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Studies on the separation of Uranium from Algerian yellow cake using different processes: impurities determination

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ABSTRACT

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Analysis of impurities in uranium materials is crucial for quality control or the purity required by standards in the nuclear industry. This study involves the analysis of impurities in a sample of uranium ore concentrate produced from Tahaggart ore. The instrumental techniques used for the analysis are highly sensitive and susceptible to much interference following the uranium spectrum. Different processes for separating uranium from impurities for comparison were used to quantify them. The uranium sample is an Algerian yellow cake that was digested in nitric acid, separated by solvent extraction using the $TBP/CHCl₃$ system, by extraction chromatography method using tributyl phosphate (TBP) impregnated on the Amberlite XAD-7 resin and by precipitation of uranium with hydrogen peroxide. The raffinate from each process is then analyzed by flame atomic absorption spectroscopy and flame photometry. The concentration of uranium is determined by the Potassium Bichromate method in concentrated solutions and by the Arsenazo III method in raffinates and eluates. Uranium extraction yields are 99.65% using TBP-CHCl₃ and exceed 99% using TBP-impregnated XAD-7 resin. The results of the analysis of impurities in Algerian yellow cake after the separation of uranium using different processes show that the contents meet the ASTM C967-13 standard for the elements analyzed except for the iron element. A comparison of the results of the impurities analysis values in Algerian yellow cake by the three separation processes (liquid-liquid extraction (ASTM C761-11), extraction chromatography column, and precipitation) shows the absence of cadmium, titanium, lead, and chromium and the values of the manganese, zinc, and lithium are quite close. The analysis results for the solvent extraction and chromatographic column extraction processes showed that the values of magnesium, copper, and nickel are very close and identical for cobalt. The values of aluminium and iron are close.

1. Introduction

Over the last 50 years, the option of nuclear energy, and in particular its applications in the fields of health, industry, engineering, and the environment, have experienced significant development and represent a significant advance in the quality of life of the population, particularly with the use and development of new radiopharmaceuticals and radioisotopes [1].

Uranium dioxide $UO₂$ is the most widespread combustible material in nuclear reactors, mainly for the production of electricity, and in research reactors for manufacturing radioisotopes. $UO₂$ is obtained from uranium ore and has undergone a multitude of physicochemical treatments. Indeed, the first treatment comprises a uranium ore concentrate (Yellow cake) produced by extracting and concentrating uranium from ores such that the uranium concentration is upwards of 70% by mass and ultimately serves as the starting material for the fuel cycle [2]. The yellow cake does not have a sufficient degree of nuclear purity or the chemical form to be used as fuel in the reactor. It is therefore subject to an additional treatment purification. Given the composition of uranium ore, other ionic species occur alongside the uranyl ion throughout the fuel cycle, but in increasingly lower concentrations. The determination of uranium content and impurities before

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and after each stage is very necessary to maintain product purity and opt for a suitable treatment or purification process. Analysis of impurities in uranium concentrate (yellow cake) can provide information on the source, production history, and potential intended use of uranium concentrate [3].

Because of the high purity required, known as nuclear purity, it is necessary to eliminate a large number of impurities such as boron and cadmium, which are neutron adsorbents; vanadium, arsenic, and calcium, which require costly refining and conversion. Alkali metals such as calcium and magnesium, as well as iron, tend to form oxides, preferentially affecting the oxygen/metal ratio in the fuel matrix [4]. Determination of the elements in the matrix of nuclear materials is necessary before their use as nuclear fuel [5]. Several analytical techniques are used to determine these trace elements or impurities in nuclear materials; spectral methods are the most commonly adopted. Instrumental analytical techniques used for the determination of alkali or alkaline earth metals include ICP-MS, ICP-OM, flame atomic absorption spectroscopy, GFAAS, XRF, ion chromatography, etc... The previous separation of uranium from the matrix is very necessary. Such separation is carried out by liquid-liquid extraction, ion-exchange chromatography [6], precipitation, distillation, and electrolysis, in order to minimize the problem of interference. The main problem associated with the use of emission techniques is spectral interference in the emission spectrum of the uranium complex [7]. The elemental determination of impurities in solid samples is divided into three main stages: dissolution of the material, separation of impurities from the matrix, pre-concentration, and basic determination. Materials are dissolved in several types of acids [8]. The challenge is therefore to develop separation techniques making it possible to compose both the chemical complexity of the sample and the range of concentrations encountered. Several radiochemical separation methods have been developed to extract uranium from the matrix; the most widely used are the solvent extraction technique using a $TBP/CCl₄$ system, extraction chromatography using resins, and hydroxide precipitation in the presence of complexing agents. Liquidliquid extraction (SX) is the oldest separation method and still the most widely used industrially. Tributyl phosphate is used industrially for uranium extraction. The advantages of this technique are its low operating costs and its simplicity. However, this technique has the disadvantage of the use of flammable solvents, the generation of waste, the increased exposure of workers to sometimes high levels of radiation, and constitutes a greater environmental problem [9]. Extraction chromatography is widely used for the

