稀土矿石化学分析方法 第3部分：锂、铍、钪、锰、钴、镍、铜、锌、镓、铷、铌、钼、铟、铯、钽、钨、铊、铅、铋、钍、铀及15个稀土元素含量的测定 混合酸分解—电感耦合等离子体质谱法（报批稿）

编制说明

国家地质实验测试中心
二〇二二年九月
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第一章 任务来源和编制过程

1.1 任务来源

工作内容名称：铌钽、稀土、铍矿石分析标准方法研究

所属子项目：铝土矿、典型轻稀土等 17 种标准物质和铌钽铍稀土等 8 项标准方法研制

子项目编码：DD20160095-3

任务书编号：[2015]05-03-02-081

工作起止年限：2015 年—2016 年

组织审查单位：地调局总工室

所属项目名称：地质调查标准制修订与升级推广项目

实施单位：中国地质调查局发展研究中心

所属工程名称：地质矿产调查战略与规划支撑工程

工程牵头单位：中国地质调查局发展研究中心

所属计划名称：国土资源开发与保护基础地质支撑计划

子项目承担单位：国家地质实验测试中心

本部分的研究工作主要由刘贵磊、温宏利、马生凤、许俊玉等人承担完成；分析方法主要起草人：刘贵磊、朱云、芦苒、王蕾、马生凤、张欣、许俊玉、温宏利、安子怡、屈文俊。

本部分在 2019 年 11 月列入标准制修订计划，文件号：自然资办发【2019】49 号；文件名称：《自然资源部办公厅关于印发 2019 年度自然资源标准制修订计划的通知》；标准计划号 201913033。

1.2 编制过程

1.2.1 预研阶段

本项目标准方法研究是在地调工作项目《现代光质谱技术在钨铁铜等重要矿种成矿及伴生元素同时分析中的研究与应用示范》的研究任务基础上，于 2015 年 1 月开始启动，根据任务书的要求，查询了稀土矿石检测相关的标准和文献资料，明确了本标准制定的主要问题、技术难点、技术路线和实验方案，组织内部专家论证，指出在标准制定过程中应检出限、测定限、分析步骤等内
容进一步细化，使其更具有可操作性。编制了项目工作内容设计书，设计书于2015年6月通过了中国地质调查局组织有关专家的审查，根据专家审查意见完成了对设计书的修改，同时向项目组织实施单位中国地质调查局发展研究中心提交了修改后的工作内容设计书和设计修改说明。确定了标准的工作内容为五个新增标准方法：

1. 《铌矿石 钽矿石化学分析方法 第1部分：铌、钽、钨含量的测定 封闭酸溶-电感耦合等离子体原子发射光谱法》;

2. 《铌矿石 钽矿石化学分析方法 第2部分：铌、钽、钨、罂粟、铜、锌、钽，铌和钇元素含量的测定 封闭酸溶-电感耦合等离子体原子发射光谱法》;

3. 《稀矿石化学分析方法 第1部分：二氧化硅、三氧化二铝、三氧化二铁、氧化钙、氧化镁、氧化钾、氧化钠、二氧化钛、氧化锰、五氧化二磷、锶和钡含量的测定 偏硼酸锂熔融-电感耦合等离子体原子发射光谱法》;

4. 《稀土矿石化学分析方法 第2部分：铝、铁、钙、镁、钾、钠、钛、锰，磷及15个稀土元素含量的测定 混合酸分解-电感耦合等离子体原子发射光谱法》;

5. 《稀土矿石化学分析方法 第3部分：锂、铷、钪、锰、钴、镍、铜、锌、镓、铷、铌、钼、铟、铯、钽、钨、铊、铅、铋、钍、铀及15个稀土元素含量的测定 混合酸分解-电感耦合等离子体质谱法》。

本标准方法是其中之一：稀土矿石化学分析方法 第3部分：锂、铷、钪、锰、钴、镍、铜、锌、镓、铷、铌、钼、铟、铯、钽、钨、铊、铅、铋、钍、铀及15个稀土元素含量的测定 混合酸分解-电感耦合等离子体质谱法。

根据工作部署，2016年3月完成了方法试验研究工作（包括样品分解方法的确定，质谱干扰及干扰扣除，干扰扣除对测定结果不确定度的影响，稀土元素的线性实验，介质对质谱测定的影响，分析方法测定参数等），并请地质专家协助进行了方法精密度协作试验稀土矿石样品的筛选工作，编制了标准方法草案。

