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A new desorption method of polyurethane foam for the determination of gold and a comparative study on four desorption methods based on meta-analysis

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Accurate determination of gold in geological samples is an important prerequisite and guarantee for studying geological problems. There are many methods for digestion and enrichment of gold among which polyurethane foam (PUF) enrichment after aqua regia digestion is the most commonly used in the experiments. A new method to help the relief of gold from the PUF was put forward in this study, and it was applied to four certified reference materials (CRMs) together with three previously used methods, and the optimal extraction and enrichment conditions were determined through experiments. The four methods were compared by meta-analysis, and the thiourea liberation method was superior to the other three methods because of its simple operation and high accuracy. Out of consideration for the incomplete adsorption of gold in the solution by only one piece of PUF, repetitive adsorption of gold with a second and a third piece of PUF in the solution was proposed in this study. Results show that the gold content obtained by secondary and tertiary adsorption accounts for 11.03% of the total content, and the highest can reach 20.74%. When the third adsorption was carried out, the gold content in several samples was below the detection limit. Therefore, repeated adsorption of gold in the solution is necessary, and three times of adsorption is necessary.

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1. Introduction

Gold is a noble metal with high ductility, corrosion resistance and chemical stability. It has important applications in geology, materials, chemistry, biology, medicine and other fields because of its special properties. Studies of the source, enrichment process, and state of gold are of great guiding significance in gold exploration. However, the distribution of gold in various geological samples is uneven and often exists in a low content which makes the separation, enrichment,¹ and accurate determination of gold a difficult problem in geological analysis.

The treatment methods before gold determination include dry ashing,^{2–4} and wet acid digestion. Enrichment, such as precipitation,⁵ ion exchange resin,⁶ solvent extraction,^{7–9} PUF adsorption,^{10,11} extraction chromatography,¹² is followed. There are many testing methods for gold, including atomic absorption spectrometry,¹³ inductively coupled plasma mass spectrometry^{14,15} and so on. However, the uncertainty of gold determination is often high due to incomplete sample digestion, incomplete adsorption and desorption process of gold, high

detection limit of instruments, and interference of instrument matrix.

PUF was widely used in various field^{16–18} and was first applied to adsorb gold in acidic medium in 1970 by Bowen.¹⁹ PUF is made of toluene diisocyanate and polyether polyol. The carbon dioxide generated in the synthesis process is left in the polymer to form spongy foam plastic with a certain degree of cross-chain. When the gold in the solution meets the foam, adsorption and exchange occur on the foam membrane potential. The coordination anions of gold would be bonded with the active groups of $-\text{CH}_2-\text{O}-\text{CH}_2$ and $-\text{O}-\text{C}-\text{NHR}$ on the foam skeleton, so that the two would combine to form a stable ionic association.^{20–23} In recent years, PUF has been widely used in the determination of trace gold in geological samples because of its strong adsorption performance and low price. Desorption is required before determination. There are several methods of desorption of gold, so it is of great significance to choose the best way among them.^{24–27}

Meta-analysis is widely used in medicine, psychology and other fields to conduct comprehensive quantitative analysis of many research results of the same subject with specific conditions, which is superior to the comprehensive analysis ability of conventional literature review.²⁸ However, it is rare in the study of geological phenomena.^{29–32} Meta-analysis is applied to the correlation between a specific exposure factor and a specific

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outcome in medical analysis.^{33–35} It is feasible to transform the original geochemical data into valid data which can be identified by meta-analysis, and to obtain the mean differences of four different methods so as to compare different methods.

The desorption methods of PUF were discussed and a new method of desorption of PUF was proposed. Meta-analysis was applied to compare the four desorption methods of PUF. Considering the incomplete adsorption of one piece of PUF, the necessity of repeated adsorption was put forward and verified. This study aims to establish an accurate, simple, and rapid process for the determination of gold in geological samples through a comparative study of sample digestion method, the time of PUF adsorption, the way of PUF desorption.

2. Materials and methods

2.1. Reagents and standards

Hydrochloric acid (HCl), nitric acid (HNO₃) of analytical grade and deionized water were used in sample digestion. 250 g L⁻¹ ferric chloride solution with 1% HCl was used during adsorption. 10 g L⁻¹ thiourea solution, 200 g L⁻¹ potassium chloride solution and 500 g L⁻¹ potassium bromide solution, methyl isobutyl ketone (MIBK), and potassium chlorate of analytical grade were employed in the desorption process.

