Rapid Determination of Uranium in Ore Samples by Online Extraction Coupled ICP-MS

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ABSTRACT: Rapid analysis of uranium is useful for the exploration and exploitation of uranium ore, while traditional methods always require several hours for a single analysis procedure. Herein, a novel online extraction device that incorporates a heating unit was fabricated to efficiently extract the uranium from ore, followed by real-time ICP-MS detection. As a result, without sample pretreatment, the uranium content in two kinds of uranium ore can be obtained directly within 15 min with low sample consumption (1.0 mg), low energy consumption, and high recovery (90%). The present method provided a useful platform for the rapid quantitative analysis of uranium, which is beneficial for the improvement of the efficiency of exploration and exploitation of uranium.

INTRODUCTION

As a crucial metallic element, uranium plays significant roles in the fields of energy, medicine, military, etc. The global annual consumption of uranium is approximately 50,000 tons, while the currently identified resources recoverable are around 8 million tons worldwide.[1] Therefore, enhancing the exploration and exploitation efficiency of uranium mines is essential for the development of the global uranium industry. Advanced analytical methods are indispensable for improving the efficiency of exploration and exploitation, which can be used for determining the grade of uranium in ore samples, residual content in tailings as well and product quality.[2] Moreover, direct analysis of uranium can reflect changes in ore type and redox potential within an environment which directly affects leaching difficulty; thus, it holds great significance for mining operations, extraction processes, and environmental protection.[3, 4] Currently, uranium ore are normally dissolved using various chemical reagents to form analyte solutions prior to elemental analysis, The sample solutions are usually offline analyzed by radiological and chemical approaches, and mass spectrometry (MS). The radiological analysis encompasses α energy spectroscopy,[5,6] γ energy spectroscopy,[7, 8] neutron activation method,[9] *etc.*, while chemical analysis methods include volumetric method,[10,11]

spectrophotometry,[12,13] fluorescence spectroscopy,[14] X-ray fluorescence spectroscopy,[15-17] atomic spectroscopy,[18-20] and mass spectrometry.[21-24] Among these techniques, the volumetric method and spectrophotometry are cost-effective but exhibit relatively low detection sensitivity, while fluorescence spectrophotometry and atomic spectrometry are susceptible to chemical interference as well as spectral interference. Mass spectrometry is widely recognized as a standard method for ore analysis due to its outstanding advantages of high accuracy and sensitivity. However, traditional standard mass spectrometry methods required complex sample pretreatment, resulting in long analysis time, low efficiency, high cost, and energy consumption. Recently, our team has developed a direct analysis strategy for rare earth ore by mass spectrometry which enables rapid acquisition of component information without sample pretreatment.[25-28] This provides an important analytical tool for geological and mineral samples. However, the harsh extraction conditions for uranium in uranium ore make it difficult to achieve direct analysis using reported analytical methods.

Therefore, based on our previous research, a novel online extraction device that incorporates a heating unit was fabricated to extract the uranium from ore. The extraction was regulated by precisely controlling the extraction temperature. The extracted uranium transported online to ICP-MS for quantitative analysis by

coupling the ICP-MS with the extraction device. The present method has been demonstrated to enable the determination of uranium content in ore within 15 minutes without any sample pretreatment with a recovery above 90%. Compared to traditional mass spectrometry methods, our approach maintained analytical recovery and reduced analysis time and energy consumption by more than ten-fold and 100 times.

EXPERIMENTAL

Reagents, materials, and equipment. The high-resolution inductively coupled plasma mass spectrometer used in the test is Element II of Thermo Fisher Scientific Co., Ltd. And the instrument equipped with a standard glass nebulizer, quartz spray chamber. The instrument conditions were: atomizer gas flow rate of 1.1 $L \cdot min^{-1}$, auxiliary gas flow rate of 0.9 $L \cdot min^{-1}$, plasma gas flow rate 16.0 $L \cdot min^{-1}$, RF power 1350 W, sampling cone aperture of 1.2 mm, interception cone aperture of 1.0 mm. The sample is weighed using an analytical balance (MS105DU of Mettler Toledo) with an accuracy of 0.01mg.