1.2.2 起草阶段

2016年6月在北京召开了“稀土矿石标准方法协作试验工作会议”组织实施13家有相关测试经验的实验室参加方法精密度试验和正确度试验（在数据统计之时，收回12家实验室数据），标准编制人就方法草案技术细节作报告进行了解读，发放了协作试验样品，并组织协作单位的专家进行了研讨。
2017 年 5 月至 7 月对 12 家实验室提交的实验数据进行统计分析，对部分离
群数据进行复核，确定分析方法重复性限与再现性限，于 2017 年 8 月完成了标
准方法文本和标准编制说明征求意见稿。

1.2.3 征求意见阶段
2018 年 5 月，向地质矿产实验测试分技术委员会委员及相关实验室，发送
《稀土矿石化学分析方法 第 3 部分：锂、铍、钪、锰、钴、镍、铜、锌、镓、
铷、铌、钼、铟、铯、钽、钨、铊、铅、铋、钍、铀及 15 个稀土元素含量的测
定 混合酸分解-电感耦合等离子体质谱法》征求意见稿及编制说明，广泛征求
意见。
2018 年 7 月～2019 年 9 月，根据专家的意见，对征求意见稿进行了修改完
善。
2019 年 10 月 9 日，组织专家在北京铁道大厦召开现场技术研讨会，对本标
准方法的征求意见稿进行了研讨。
2019 年 10 月至 2022 年 8 月，对照收集的各条意见建议，对标准征求意见
g及编制说明进行修改完善，形成了标准送审稿及相关送审材料，提交至全国自然
资源标准化技术委员会勘查技术与实验测试分技术委员会，准备审查。

1.2.4 审查阶段
2022 年 8 月 25-26 日，全国自然资源与国土空间规划标准化技术委员会勘
查技术与实验测试分技术委员会组织实验测试技术专家组在北京召开标准审查
会，对本标准送审稿进行审查。与会专家一致同意修改完善后，作为行业标准上
报。
根据专家的意见，起草组对相关内容进一步验证，对文本进行了修改。2022
年 9 月完成归口技术委员会的全体委员投票，修改形成报批稿。

1.2.5 报批阶段
2022 年 9 月，完成标准报批。

1.3 参加方法精密度协作试验的单位
参加方法精密度协作试验的有 12 家单位（见表 1），代表着地质行业的实验
室的平均测试水平：

<p>| 表 1 方法精密度协作试验实验室 |</p>
<table>
<thead>
<tr>
<th>序号</th>
<th>姓名</th>
<th>学历</th>
<th>专业</th>
<th>职称</th>
<th>专业工作年限</th>
<th>对制定标准的具体贡献</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>刘贵磊</td>
<td>博士</td>
<td>分析化学</td>
<td>副研究员</td>
<td>8</td>
<td>参与方法试验和方法验证工作，标准文本及编制说明编写以及修改。</td>
</tr>
<tr>
<td>2</td>
<td>朱 云</td>
<td>博士</td>
<td>环境矿物学</td>
<td>副研究员</td>
<td>9</td>
<td>参与方法条件实验。</td>
</tr>
<tr>
<td>3</td>
<td>芦苒</td>
<td>本科</td>
<td>应用化学</td>
<td>工程师</td>
<td>9</td>
<td>参与方法条件实验。</td>
</tr>
<tr>
<td>4</td>
<td>王蕾</td>
<td>本科</td>
<td>分析化学</td>
<td>高工</td>
<td>20</td>
<td>参与方法试验。</td>
</tr>
<tr>
<td>5</td>
<td>马生凤</td>
<td>硕士</td>
<td>分析化学</td>
<td>教授级高工</td>
<td>17</td>
<td>参与方法试验和方法验证工作。</td>
</tr>
<tr>
<td>6</td>
<td>张 欣</td>
<td>硕士</td>
<td>地球化学</td>
<td>工程师</td>
<td>11</td>
<td>参与方法试验和方法验证工作。</td>
</tr>
<tr>
<td>7</td>
<td>许俊玉</td>
<td>本科</td>
<td>分析化学</td>
<td>教授级高工</td>
<td>40</td>
<td>项目负责人，制定项目设计书，指导方法试验。</td>
</tr>
<tr>
<td>8</td>
<td>温宏利</td>
<td>本科</td>
<td>分析化学</td>
<td>教授级高工</td>
<td>40</td>
<td>制定工作内容设计书，指导方法试验、组织方法精密度协作试验与数据统计分析。</td>
</tr>
<tr>
<td>9</td>
<td>安子怡</td>
<td>硕士</td>
<td>分析化学</td>
<td>副研究员</td>
<td>11</td>
<td>指导方法试验、组织方法精密度协作试验。</td>
</tr>
<tr>
<td>10</td>
<td>屈文俊</td>
<td>博士</td>
<td>地球化学</td>
<td>研究员</td>
<td>32</td>
<td>指导方法试验、组织方法精密度协作试验。</td>
</tr>
</tbody>
</table>

