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Inclusion and Trace Elements in Quartz LA-ICP-MS Analysis and Research

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ABSTRACT

This article conducts quartz inclusion and trace element testing and analysis on quartzite in a certain area of Henan Province, and preliminarily determines the occurrence state of impurity elements inside the quartzite. The main impurity elements are mainly associated minerals, inclusions, and lattice impurities that exist inside the quartz. By observing the mineral composition inside quartzite under a microscope, the type of impurity minerals can be preliminarily determined; In situ determination of the composition of gas-liquid inclusions inside quartz using a high-resolution confocal laser Raman spectroscopy imaging system; By using a laser ablation inductively coupled plasma mass spectrometer, the trace element composition of quartz is determined to determine the content and occurrence state of impurity elements.

KEYWORDS

Quartzite; Inclusion; Trace elements; LA-ICP-MS.

1. INTRODUCTION

Accurately analyzing the composition of fluid and mineral inclusions in quartz, conducting research on high-purity quartz raw material mineral exploration, and exploring the mineralization potential of quartz are of great significance. In recent years, laser ablation inductively coupled plasma mass spectrometer (LA-ICP-MS) has become one of the main methods for trace element determination, which can quantitatively analyze major trace elements in solid samples. It has the advantages of high sensitivity, less interference from multi atomic ions, low sample consumption, and simple sample pretreatment [1-6].This method has the characteristics of high accuracy, low detection limit, and rapid detection of multiple elements while analyzing individual inclusions, overcoming the shortcomings of traditional group inclusion analysis where multiple inclusions are detected simultaneously. Therefore, it has unparalleled advantages over traditional methods in accurately tracing the source of ore-forming materials, finely characterizing the mineralization process, and deeply revealing the mineralization mechanism, and has become one of the best means for analyzing the composition of fluid inclusions today. There have been many studies and applications of this method internationally, but current research in this area in China is still very weak and requires continuous research.

At present, there is no 100% pure quartz in the world. Quartz in nature contains different types of impurities, and the distribution of impurities is also different. They are mainly distributed as independent minerals in cracks, exist in inclusions inside quartz, and exist in the quartz lattice in the form of isomorphism. In order to study the inclusions and trace elements in quartz, we used the alpha300R high-resolution micro confocal laser Raman spectral imaging system equipped by the State Key Laboratory to preliminarily analyze the gas-liquid inclusions in quartz, and used the Analyze HE excimer laser ablation system and AGilent 7700 inductively coupled plasma mass spectrometer to carry out LA-ICP-MS research on inclusions and trace elements in quartz, and established relevant analysis methods. In this paper, quartz sandstone from a certain place in Henan will be tested and analyzed.

2. HISTORY AND PROGRESS OF LA-ICP-MS RESEARCH ON INCLUSIONS

Fluid inclusions of solid, liquid, and gas mixed phases are widely distributed in transparent quartz minerals. Among them, the solid phase mainly consists of inorganic salt minerals such as NaCl, CaCl2, KCl, etc., the liquid phase mainly contains H2O and some dissolved inorganic salts, and the gas phase mainly consists of small molecules such as H2O, CO2, N2, SO2, SO3, CH4, etc. These mixed fluids are important sources of impurities that affect the quality of quartz sand.

2.1. Research progress on fluid inclusions

The study of geological fluids plays an important role and significance in the development of Earth science. The initial observation of inclusions was made by Boyle [7] in 1672, who observed the phenomenon of "moving bubbles" in quartz. Davy first attempted to determine the chemical composition of inclusions within quartz crystals in 1822. The book "Fluid Inclusion" written by Roedder[8] in 1984 systematically summarized the achievements of European and American countries since 1958. The publication of this book gradually popularized the study of fluid inclusions in the West, and more papers were published. In the second half of the 20th century, analytical techniques for the determination of major and trace elements were developed. In the early 1970s, Anderson (1974) and Clocchiatti (1975) first utilized electron microprobes. Anderson et al. (1989) analyzed melt inclusions using infrared spectroscopy, while Kamenetsky[9] (1997) were the first to use laser dissolution inductively coupled plasma mass spectrometry to determine trace elements within inclusions.

