Research progress of modern inspection technical method of rock and mineral identification

Reported by: Du Gu
Chengdu Geologic Survey Center of China Geological Survey
September 2017
Application of modern analysis instrument in sedimentary rock rock-mineral determination

Research on status of occurrence of platinum family of minerals in Sichuan Danba Cu-Ni sulfide mineral
Utilize modern experimental analysis equipment and related experimental analysis technology to research characteristic components, structures and related information of main minerals and auxiliary minerals in sedimentary rock; in addition, exert advantages of various instrument equipment in rock-mineral determination inspection, solve qualitative and quantitative analysis problem of sedimentary rock components as well as correlations among minerals and establish systematical inspection technical method.
Technical method of sedimentary rock rock-mineral determination

Research method

Sample to be inspected

Quantitative analysis

Electron probe microanalysis (low content mineral)

Infrared characteristic absorption peak (mineral infrared absorption)

X diffraction analysis (X diffraction peak of characteristics of crystalline minerals)

Noncrystalline quantitative analysis

Infrared/Raman spectrum peak (content of peak intensity reaction mineral)

SEM analysis (estimation of content by visual method)

X diffraction (content of corresponding mineral of diffracted intensity)

Fabric characteristics

SEM analysis (structure and construction observation)

Analysis of the formation of stone

Laser Raman REM (fluid inclusion test and post-sedimentation role observation)

Rock nomenclature
Develop observation and research of rock fabric characteristics, heavy mineral analysis, cementation type, particle support property, diagenesis stage and other contents based on Classification and nomenclature schemes of the rocks Classification and nomenclature schemes of igneous rock (GB/T17412[1].2-1998), gain accurate and systematic microscopic information of rock and mineral to conduct appraisal and analysis of sedimentary rock.
Nomination of carbonate rock: **diagenesis change + structure + auxiliary minerals + basic name**

Production of carbonate rocks change (minerals after diagenesis)
- When content is less than 25% to 5%, it is called xx or weakening xx as additional modifier;
- When content is less than 50% to 25%, it is called xx or weakening xx as additional modifier;

**Structure:**
- When overall content is over 50%, the main structure is grained clast;
- When overall content is 50% to 25%, grained clast is auxiliary structure and interstitial material is main structure;
- When overall content is 25% to 10%, grained clast doesn’t participate into nomination.

**Auxiliary minerals:**
- When auxiliary mineral is less than 5%, it won’t participate into nomination. When it has special geologic meaning, the micro including of xx shall be regarded as additional modifier;
- When auxiliary mineral is between 5%-25%, the including of xx shall be regarded as additional modifier;
- When auxiliary mineral is between 25%-50%, the xx shall be regarded as additional modifier;
Qualitative analysis of mineral component and determine basic name of minerals

Quantitative analysis of mineral component, mineral composition content, distinguish host mineral and accessory mineral and determine basic name of rock
Calcite: 70.4%; dolomite: 11.0%; muscovite: 10.9%; quartz: 4.6%; montmorillonite: 3.1%. It is named as: dolomitic limestone.
Technical method of sedimentary rock rock-mineral determination

Infrared spectrum analysis

Test of symmetrical stretching vibration, out-of-plane bending vibration, asymmetric vibration and in-plane bending vibration

Molecular structure information of mineral, complete semi-quantitative analysis of mineral in rock and determine basic name of rock

Mutual corroboration of X-diffraction result
The host mineral is calcite based on absorption characteristic peak, including dolomite, quartz and other silicate minerals. It is named as: dolomitic limestone.