1.4 主要编制人员

主要编制人员情况，见表 2。
第二章 标准编制原则和确定标准主要内容的依据

2.1 标准编制的主要原则

研制的标准方法技术成熟可靠，有广泛的应用基础；分析技术先进，分析方法简单，易于掌握，能够提高分析测试水平，有助于先进技术方法的推广应用；考虑多元素同时测定，提高工作效率并尽量降低使用成本。

2.2 确定标准主要内容依据

标准方法的整体结构和内容编写方法国家标准有统一的要求和规定。我国各级标准按照我国最新发布的国家标准：标准化工作导则、指南和编写规则的規定进行编写。尤其应遵循《GB/T 1.1-2020 标准化工作导则第1部分：标准的结构和编写规则》的规定编写。方法标准还要参照《GB/T 20001.4-2015 标准编写规则第4部分：试验方法标准》、《GB/T 14505-2010 岩石和矿石化学分析方法总则及一般规定》等国家标准进行编写，尽量做到编写的标准合乎规范。

本方法的主要实验参数是通过相关的条件试验来确定的。

本标准的主要技术指标包括方法检出限、测定范围（方法定量限～方法测定上限）、精密度、正确度等。

1、方法检出限应该是指特定分析方法中，分析物能够被识别和检测的最低浓度。目前方法检出限一般采用10个全流程试剂空白，按照方法中规定的仪器条件，将仪器调整到最佳状态，连续测定值的3倍标准偏差所相当的分析物浓度。

2、方法定量限（测定范围下限）指特定分析方法中，分析物能够被识别、检测并报出数据的最低浓度，也就是说其置信度要比方法检出限更高，就是测定范围的下限。目前采用10个实验室全流程试剂空白，连续测定值的10倍标准偏差所相当的分析物浓度，作为定量分析下限的估计值。

3、方法测定范围上限一般是通过用相当于样品溶液中分析物浓度范围的标准溶液，按照方法中规定的实验条件，测定方法的光谱强度—浓度校准曲线，测定该实验条件下被测物质符合 Beer 定律的浓度范围，通过线性回归方程拟合度检验，本方法线性范围的评价参数为曲线的相关系数 $R^2 \geq 0.999$。测定上限的浓度是根据常规稀土矿石样品中痕量元素的测定范围，根据校准曲线线性范围上
限乘以稀释倍数 2000 并参照稀土矿石标准物质常规含量确定的。4、精密度和正
确度是通过按照 GB/T 6379.1—2004《测量方法与结果的准确度（正确度与精密度）
第 1 部分：总则与定义》的要求，邀请了 10 个实验室参加方法准确度协作
试验，将 5 个精密度协作试验样品发放到 10 家实验室，要求对所接受的精密度
试验样品所测试的元素提供 4 个独立分析数据，然后根据 GB/T 6379.2—2004《测
量方法与结果的准确度（正确度与精密度）第 2 部分：确定标准测量方法重复性
与再现性的基本方法》来对数据统计计算，计算出各元素重复性标准差 Sr 和重
复性限 r、再现性标准差 SR 和再现性限 R，以及它们和含量水平 m 之间的函数关
系式。正确度是依据《测量方法与结果的准确度（正确度与精密度）第 4 部分：
确定标准测量方法准确度的基本方法》计算出测量方法的偏倚 δ。
第三章  标准方法主要条件实验研究

3.1 样品分解方法的研究

本分析方法是借鉴盐酸+硝酸+氢氟酸和高氯酸（或硫酸）分解化探样品的方法，经过实验对分析方法进行了改进，使之适用稀土矿石样品的分解工作。

3.1.1 硫酸加入对稀土矿石样品分解的影响

分别使用盐酸+硝酸+氢氟酸和高氯酸分解两组稀土矿石物质 GBW07160、GBW07161、GBW07187 和 GBW07188，一组加入（1+1）硫酸 1mL，而另一组未加进行对比实验，考察酸溶中硫酸对稀土矿石分解的影响。

实验数据（ICP-MS 测定）见表 3。数据表明：加入硫酸的稀土元素的测定结果高，并与标准物质推荐值吻合，而有些未加入硫酸的稀土元素测定结果明显偏低，因此样品分解时需要加入一定量的硫酸。