Fluid inclusions are the most common and abundant inclusions in quartz [10-13]. They can be captured by quartz during the growth of quartz crystals, forming primary fluid inclusions, or they can be formed by fluid infiltration along microcracks of quartz in later stages, forming secondary inclusions during the healing of quartz crystals. Common fluid inclusion components include H2O, H2O,CO2,CH4,N2,H2S,H2,O2,Na+,K+,Ca2+,Mg2+,Cl-,SO2-4,F-, as well as various hydrated metal ions and their mixtures [14].

Table 1. Forms and Main Components of Fluid Impurities in Quartz[15]

Name	Form of existence	Material terms (molecular formula)
Fluid inclusion	Uniform distribution or distribution along the cleavage plane	Gas phase: H_2O,CO_2,CO,C_xC_y,C_xC_yOH,H_2,H_2S, SO_2, HCl,Cl_2,F_2 liquid phase: Na^+,K^+,Ca^{2+},Mg^{2+},Cl^-,SO_2^{-4},F^-,Cr^-, HCO_3^-,C_xH_x

2.2. Research progress on mineral inclusions

In theory, all impurity minerals present in quartz exist in the form of tiny inclusions. The main forms of inclusion of impurity silicate minerals are shown in the table below:

Table 2 Main forms of impurities in silicate mineral inclusions

Types of quartz minerals	Types of silicate mineral inclusions
magmatic rock	Feldspar, Rutile, Biotite, Muscovite, Zircon, Apatite
Low-grade metamorphic rock	Green mudstone, white mica (sericite), red columnar stone
High-grade metamorphic rock	Cross shaped stone, Blue sapphire, Garnet
Sedimentary rock	Gypsum, Calcite,Salt minerals and organic matter

Mineral inclusions are vein minerals enclosed by the main mineral. The mineral inclusions in quartz mainly come from the displacement of crystal interfaces during the cooling and metamorphism processes of the melt. There are many reasons for the formation of solid inclusions, on the one hand, it is the displacement of grain boundaries during the process of melting and solid cooling, and on the other hand, it is the displacement of lattice during the metamorphism process. Adachi [16] studied the formation of needle shaped hematite due to the high Ti content of quartz melt during the cooling process. The common mineral inclusions in quartz mainly include mica, feldspar, kaolinite, hematite, pyrite, calcite, rutile, etc.

3. RESEARCH ON TRACE ELEMENTS IN QUARTZ AND LA-ICP-MS ANALYSIS

As a typical Si-O tetrahedral structure, few elements can replace Si4+in quartz to make it one of the purest minerals on Earth. However, some elements can still enter quartz, although most of the content may be below 1×10-6. Elements with a content greater than 1×10-6 in quartz usually include Al, Ti, Na, Ca, K, Li, Fe, Cl, P, B, and Ge. Elements with a content between1×10-9 and 1×10-6 include Pb,Br,Mn, Ｒb,Sr,Be,Ba,Zn,As,Ce,Cr,Cs,La,Ga,V,Nd,W,I,Co,Th,U,Ta,Ag,Sc,Sm,Dy,Yb, Eu And Hg, while Hf, In, Tb, Lu, and Au are less than1× 10-9, and trace elements mainly exist in the lattice. Generally speaking, Al, Ti, Fe, Li, Na, and K,Considered as the most common trace element replacing silicon in hydrothermal quartz[17].

Lattice impurities refer to impurity ions entering the lattice of quartz crystals in the form of isomorphic substitution. The lattice impurity elements are closely related to the point defects of quartz crystals.The point defects of quartz crystals include vacancies, displaced atoms, and interstitial atoms, among which the latter two are the main ones that can introduce impurity elements. Foreign ions (such as P5+, Ti4+, Ge4+, Al3+, Fe3+, B3+, etc.) displace Si4+and occupy the position of Si4+, forming substitutional impurity elements; At the same time, certain ions (such as Al3+, Fe3+, etc.) may introduce Na+, K+and other valence compensating ions between atoms as interstitial impurity elements in order to maintain electricity balance when replacing Si4+.Impurities are mainly formed in quartz crystals through three forms [18-20]: first, single ions such as Al3+, Fe3+, B3+, Ti4+, Ge4+, P5+, etc. directly replace the Si4+position in the lattice; The second is to replace adjacent Si4+with coupled ion groups (such as Al3+and P5+combination replacing Si4+); The third is that large radius ions (such as Li+, K+, Na+, H+, OH-, etc.) act as compensating coordination ions for lattice substitution ions and enter the gaps between atomic connections[21].

