

Determination of Heavy Metal Content in Lapis Lazuli (Complex Processing)

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Abstract [Objectives] To determine the heavy metal content in lapis lazuli (complex processing) and provide a reference for establishing its heavy metal limit standards. [Methods] Seven batches of lapis lazuli (complex processing) samples were collected from Hongyuan County Tibetan Medicine Hospital, Aba Tibetan and Qiang Autonomous Prefecture Tibetan Medicine Hospital, and Aba County Tibetan Medicine Hospital. The contents of Hg, As, Pb, Cd, and Cu were determined using inductively coupled plasma mass spectrometry in accordance with General Rule 2321 of the *Pharmacopoeia of the People's Republic of China (2020 Edition)*. Tests for heavy metal limits and arsenic salt limits were conducted according to General Rules 0821 and 0822, respectively. [Results] The Cu content in the seven batches of samples ranged from 8.49 to 21.41 mg/kg, As content ranged from 6.82 to 16.87 mg/kg, Hg content ranged from 0.03 to 0.62 mg/kg, and Pb content ranged from 3.66 to 26.98 mg/kg. Cd was not detected in any of the samples. The contents of heavy metals varied considerably among samples from different origins. The heavy metal limit test yielded a result of ≤ 50 mg/kg, while the arsenic salt limit test produced a result of ≤ 40 mg/kg. [Conclusions] The heavy metal content in various processed lapis lazuli products varies significantly. Processing technology is an important factor influencing the heavy metal content. This study provides fundamental data for the development of processing specifications and quality standards for lapis lazuli.

Key words Lapis lazuli, Heavy metal content, Complex processing

1 Introduction

Lapis lazuli, referred to as Qingjinshi in Pinyin, Lazurlite in Latin, and ལྷ་མོ་ལོ་ (transliterated as Muman) in Tibetan, is a naturally occurring deep blue gemstone^[1]. This product is a silicate mineral belonging to the sodalite group, primarily composed of sodium-calcium aluminosilicate $[(\text{Na}, \text{Ca})_8(\text{AlSiO}_4)_6(\text{SO}_4, \text{S}, \text{Cl})_2]$. The medicinal material appears as irregular fragments exhibiting blue or blue-purple coloration, a vitreous luster, a relatively smooth surface, and a hard texture. It is traditionally attributed with properties such as clearing heat and detoxification, alleviating irritability and depression, refreshing the mind, and invigorating the spirit. Its primary therapeutic applications include the treatment of depression and restlessness, palpitations and shortness of breath, flat warts, vitiligo, and persistent bleeding^[2].

Lapis lazuli is documented in several official standards, including the *Drug Standards of the Ministry of Health: Uyur Medicine Volume*^[3], the *Processing Specifications for Tibetan Medicinal Materials of Qinghai Province*^[1], the *Processing Specifications for Chinese Medicinal and Uyur Medicinal Decoction Pieces of Xinjiang*^[2], the *Quality Standards for Local Tibetan Medicinal Materials*, and the *Processing Specifications for Tibetan Medicinal Materials of Tibet Autonomous Region*^[4]. However, the processing methods of lapis lazuli differ across regions. To date, there is a lack of literature addressing whether the heavy metal content in lapis lazuli exceeds standards under various processing methods. In our series of studies on lapis lazuli, we observed that different processing methods may influence the heavy metal composition of lapis la-

zuli, which in turn could affect its clinical efficacy. For this purpose, we employed inductively coupled plasma mass spectrometry in accordance with General Rule 2321 of the *Pharmacopoeia of the People's Republic of China (2020 Edition)*^[5] to determine heavy metal content. This approach enabled a systematic comparison of the variations in heavy metal contents in lapis lazuli subjected to complex processing. The study addresses a research gap concerning the impact of complex processing on the heavy metal content of lapis lazuli and provides valuable data for future related investigations.

2 Materials

2.1 Instruments LabMS 3000 inductively coupled plasma mass spectrometer (Beijing LabTech Instruments Co., Ltd.), MASTER 40 high-throughput closed microwave digestion/extraction workstation (Shanghai Sineo Microwave Chemistry Technology Co., Ltd.), TK20 vapor block (Shanghai Sineo Microwave Chemistry Technology Co., Ltd.), ME204E electronic balance (Mettler Toledo, Germany), and Puxi GWB-1T/2T ultrapure water purifier (Beijing Purkinje General Instrument Co., Ltd.) were utilized in this study.

