\text{g/L}$ )	103,8	Undetectable
Ni ( $\mu\text{g/L}$ )	7,10	5,53
Cu ( $\mu\text{g/L}$ )	20,51	11,59
Zn ( $\mu\text{g/L}$ )	158,70	25,09
As ( $\mu\text{g/L}$ )	2,24	2,18
Se ( $\mu\text{g/L}$ )	1,90	6,03
Mo ( $\mu\text{g/L}$ )	0,68	4,77
Sb ( $\mu\text{g/L}$ )	Undetectable	Undetectable

In Table 2 we present the complementation of the characterization of the waters carried out by ICP-MS, with data from other physicochemical parameters relevant to studies of speciation of the elements in the environment.

Table 2.- Physicochemical parameters of the samples.

	<b>G21</b>	<b>G42</b>
Fluorides (mg/L)	1,023	1,000
Chlorides (mg/L)	6,33	4,11
Nitrites (mg/L)	2,24	8,84
Bromides (mg/L)	3,54	3,54
Nitrates (mg/L)	3,63	14,09
Phosphates (mg/L)	3,28	3,27
Sulfate (mg/L)	21,40	3,88
Ammonium (mg/L)	0,26	Undetectable
pH	6,73	7,25
Conductivity ( $\mu\text{s/cm}$ )	232	184,5
Organic Matter (mg/L $\text{O}_2$ )	14,4	12,8
Carbonates (meq/L $\text{CO}_3$ )	Undetectable	Undetectable
Bicarbonates (meq /L $\text{HCO}_3$ )	0,40	0,40

For the simulations we used the characterization data of the samples, but we varied the pH value. By varying the pH values, we intend to consider different techniques for selective

extraction of elements and remediation of contaminated environments, since the manipulation of the acidity of the medium is a resource used to control the elements made available in the medium [Migeon *et al.* 2020; Frascoli and Hudson-Edwards 2018].

The simulations also considered the presence or absence of organic matter. This is because this parameter can affect the saturation index (SI) of some elements present. The supersaturated minerals, that is, which presented a positive index and, therefore, an indication of the possibility of formation of the mineral solid phase from the aqueous solution, vary according to the samples, acidity of the medium and the presence of organic matter. The simulation results are presented in three tables. In Table 3 we present the simulation results for the G21 sample, in Table 4 the simulation results for the G42 sample. In Table 5, we present data on the saturation index found for some elements, both from sample G21 and from sample G42.

Table 3.- Molybdenum and uranium concentration data for sample G21 considering the presence or absence of organic matter [OM], varying the pH.

<b>G21</b> <b>[mol/kg]</b>	<b>pH/OM</b> <b>3 / N</b>	<b>pH/OM</b> <b>3 / Y</b>	<b>pH/OM</b> <b>7 / N</b>	<b>pH/OM</b> <b>7 / Y</b>	<b>pH/OM</b> <b>9 / N</b>	<b>pH/OM</b> <b>9 / Y</b>
UF <sub>3</sub> <sup>+</sup>	8.431e-12	4.606e-08				
<b>U(OH)<sub>4</sub></b>	<b>6.058e-12</b>	<b>5.713e-08</b>	<b>1.844e-15</b>	<b>1.229e-07</b>	<b>3.234e-19</b>	<b>1.229e-07</b>
UF <sub>2</sub> <sup>+2</sup>	2.229e-12	1.065e-08				
UF <sub>4</sub>	1.418e-12					
<b>UO<sub>2</sub><sup>+</sup></b>	<b>6.811e-11</b>	<b>9.061e-11</b>	<b>1.819e-14</b>	<b>9.061e-15</b>	<b>3.182e-18</b>	<b>3.531e-15</b>
UO <sub>2</sub> (CO <sub>3</sub> ) <sub>3</sub> <sup>-5</sup>	2.147e-33					
UO <sub>2</sub> F <sup>+</sup>	5.424e-08	1.159e-11				
UO <sub>2</sub> <sup>+2</sup>	2.733e-08	5.108e-12				
UO <sub>2</sub> H <sub>2</sub> PO <sub>4</sub> <sup>+</sup>	1.762e-08	3.411e-12				
UO <sub>2</sub> HPO <sub>4</sub>		2.366e-12	1.337e-08			
UO <sub>2</sub> CO <sub>3</sub>		1.973e-12				
UO <sub>2</sub> (OH) <sub>3</sub> <sup>-</sup>					6.311e-08	
UO <sub>2</sub> (OH) <sub>2</sub>			2.756e-08		4.834e-08	4.266e-17
UO <sub>2</sub> (CO <sub>3</sub> ) <sub>2</sub> <sup>-2</sup>			1.390e-08	1.899e-16	5.295e-09	1.333e-16