Positive tetravalent ions Ti4+and Ge4+can directly replace Si4+in silicon oxygen tetrahedra,for example:

$$Ti4+,Ge4+\rightarrow Si4+ \qquad (1)$$

Al3+,Fe3+,B3+ When trivalent ions enter the quartz lattice, the principle of charge compensation must be followed, and the charge must reach equilibrium,for example:

$$Al3+(Fe3+,B3+)+Li+(K+,Na+,H+)\rightarrow Si4+ \qquad (2)$$

Al3+and P5+can couple and replace Si4+into the quartz lattice,for example:

$$Al^{3+}+P^{5+}\rightarrow 2Si^{4+} \quad (3)$$

With the development of modern analytical testing technology, more and more instruments and methods (such as ICP-MS, LA-LCP-MS, SIMS, SEM-CL, TEM, etc.) are used to analyze the content and occurrence state of impurity elements in quartz lattice, and a large amount of mineralogical and geochemical data has been accumulated[22-24].

4. EXPERIMENTAL SECTION

4.1. Research progress on mineral inclusions

The samples used in this experiment were collected from quartzite in Luanchuan, Henan Province. The sample is mainly composed of quartz, with a small amount of sericite and hematite. Quartz with good sorting is gray white, granular, with particle size mainly ranging from 0.2-0.4mm, and visible wavy extinction. Sericite is distributed between quartz particles and appears as cryptocrystalline scales.

Figure 1: Photo of Experimental Sample Hand Specimen

4.2. Experimental Instruments

This experiment uses a high-resolution confocal laser Raman spectroscopy imaging system to test and analyze the gas-liquid inclusions inside quartz sandstone, and preliminarily determine the composition of impurity elements contained in the gas-liquid inclusions; Quantitative analysis of solid inclusions and major trace elements inside quartz sandstone was conducted using a laser ablation inductively coupled plasma mass spectrometer, and the forms of impurity elements were preliminarily determined.

Table 3 Relevant parameters of high-resolution confocal laser Raman spectroscopy imaging system

High resolution confocal laser Raman spectroscopy imaging system	alpha300R
Manufacturer	WITec Company(German)
Laser device	532nm and 785nm,Power regulation accuracy,0.1mW
Spectrometer	300g/mm,600g/mm,1800g/mm
Zeiss research grade optical microscope from Germany, objective lens	×5,×10,×50(Short Focus),×50（Telephoto), ×100 LWD
Horizontal spatial resolution	350nm
Vertical spatial resolution	850nm
Spectral resolution (full spectrum)	<1.5cm^{-1}
Spectral range	10-3900cm^{-1}

Table 4 Instruments and analytical conditions used for the LA-ICP-MS measurements

Laser ablation system	
Instrument model	Analyte HE Excimer laser ablation system
Laser device	ArF excimer laser
Excimer laser pulse energy	100mJ
Energy density	3.5J/cm2
Pulse frequency	5HZ
Beam spot size	50μm/15μm
Sampling method	Single point erosion
Carrier gas	He
He gas flow	1.1L/min
Duration of erosion	75S
Detecting elements	^{7}Li,^{11}B,^{23}Na,^{24}Mg,^{27}Al,^{29}Si,^{31}P,^{39}K,^{42}Ca,^{45}Sc,^{49}Ti,^{52}Cr,^{55}Mn,^{56}Fe,^{59}Co ,^{60}Ni,^{63}Cu,^{66}Zn,^{71}Ga,^{85}Rb,^{88}Sr,^{89}Y,^{90}Zr,^{93}Nb,^{139}La,^{140}Ce,^{141}Pr,^{146}Nd, ^{147}Sm,^{153}Eu,^{157}Gd,^{159}Tb,^{163}Dy,^{165}Ho,^{166}Er,^{169}Tm,^{172}Yb,^{175}Lu,^{178}Hf, ^{181}Ta,^{182}W,^{208}Pb,^{232}Th,^{238}U