2.2 Test drugs and reagents Hg reference material (No.: GSB 04-1729-2004, 1 000 $\mu\text{g/mL}$), As (No.: GSB 04-1714-2004, 1 000 $\mu\text{g/mL}$), Pb (No.: GSB 04-1742-2004, 1 000 $\mu\text{g/mL}$), Cu (No.: GSB 04-1725-2004, 1 000 $\mu\text{g/mL}$), Cd (No.: GSB 04-1721-2004, 1 000 $\mu\text{g/mL}$), Bi (No.: GSB 04-1719-2004, 1 000 $\mu\text{g/mL}$), In (No.: GSB 04-1731-2004, 1 000 $\mu\text{g/mL}$), Au (No.: GSB 04-1715-2004, 1 000 $\mu\text{g/mL}$), and Ge (No.: GSB 04-1728-2004, 1 000 $\mu\text{g/mL}$) were obtained from the National Nonferrous Metals and Electronic Materials Analysis and Testing Center. Nitric acid (batch No.: 20200810) of superior purity was procured from Chengdu Jinshan Chemical Reagent Co., Ltd. Ul-

Received: May 15, 2026 Accepted: July 3, 2026

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trapure water was used throughout the study.

2.3 Samples Lapis lazuli (complex processing) decoction pieces QJS-1 and QJS-2 were supplied by Hongyuan County Tibetan Medicine Hospital; QJS-3 and QJS-4 were obtained from Aba Tibetan and Qiang Autonomous Prefecture Tibetan Medicine Hospital; and QJS-5, QJS-6, and QJS-7 were provided by Aba County Tibetan Medicine Hospital.

Based on the characteristics of the lapis lazuli (complex pro-

cessing) sample, its properties are described as follows; the product is an irregular, flat block exhibiting colors ranging from sky blue to dark blue, with interspersed white spots. It possesses an oily luster and varies from slightly transparent to opaque, featuring white or light blue streaks. The material is brittle yet resistant to breakage and displays a rough fracture surface. It is odorless and tasteless. Fig. 1 presents seven batches of lapis lazuli (complex processing) samples.