$\text{UO}_2(\text{CO}_3)_3^{-4}$						1.284e-09
$\text{UO}_2\text{PO}_4^-$			6.169e-08	1.411e-16	1.106e-09	6.964e-17
$\text{MoO}_4^{-2}$	<b>7.089e-09</b>	<b>7.088e-09</b>	<b>7.088e-09</b>	<b>7.088e-09</b>	<b>7.088e-09</b>	<b>7.089e-09</b>

We observe in Table 3 that, in all simulation conditions, molybdate [ $\text{MoO}_4^{-2}$ ] and uranium species  $\text{U}(\text{OH})_4$  and  $\text{UO}_2^+$  are formed. Considering the selective removal of elements by ion exchange, using resin, for example, we observed that acidic conditions favor the selectivity between uranium and molybdenum. This is because at pH 3 the formation of uranium cations prevails, while molybdate is an anion. At neutral and basic pH, anionic formations of uranium occur, demonstrating the chemical similarity between the two elements under these conditions. On the other hand, considering the expressive formation of  $\text{UO}_2^+$  in all simulated conditions and, being a cation, the use of ion exchange resin for the selective removal of uranium using cationic resin is justified. The same cannot be considered for the selective removal of molybdenum, being only effective the use of anionic resin and in acidic conditions.

Table 4.- Molybdenum and uranium concentration data for sample G42 considering the presence or absence of organic matter [OM], varying the pH.

<b>G42</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>
<b>[mol/kg]</b>	<b>3 / N</b>	<b>3 / Y</b>	<b>7 / N</b>	<b>7 / Y</b>	<b>9 / N</b>	<b>9 / Y</b>
$\text{UF}_3^+$	5.373e-12	2.675e-08				
$\text{U}(\text{OH})_4$	<b>3.983e-12</b>	<b>3.830e-08</b>	<b>1.126e-15</b>	<b>7.857e-08</b>	<b>1.970e-19</b>	<b>7.857e-08</b>
$\text{UF}_2^{+2}$	1.422e-12					
$\text{UO}_2^+$	<b>4.490e-11</b>	<b>5.849e-10</b>	<b>1.105e-14</b>	<b>1.563e-15</b>	<b>1.930e-18</b>	<b>1.178e-16</b>
$\text{UO}_2\text{F}^+$	3.527e-08	7.244e-10				
$\text{UO}_2^{+2}$	1.778e-08	3.146e-10				
$\text{UO}_2\text{H}_2\text{PO}_4^+$	1.164e-08	2.115e-10				
$\text{UO}_2\text{HPO}_4$		1.541e-10				
$\text{UO}_2\text{CO}_3$		1.416e-10				
$\text{UO}_2(\text{OH})_3^-$					3.827e-08	
$\text{UO}_2(\text{OH})_2$			1.684e-08	1.012e-16	2.945e-08	
$\text{UO}_2(\text{CO}_3)_2^{-2}$				5.821e-16	5.331e-09	1.311e-16

$\text{UO}_2(\text{CO}_3)_3^{-4}$				1.106e-16	3.709e-09	1.535e-16
$\text{UO}_2\text{PO}_4^-$				4.067e-08		
<b><math>\text{MoO}_4^{-2}</math></b>	<b>4.972e-08</b>	<b>4.972e-08</b>	<b>4.972e-08</b>	<b>4.972e-08</b>	<b>4.972e-08</b>	<b>4.972e-08</b>

We observed a similarity in the results obtained by the simulation with data from the G21 sample in relation to those obtained with data from the G42 sample. Once again, the formation of molybdate and uranium species  $\text{U}(\text{OH})_4$  and  $\text{UO}_2^+$  is observed. However, we note the absence of  $\text{UF}_4$  and  $\text{UO}_2(\text{CO}_3)_3^{-5}$  formation.

Considering the possibility of immobilizing elements by precipitation as a resource to control environmental contamination by uranium and/or molybdenum, we present results of the saturation index [SI] in Table 5. We observed a very similar behavior between the samples with regard to the elements that precipitate under the various simulation conditions. It is interesting to note, for example, that although the concentration of selenium in the two samples is different [Table 1], this element is important in the formation of the  $\text{MoSe}_2$  precipitate for the two samples. We note that when considering the contribution of organic matter in the environment, the simulation indicates precipitation of  $\text{UO}_2$ .