Proof qualitative analysis of mineral in X-diffraction rock
Technical method of sedimentary rock rock-mineral determination

Electron probe microanalysis

Analysis of variety of mineral in rock and component of micro fine mineral, gain quantitative information of mineral component elements

Conduct accurate nomination of mineral and its variety

Analyze component mineral type of fine agglutinate and base material to provide basis for research of diagenetic environment and division of diagenetic stage.
No.1-5: common dolomite; No.6-10: ankerite

<table>
<thead>
<tr>
<th>Sample</th>
<th>Na$_2$O</th>
<th>MgO</th>
<th>K$_2$O</th>
<th>CaO</th>
<th>MnO</th>
<th>FeO</th>
<th>SrO</th>
<th>BaO</th>
<th>CO$_2$</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.017</td>
<td>21.699</td>
<td>0.006</td>
<td>30.885</td>
<td>0.011</td>
<td>0.033</td>
<td>0.065</td>
<td>0.008</td>
<td>47.276</td>
<td>100.000</td>
</tr>
<tr>
<td>2</td>
<td>0.000</td>
<td>21.849</td>
<td>0.000</td>
<td>31.031</td>
<td>0.044</td>
<td>0.013</td>
<td>0.000</td>
<td>0.000</td>
<td>47.063</td>
<td>100.000</td>
</tr>
<tr>
<td>3</td>
<td>0.008</td>
<td>21.617</td>
<td>0.011</td>
<td>31.283</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>47.081</td>
<td>100.000</td>
</tr>
<tr>
<td>4</td>
<td>0.007</td>
<td>21.580</td>
<td>0.000</td>
<td>31.186</td>
<td>0.005</td>
<td>0.028</td>
<td>0.000</td>
<td>0.000</td>
<td>47.194</td>
<td>100.000</td>
</tr>
<tr>
<td>5</td>
<td>0.000</td>
<td>21.823</td>
<td>0.000</td>
<td>30.783</td>
<td>0.003</td>
<td>0.094</td>
<td>0.020</td>
<td>0.000</td>
<td>47.277</td>
<td>100.000</td>
</tr>
<tr>
<td>6</td>
<td>0.005</td>
<td>10.451</td>
<td>0.000</td>
<td>26.047</td>
<td>0.000</td>
<td>15.024</td>
<td>0.000</td>
<td>0.000</td>
<td>48.473</td>
<td>100.000</td>
</tr>
<tr>
<td>7</td>
<td>0.009</td>
<td>11.661</td>
<td>0.000</td>
<td>29.351</td>
<td>0.013</td>
<td>10.045</td>
<td>0.000</td>
<td>0.000</td>
<td>48.921</td>
<td>100.000</td>
</tr>
<tr>
<td>8</td>
<td>0.000</td>
<td>12.729</td>
<td>0.000</td>
<td>27.762</td>
<td>0.088</td>
<td>16.207</td>
<td>0.000</td>
<td>0.000</td>
<td>43.214</td>
<td>100.000</td>
</tr>
<tr>
<td>9</td>
<td>0.000</td>
<td>9.851</td>
<td>0.008</td>
<td>26.093</td>
<td>0.038</td>
<td>13.095</td>
<td>0.000</td>
<td>0.026</td>
<td>50.889</td>
<td>100.000</td>
</tr>
<tr>
<td>10</td>
<td>0.000</td>
<td>10.667</td>
<td>0.004</td>
<td>27.710</td>
<td>0.000</td>
<td>15.123</td>
<td>0.033</td>
<td>0.000</td>
<td>46.463</td>
<td>100.000</td>
</tr>
</tbody>
</table>
Analysis of pattern characteristic of micron-level mineral and microfabric characteristic in rock

Pattern of micro fine mineral and co-relation information
The grain diameter is in 90µm-1mm with separation error, different sizes and random distribution. The interstitial material has uneven isolation, structure and cementation.
Analysis of heavy mineral

Accessory mineral: eremite.
Observation of agglutinate

Agglutinate is in fine and long slice so it can be siliceous cementation including iron and magnesium through qualitative analysis of energy spectrum.
Fluid inclusion component analysis

Heating and freezing stage temperature and salinity analysis

Divide diagenesis stage based on pressure, burial depth, mineral stage and other information
Research on diagenetic stage

Component: CH$_4$

Even temperature scope: 60-195(℃)