5. DATA PROCESSING

5.1. Observation under a microscope

Using a polarizing microscope to observe quartzite probe pieces under the microscope, analyze opaque minerals and fluid inclusions. The quartz particles are tightly embedded, and there is sticky dust distributed at the interfaces and cracks of the quartz particles. There are also a small amount of opaque impurity mineral inclusions inside the quartz, which run through the entire quartz particle. The sample is mainly composed of quartz, with a small amount of sericite and hematite, and the accessory minerals contain hematite and zircon.Under the microscope, it can be seen that the surface of quartz contains mineral inclusions and some micro scale gas-liquid inclusions. The morphology of gas-liquid inclusions mainly includes three types: round, triangular, and irregular, with particle sizes ranging from 0.5 to 25μm, mostly concentrated in the range of 2 to 10 μm; Most solid inclusions may be syngenetic inclusions, containing minerals such as hematite and iron minerals.

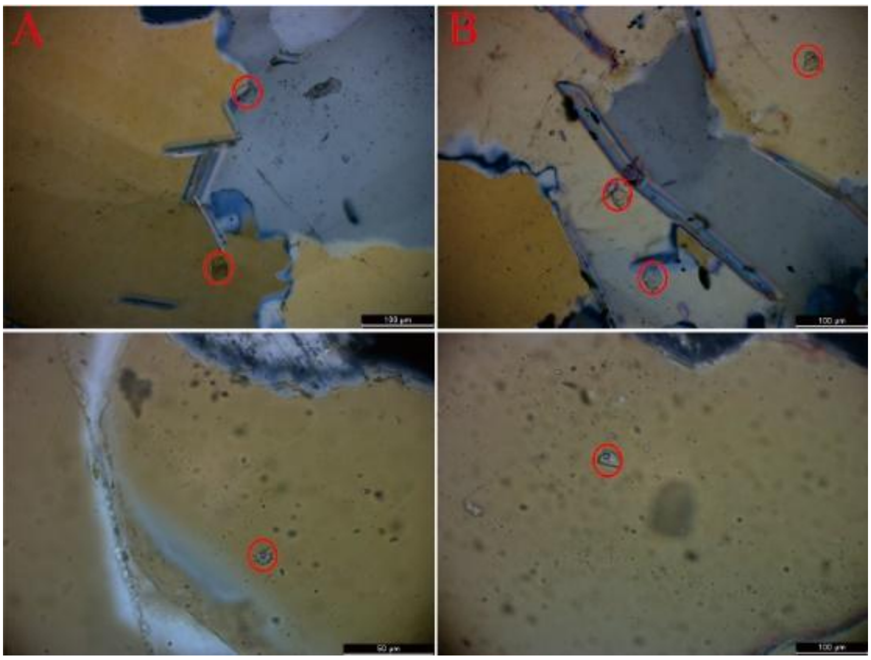

Figure 2 Photo of Quartz and Inclusion under Polarized Microscope

5.2. 5.2 Laser Raman Testing Analysis

More comprehensive research on the application of laser micro Raman spectroscopy technology in fluid inclusion analysis was mainly conducted in the 1980s, such as Beny et al. (1982) [25] and Burke and Lustenhouwer (1987) [26] who published relevant articles in this area. Usually, the gas-liquid ratio is V=V gas/(V gas+V liquid) × 100%. If V is less than 30%, it is a liquid inclusion; V is equal to 30%~50% for gas-liquid inclusions; V greater than 50% is a gaseous inclusion. Gas is composed of water vapor, carbon dioxide, or methane; Liquids are generally water or hydrocarbons containing salts or carbonates; Solids are mainly fluorides, chlorides, carbonates, or sulfates of Na/K/Ca/Mg.