National standard gold single element solution (GSB-1715-2004, 1000 mg L⁻¹) was acquired from the National Center of Analysis and Testing for Nonferrous Metals and Electronic Materials. CRMs (GBW07248a, GBW07808b, GBW07809b) were obtained from the Institute of Geophysical and Geochemical Exploration, Chinese Academy of Geological Sciences (Langfang, China). CRM GBW07192 was obtained from the Central South Institute of Metallurgical Geology (Yichang, China).

2.2. Instrumentation

Samples were triturated by a planetary ball mill (QM-3SP4, Laibu, China) and dried in an air-dry oven (DHG-9423A, Shanghai Jinghong, China) at 105 °C. Cooled sample powder was weighed with an electronic balance (ATY124, Shimadzu, Japan) and ashed in a muffle furnace (SXL-1008, Shanghai Jinghong, China). The electric heating plate (SB-1.8-4, Shanghai Shiyan, China) was used in sample digestion. The cyclotron oscillator (HY-8A, Jintan Jingda, China) was used to ensure the gold was absorbed by PUF completely. The constant temperature water bath (HH-S26S, Jintan Instruments, China) was employed in the desorption of gold. Gold determination was performed on an inductively coupled plasma mass spectrometry (ICP-MS) (Nexion 350D, PerkinElmer, USA). The operating parameters of ICP-MS were listed in Table 1.

3. Experiments

3.1. Pretreatment of PUF

The internal residues left during the production of PUF such as carbamido, allophanate, biuret, and isocyanate will greatly reduce the adsorption performance of the PUF which leads to the degradation of the adsorption performance. Hence, it is necessary to pretreat the PUF before the adsorption experiment. First, the PUF was cut into small squares of about 0.2 g (3 × 2 × 1 cm³), soaked into sodium hydroxide solution and boiled for 30 minutes, washed with deionized water until neutral. Then, it was transferred into 10% HCl and soaked for 2 h, washed again and dipped in deionized water for 2 h, squeeze the water out, dried naturally for later use.³⁶

3.2. Sample digestion

Herein, the high-temperature ashing-acid digestion and PUF adsorption were performed to determine the content of gold in rock samples according to the geological and mineral industry standard DZ/T 0279.4-2016 of the People's Republic of China. 10.0000 g of sample was accurately weighed into a porcelain crucible and sent to a muffle furnace for 2 h at 700 °C, during which the furnace was opened twice for oxygen supplement to ensure that the sample powder was ashed completely. After cooling, the sample was transferred to a conical flask, several drops of water were added to moisten the powder, 30 mL 50% aqua regia was added to dissolve the sample, place the conical flask on the electric heating plate with the surface dish covered. Remove the surface dish one hour later and continue to evaporate until the solution is about 10 mL. 70 mL water and 3 mL FeCl₃ were added with a PUF to the cooling solution. The conical flask was then placed on the cyclotron oscillator for 30 minutes.^{10,11} The PUF was taken out, the residue was washed and squeezed for later use.

3.3. Desorption of gold

Four methods of desorption of gold from PUF were performed in the experiment, among which method III was a new method proposed in this study.

3.3.1. Method I (HNO₃-KClO₃ decomposition). A new method proposed in this study: PUF could be decomposed by inorganic acid and oxidizing agent, among which nitric acid and potassium chlorate have the best decomposition effect.³⁷ Specific operations are as follows: the PUF after adsorption of gold was placed into a glass beaker where 0.1 g KClO₃ and 10 mL HNO₃ were added. The beaker was then placed on a hot plate until the solution evaporates to dry during which a lot of

Table 1 Operating parameters of ICP-MS

Parameter	Setting	Parameter	Setting
Radiofrequency power/W	1150	Sampling cone aperture/mm	1.2
Cooling gas flow/(L min ⁻¹)	18	Skimmer cone aperture/mm	1
Auxiliary gas flow/(L min ⁻¹)	1.2	Scanning times	20
Nebulizer gas flow/(L min ⁻¹)	0.92	Sampling time/s	60

NO₂ gas was released. 10 mL 50% HCl was added and continued heating until solids dissolve, the solution was transferred into a 50 mL colorimetric tube after cooling, deionized water was added to scale for direct determination.