Salinity: 3.23-5.12 (wt% NaCl)

Diagenetic stage: late diagenetic stage
## Inspection report of rock mineral

<table>
<thead>
<tr>
<th>Field name: carbonate rock</th>
<th>Inspection name:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sample No.</td>
</tr>
<tr>
<td></td>
<td>20130503</td>
</tr>
<tr>
<td></td>
<td>Level:</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Mineral component (%)</th>
<th>X-ray diffraction analysis</th>
<th>Infrared spectrum analysis</th>
<th>Electron probe microanalysis</th>
<th>SEM analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Through spectral analysis, calcite characteristic peak d: 3.02564, 2.77784, 2.08814; dolomite characteristic peak d: 2.88326, 2.18962, 1.7852; quartz characteristic peak d: 3.34547, 1.8028, 1.54106; muscovite characteristic peak d: 10.05956, 3.3300, 2.3700; montmorillonite characteristic peak d: 13.12150, 4.5000, 3.0200 (Appendix 1)</td>
<td>Through spectral analysis, spectrogram includes carbonate group absorption peak: 1465cm⁻¹; calcite characteristic absorption peak: 714cm⁻¹, 883cm⁻¹, and base on (Appendix 2)</td>
<td>Based on the test result of carbonate mineral, content of FeO is (10%-25%), and it is determined as ankerite. (Appendix 3)</td>
<td>Grain size, rounding and other characteristics of structural constituent in carbonate rocks; truly present filling method of interstitial material among calcite grains and pattern characteristic of mineral (Appendix 4)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Calcite</th>
<th>CO₃</th>
<th>1465cm⁻¹</th>
<th>FeO</th>
<th>Fluid composition</th>
<th>CH₄</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>70.4</td>
<td>1465cm⁻¹</td>
<td></td>
<td></td>
<td>Fluid composition</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Calcite</td>
<td>883cm⁻¹</td>
<td>15.024</td>
<td></td>
<td>Even temperature</td>
<td>60-195</td>
</tr>
<tr>
<td>Dolomite</td>
<td>11</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Muscovite</td>
<td>10.9</td>
<td>714cm⁻¹</td>
<td>10.045</td>
<td></td>
<td>Salinity</td>
<td>3.23-5.12</td>
</tr>
<tr>
<td>Quartz</td>
<td>4.6</td>
<td></td>
<td>16.207</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Montmorillonite</td>
<td>3.1</td>
<td></td>
<td>13.095</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Content description

Rock is mainly the calcite group, including small amount of dolomite, quartz, muscovite and montmorillonite; the grain particle is 90µm-1mm; calcite grain is in subangular shape with poor grade. Dolomite is mainly self-shaped, which is mainly divided into dolomite and ankerite. Interstitial materials are mainly dolomite in scale shape with uneven separation and cementation. The terrigenous fragment is mainly quartz with the grain diameter of 90µm-1mm and poor grade in xenomorphic granular and it is randomly distributed. The heavy mineral is mainly rutile; then, it is zircon and small amount of eremite which is in sporadic distribution. Fluid phase component is mainly methane and even temperature is 60-195, indicating it is late diagenetic stage.
### Technical method of sedimentary rock rock-mineral determination