Compared to the qualitative and quantitative analysis of mass spectrometers, laser Raman spectroscopy can only qualitatively analyze individual gas-liquid inclusions, but can qualitatively analyze the composition of solution or sub minerals within the inclusions. The internal inclusions of quartz are mainly gas-liquid two-phase inclusions, with the main components being C2H6 and H2O. The laser Raman spectra of gas-liquid inclusions inside quartz show a clear Raman peak of H2O at 3429cm-1 and a clear Raman peak of C2H6 at 3056cm-1. The specific measurement results are shown in the following figure:

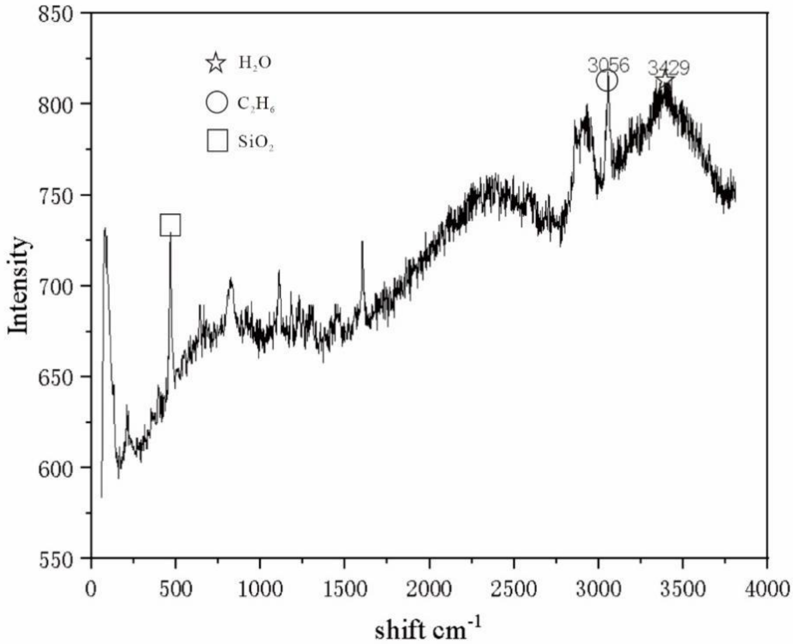

Figure 3 Raman spectrum of quartzite

5.3. 5.3 LA-ICP-MS analysis

In recent years, different scholars at home and abroad have conducted research on the occurrence forms of trace elements in quartz. Research has found that trace elements in quartz mainly exist in three forms: ① entering the quartz lattice as solid solutions; ② Entering quartz in the form of visible micrometer sized micro mineral inclusions; ③ There are invisible nanoscale mineral particles that enter the lattice defects of quartz. When conducting LA-ICP-MS testing and analysis, quartz is prone to fragmentation during the 193nm excimer laser ablation process. To avoid fragmentation, some laboratories internationally adopt a stepwise ablation method [27-28], which first uses a small beam spot laser to preliminarily open the fluid inclusion, and then uses a large beam spot laser to wrap the entire inclusion for ablation.This method greatly improves the controllability of fluid inclusion erosion and the reproducibility of data. This experiment tested the mineral inclusions and quartz inside quartzite to analyze the occurrence state of elements.

Table 5 LA-ICP-MS test results of trace elements in quartz ($\times 10^{-6}$)