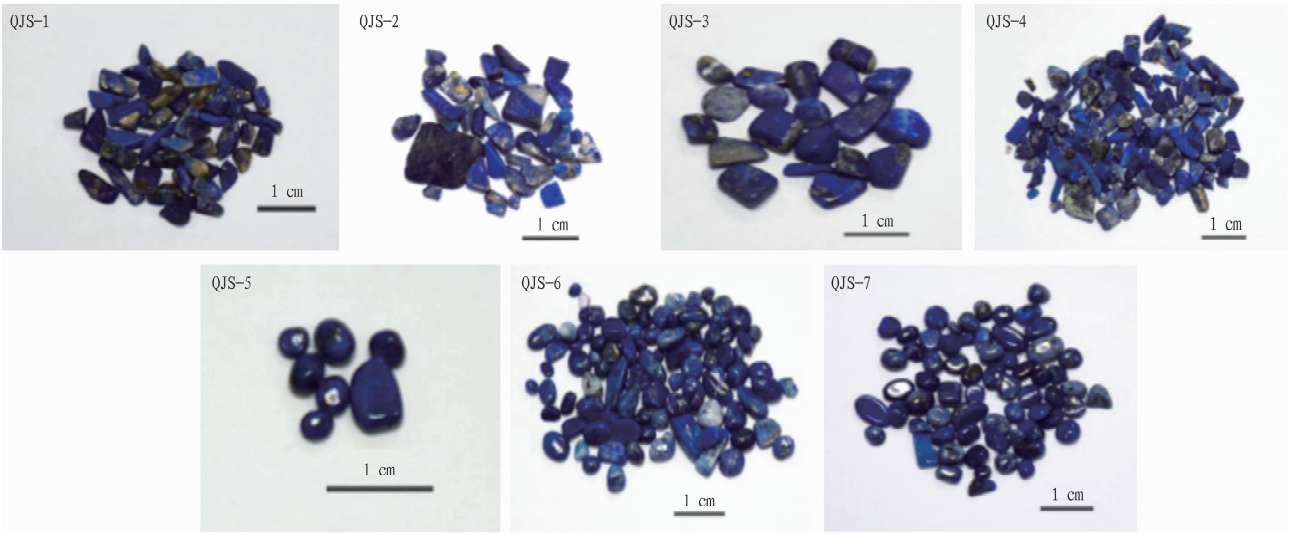


Fig. 1 Seven batches of lapis lazuli (complex processing) samples

3 Results and analysis

Heavy metals and harmful elements were quantified using inductively coupled plasma mass spectrometry in accordance with General Rule 2321 of the *Pharmacopoeia of the People’s Republic of China (2020 Edition)*.

3.1 ICP-MS instrument parameters ICP-MS instrument was operated under the following conditions; radio frequency power set at 1 240 W; rectangular tube depth of 6.95 mm; plasma gas flow rate of 13.0 L/min; atomization gas flow rate of 1.06 L/min; atomization chamber temperature maintained at 5 °C; collision mode utilizing helium gas; collision gas flow rate of 4.5 mL/min; peristaltic pump speed at 40 rpm; three measurement points or peaks; and data acquisition performed using peak-jumping mode with three repeated collections.

3.2 Preparation of test solution The lapis lazuli sample was ground into powder, thoroughly mixed, and accurately weighed to approximately 0.2 g. It was then placed in a microwave digestion vessel resistant to high pressure and high temperature, to which 7 mL of nitric acid was added. The mixture was shaken well, the vessel lid was securely closed, and the vessel was placed in the microwave digestion instrument. Digestion was carried out according to the procedure in Table 1. After complete digestion, the solution was allowed to cool to below 60 °C. The digestion solution was then transferred to a 50 mL volumetric flask. The digestion vessel was rinsed three times with small volumes of water, and the

rinses were combined in the volumetric flask. Subsequently, 200 μL of a gold single-element standard solution (1 μg/mL) was added, and the volume was adjusted to the mark with water. The solution was thoroughly mixed, resulting in the preparation of the test solution. Reagent blank solutions were prepared using the same procedure, except without the addition of the gold single-element standard solution.

Table 1 Microwave digestion conditions

Step	Control temperature//°C	Constant temperature time//min
1	120	10
2	120	10
3	180	20

3.3 Preparation of internal standard solution An appropriate volume of standard solutions of the single elements Ge, In, and Bi was accurately measured and diluted with water to prepare a mixed solution containing 1 μg of each element per mL. This solution was used as the internal standard.

3.4 Preparation of reference substance stock solution Appropriate volumes of single-element standard solutions of Pb, As, Cd, Hg and Cu were accurately measured and subsequently diluted with a 10% nitric acid solution to prepare solutions containing 1 μg Pb, 0.5 μg As, 1 μg Cd, 1 μg Hg, and 10 μg Cu per mL, respectively.

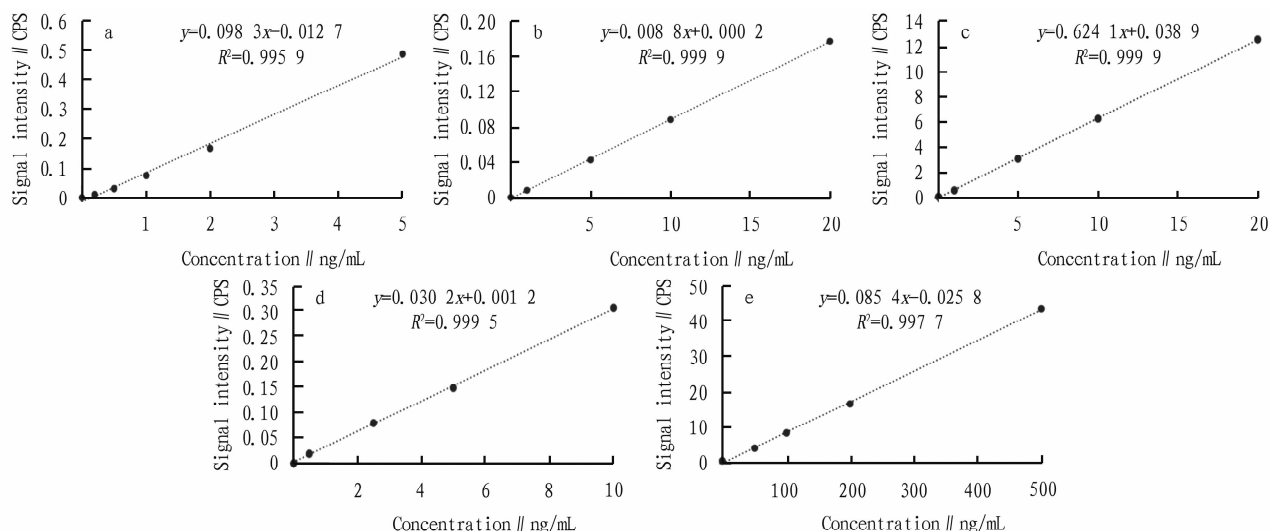
3.5 Standard curve plotting Preparation of standard solutions;