3.3.2. Method II (thiourea liberation). Au (III) could be reduced to Au (I) by hot thiourea solution and combined to produce the Au (I) – thiourea complex in which process the gold ions can be liberated from the PUF. The specific operation is as follows: the PUF was transferred into a colorimetric tube where 10 mL thiourea solution was added, placed the tube in a boiling water bath for 30 minutes, during which the PUF was continuously extruded with a rubber-tipped glass rod so that the gold could be liberated completely. Squeeze the PUF as many times as possible but be careful not to break the bottom of the colorimetric tube which may lead to a loss of solution. Squeeze and remove the PUF immediately while it is still hot. The solution was cooled for determination.

3.3.3. Method III (ashing burning). The PUF was placed in a porcelain crucible and sent to a muffle furnace for 2 h at 600 °C after two drops of anhydrous ethanol were added to improve ashing efficiency. The residue was transferred into a beaker after being cooled and two drops of potassium chloride solution and 3 mL aqua regia were added, placed the beaker on a boiling water bath until the solution evaporated thoroughly. Ten drops of HCl were added to remove the HNO₃, the solution was transferred to a colorimetric tube for determination.³⁸

3.3.4. Method IV (organic solvent extraction). The PUF was placed in to a glass beaker where 0.1 g KClO₃ and 10 mL HNO₃ were added. The beaker was then placed on a hot plate until the solution evaporates to dryness. 10 mL 50% HCl was added to dissolve the solids, the solution was transferred into a 25 mL colorimetric tube with 1 mL 50% fresh potassium bromide solution added, dilute to 20 mL with deionized water and 5 mL MIBK was added, gold was determined in the oil layer after shaking stratification.³⁹

4. Results and discussion

4.1. Validation of methods

Four CRMs with different content of gold were digested and absorbed under the same condition and extricated with four different methods. The logarithmic deviation ($\Delta \lg \bar{C}$) between the measured mean value and the standard value of the CRMs and the relative standard deviation (RSD) between the measured value and the standard value of the CRMs were calculated to measure the accuracy and precision of the methods according to the geological and mineral industry standard DZ/T 0011-2015 of the People's Republic of China. $\Delta \lg \bar{C}$ and RSD can be evaluated by the following equations:

$$\Delta \lg \bar{C} = |\lg \bar{C}_i - \lg C_s| \quad (1)$$

$$\text{RSD} = \frac{\sqrt{\frac{\sum_{i=1}^n (C_i - C_s)^2}{(n-1)}}}{C_s \times 100\%} \quad (2)$$

where \bar{C} is the mean of parallel measurements, C_i is the value of parallel measurements, C_s is the standard value of CRM, $n = 5$ is

the number of parallel experiments. The $\Delta \lg \bar{C}$ and RSD acquired are all less than 0.11 and 10% respectively, indicating that the accuracy and precision of the methods are qualified.

4.2. Comparison of methods

The gold in the sample was absorbed three times of oscillating and the desorption of PUF was performed with the above four different methods. Parallel experiments were carried out five times under the same conditions, and the experimental results were shown in Table 2.

All the above four methods can meet the test requirements, among which the method I is highly accurate and efficient which is suitable for laboratory testing except that it requires a large amount of acid and is dangerous to operate. Method II has been widely applied in geological samples determination especially during laboratory analysis and testing with high accuracy. This method requires the use of a glass bar to squeeze the PUF which leads to the fracture of the bottom of the colorimetric tube and the loss of the sample, so it is recommended to use a glass bar with a rubber plug at one end and squeeze the PUF for more than 200 times. Method III is relatively simple and convenient to operate with small consumption of reagent which is especially suitable for the extraction of large quantities of gold during industry work but has the disadvantages of cumbersome transfer extraction, long process, high energy consumption of muffle furnace. It is easy to cause the problem of unqualified accuracy and precision of method IV due to the unstable absorbance of the instrument of organic solvents and the use of organic solvents in the process is not environmentally friendly. In addition, it is unkind for ICP-MS to determine the content of solution with organic reagent as solvent.