	JW-1-1	JW-1-2	JW-1-3	JW-1-4	JW-1-5
Li	3.626397376	7.289165304	6.367802061	9.695860389	6.42990548
B	10.60259802	2.740814044	16.6180641	15.22079368	13.40415341
Na	82686.89385	42179.6985	139504.8073	145257.0954	139517.2894
Mg	16940.45093	8462.123956	27375.99842	28379.46217	27455.76128
Al	1895.855436	941.2349583	2737.528683	2821.307597	2741.774909
Si	306975.6778	299045.2248	431737.8823	443836.1883	429832.8115
P	35.7158483	15.88615882	44.41304382	61.31787978	60.8816252
K	76.16489954	44.12715081	130.0250508	141.4032712	137.561148
Ca	58014.00239	27706.29344	87585.58923	88503.39163	86733.63733
Sc	1.014797406	1.168317033	1.892707428	2.150333503	1.975077132
Ti	34.4352201	19.28227613	51.39237514	66.22784002	57.47998824
Cr	5.870699531	7.99238093	6.582672297	5.256249984	5.213479602
Mn	4.322019631	1.740920808	5.129686501	6.442763458	5.970595148
Fe	34.28753141	25.88413116	113.4690882	98.59614458	87.48905287
Co	0.205412019	0.000428614	0.231135294	0.218434387	0.223317279
Ni	0.185988394	-	0.224669287	0.785604657	0.73415632
Cu	5.660014416	2.641428098	10.8721316	10.42990555	9.938964309
Zn	1.368283197	0.922155746	3.054192712	3.952527979	4.372702471
Ga	0.24964781	0.048230525	0.381775443	0.418481953	0.518107431
Rb	0.349689206	0.258345546	0.737760531	0.847285518	0.715427033
Sr	61.59368769	30.977382	92.03332976	93.64695573	93.19953641
Y	1.358164376	0.763044265	2.025806326	2.017078714	2.075646108
Zr	52.86633482	26.86538555	74.90678038	79.49772361	76.59278106
Nb	0.133766215	0.022784255	0.264973335	0.241492098	0.238715554
La	1.147158016	0.509870166	1.530423071	1.797452122	1.577706785
Ce	4.641153626	2.145265018	7.563917028	7.555462498	7.514115502
Pr	0.199051169	0.086326413	0.292360779	0.319877514	0.260934472
Nd	0.589038289	0.347724703	0.993602301	1.137491252	1.17939684
Sm	0.06397579	0.03828439	0.174307149	0.216656221	0.229331086
Eu	-	-	0.008460843	0.003747729	0.011391181
Gd	0.056000239	1.45695E-09	0.082162011	0.217977446	0.377156303
Tb	0.002928476	0.008845566	0.035928147	0.021776314	0.032570921
Dy	0.034804042	0.115964607	0.235304512	0.276593948	0.281227707
Ho	0.069669914	1.13255E-17	0.051818044	0.055997393	0.057947434
Er	0.14710059	0.017214256	0.169573836	0.232871949	0.131705503
Tm	0.017802099	0.003236711	0.027801948	0.032735093	0.02270137
Yb	0.122899303	0.050611478	0.198139449	0.294706706	0.2189432
Lu	0.014754801	-	0.04399523	0.043474679	0.039316638
Hf	1.829442465	0.84881567	2.395962299	2.311004497	2.280270022
Ta	0.007791426	-	0.037776554	0.018564519	0.051994043
W	0.009406091	-	0.061671834	0.113376804	0.020192801
Pb	0.470763143	0.219631213	0.898591272	0.909501197	0.834438299
Th	0.335489627	0.20275787	0.549892741	0.577094395	0.533561027
U	0.316330507	0.153093639	0.494182853	0.495274133	0.499428916

According to LA-ICP-MS testing analysis, the true values of trace elements in quartz were obtained. As shown in the table above, the contents of Na, Mg, Al, Si, and Ca are relatively high. It is preliminarily determined that this is caused by minerals such as feldspar and mica, which are associated with them. A small portion exists in nanoscale inclusions and may also exist in the crystal lattice in the form of homomorphic phenomena. The low content of K and Fe may be related to the small amount of hematite and sericite in quartz. The low content of Ti may be related to the small amount of rutile in quartz, or it exists in the quartz lattice as a isomorphism.

Table 6 LA-ICP-MS test results of trace elements in quartz internal inclusions ($\times 10^{-6}$)