selective separation of radiochemical impurities in a wide variety of samples. Extraction chromatography (EXC) is based on the same principles as solvent extraction, but the separation is carried out using a chromatographic column. The advantages of EXC over SX include [10] the elimination of the need for mixing and separation of phases and associated issues regarding ligand solubility and phase transfer kinetics, the removal of the possibility of formation of a third phase and reduction, or even the total absence of radioactive organic waste [11]. The precipitation of uranium by hydrogen peroxide is an excellent method that occupies an important place in the separation of uranium impurities. However, this technique is not directly applicable to yellow cake because of the presence of alkali metals (Na, Li, and K) and alkaline earth metals (Mg and Ca) as well as sulfates. A hydroxide precipitation is carried out before the precipitation of the uranium with hydrogen peroxide to eliminate the aforementioned elements [12].

The separation of uranium from uranyl nitrate matrix is essential prior to the determination of impurities and establishment of the technical sheet of the uranium ore concentrate in order to decide on its accordance with the (ASTMC 967-13) standard. In this article, we propose three uranium separation procedures in order to quantify impurities in an Algerian yellow cake resulting from the processing of Tahaggart ore. The first process is the solvent extraction system TBP-6M nitric acid (ASTM C761-11) [13] for separation of uranium from its impurities in Algeria yellow cake. After extraction of the aqueous phase with 100 % TBP a second extraction is made with 20% TBP in chloroform to improve the yield of extraction and allow better phase separation. Finally, the aqueous phase is washed with chloroform to remove residual TBP. The second process is chromatographic extraction, this separation technique uses an inert macroporous polymeric support (Amberlite XAD-7) impregnated with a selective extractant tri-n-butyl phosphate (TBP). Columns made with this resin selectively adsorb the uranium from acid media. The study pointed out that, the main parameters affecting the chromatography extraction are mainly washing reagents, impregnation time of the resin and height of the column bed. A Chemical precipitation process of recovering uranium from the dissolving solution of uranium ore concentrate was investigated. The process includes primarily precipitation of uranium with ammonium hydroxide for Na, K, Ca and Mg determination and precipitation of uranium with H_2O_2 for Al, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Ti, V, Pb and Zn determination [12]. A flame atomic absorption spectrometry (FAAS) and flame photometry have been used for the determination of

impurities concentrations after uranium chemical separation. Correlations between solvent extraction, column separation and precipitation are compared and discussed.

2. Materials and Methods

2.1 Instrumentation

A GBC AVANTA Σ , an Australia-made model of atomic absorption spectrophotometer (FAAS) is used for flame measurements. A GBC Cintra 40, Australia-made model UV-visible spectrophotometer was used to determine the concentration of uranium in the raffinate.

A Jenuray PFR7 flame photometry was used to analyze alkaline and alkaline earth elements