As shown in Fig.1(a), the sample chamber consists of five major parts: filter, filter paper, gasket, fastening, and adapter. Except for the filter paper and gasket, which are made of Polytetrafluoroethylene (PTFE), the other three parts are made of poly(ether-ether-ketone) (PEEK). Briefly, the inner and outer diameters of the filter body were 8.00 and 14.00 mm, and the inner and outer diameters of the filter tip were 1.80 and 0.80 mm. The diameter of the filter paper was 8.00 mm. The inner and outer diameters of the gasket were 4.00 and 8.00 mm. The height of the gasket was 2.00 mm. The internal diameter of the adapter was 1.20 mm. The sample chamber is connected via threading, and the analysis concludes with a replacement of the sample chamber. The membrane filter is made of Teflon and cut with an 8 mm circle hole puncher. The pore size of the membrane filter is 0.45μm.

The reagents H_2O_2 (68%), HNO_3 (68%), HCl (37%), $HClO_4$ (71%) and HF (40%) used in the experiment are guaranteed reagent and purchased from Sinopharm Chemical Reagent Co., Ltd. Uranium standard solution (GBW(E)080173) was purchased from the Beijing Institute of Chemical Metallurgy of Nuclear Industry. Two ores were used in this work, including a standard sample of granite (GWB04122) and sample of alaskites from Husab uranium deposit in Namibia (nami-01). The component of the GBW04122 was presented in the Table S1. The SiO_2 content of GBW04122 is 75.18%. The SiO_2 content of Nami-01 sample is above 75%. The ultrapure water (>18 $M\Omega \cdot cm^{-1}$) was prepared by the Millipore ultrapure water system. Glassware for analysis was first soaked in 30% nitric acid solution for 24 h and then washed ultrasound with ultrapure water. The plastic container was used for the sample solution in the experiment. The standard solution was stored in a PFA bottle and was diluted in plastic containers.

Fig. 1 The strategy for uranium rapid analysis of ore sample. a) Major parts of the sample chamber for online extraction of ore samples for ICP-MS detection; b) The schematic diagram of the procedure for the rapid analysis.

Calibration curve. Calibration solutions of uranium with concentrations 2, 6, 10, 20, 60, and 100 $\mu g \cdot L^{-1}$ were prepared in 2% HNO_3 from standard solution. The ICP-MS signals of ^{238}U were used for quantitative analysis. The calibration curve was prepared in two modes respectively, including signal intensity vs. uranium concentration and signal area vs. uranium mass. In signal intensity vs. concentration mode, the concentration of each calibration solution of uranium was set as horizontal coordinates and the corresponding intensity was set as vertical coordinates. In signal area vs. mass mode, 200 μL calibration solutions with different concentrations were successively introduced to the ICP-MS with a flow rate of 0.3 $mL \cdot min^{-1}$, and the ^{238}U was recorded in real time. The area of the ^{238}U signal was calculated by the integration method in Origin 9.0 software. The mass of the U of the corresponding ^{238}U signal was calculated as M=C*V, where "C" was the concentration of the U, and "V" was the dosage of the calibration solution. Then, the mass of uranium was set as horizontal coordinates, and the corresponding signal area was set as vertical coordinates.