4.3. Meta-analysis

In the meta-analysis, when the probability of heterogeneity test is $P > 0.05$, multiple independent studies can be considered to

Table 2 Measurement results of gold in CRMs

CRM	Standard value ($\mu\text{g g}^{-1}$)	Method	Content ($\mu\text{g g}^{-1}$)	Recovery rate (%)	Relative error (%)
GBW07192	15.100	I	15.278	101.18	1.18
		II	14.130	93.58	6.42
		III	14.418	95.48	4.52
		IV	14.298	94.69	5.31
GBW07248a	0.101	I	0.107	105.96	5.96
		II	0.102	101.41	1.41
		III	0.105	103.80	3.80
		IV	0.105	103.96	3.96
GBW07808b	3.300	I	3.353	101.61	1.62
		II	3.253	98.58	1.42
		III	3.280	99.39	0.61
		IV	3.245	98.32	1.68
GBW07809a	10.800	I	10.771	99.73	0.27
		II	10.706	99.13	0.87
		III	10.744	99.48	0.52
		IV	10.443	96.69	3.31

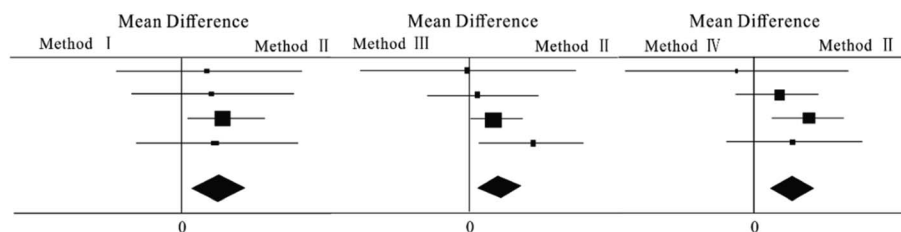


Fig. 1 Forest plots of meta-analysis for four desorption methods.

Table 3 Meta-analysis results of four methods^a

Treatment group	Control group	Heterogeneity test	Overall effect test	Significance
Method I	Method II	$P = 0.52$; $I^2 = 0\%$	$P = 0.006$; MF = 0.02	Significant
Method III	Method II	$P = 0.99$; $I^2 = 0\%$	$P = 0.006$; MF = 0.02	Significant
Method IV	Method II	$P = 0.57$; $I^2 = 0\%$	$P = 0.004$; MF = 0.03	Significant

^a I^2 is the statistic; P is the probability of the statistic.

be homogenous, and a fixed effect model can be selected to calculate their combined statistics. When the probability of the population effect test is $P < 0.05$, it is statistically significant. The diamond at the bottom of the forest plot represents the combined results of multiple randomized controlled trials (RCTs). The figure was divided into two halves, left and right, to judge whether the difference is statistically significant. The left side of the line is the experimental group, namely the treatment group, and the right side is the control group. When the diamond intersects the vertical line, there is no statistical significance between different methods in the RCT. The position and meaning of the diamond are as follows: for adverse outcomes such as disease events and death, when the diamond is completely on the left of the vertical line, the treatment group is more effective; when the diamond is completely on the right,

the control group is more effective; For favorable outcomes such as remission and cure, the position and meaning of the diamond are opposite; The greater the distance of the rhombus from the vertical line, the more obvious the effectiveness difference between the two schemes.

In this study, four methods were used to test the relative errors and relative standard deviations of four samples for meta-analysis. The forest map obtained is shown in Fig. 1. The analysis results are shown in Table 3. Results indicated that the four independent methods were homogeneous, and the probability of the overall effect test was less than 0.05, which had significant statistical significance. All the three mean differences were greater than 0, the mean difference of the method II and method III are equal in value and both less than method IV, which indicated that the effect of method I in the control group