2.2 Reagents

Nitric acid (HNO₃ 65 %, Panreac), perchloric acid (HClO₄, 60%, Analar), sulfuric acid $(H_2SO_4 \ 95 \ %$, Prolabo), hydrofluoric acid (HF, 40 %, Riedel-de Haën), peroxide hydrogen $(H_2O_2, 30 \%)$, VWR chemicals), acetone (VWR chemicals), methanol (fisher chemicals), and hydrochloric acid (37 % VWR chemicals) used are of analytical grade, sodium carbonate (fisher chemicals) and sodium bisulfate (Sigma Aldrich) are also used for the treatment of the residue resulting from the dissolution. Tributyl phosphate (99%, Merck-Schuchart) and chloroform (99 %, Labosi) are purified by deionized water to remove sodium and orthophosphates for uranium extraction. XAD-7 extraction resin. FAAS impurity element standard solutions were obtained from Fluka (Fe, Ni, Cd, Zn, Mn, Li, Ca, K, V, Na, Pb, Co, Cu, Cd, Mg, Ti, 1g/L, High purity), Analar (Cr, $1g/L$, >99%) or Merck (Al, $1g/L$, >99%). Standard uranium solution (uranyl nitrate hexahydrate, > 99%, Merck). Ammonium hydroxide NH4OH (32 %, Scharlau) as well as Ammonium nitrate (BDH). The products used in the determination of uranium are potassium dichromate (BDH, 99 %), Mohr's salt (BDH, 98.50 %), iron sulfate heptahydrate (Merck, 99.50 %), sodium nitrite (Analar, 98 %) and urea (Fisher chemicals, 99.7 %), phosphoric acid (Sigma-Aldrich, 85 %) and the sodium diphenylamine sulfonated indicator (BDH). Chloroacetic acid (99 %, Sigma Aldrich), ethylene diamine tetra-acetic acid (99 %, Labosi), para nitrophenol (Aldrich), and arsenazo III (Fluka) were used in the determination of uranium by arsanazo III.

2.3 Materials

A branded peristaltic pump was used for filtration, and a

chromatographic column with a height of 25 cm and a diameter of 1.7 cm, with glass stopcocks, was used. A Janke & Kunkel Ika-Werk HS 500 shaker. The pH measurement was carried out by using a Hanna Instrument model 2210. A centrifuge Heraeus Lobofuge 601 model is used to separate liquid from material. A Prolabo oven is used for drying.

2.4 Procedure

Processes for separating uranium from impurities such as liquid-liquid extraction (ASTM C761-11) and extraction chromatography require the yellow cake to be dissolved and for the precipitation process, another dissolution is carried out.

2.4.1 Dissolution of Algerian yellow cake (ASTM C1347- 08) for SX and EXC

A portion of the Algerian yellow cake sample was taken in a 250-mL beaker, and moistened with distilled water; the latter was placed in a sand bath, and the nitric acid 8 M $HNO₃$ (50 mL) was added to the sample until dissolution. After 1 hour and a half of heating, the appearance of white particles in the solution shows that the latter is filtered (with Whatman ashless). The filtrate was placed in a 250 mL flask, and the residue on the filter paper was further processed [14].

2.4.1.1 Residue treatment

The collected filter paper was put into a platinum crucible, dried on a hot plate, and brought to a muffle furnace at 700°C for one hour for calcination. After one hour, the crucible is removed from the oven and allowed to cool. concentrated $HNO₃$, $H₂SO₄$ (9 M), and HF are added, and the crucible is filled with H_2O and heated until smoke $(SO₃)$ is released. After cooling, 2 mL of distilled water and 1 mL of concentrated $HNO₃$ are added. We notice that the solution is still cloudy and that there is a precipitate present. The treatment of the residue is continued by dry evaporation of the sample (platinum crucible); the crucible is removed and 0.5 g of NaHSO₄ is added, all in the muffle furnace until total melting, obtaining a white fusion. The crucible is cooled, dissolved with distilled water, and the contents are transferred to a beaker. Concentrated $HNO₃$ is added and heated until complete dissolution. After heating, a cloudy solution is obtained, which is filtered into the flask that contains the dissolved sample. The filter paper is collected, placed in a platinum crucible, dried on a hot plate, and then introduced into a muffle furnace at 700°C for one hour for calcination. We remove the crucible from the oven, cool it, add a volume of $Na₂CO₃$, which is 10 times the residue, and melt it in a muffle furnace until complete fusion. We remove the platinum crucible, place it in a beaker, cover it with distilled water, and slowly add concentrated $HNO₃$ until the reaction is complete. Remove the crucible from the beaker, using $HNO₃ (8M)$ to wash it. At the end, add 10 mL of concentrated $HNO₃$. It is heated until complete dissolution [14]. We note that the solution obtained is clear; the latter is transferred to the flask that contains the dissolved sample and a solution of impure uranyl nitrate (UNH) and uranium is analyzed by the potassium dichromate method [15].