Direct determination of uranium in an ore sample. The ore samples were grinded and sieved using a 200-mesh sieve with a maximum diameter of 74 μm, following the standard protocol (DZ/T 0279.6-2016). [29] As the sample diameter decreases, there is an increased likelihood of contact with the reagent and a greater ease in eluting the element for measurement. However, excessively small particle diameters can lead to filter paper clogging and elevated pressure within the self-made reactor, which may impede reaction efficiency. After that, the execution of sample loading procedure was conducted. As shown in Fig. 1a, the filter paper was initially placed into the filter and securely attached to the pre-moistened sieve. Subsequently, the gasket was carefully positioned onto the filter paper. A quadrate shaped weighing paper was placed on top of the measuring plate of the analytical balance. Then the sample of 1.00 mg was measured out by placing the sample on the paper. After that, the weighing paper was curled up to form a sharp conical tip. The paper was then tilted up and the sample was transferred onto the filter paper by tapping the paper. Following this step, the fastener was inserted into the filter to firmly secure the gasket in place. Lastly, an adapter was affixed to complete the assembly of the entire device.

Determination of the uranium. The instrument's baseline was initially adjusted before commencing the analysis of the mine sample. Subsequently, the experimental apparatus was arranged as depicted in Fig. 1b. The eluent passed through the heater and entered the sample chamber to facilitate U extraction from the ore sample with a flow rate of 0.3 $mL \cdot min^{-1}$. The temperature inside the elution device can be assessed by measuring the temperature of the eluent in the exit of the sample chamber, and the heater was adjusted to make the temperature of the eluent in the exit of the sample chamber was 90 °C. Non-spectral interference can be caused by difference in viscosity, surface tension, density, and in the concentrations of matrix elements in the individual samples, and it can be reduced by diluting sample, internal standard and removing matrix etc.[30] Thus, in this work, a simple method on-line dilution was used to reduce the non-spectral interferences. The extracted uranium was mixed online with 2% HNO_3 through a Y connector (PEEK), followed by being transported to a nebulizer for ICP-MS detection. The signal of ^{238}U was recorded in real-time. Though other elements like silicon, alkali and alkaline earth metals, transition elements, lanthanides, etc may be extracted along with uranium, the relative atomic mass of ^{238}U mass is larger than that of these elements, and the interference caused by these elements were negligible. The determination was completed until the signal of ^{238}U decreased to baseline. The chamber can be reused by changing the filter paper and cleaning the chamber adequately with aqua regia.

The results achieved by the present method are compared with those obtained by the standard method (DZ/T 0279.6-2016).[29] The standard method is as follows. Accurately weigh 0.1 g (with an accuracy of 0.1 mg) powder sample with particle size less than 74 μm. Then place it in a 50 mL Teflon beaker followed by the addition of 10 mL nitric acid, 5 mL hydrofluoric acid, 2 mL perchloric acid, and a small amount of water. After that, the beaker is covered using a crucible cover and heated up for 1 h using a temperature-controlling electric rod at 150 °C. Following that, the crucible cover is taken away and cleaned using water. The open beaker is then heated up to 240 °C and kept heated until the white perchlorate smoke stops coming out. While hot, the beaker walls are flushed using 6 mL 5% nitric acid and then kept warm for 5-10 min until the solution turns clear. Then the solution is cooled down and transferred to a 50 mL volumetric flask for test with the volume brought by using high-purity water.

RESULTS AND DISCUSSION

Calibration curve. According to the methodology described in section 2.2, two kinds of standard curves were generated. Fig. 2

Fig. 2 Calibration curve of U prepared in signal intensity vs. concentration mode.

displays the calibration curve prepared in signal intensity vs. concentration mode. The equation for the calibration curve is $y = 26598x + 1751$, with a linear range of 0-100 $\mu g \cdot L^{-1}$ and a correlation coefficient of 0.9997 within the range. The detection limit was calculated to be 12.1 ng· L^{-1} according to LOD=3 σ/a, where "σ" was the standard deviation of the blank solution and the "a" was the slope of the calibration curve.

Figure S1a displayed the extracted ion chromatogram of ^{238}U with different concentrations and a calibration curve in signal area vs. mass mode was displayed in Fig. S1b. The linear equation was $y = 6.88 \times 10^7 x + 5.12 \times 10^6$ with a linear range of 0-20 ng and a correlation coefficient of 0.9999 within the range. The detection limit (LOD) was calculated to be 25.4 pg according to LOD=3σ/a, and the limit of quantitation (LOQ) calculated to be 84.7 pg according to LOQ=10σ/a. These two kinds of calibration curves demonstrated that the ICP-MS has a high sensitivity for ^{238}U detection.