表 3  加入硫酸和不加硫酸对比数据表

<table>
<thead>
<tr>
<th>检测编号</th>
<th>GBW07160 检测结果</th>
<th>GBW07161 检测结果</th>
<th>μg/g</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>加硫酸</td>
<td>未加硫酸</td>
<td>推荐值</td>
</tr>
<tr>
<td>La</td>
<td>92.2</td>
<td>68</td>
<td>93.8</td>
</tr>
<tr>
<td>Ce</td>
<td>25.8</td>
<td>18.3</td>
<td>28.3</td>
</tr>
<tr>
<td>Pr</td>
<td>37.2</td>
<td>28.5</td>
<td>37</td>
</tr>
<tr>
<td>Nd</td>
<td>189</td>
<td>148</td>
<td>189</td>
</tr>
<tr>
<td>Sm</td>
<td>126</td>
<td>101</td>
<td>129</td>
</tr>
<tr>
<td>Eu</td>
<td>1.13</td>
<td>0.97</td>
<td>1.6</td>
</tr>
<tr>
<td>Gd</td>
<td>251</td>
<td>209</td>
<td>234</td>
</tr>
<tr>
<td>Tb</td>
<td>50.7</td>
<td>43.3</td>
<td>49.1</td>
</tr>
<tr>
<td>Dy</td>
<td>326</td>
<td>282</td>
<td>314</td>
</tr>
<tr>
<td>Ho</td>
<td>72.5</td>
<td>63.4</td>
<td>65.5</td>
</tr>
<tr>
<td>Er</td>
<td>217</td>
<td>191</td>
<td>192</td>
</tr>
<tr>
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<td>µg/g</td>
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<td>未加硫酸</td>
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<td>553</td>
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<td>89.3</td>
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<td>1670</td>
<td>2000</td>
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<tr>
<td>Er</td>
<td>59.3</td>
<td>57.8</td>
<td>56.7</td>
</tr>
</tbody>
</table>

GBW07187检测结果

检测编号：加硫酸 | 未加硫酸 | 推荐值 | s
La：2054 | 1939 | 2132 | 85
Ce：422 | 402 | 431 | 33
Pr：714 | 701 | 737 | 33
Nd：1960 | 1897 | 2060 | 86
Sm：562 | 553 | 569 | 52
Eu：6.93 | 6.67 | 8.3 | 0
Gd：786 | 768 | 790 | 17
Tb：162 | 157 | 162 | 9
Dy：992 | 962 | 1046 | 87
Ho：213 | 206 | 201 |
Er：593 | 572 | 595 | 17
Tm：74.3 | 71.3 | 72.6 | 7.3
Yb：453 | 441 | 450 | 26
Lu：59.3 | 57.8 | 56.7 | 4.4

GBW07188检测结果

检测编号：加硫酸 | 未加硫酸 | 推荐值 | s
La：1900 | 1670 | 2000 | 170
3.1.2 高氯酸加入对稀土矿石样品分解的影响

分别使用盐酸+硝酸+氢氟酸和硫酸分解两组稀土矿石物质 GBW07160、GBW07161、GBW07187 和 GBW07188，一组加入高氯酸 1mL，而另一组未加进行对比实验，考察酸溶中高氯酸对稀土矿石分解的影响。

实验数据（ICP-MS 测定）见表 4。数据表明：加不加高氯酸对稀土矿石的测定结果无明显的影响，测定结果与标准物质的推荐值吻合的很好；但在样品含有机质比较多时，未加高氯酸制备的样品溶液中会含有比较多的碳，所以本方法确定采用盐酸+硝酸+氢氟酸+高氯酸和硫酸分解稀土矿石样品。

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<tr>
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<tr>
<td>Sm</td>
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<td>Eu</td>
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<td>加高氯酸</td>
</tr>
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<td>------</td>
<td>-----------</td>
</tr>
<tr>
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<tr>
<td>Y</td>
<td>978</td>
</tr>
<tr>
<td>La</td>
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</tr>
<tr>
<td>Ce</td>
<td>187</td>
</tr>
<tr>
<td>Pr</td>
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<tr>
<td>Nd</td>
<td>1652</td>
</tr>
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<td>Sm</td>
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</tr>
<tr>
<td>Eu</td>
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<td>Gd</td>
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<td>39.7</td>
</tr>
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<td>Ho</td>
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<td>Yb</td>
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<tr>
<td>Lu</td>
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<th>s</th>
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<td>2211</td>
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<td>90</td>
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3.1.3 硫酸加入量

采用标准物质 GBW07159 为实验样品，在样品分解时分别加入（1+1）硫酸 1.00mL、0.75mL、0.50mL、0.25mL，进行对比实验，实验数据（ICP-AES 测定）见表 5。数据表明：当硫酸加入量为 0.25mL 时，Al、Fe、Ca、Mg、Mn 的结果明显偏低，而当硫酸加入量大于 0.5mL 以上时，结果基本一致。
### 表 5  硫酸加入量对比数据表