appropriate volumes of Pb, As, Cd, and Cu standard stock solutions were accurately measured and diluted with 10% nitric acid to prepare a series of mixed solutions with concentrations of 0, 1, 5, 10, and 20 ng/mL for Pb and As; 0, 0.5, 2.5, 5, and 10 ng/mL for Cd; and 0, 50, 100, 200, and 500 ng/mL for Cu. Additionally, an appropriate volume of Hg standard stock solution was accurately measured and diluted with 10% nitric acid to prepare solutions containing 0, 0.2, 0.5, 1, 2, and 5 ng/mL of Hg. These solutions were prepared immediately prior to use. Under the specified instrumental parameters, the series of concentrations were measured accordingly. Standard curves were constructed by plotting the concentrations (X-axis, ng/mL) against the corresponding measured signal intensities (Y-axis, 1×10^5 CPS). The results

are presented in Table 2 and Fig. 2.

Table 2 Linear regression equations for five heavy metals and harmful elements

No.	Element	Regression equation	r	Linear range ng/mL	Detection limit ng/mL
1	Hg	$y = 0.0983x - 0.0127$	0.9979	0–5	0.04
2	As	$y = 0.0088x + 0.0002$	0.9999	0–20	0.07
3	Pb	$y = 0.6241x + 0.0389$	0.9999	0–20	0.09
4	Cd	$y = 0.0302x + 0.0012$	0.9998	0–10	0.08
5	Cu	$y = 0.0854x - 0.0258$	0.9988	0–500	0.12



NOTE a. Standard curve of Hg; b. Standard curve of As; c. Standard curve of Pb; d. Standard curve of Cd; e. Standard curve of Cu.

Fig. 2 Standard curve of elements

3.6 Methodological verification

3.6.1 Precision test. The sample solution (batch No. : QJS-1) was collected and injected consecutively six times. The relative standard deviation (*RSD*) of the content for each element ranged from 0.87% to 2.68%, all below 3%, indicating that, under the selected conditions, the instrument demonstrated good precision.

3.6.2 Repeatability test. Six copies of sample powder (batch No. : QJS-1) were prepared into test solutions following the procedure described in Section 3.2. These solutions were then injected and analyzed under the ICP-MS conditions in Section 3.1 to determine and quantify the contents of five heavy metals and harmful elements. The results from six replicate analyses of the same batch demonstrated *RSDs* ranging from 1.76% to 3.33%, indicating that the method exhibited good repeatability.

3.6.3 Stability test. An appropriate quantity of sample powder (batch No. : QJS-1) was taken and prepared into the test solution following the procedure in Section 3.2. Measurements were conducted once at 0, 1, 2, 4, and 8 h, resulting in a total of five measurements. The *RSD* of the response values for the five heavy metals and harmful elements ranged from 2.16% to 3.78%, all

below 5%, indicating that the test solution remained stable within 8 h.

3.6.4 Detection limit. The detection limits were established at concentrations of the five heavy metals and harmful elements corresponding to three times the baseline signal of the blank solution (*i. e.*, a signal-to-noise ratio of 3 : 1). The results indicated that the detection limits for these five heavy metals and harmful elements ranged from 0.04 to 0.12 ng/mL.