2.4.2 Dissolution of yellow cake for the precipitation process

A 1.2594 g portion of the Algerian yellow cake sample was taken in a 250 mL beaker and treated to dryness twice with 5 mL of 8M HNO₃ nitric acid and 0.5 mL of perchloric acid on a sand bath. The content was digested using 1M nitric acid (50 mL), and it was filtered (after adding half a Whatman ashless tablet to prevent clogging of the filter paper due to silica). The filter paper was washed with hot 1M $HNO₃$ nitric acid (4 x 5 mL). The filtrate was collected in a flask and then adjusted to 125 mL with distilled water, and the filter paper was reserved for silica analysis [12]. The solution obtained has an acidic pH of 0.38.

2.5 Solvent extraction process

The sample solutions prepared are subjected to solvent extraction separation. 100 mL of nitrate d'uranyle impure solution of Algerian yellow cake is evaporated to dryness then the residue is redissolved in a solution of $6N HNO₃$ (100 mL). The sample solution was transferred to a 100 mL separating funnel and 50 mL of TBP and extracted for about 5 min. The phases were allowed to separate for a few minutes and the organic layer was separated and washed twice with 30 mL of 6 N $HNO₃$. The aqueous phase is transferred to a second separating funnel. The extraction process was repeated twice more. The second was with 50 mL of TBP-CHCl₃ 20%. Allow the phases to separate and remove the lower organic phase. Washed again the aqueous phase with 25 mL of CHCl_3 to remove residual TBP. The $HNO₃$ solution containing the elements to be determined is evaporated to dryness, and the residue is redissolved with 0.2 N HCl (fig.1). Any or all of the elements can then be determined in this one solution by standard atomic absorption spectrometry procedures [12].

2.6 *Chromatographic column extraction process*

A portion of the impure uranyl nitrate solution resulting from the dissolution must contain ≈ 1 g of uranium and be placed in a 50-mL beaker heated under a sand bath and evaporated to dryness. The latter is dissolved in 5 to 10 mL of 5.5M $HNO₃$, and in parallel, the blank is prepared in the same way, following the procedure for the sample. The sample and the blank will pass through the bed of the resin impregnated with TBP and elute with a 5.5 M HNO₃ solution to recover eluates E1 and E2 of 50 mL. The concentration in the initial solution was determined with the potassium dichromate method [14], and in the blank and eluates E1 and E2, it was carried out by the Arsenazo III method [16, 17] at λmax of 652 nm for uranium. The extraction efficiency of uranium Rext is calculated by the following equation:

$$
\text{Re}x = \left[\frac{Q_0 - Q_f}{Q_0}\right] \times 100\tag{1}
$$

 Q_0 : initial quantity determined by the potassium dichromate method in grams

 Q_f : final quantity of uranium in the Eluates or raffinate in grams determined by Arsenazo III.

2.6.1 Resin treatment

The resin used is Amberlite XAD-7, with 20-60 mesh purchased from Thermo Scientific. An optimization study of the parameters influencing the chromatographic separation, such as the resin washing reagents, impregnation time of the resin with TBP, drying time of the TBP in the oven, conditioning time of the resin in HNO3, and the height of the column bed was achieved. A portion of the resin was transferred into a glass funnel with filter paper, wetted with distilled water, it was washed with 1 M HNO₃, water, and finally washed with acetone, then dried in the oven at 50°C for 48 h. A 10 g amount of the washed XAD-7 was impregnated with TBP. The mixture was allowed to stand for at least 24 h to ensure good diffusion of the TBP inside the polymeric material, the excess TBP was removed using a pump and the resin was dried at 50 $^{\circ}$ C. The latter is conditioned in 5.5 M HNO₃ for 2 hours.