<table>
<thead>
<tr>
<th>硫酸加入量（mL）</th>
<th>Al</th>
<th>Fe</th>
<th>Ca</th>
<th>Mg</th>
<th>K</th>
<th>Na</th>
<th>Ti</th>
<th>Mn</th>
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<td>79310</td>
<td>8166</td>
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<td>472</td>
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<td>1265</td>
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<td>130</td>
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<td>s</td>
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<td>1000</td>
<td>104</td>
<td>12</td>
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### 3.1.4 盐酸提取实验

用标准物质 GBW07160、GBW07161、GBW07187 和 GBW07188 为实验样品，在样品分解过程中，分别采用浓盐酸和（1+1）盐酸进行复溶对比实验，实验数据 (ICP-MS 测定) 见表 6，浓盐酸复溶结果明显偏低，而 (1+1) 盐酸复溶结果基本与标准物质推荐值吻合。

### 表 6 浓盐酸和（1+1）盐酸提取实验数据对比表

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<th>GBW07160 检测结果</th>
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检测原始浓盐酸（1+1）盐酸推荐值s

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检测原始浓盐酸（1+1）盐酸推荐值s

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<th>GBW07188检测结果</th>
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### 3.2 质谱干扰及干扰扣除

质谱分析方法的特点是：谱线简单、干扰相对比较小，但还是存在着氧化物干扰、复合粒子干扰、同质异位素干扰和二次离子等干扰，特别是轻稀土含量很高，而重稀土含量很低的样品（白云鄂博稀土矿），其轻稀土对重稀土的氧化物干扰是非常严重的，以至于（如 Gd、Tb）无法准确测定。应用质谱测定稀土矿石中常见的干扰元素和被干扰元素见列表 7。

<p>| 表 7 常见干扰元素和被干扰元素 |</p>
<table>
<thead>
<tr>
<th>干扰元素</th>
<th>干扰元素丰度</th>
<th>被干扰元素</th>
<th>被测元素丰度</th>
<th>干扰的离子</th>
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<td>11.232</td>
<td>$^{153}$Eu</td>
<td>24.84</td>
<td>$^{137}$Ba$^{16}$O$^+$</td>
</tr>
<tr>
<td>$^{142}$Ce</td>
<td>11.114</td>
<td>$^{158}$Gd</td>
<td>24.84</td>
<td>$^{142}$Ce$^{16}$O$^+$</td>
</tr>
<tr>
<td>$^{141}$Pr</td>
<td>100</td>
<td>$^{158}$Gd</td>
<td>24.84</td>
<td>$^{141}$Pr$^{16}$OH$^+$</td>
</tr>
<tr>
<td>$^{142}$Nd</td>
<td>27.2</td>
<td>$^{158}$Gd</td>
<td>24.84</td>
<td>$^{142}$Nd$^{16}$O$^+$</td>
</tr>
<tr>
<td>$^{143}$Nd</td>
<td>12.2</td>
<td>$^{159}$Tb</td>
<td>100</td>
<td>$^{143}$Nd$^{16}$O$^+$</td>
</tr>
<tr>
<td>$^{148}$Nd</td>
<td>5.7</td>
<td>$^{164}$Dy</td>
<td>28.18</td>
<td>$^{148}$Nd$^{16}$O$^+$</td>
</tr>
<tr>
<td>$^{148}$Sm</td>
<td>11.24</td>
<td>$^{164}$Dy</td>
<td>28.18</td>
<td>$^{148}$Sm$^{16}$O$^+$</td>
</tr>
<tr>
<td>$^{149}$Sm</td>
<td>13.82</td>
<td>$^{165}$Ho</td>
<td>100</td>
<td>$^{149}$Sm$^{16}$O$^+$</td>
</tr>
<tr>
<td>$^{150}$Nd</td>
<td>5.6</td>
<td>$^{166}$Er</td>
<td>33.61</td>
<td>$^{150}$Nd$^{16}$O$^+$</td>
</tr>
<tr>
<td>$^{150}$Sm</td>
<td>7.8</td>
<td>$^{166}$Er</td>
<td>33.61</td>
<td>$^{150}$Sm$^{16}$O$^+$</td>
</tr>
<tr>
<td>$^{142}$Ce</td>
<td>11.114</td>
<td>$^{71}$Ga</td>
<td>39.892</td>
<td>$^{142}$Ce$^{2+}$</td>
</tr>
<tr>
<td>$^{142}$Nd</td>
<td>27.2</td>
<td>$^{71}$Ga</td>
<td>39.892</td>
<td>$^{142}$Nd$^{2+}$</td>
</tr>
<tr>
<td>$^{148}$Nd</td>
<td>5.7</td>
<td>$^{74}$Ge</td>
<td>36.28</td>
<td>$^{148}$Nd$^{2+}$</td>
</tr>
<tr>
<td>$^{148}$Sm</td>
<td>11.24</td>
<td>$^{74}$Ge</td>
<td>36.28</td>
<td>$^{148}$Sm$^{3+}$</td>
</tr>
</tbody>
</table>