Table 4 Measurement results of gold in CRMs

CRM	Method	Time of adsorption				Sum ($\mu\text{g g}^{-1}$)		
		1 ($\mu\text{g g}^{-1}$)	ratio (%)	2 ($\mu\text{g g}^{-1}$)	ratio (%)			
GBW07192	I	14.280	93.47	0.768	5.03	0.230	1.51	15.278
	II	13.669	96.74	0.443	3.14	0.018	0.13	14.130
	III	13.560	94.05	0.630	4.37	0.228	1.58	14.418
	IV	12.961	90.65	0.947	6.62	0.390	2.73	14.298
GBW07248a	I	0.086	80.37	0.015	14.02	0.006	5.61	0.107
	II	0.086	84.31	0.014	13.73	0.002	1.96	0.102
	III	0.083	79.05	0.019	18.10	0.003	2.86	0.105
	IV	0.084	80.00	0.017	16.19	0.004	3.81	0.105
GBW07808b	I	2.992	89.23	0.320	9.54	0.041	1.22	3.353
	II	2.883	88.63	0.342	10.51	0.028	0.86	3.253
	III	2.840	86.59	0.359	10.95	0.081	2.47	3.280
	IV	2.831	87.24	0.392	12.08	0.022	0.68	3.245
GBW07809b	I	10.102	93.79	0.554	5.14	0.115	1.07	10.771
	II	10.281	96.03	0.339	3.17	0.086	0.80	10.706
	III	9.684	90.13	0.948	8.82	0.112	1.04	10.744
	IV	9.713	93.00	0.689	6.60	0.041	0.39	10.443

was more significant than that in the experimental groups. Method II and method III are more accurate and effective than method IV under the experimental conditions set up in this study.

Of course, the experimental conditions employed in this study including the time and temperature of adsorption, desorption, and ashing burning, the amount of oxidant may introduce a certain degree of impact on the measurement results, which deserved to be explored and optimized in the subsequent study.

5. Repetitive adsorption

In order to avoid the incompleteness of single adsorption of PUF which led to the inaccurate determination of gold, the gold in the solution was adsorbed three times under the premise of ensuring the same PUF treatment mode, adsorption mode and foam treatment mode in this experiment, and finally, the sum of the three adsorption times was obtained as the total gold content.

It can be seen from Table 4 that the gold content determined after the second and third adsorption accounted for more than 3.26% of the total gold content, with an average of 11.03% and a maximum of 20.74%. Among them, the gold content determined after the second adsorption accounted for more than 3.14% of the total gold content, with an average of 9.23% and the highest of 16.19%. After the third adsorption, the determined gold content decreased significantly, with an average value of 1.79%. When the third adsorption was carried out, the gold content of some samples was already below the detection limit.

6. Conclusions

The new method of the desorption of gold proposed in this study has the advantages of simple operation and time-saving. The accuracy and precision of all these four methods can meet the test requirements. The reagent dosage, proportion and detailed operation process of each method were proposed, which can provide a basis for the determination of gold in geological samples.

The four PUF desorption methods were compared and the thiourea liberation method was determined as the best method to help gold relieve from the PUF by meta-analysis. The advantages of this method are high accuracy, high precision, and simple operation. Ashing burning method and HNO_3 - KClO_3 decomposition method followed, while the organic solvent extraction method is not stable enough and the introduction of organic solvents was not environmentally friendly. The thiourea liberation method and HNO_3 - KClO_3 decomposition method with high accuracy and efficiency were suitable for laboratory analysis and determination while the ashing burning method can be employed in industry work. Meta-analysis can quantitatively compare the results of different methods which has immeasurable application potential in the geochemical field.

In this study, repeated adsorption of gold in the solution was proposed. Repeated adsorption experiments were carried out for each desorption method. Experiment results showed that the gold content of secondary adsorption and tertiary adsorption accounted for 11.03% of the total content, and the highest was 20.74%. Moreover, the lower the gold content in the sample, the higher the ratio of determining gold content to total content after repeated adsorption. Therefore, it is necessary to carry out repeated adsorption of PUF under the adsorption and desorption conditions of setting according to this experiment, and three times of adsorption is enough.

Author contributions

Yaru Hou: Conceptualization, methodology, reparation, data processing, experimenting, validation, writing-reviewing. Jilong Lu: conceptualization, project administration, funding acquisition, reviewing. Mao Li: software, validation, investigation, writing-reviewing. Qiaoqiao Wei: methodology, reparation. Yuchao Fan: reparation, reviewing. Yongzhi Wang: data processing, writing-reviewing.

Conflicts of interest

There are no conflicts to declare.

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