3.7 Determination results of heavy metal content in lapis lazuli (complex processing) from different manufacturers The aforementioned method was employed to accurately weigh seven lapis lazuli (complex processing) samples obtained from various manufacturers. The concentrations of five heavy metals and harmful elements, namely Hg, As, Pb, Cd, and Cu, were individually measured. The results of these determinations are presented in Table 3.

Based on the above results of heavy metal analysis, tests were conducted to determine the limits of heavy metals and arsenic salts in lapis lazuli.

Table 3 Contents of heavy metals and harmful elements in different batches of lapis lazuli ($n=3$, mg/kg)

No.	Batch No.	Cu	As	Cd	Hg	Pb
1	QJS-1	18.44	14.65	–	0.19	26.98
2	QJS-2	21.41	16.87	–	0.62	16.13
3	QJS-3	9.12	12.96	–	0.22	22.85
4	QJS-4	18.05	11.42	–	0.23	15.46
5	QJS-5	9.91	13.60	–	0.10	7.83
6	QJS-6	9.19	16.79	–	0.11	38.81
7	QJS-7	8.49	6.82	–	0.03	3.66

NOTE "–" denotes that the element was not detected.

3.8 Determination of heavy metal limits The limits of heavy metals were determined in accordance with Method 1 of General Rule 0821 from the *Pharmacopoeia of the People's Republic of China (2020 Edition)*^[5]. For the preparation of the test sample, 0.2 g of the product was dissolved in 7 mL of nitric acid, followed by the addition of water to a final volume of 50 mL. To prepare the standard Pb solution, 0.159 g of lead nitrate was accurately weighed and transferred into a 1 000 mL volumetric flask. Subsequently, 5 mL of nitric acid and 50 mL of water were added to dissolve the compound, and the solution was then diluted to the mark with water, mixed thoroughly, and used as the stock solution. 10 mL of the stock solution were accurately measured and transferred into a 100 mL volumetric flask. The solution was then diluted to the mark with water and mixed thoroughly to obtain the standard Pb solution, wherein each mL corresponds to 10 μg of Pb. The determination procedure involved the use of three 25 mL Nessler colorimetric tubes. In tube A, a specified volume of standard Pb solution and 2 mL of acetate buffer solution (pH 3.5) were added, followed by dilution with water to a final volume of 25 mL. Tube B contained 25 mL of the test solution prepared according to the prescribed method. In tube C, an amount of the test solution equivalent in weight to that in tube B was introduced, after which an appropriate volume of the solvent used for preparing the test solution was added to dissolve it. Subsequently, 2 mL of the same standard Pb solution and acetate buffer (pH 3.5) as in tube A were added, and the mixture was diluted with the solvent to 25 mL. If the test solution exhibited coloration, a small quantity of dilute caramel solution could be added to tube A to ensure consistency in color with tubes B and C. Thereafter, 2 mL of thioacetamide solution was added to each of tubes A, B, and C, followed by thorough mixing and a standing period of 2 min. The tubes were then placed on white paper and observed vertically. The criterion for evaluation was that when the color intensity in tube C was not lighter than that in tube A, the color in tube B should not be darker than that in tube A.

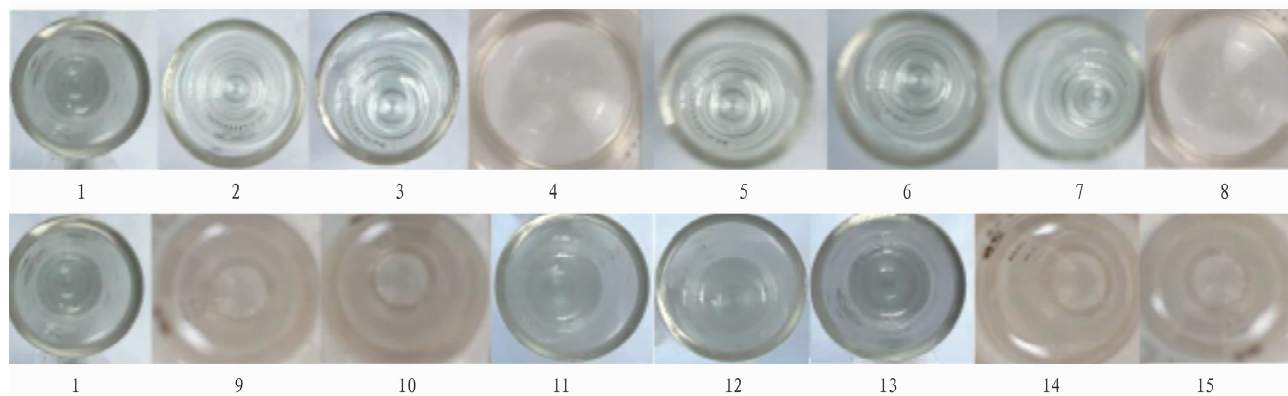
As shown in Fig. 3, the color of tube C was not lighter than that of tube A, and the color of tube B was not darker than that of tube A. This observation suggested that the concentration of heavy metals in lapis lazuli did not exceed 50 mg/kg.