用 Ce 的溶液为调试液（测定 $^{142}$Ce 和 $^{142}$Ce$^{16}$O$^+$ 的计数，计算仪器氧化物产率），调整仪器的氧化物产率从 1.4%～2.6%，然后用 1000ng/mL 单元素标准实测各氧化物干扰系数的变化情况，详见表 8。

| 表 8 实测各氧化物干扰系数随氧化物产率的变化情况 |
| 仪器调试的氧化物产率 % | 1.40 | 1.90 | 2.30 | 2.50 | 2.60 |
用 Ce 的溶液为调试液（测定 $^{142}$Ce 和 $^{144}$Ce$^{++}$的计数），计算仪器二次离子产率，调整仪器的二次离子产率从 1.8%~3.0%，然后用 1000ng/mL 单元素标准实测各二次离子干扰系数的变化情况，详见表 9。

<table>
<thead>
<tr>
<th>干扰元素</th>
<th>被干扰元素</th>
<th>干扰系数</th>
<th>干扰系数</th>
<th>干扰系数</th>
<th>干扰系数</th>
<th>干扰系数</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ba</td>
<td>Eu $^{153}$</td>
<td>$0.313 \times 10^{-3}$</td>
<td>$0.354 \times 10^{-3}$</td>
<td>$0.394 \times 10^{-3}$</td>
<td>$0.514 \times 10^{-3}$</td>
<td>$0.346 \times 10^{-3}$</td>
</tr>
<tr>
<td>Ce</td>
<td>Gd $^{158}$</td>
<td>$8.496 \times 10^{-3}$</td>
<td>$10.799 \times 10^{-3}$</td>
<td>$10.157 \times 10^{-3}$</td>
<td>$16.333 \times 10^{-3}$</td>
<td>$17.802 \times 10^{-3}$</td>
</tr>
<tr>
<td>Zr</td>
<td>Ag $^{199}$</td>
<td>$1.748 \times 10^{-3}$</td>
<td>$1.637 \times 10^{-3}$</td>
<td>$1.695 \times 10^{-3}$</td>
<td>$1.681 \times 10^{-3}$</td>
<td>$1.431 \times 10^{-3}$</td>
</tr>
<tr>
<td>Nd</td>
<td>Gd $^{158}$</td>
<td>$21.691 \times 10^{-3}$</td>
<td>$23.006 \times 10^{-3}$</td>
<td>$23.161 \times 10^{-3}$</td>
<td>$29.786 \times 10^{-3}$</td>
<td>$30.092 \times 10^{-3}$</td>
</tr>
<tr>
<td>Nd</td>
<td>Tb $^{159}$</td>
<td>$2.48 \times 10^{-3}$</td>
<td>$2.557 \times 10^{-3}$</td>
<td>$2.64 \times 10^{-3}$</td>
<td>$3.291 \times 10^{-3}$</td>
<td>$3.182 \times 10^{-3}$</td>
</tr>
<tr>
<td>Nd</td>
<td>Dy $^{164}$</td>
<td>$4.035 \times 10^{-3}$</td>
<td>$4.304 \times 10^{-3}$</td>
<td>$4.471 \times 10^{-3}$</td>
<td>$5.429 \times 10^{-3}$</td>
<td>$5.364 \times 10^{-3}$</td>
</tr>
<tr>
<td>Nd</td>
<td>Er $^{166}$</td>
<td>$3.871 \times 10^{-3}$</td>
<td>$4.079 \times 10^{-3}$</td>
<td>$4.093 \times 10^{-3}$</td>
<td>$5.032 \times 10^{-3}$</td>
<td>$4.942 \times 10^{-3}$</td>
</tr>
<tr>
<td>Sm</td>
<td>Dy $^{164}$</td>
<td>$0.939 \times 10^{-3}$</td>
<td>$1.229 \times 10^{-3}$</td>
<td>$1.166 \times 10^{-3}$</td>
<td>$1.819 \times 10^{-3}$</td>
<td>$1.774 \times 10^{-3}$</td>
</tr>
</tbody>
</table>

由表 8 和表 9 数据可以看出，由稀土产生的干扰还是比较多的，有些干扰也是比较严重的，如 Ce、Nd 对 Gd 的干扰，在数据处理时需要扣除干扰。一般样品需要扣除 Ba 对 Eu 的干扰，扣除 Ce、Nd 对 Gd 的干扰，扣除 Nd 对 Tb 的干扰，其余的干扰量非常小，不需要进行扣除校正。但对于白云鄂博的稀土矿石样品，其轻稀土含量非常高（La、Ce、Pr、Nd 可达到百分含量），而 Gd、Tb、Dy、Ho、Er、Ga 和 Ge 等元素含量非常低，其干扰也需要扣除；当干扰特别严重时，上述元素无法直接测定，需要分离干扰之后才能测定。