	JW-1B-1	JW-1B-2	JW-1B-3	JW-1B-4	JW-1B-5
B	-	0.079695349	0.388991374	0.043724588	0.17147283
Na	11867.87018	0.859436361	-	-	-
Mg	1851.167621	-	-	3.276705747	0.543431735
Al	169148.7055	7.511868567	-	-	-
Si	187632.5018	2502.623269	2855.773483	294.3226468	-
P	46.18981519	64.92840017	62.2585877	32.2850013	60.19231118
K	61518.83737	23.74306731	13.69252008	23.02651258	-
Ca	-	48.95836715	-	-	7.04816703
Sc	7.072299711	-	-	-	-
Ti	-	0.042607382	-	-	1.551422551
Cr	86.07098806	7.448751403	36.8153158	13.8502676	7.508308361
Mn	1.664414077	-	5.074675394	4.076425038	2.023851054
Fe	4543.805618	9.117744045	26.66474176	-	-
Co	0.010250905	-	-	-	-
Ni	0.386230597	-	0.069192452	0.802195843	0.194063244
Cu	0.385185104	1.039969929	1.442332939	-	1.65707195
Zn	-	-	0.020897094	-	0.039919892
Ga	35.13221676	-	-	0.000195947	-
Rb	165.7893019	-	0.473446366	-	0.195042679
Sr	95.38017192	-	-	-	-
Nb	15.89153495	-	-	-	-

According to LA-ICP-MS testing analysis, the true values of trace elements in the internal inclusions of quartz are obtained. As shown in the table above, the contents of Na, Mg, Al, Si, K, and Fe are relatively high, indicating that the mineral inclusions mainly contain minerals such as feldspar, quartz, and hematite.

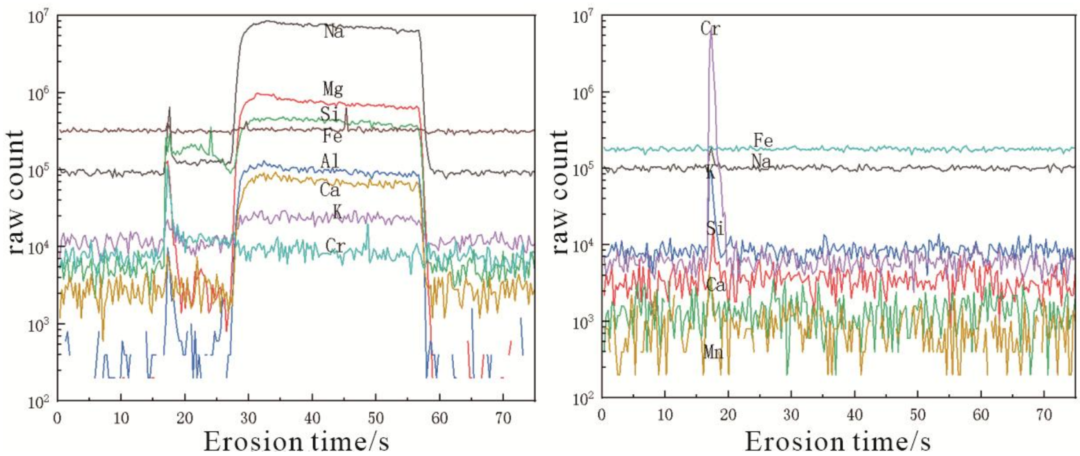

Figure 4 Typical output signal diagram of plasma mass spectrometer during quartz and inclusion LA-ICP-MS analysis

6. CONCLUSION

This article uses laser ablation inductively coupled plasma mass spectrometer (LA-ICP-MS) and high-resolution confocal laser Raman spectroscopy imaging system (LRS) to analyze the inclusion composition and trace element testing of quartzite in a certain area of Henan province. The main impurities inside quartz are distributed in quartz cracks, or exist in the form of inclusions on quartz, and a small amount of impurities are assigned to quartz as lattice impurities. Through microscopic observation and LA-ICP-MS analysis, it was found that the quartzite studied in this study is mainly associated with minerals such as feldspar, sericite, hematite, and hematite. The internal inclusions of quartz are mainly mineral inclusions and gas-liquid inclusions, with C2H6 and water being the main gas-liquid inclusions. The quartzite was analyzed by LA-ICP-MS testing, and the contents of Na, Mg, Al, Si, and Ca were high, while the contents of K and Fe were low. The content of Na, Mg, Al, Si, K, and Fe in the mineral inclusions was high. It is preliminarily speculated that impurity elements mainly exist in the associated minerals and mineral inclusions, and a small part exists in the quartz lattice in the form of isomorphism.

CONFLICTS OF INTEREST

The authors declare that they have no conflict of interest.

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