3.9 Determination of arsenic salt limits The arsenic salt

limit was determined in accordance with Method 1 of General Rule 0822 from the *Pharmacopoeia of the People's Republic of China (2020 Edition)*^[5]. For the preparation of the test sample, 0.2 g of the product was dissolved in 7 mL of nitric acid, followed by dilution with water to a final volume of 50 mL. To prepare the standard As solution, 0.132 g of arsenic trioxide was accurately weighed and transferred into a 1 000 mL volumetric flask. Subsequently, 5 mL of 20% sodium hydroxide solution was added to dissolve the arsenic trioxide, after which the solution was neutralized with an appropriate amount of dilute sulfuric acid. An additional 10 mL of dilute sulfuric acid was then added, and the solution was diluted to the mark with water, mixed thoroughly, and used as the stock solution. Prior to use, 50 mL of the stock solution was accurately measured and transferred into a 100 mL volumetric flask. Subsequently, 10 mL of dilute sulfuric acid was added, and the volume was adjusted to the mark with water. The mixture was then thoroughly shaken to prepare the standard As solution, with each mL corresponding to 50 μg of As. For the preparation of standard As spots, 2 mL of standard As solution were accurately measured and transferred into bottle A. Then, 5 mL of hydrochloric acid and 21 mL of water were added, followed by the addition of 5 mL of potassium iodide solution and five drops of acidic stannous chloride solution. After allowing the mixture to stand at room temperature for 10 min, 2 g of Zn granules were introduced. Immediately thereafter, the gas guide tube C, prepared as described above, was securely attached to bottle A. Bottle A was then placed in a water bath maintained at 25 to 40 $^{\circ}\text{C}$ and allowed to react for 45 min. Finally, the mercury bromide test strip was taken out to obtain the standard As spots. The inspection method involved preparing the test solution according to the prescribed sample preparation procedure. The solution was placed in bottle A and subjected to the standard As spot preparation procedure, beginning with "the addition of 5 mL of potassium iodide solution".

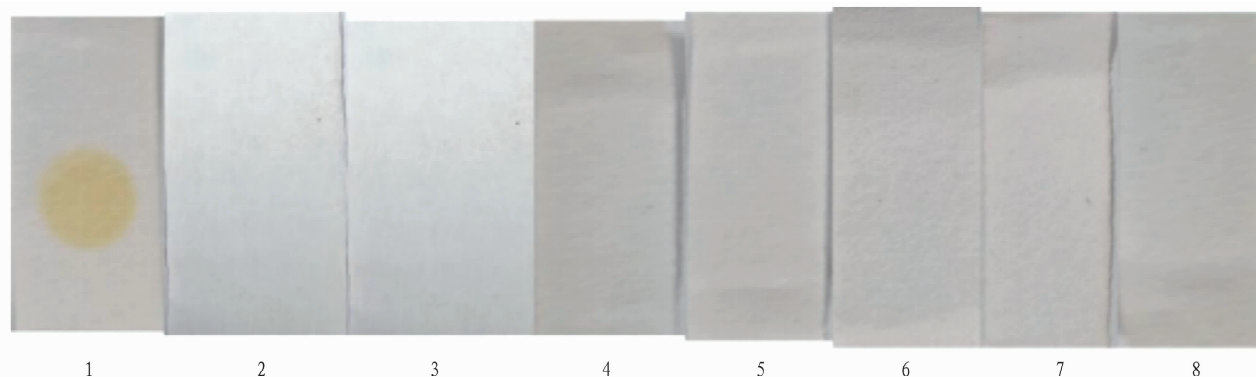
As illustrated in Fig. 4, the color intensity of the As spot on the sample was not darker than that of the standard As spot, suggesting that the arsenic salt content in the lapis lazuli did not exceed 40 mg/kg.