一般在测定稀土矿石的分析过程中，仪器调试时尽可能降低氧化物产率，以降低氧化物对被测元素干扰的影响，保证被测元素测定结果的准确度。
3.3 干扰扣除对测定结果的不确定度的影响

如何判定扣除干扰后的测定结果的准确性是非常重要的问题，探讨这个问题首先解析扣除干扰后的测定结果表示公式:

\[ \omega_{\text{结果}} = \omega_{\text{测定}} - \omega_{\text{C}} \cdot k = \omega_{\text{A}} + \omega_{\text{B}} - \omega_{\text{C}} \cdot k \]

注：\( \omega_{\text{结果}} \) — 被测元素的测定结果；\( \omega_{\text{测定}} \) — 被测元素的测定值，其包含 \( \omega_{\text{A}} \) — 被测元素的贡献量和 \( \omega_{\text{B}} \) — 干扰元素对被测元素的贡献量；\( \omega_{\text{C}} \) — 干扰元素测定量；\( k \) — 干扰系数

则扣除干扰后测定结果的不确定度用公式表示为:

\[ S^2 = S_A^2 + S_B^2 + S_C^2 \]

注：\( S \) — 被测元素在扣除干扰后测定结果的不确定度；\( S_A \) — 被测元素贡献量的不确定度；\( S_B \) — 干扰元素对被测元素贡献量的不确定度；\( S_C \) — 干扰元素测定量的不确定度

由于只有测定值 \( \omega_{\text{测定}} \) 和 \( \omega_{\text{C}} \)，而并不知道 \( \omega_{\text{A}} \) 和 \( \omega_{\text{B}} \) 值，所以假设测定中各量的相对不确定度相当，则有 \( S_B \approx S_C \)，并且干扰系数求得准确。

当 \( \omega_A=6\omega_B \) 时，即被测元素贡献量是干扰贡献量的 6 倍以上，则 \( S_A^2 + S_B^2 + S_C^2=1.055 S_A^2 \)，测定结果的合成不确定度 \( S=1.027S_A \)，即表示干扰及扣除后测定结果准确度增加了 2.7%。

当 \( \omega_A=5\omega_B \) 时，即被测元素贡献量是干扰贡献量的 5 倍以上，则 \( S_A^2 + S_B^2 + S_C^2=1.08 S_A^2 \)，测定结果的合成不确定度 \( S=1.039S_A \)，即表示干扰及扣除后测定结果准确度增加了 3.9%。

当 \( \omega_A=4\omega_B \) 时，即被测元素贡献量是干扰贡献量的 4 倍，则 \( S_A^2 + S_B^2 + S_C^2=1.125 S_A^2 \)，合成不确定度 \( S=1.061S_A \)，则表示扣除干扰后对测定结果不确定度增加了 6.1%。

当 \( \omega_A=3\omega_B \) 时，即被测元素贡献量是干扰贡献量的 3 倍，则 \( S_A^2 + S_B^2 + S_C^2=1.222 S_A^2 \)，合成不确定度 \( S=1.106S_A \)，则表示扣除干扰后对测定结果不确定度增加了 10.6%。

当 \( \omega_A=2\omega_B \) 时，即被测元素贡献量是干扰贡献量的 2 倍，则 \( S_A^2 + S_B^2 + S_C^2=1.5 S_A^2 \)，合成不确定度 \( S=1.225S_A \)，则表示扣除干扰后对测定结果不确定度
增加了 22.5%。

当 $\omega_A=\omega_B$ 时，即被测元素贡献量是干扰贡献量的相当，$S_A^2+S_B^2+S_C^2=3S_A^2$，合成不确定度 $S=1.732S_A$，则表示扣除干扰后对测定结果不确定度增加了 73.2%。

表 10 干扰扣除后对合成不确定度的影响量

<table>
<thead>
<tr>
<th>扣除量/元素贡献量</th>
<th>1/10</th>
<th>1/9</th>
<th>1/8</th>
<th>1/7</th>
<th>1/6</th>
<th>1/5</th>
<th>1/4</th>
<th>1/3</th>
<th>1/2</th>
<th>1/1</th>
</tr>
</thead>
<tbody>
<tr>
<td>$S_A$ 的增加量（%）</td>
<td>1.00</td>
<td>1.23</td>
<td>1.55</td>
<td>2.02</td>
<td>2.7</td>
<td>3.9</td>
<td>6.1</td>
<td>10.6</td>
<td>22.5</td>
<td>73.2</td>
</tr>
</tbody>
</table>