<table>
<thead>
<tr>
<th>Observation content</th>
<th>Optical microscope</th>
<th>Modern large instrument</th>
<th>Comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral component research</td>
<td>It is uneasy to distinguish constituent minerals of carbonate rock through optical characteristic; in addition, large subjectivity exists in the component analysis.</td>
<td>Gain accurate and scientific mineral content through semi quantitative analysis by X-ray diffraction instrument and infrared spectrometer.</td>
<td>Large instrument specifies content of mineral as nomination prefix.</td>
</tr>
<tr>
<td>Observation of fabric characteristic</td>
<td>Due to the restriction of resolution ratio, pattern characteristic of micron agglutinate cannot be observed, which is collectively called silt in inspection.</td>
<td>Clearly observe pattern characteristic of various clay minerals through SEM which can provide basis for division of diagenetic stage.</td>
<td>Large instrument specifies iron silt in inspection of optical microscope to accurately analyze pattern characteristic and type of clay mineral to provide basis for diagenesis research.</td>
</tr>
<tr>
<td>Observation of heavy mineral</td>
<td>Due to grain diameter of heavy mineral is small and it is uneasy to be inspected in optical microscope, so omission of judge may exist.</td>
<td>It can clearly present distribution form of heavy mineral through SEM back scattering image; type of heavy mineral can be accurately inspected by combining X-ray energy spectrum.</td>
<td>Large instrument avoids omission and wrong judgment of mineral so as to guarantee systematicness and comprehensiveness of inspection result.</td>
</tr>
</tbody>
</table>
1. The introduction of modern analysis instrument improves accuracy, objectiveness and reliability of rock mineral inspection.

2. Test result in different analysis instruments can verify each other to guarantee quality of rock mineral inspection result.
Report outline

Application of modern analysis instrument in sedimentary rock rock-mineral determination

Research on status of occurrence of platinum family of minerals in Sichuan Danba Cu-Ni sulfide mineral
Establish technical method of research and analysis of status of occurrence of platinum family of minerals in Sichuan Danba Cu-Ni sulfide mineral

Gain fabric characteristics, distribution discipline and other mineralogy characteristics in platinum family minerals.
Sample selection

Pt content: low → high.

$22.7 \times 10^{-9}$  $487 \times 10^{-9}$  $1670 \times 10^{-9}$
Technical method

Analysis condition selection

- 15KV
- 20KV
- 25KV
Research on status of occurrence of platinum family of minerals

Technical method

Analysis condition selection

The same area

Capture platinum family mineral information through abnormal gray level of platinum family mineral by selecting matching multiples.
The abnormal gray level of platinum family mineral may be lost if low enlargement factor is selected and platinum family mineral in rock may not be observed.
Radiation current: 10μA~15μA

Accurately gain information of isomorphism element in low-content platinum family element mineral
Instrument condition:
Acceleration voltage 20kV, extraction voltage 4.9kV, radiation current 10 μA; work distance 15mm; signal receiver: back scattering electronic receiving probe; collecting mirror current 5; objective aperture 100μm

Select ×300± for enlargement factor in low multiple mode and capture abnormal gray level in platinum family mineral and find out platinum family mineral
Adjust contrast ratio and luminance by referring to internal standard atlas to gain high quality image with clear color gradation, medium gray level and clear detail.
Research on status of occurrence of platinum family of minerals

Grain diameter, dissemination characteristic and relation with carry mineral of sperrylite.
Occurrence status of platinum family element mineral:
Main mineral is sperrylite, stibiopalladinite and tellurium antimony palladium ore
Submineral is native platinum, sulfur arsenic rhodium ore and sulfur arsenic iridium ore
It occurs in iron pyrite, magnetic pyrite and serpentine in elliptical and spindle shape
Some parts exist in isomorphism, and extremely small amount of platinum and palladium element exists in solid solution

Occurrence status of platinum family element:
It is mainly distributed in sulfide containing iron,
The content of Pt, Pb and platinum element and the content of Fe and Ni are in positive correlation
Geochemical characteristics of Fe, Ni and S pro in platinum family element in Danba area
Research on status of occurrence of platinum family of minerals

Summary

The established technical method of research and analysis of status of occurrence of platinum family of minerals in Sichuan Danba Cu-Ni sulfide mineral avoids omission and wrong judgment of mineral and provides comprehensive and accurate micro information. Currently, there is no similar research method in China; although there are mineral analysis softwares in foreign countries, they cannot solve all minerals in rock due to restriction of method. So, this method is advanced and practical.
Chengdu Geologic Survey Center of China Geological Survey:
Wang Kunyang, Xu Jinsha and Pan Zhongxi, etc.
Thank You!