Table 5.- Results of saturation index [SI] obtained by simulation.

<b>G21</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>
<b>[mol/kg]</b>	<b>3 / N</b>	<b>3 / Y</b>	<b>7 / N</b>	<b>7 / Y</b>	<b>9 / N</b>	<b>9 / Y</b>
Mo	-36,44	-14,14	-68,53	-18,63	-84,52	-19,06
$\text{MoSe}_2$	<b>17,06</b>	<b>24,33</b>	-47,96	<b>11,80</b>	-87,09	<b>10,46</b>
$\text{UO}_2$	-1,79	<b>2,18</b>	-5,36	<b>2,46</b>	-9,12	<b>2,46</b>
$\text{U}(\text{HPO}_4)_2 \cdot 4\text{H}_2\text{O}$		0,49				
$\text{UOF}_2$		0,06				
$\text{UOF}_2 \cdot \text{H}_2\text{O}$		0,59				
$\text{UP}_2\text{O}_7$		0,76				
<b>G42</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>	<b>pH/OM</b>
<b>[mol/kg]</b>	<b>3 / N</b>	<b>3 / Y</b>	<b>7 / N</b>	<b>7 / Y</b>	<b>9 / N</b>	<b>9 / Y</b>

Mo	-35,58	-19,34	-67,67	-20,81	-83,67	-21,06
MoSe <sub>2</sub>	<b>18,92</b>	<b>24,13</b>	-46,09	<b>9,97</b>	-85,21	<b>6,97</b>
UO <sub>2</sub>	-1,97	<b>2,01</b>	-5,58	<b>2,27</b>	-9,33	<b>2,27</b>
U(HPO <sub>4</sub> ) <sub>2</sub> : 4H <sub>2</sub> O		0,28				
UOF <sub>2</sub> :H <sub>2</sub> O		0,38				
UP <sub>2</sub> O <sub>7</sub>		0,56				

## 4.- DISCUSSION

After analyzing the data obtained by simulating chemical speciation using the PHREEQC software, it was noticed that the saturated minerals based on the results for the conditions entered vary for the same sample considering or not the degradation reaction of organic matter. Only for acidic pH is this difference not noticed, with regard to precipitation of MoSe<sub>2</sub>. It is important to remember that the pH value obtained experimentally for the samples was close to the neutrality value. Comparing the analysis considering the degradation reaction of organic matter and disregarding it, it is noted that taking into account the degradation reaction, the amount of minerals that become supersaturated increases, and a particular interest occurs in the emergence of Uraninite [UO<sub>2</sub>] as a supersaturated mineral, for all samples, with a saturation index close to 2.0.

With regard to chemical speciation, analyzing the concentration of uranium species, it can be seen that for the two samples analyzed and at all simulated pH values, the concentration of U(VI) is the highest among the species when not the degradation of organic matter is considered. Considering the simulation of the complete degradation of the organic matter present in the analyzed samples, the predominant uranium species becomes U(IV), more specifically U(OH)<sub>4</sub>. In all simulated cases, molybdenum is in the form of molybdate. [MoO<sub>4</sub><sup>-2</sup>]. The simulation data regarding the predominance of the U(VI) species corroborates what was found in the literature, disregarding the complete degradation of the

organic matter in the environment [Bachmaf and Merkel 2011]. Knowing that such species are strongly retained in ion exchange resins, this is a promising finding for the application of the method to capture uranium from the medium. The formation of neutral species, however, impairs the sorption of uranium in ion exchange resin, which points to the need to use different strategies for recovery from contaminated environment and radioprotection.

## 5.- CONCLUSIONS

Samples of water that were in contact with soil from a deactivated uranium mine region were experimentally obtained and analyzed to compose input data to simulate the physico-chemical conditions of uranium and molybdenum in the medium. The simulations considered the variation of pH and degradation of organic matter. Uranium and molybdenum ionic formations were observed, indicating the feasibility of selective separation of these elements using ion exchangers. In this case, as molybdenum always forms molybdate  $[\text{MoO}_4^{-2}]$ , it could be separated from uranium especially under acidic conditions. On the other hand, if the interest is only in uranium recovery, one can alternate the use of cationic and anionic resins. To control the mobility of these elements in the medium, the saturation index was analyzed under the different simulation conditions. Considering the degradation of organic matter, precipitation of  $\text{UO}_2$  is observed, favoring its immobilization. Regarding molybdenum, precipitation in the form of  $\text{MoSe}_2$  is observed, also considering the degradation of organic matter.

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