This study determined the heavy metal content in seven samples of lapis lazuli (complex processing). The concentrations of Cu ranged from 8.49 to 21.41 mg/kg; As ranged from 6.82 to 16.87 mg/kg; Hg ranged from 0.03 to 0.62 mg/kg; and Pb ranged from 3.66 to 26.98 mg/kg. Cd was not detected in any of the samples. Among the samples, QJS-2 exhibited the highest concentrations of Cu, As, and Hg, whereas QJS-7 showed the lowest levels of these metals. The Pb content was highest in QJS-1 and lowest in QJS-7. According to heavy metal limit standards, the total heavy metal content in lapis lazuli should not exceed 50 mg/kg. Additionally, arsenic salt content must not exceed 40 mg/kg according to the arsenic salt limit test results.



NOTE 1. 50 mg/kg Pb standard solution; 2 – 8. Tube B samples; 9 – 15. Tube C samples.

Fig.3 Inspection chart of heavy metals



NOTE 1. 40 mg/kg As standard solution; 2 – 8. Samples.

Fig.4 Inspection chart of arsenic salt

4 Conclusions and discussion

The heavy metal content in mineral medicinal materials directly influences their quality. Complex processing significantly impacts the quality of these materials, thereby affecting their clinical efficacy^[6]. In this study, the heavy metal content of lapis lazuli (complex processing) was determined using inductively coupled plasma mass spectrometry, following the General Rule 2321 of the *Pharmacopoeia of the People's Republic of China (2020 Edition)*. This method offers advantages such as simplicity, rapidity, and accuracy, enabling precise quantification of heavy metal content.

After reviewing the *Pharmacopoeia of the People's Republic of China* and the current local standards for medicinal materials, including those pertaining to Tibetan, Mongolian, and Uighur medicine, it was determined that the relevant heavy metal limit values for lapis lazuli were not specified. Consequently, this study referenced the heavy metal limit standards established for botanical medicinal materials and decoction pieces in General Rule 0212 of the *Pharmacopoeia of the People's Republic of China (2020 Edition)*^[5,7]. The results indicated that, among the seven samples analyzed, the Hg content in three samples exceeded 0.2 mg/kg, whereas the Hg levels in the remaining samples complied with the current limit standard for Hg in botanical medicines (≤ 0.2 mg/kg). Cd was undetectable in all seven samples, consistent with the existing

standard for Cd content in botanical medicines (≤ 1 mg/kg). Cu concentrations in six samples adhered to the limit standard for Cu in botanical medicines (≤ 20 mg/kg). As content in one sample conformed to the established limit for As in botanical medicines (≤ 2 mg/kg). Additionally, one sample exhibited Pb levels within the acceptable range for Pb in botanical medicines (≤ 5 mg/kg). In summary, given that lapis lazuli is a mineral-based medicine, its natural metal element content is much higher than that typically found in botanical medicines. Therefore, the general limits established for botanical medicines serve only as a reference and should not be directly applied as evaluative criteria for mineral medicines such as lapis lazuli.

Currently, neither the *Pharmacopoeia of the People's Republic of China* nor existing local standards for medicinal materials specify heavy metal limit values for processed lapis lazuli products. Furthermore, there are no standardized quality control criteria for its complex processing techniques. This study offers a methodological reference for the quality evaluation of lapis lazuli (complex processing) and addresses the research gap concerning the impact of such processing on its heavy metal content. The findings provide valuable data to inform future investigations. Lapis lazuli is a mineral used in medicinal applications, and its heavy metal con-

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tent is influenced by various factors. This article offers a foundational reference for further research on the processing techniques of the mineral medicine lapis lazuli.

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