2.6.2 Column preparation

A glass column (25 cm x 1.7 cm I.D.) equipped with glass stopcock was employed. After closing the Teflon tap, a piece of glass wool is gently inserted into the end of the column without packing it. Enough resin treated and impregnated with TBP (XAD-7) was added to the column

to give a resin height of approximately 23 cm. The resin was allowed to settle in the column. This helped to remove any air bubbles formed when loading the column and allowed the resin beads to settle uniformly in the column. The resin bed was conditioned by adding 50 mL of the appropriate acid 5.5 M HNO₃ to the column with a transfer pipet and collecting the blank in a flask for analysis. The sample solution was slowly and carefully poured, without loss, onto the resin bed and the effluent was collected in a plastic bottle, the eluted with 50 mL 5.5M HNO₃ (E1). 5.5 M HNO₃ is then added until a second eluent of 50mL is obtained and collected in a second plastic bottle (E2). Uranium (VI) analysis in the blank and Eluates E1 and E2 by Arsenazo III are mentioned in the table 2.

2.7 Precipitation process

The hydrogen peroxide precipitation of uranium is an excellent method that occupies an important place in the separation of uranium from impurities. However, this method cannot be directly applied to yellow cake due to the presence of alkali metals (Na, Li, and K) and alkaline earth metals (Mg and Ca) as well as sulfate. Therefore, hydroxide precipitation is carried out before the peroxide precipitation for the removal of the above elements.

2.7.1 Determination of Na, K, Li, Ca, and Mg

A volume of 50 mL of the filtered solution above was taken into a beaker and diluted to 100 mL. The solution obtained has an acidic character. The precipitation of the hydroxide was carried out at pH 9 with an ammonium hydroxide solution. The solution is heated and then filtered through the Whatman No. 42 filter paper, and the filter paper is washed three times with a hot solution of 2% ammonium hydroxide in 3% ammonium nitrate (15 mL for each wash). The precipitate was redissolved with a minimal amount of nitric acid, and the filter paper containing the residue was reserved for uranium analysis. The filtrates and washes were collected in a beaker (250 mL) and then evaporated with wet salt in a sand bath (the ammonium salts were destroyed). The residue obtained is taken up with 0.5 M nitric acid (50 mL) (Solution F1) for the determination of the elements sodium, potassium, calcium, and lithium by flame photometry and magnesium by flame atomic absorption spectrophotometer (FAAS).

II.7.2 Determination of Al, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, V, Cu, Ti, Mo, and Zn

The second part of the Algerian yellow cake sample solution (50 mL) was taken into a beaker, and evaporated

with wet salt in a water bath, and the precipitation of uranium with hydrogen peroxide was carried out as described previously. The filter paper containing the residue obtained during the previous step (determination of the elements Na, K, Li, Ca, and Mg) was washed with a jet of water, and then with 15 mL of hot nitric acid (2 M). The solution becomes clear after heating. It is evaporated to dryness in a water bath. The residue was diluted with water, and 0.5 mL of glacial acetic acid was added. The pH of the solution obtained is 0.98; it was adjusted to 3 using diluted ammonia. The solution was then cooled in ice water (~2 °C), and 8 mL of hydrogen peroxide (H_2O_2) was added drop-wise, with constant stirring. After keeping the precipitate for one hour in ice water, it was filtered through Whatman 42, and the residue was washed three times with a 15 mL fraction of an 8% hydrogen peroxide solution. The filtrate was evaporated to dryness in a water bath, and the residue was dissolved in 50 mL of 0.5 M nitric acid. This solution is intended for the analysis of the elements Al, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Mo, V, Ti, and Zn by flame atomic absorption spectrophotometry

3. Results and Discussion

3.1 Determination of the efficiency of the solvent extraction process and results of an analysis of impurities in the raffinate

The extraction efficiency of uranium from the solution of impure UNH resulting from the dissolution of Algerian yellow cake was determined by analysis of uranium in impure uranyl nitrate by the potassium dichromate method and in the raffinate by the Arsenazo III method in triplicate, and the results are mentioned in Table 1.

Table 1: Uranium extraction efficiency from impure uranyl nitrate solution

Sample	$\mathbf{Q_0}$	Qf	Rext
	$\left(\mathbf{g} \right)$	$\left(\mathbf{g} \right)$	(%)
Algerian Yellow cake	1.9470	0.0085	99.65

The extraction efficiency obtained reveals that the extraction operation worked very well, the yield result is quite close previous work [6, 18] work with a uranium extraction yield of 99.9%. The analytical results obtained for elements in the raffinate obtained after uranium extraction by FAAS and Flame photometers are shown in Table 3.