On-line analysis of uranium ore. The national standard sample (uranium-producing rock composition analysis standard GBW 04122) and Namibia Husab Uranium Deposit white granite uranium ore were employed to characterize the performance of the present method. According to the methodology described in section 2.3, 5% nitric acid (0.80 mol·L^{-1}) + 5% hydrogen peroxide (0.33 mol·L^{-1}) was first used as the eluent to extract uranium from the ore sample for ICP-MS detection. The eluent was selected according to the previous reports.[31, 32] Santos et al.[31] reported that the most ordinary way for decomposition of solid samples for uranium determination is employing acids such as nitric acid, hydrofluoric acid and hydrogen peroxide, as well as mixtures of such reagents, and a maximum concentration of 5% nitric acid was recommend. Hickam et al.[32] reported that uranium nitride and dioxide uranium dissolution in aqueous solutions containing hydrogen peroxide is extensive and rapid, but when the concentration is too large, it is easy to form uranyl peroxide cluster. Herin, combination of the previous reports, 5% nitric acid and 5% hydrogen peroxide was selected as eluent.

Figures 3a and 3b displayed the detection results of uranium in three parallel GBW 04122 and Namibian uranium white granite ore samples, respectively. Due to the micro-scaled device and online process, the speed and efficiency of extraction were enhanced. Consequently, the sample consumption and/or sample loss were both minimized referring to conventional offline methods. As shown in Figs. 3a and 3b, the signal increased sharply to the apex within about 2 s and then decreased slowly to the baseline within about 700 s, the single experimental procedure can be completed within 15 min with only a small volume of extraction solvent (~4.5 mL), resulting in a relatively high concentration and a strong signal although only a small amount of sample (1.0 mg) was used. In comparison to a previously reported online extraction-based ICP-MS method,[33] where a complex

Fig. 3 Direct analysis spectrum of ore samples. a) The uranium rock composition analysis reference material (GBW 04122); b) The alaskite uranium deposit in Namibia.

sample pretreatment was conducted to prepare an analyte solution followed by the utilization of liquid-liquid extraction for trace analyte element purification by eliminating matrix elements, the current approach enables direct U extraction from ore for high sensitivity ICP-MS analysis.

Obvious fluctuation can be observed in the extracted ion chromatogram, which can be ascribed to the interference of microbubbles generated during the extraction. The quantitative results of the U in both GBW 04122 and Namibian uranium white granite ore samples were displayed in Table S2 according to the linear equation shown in Fig. S1b and the signal area provided in Fig. S1a. As shown in Table 1, the content of U in GBW 04122 and the Namibian sample were detected to be 60.81 $\mu g \cdot g^{-1}$ and 1322 $\mu g \cdot g^{-1}$ with an RSD of less than 3% in three parallel tests.

In order to investigate the effect of the quantitative mode, an offline analytical mode was carried out. A mixture of 5% nitric acid + 5% hydrogen peroxide was passed through the heater and entered the sample chamber to extract U from the ore sample with a flow rate of 0.3 mL/min. The extracted uranium was then mixed online with 2% HNO_3 through a Y connector (PEEK), followed by collected in a vial. After 15 min extraction, the collected U

Table 1 Analytical result of uranium in ore samples by different methods

Method	GBW 04122		Nami-01	
	$\bar{x}$	RSD /%	$\bar{x}$	RSD /%
Online	60.81	1.21	1322	2.40
Off-line	61.57	2.41	1366	1.88
HF added	62.23	1.33	1455	2.03
DZ/T0279.6-2016	64.11	3.06	1516	2.39

extraction solution was used for ICP-MS detection. The quantitative information of the U was obtained (Table S3) according to the ICP-MS results and the linear equation shown in Fig. 2. As shown in Table 1, the content of U in GBW 04122 and the Namibian sample were detected to be 61.57 µg·g⁻¹ and 1366 µg·g⁻¹ with an RSD of less than 3% in three parallel tests. As shown in Table 1, the quantitative information respectively obtained by the two quantitative modes was almost the same, indicating that the quantitative mode almost did not affect the results.