3.2 Determination of elements after extraction chromatographic separation of uranium

A volume of the impure uranyl nitrate solution resulting from the dissolution of Algerian yellow cake equivalent to 0.5086g/U is placed in a Teflon cup; the latter is heated on

a sand bath until evaporation at dry, then dissolved with nitric acid at 5.5M \approx 5 mL; this volume will pass through the resin bed and elution with a $5.5M$ HNO₃ solution to recover eluates E1 and E2 of 50 mL and steps are already described above. The analysis of uranium in the blank and Eluates E1 and E2 by the Arsenazo III method is mentioned in the table below. The results are shown in the following table:

Table 2: Results of uranium extraction on a chromatographic column

Sample	Volume (mL)	Test sample (mL)	о (μg)	\mathbf{Q}_{UT} (g)	Extraction efficiency R(%)
Blank					
Eluats E1	50	0.1 0.1	4.700 4.405	0.0047	99.06
Eluats E ₂	50	0.1 0.1	4.843 4.981		

The various experiments carried out on the chromatographic extraction column using a pure uranyl nitrate solution at 1gU led us to achieve a yield of 99.65%

following the treatment of the resin beforehand with water, methanol, acetone and drying at 50°C, impregnation of the treated resin in TBP for 24 hours, removal of excess TBP using a pump, and drying of the resin at 50°C, conditioning of the treated and impregnated in 5.5 M $HNO₃$ for 2 hours, preparation of the sample in 5.5 M nitric acid, and the height of the column bed is 25 cm. An extraction efficiency of 99.06% is achieved with an impure uranyl nitrate solution from Algerian yellow cake under the same operating conditions. The analysis results of the different impurities in the eluates are shown in Table 3.

3.3 Determination of metallic impurities after separation of uranium by the precipitation process

The analytical results obtained for major elements, minor elements, and trace elements are shown in Table 3.

Table 3: Analytical results of elements by different processes

Table 3 compares the results obtained for the determination of impurities after separation of the matrix element by the different techniques described and the results of specific methods for each element with previous separation of uranium by solvent extraction, chromatography column extraction, and precipitation from Algeria yellow cake. The different results of the three processes were determined by comparison to ASTM C967-13 "standard Specification for

Uranium Ore Concentrate" [19]. This standard defines maximum limits for common UOC impurities relative to uranium concentration. Separate impurity concentration limits are specified without penalty and without rejection. Comparison of the results of the three separation methods used for each element is mentioned in the histogram below (fig 2).

Fig 1. Flow sheet the extraction steps according to the standard ASTM "C761-11"

Fig 2. Histogram of the levels of impurities in Algerian yellow cake after uranium separation by the three processes: solvent

extraction (ASTM C765-11), chromatography column extraction, and precipitation.

4. Conclusion

Although several preconcentration techniques are available for the enrichment of uranium (VI), the two most common traditional methods are solvent extraction and chromatography extraction. The removal of the uranium matrix serves many purposes, including the elimination of spectral interferences from OES or FAAS detection. A comprehensive study on the separation of U (VI) from a matrix by solvent extraction, resin-impregnated column and precipitation has been carried out in this study. The different processes used for the separation of uranium in order to quantify impurities in Algerian yellow cake using flame atomic absorption spectrometry (FAAS) and flame photometry (FP) were described, such as solvent extraction, chromatography column extraction, and precipitation, which showed the following results: The

efficiency of extraction was 99.65% and greater than 99 % for efficiency of extraction a chromatographic extraction column; the analysis results of elements separated by the three processes meet the contents required by standard ASTM C967-13 except for the element Iron., the analysis showed the absence of the elements Cd, Cr, Ti and Pb in the Algerian yellow cake and the values of the elements Mn, Zn and Li are quite close using the three processes, the analysis also showed that the values of the elements Mg, Cu, Ni are very close and identical for the element Co, only for the solvent extraction and chromatographic column extraction processes, the analysis showed that the value of

Al and Fe is of the same order for the two processes: solvent extraction and chromatographic column extraction. The results evaluated show that the two processes solvent extraction and chromatography column extraction are satisfactory for most of the elements analyzed.

The agreement is good for solvent extraction and chromatography column extraction.

Conflict of Interest

The authors declare that they have no conflict of interest.

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