According to the previous reports, HF can react with silicon of matrix in the ore sample and a concentration of 0.05 mol·L⁻¹ HF is conducive to the dissolution of uranium oxides.[34] Thus, a mixture of 0.2% hydrofluoric acid (0.06 mol·L⁻¹) + 5% nitric acid + 5% hydrogen peroxide was used as eluent to investigate the effect of eluent on the results. As shown in Table S4 and Table 1, the content of U in GBW 04122 and the Namibian sample was detected to be 62.23 µg·g⁻¹ and 1455 µg·g⁻¹ with an RSD of less than 3% in three parallel tests. As shown in Table 1, the extraction efficiency of U for GBW 04122 and the Namibian sample were improved 1% and 6% respectively by using an eluent contained of HF, indicating that a part of U in the sample was wrapped by silicon. Moreover, the increment of extraction efficiency of U for Nami-01 and GBW04122 consisted with their SiO₂ content.

Sample analysis. A geological industry-standard method (DZ/T 0279.6-2016) (detailed in the experimental section) was used to validate the accuracy of the present method. The results of uranium content in GBW 04122 and the Namibian sample obtained by the geological industry-standard method were displayed in Table S5. As shown in Table S5 and Table 1, the content of U in GBW 04122 and Namibian sample were detected to be 64.11 µg·g⁻¹ and 1516 µg·g⁻¹ with an RSD of about 3% in three parallel tests. Giving the results obtained by the geological industry-standard method as true value, the recovery of the present method was displayed in Fig. 4 according to the formula: recovery = the results obtained by this method/the results obtained by standard method ×100%. The recovery of the one-step extraction rate for GBW 04122 and Namibian samples without HF was 87% to 96%, whereas incorporating a small amount of hydrofluoric acid (1 mL 2%) increased the recovery to between 93% and 97%. In comparison with the reported methods (Table S6), the present method shows a satisfactory extraction efficiency for U detection in ore.

Fig. 4 The recovery of the present method compared with the geological industry-standard method. Recovery = the results obtained by this method/the results obtained by standard method ×100%.

Compared with traditional analysis methods (Table S7), the present approach obviates the requirement of pretreatment, resulting in a mere 1% energy consumption. Moreover, the time required for analysis is reduced by a factor of 10, and reagent consumption is also minimized to just 1%.

CONCLUSION

In summary, the quantitative determination of uranium in unprocessed ore samples has been successfully achieved within 15 minutes using ICP-MS combined with a custom-designed apparatus for real-time extraction of the uranium element, thereby eliminating the need for laborious offline sample preparation. In comparison with conventional methods, the present method allows significant advantages such as high speed of analysis (0.2 h vs. ~3 h), low sample consumption (1.0 mg vs. 0.5 g), high recovery (>95%), low cost, and low energy consumption. These results demonstrated that the present method is a useful platform for the rapid quantitative analysis of uranium, which is beneficial for the improvement of the efficiency of exploration and exploitation of uranium. In addition, the present method can also be used for analysis of other geological samples such as metal ore samples, fossils, soil, which is useful for geology, paleobiology, and Environics. The reason why this method has such a high extraction efficiency is that the sample amount in the experiment is small, the reactants for online analysis are constantly supplemented, the concentration is not reduced, and the products continue to flow out, and the chemical reaction in the reaction chamber is always in the direction of the forward reaction, which is the biggest advantage of this method compared with the traditional digestion method.

ASSOCIATED CONTENT

The supporting information (Fig. S1 and Table S1-S7) is available at www.at-spectrose.com/as/home.

AUTHOR INFORMATION

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Notes

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