从表 10 的计算数据可以得出以下结论：

当被测元素贡献量是干扰量的 3 倍以上时，扣除测定值的四分之一量以下时，最终结果的合成不确定度有可能增加了 10%，结果是可以接受的；

当被测元素贡献量是干扰量的 2 倍时，即扣除测定值的三分之一量时，最终结果的合成不确定度有可能增加了 22.5%，结果需谨慎的对待；

当被测元素贡献量和干扰量相当时，即扣除测定值的二分之一量时，最终结果的合成不确定度将增加了 73.2%，结果已不能接受。

3.4 稀土元素的线性实验

选择浓度为 0.01、0.1、1、10、100、1000、10000ng/mL 的稀土元素的系列标准溶液为横坐标，各稀土元素的强度计数与内标元素的强度计数比值为纵坐标，做 ICP-MS 的线性实验，各元素的线性见图 1 和图 2，具体的强度计数比值见表 11。从相关系数和强度比数据可以看，在 0.01ng/mL～10000ng/mL 浓度范围内，绝大部分元素是线性的，但 Gd 和 Tb 在 10000ng/mL 时已经弯曲，并且各元素在 10000ng/mL 时强度计数（cps）可达 $X*10^7～X*10^8$，所以确定 ICP-MS 测定稀土的上限浓度为 5000ng/mL。
图 1 轻稀土线性图

图 2 重稀土线性图

表 11 各稀土元素强度计数与内标元素强度计数比值

<table>
<thead>
<tr>
<th>浓度 (ng/mL)</th>
<th>Sc</th>
<th>Y</th>
<th>La</th>
<th>Ce</th>
<th>Pr</th>
<th>Nd</th>
<th>Sm</th>
<th>Eu</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>0.0010</td>
<td>0.00085</td>
<td>0.0022</td>
<td>0.0038</td>
<td>0.0028</td>
<td>0.0008</td>
<td>0.0007</td>
<td>0.0010</td>
</tr>
<tr>
<td>0.1</td>
<td>0.011</td>
<td>0.009</td>
<td>0.020</td>
<td>0.020</td>
<td>0.022</td>
<td>0.0058</td>
<td>0.0054</td>
<td>0.012</td>
</tr>
<tr>
<td>1</td>
<td>0.099</td>
<td>0.083</td>
<td>0.183</td>
<td>0.173</td>
<td>0.218</td>
<td>0.062</td>
<td>0.057</td>
<td>0.107</td>
</tr>
<tr>
<td>10</td>
<td>1.04</td>
<td>0.87</td>
<td>1.82</td>
<td>1.73</td>
<td>2.18</td>
<td>0.59</td>
<td>0.55</td>
<td>1.08</td>
</tr>
<tr>
<td>100</td>
<td>10.5</td>
<td>9.13</td>
<td>18.3</td>
<td>16.8</td>
<td>23.6</td>
<td>5.67</td>
<td>5.46</td>
<td>10.6</td>
</tr>
<tr>
<td>1000</td>
<td>102.9</td>
<td>86.4</td>
<td>197.9</td>
<td>187.4</td>
<td>235.9</td>
<td>63.7</td>
<td>62.2</td>
<td>121.7</td>
</tr>
<tr>
<td>10000</td>
<td>1055</td>
<td>837</td>
<td>2024</td>
<td>1953</td>
<td>2442</td>
<td>654</td>
<td>644</td>
<td>1258</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>浓度 (ng/mL)</th>
<th>Gd</th>
<th>Tb</th>
<th>Dy</th>
<th>Ho</th>
<th>Er</th>
<th>Tm</th>
<th>Yb</th>
<th>Lu</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>0.00050</td>
<td>0.00227</td>
<td>0.00044</td>
<td>0.00181</td>
<td>0.00081</td>
<td>0.00190</td>
<td>0.00069</td>
<td>0.00217</td>
</tr>
<tr>
<td>0.1</td>
<td>0.00496</td>
<td>0.01862</td>
<td>0.00569</td>
<td>0.01732</td>
<td>0.00595</td>
<td>0.01776</td>
<td>0.00600</td>
<td>0.01638</td>
</tr>
</tbody>
</table>

浓度 (ng/mL)

计数比值

强度计数比值

表 11 各稀土元素强度计数与内标元素强度计数比值

浓度 (ng/mL)
3.5 介质对质谱测定的影响

实验分别采用硝酸、盐酸不同介质的校准标准溶液和内标溶液，使用 ICP-MS 进行测定，考查介质对各元素测定强度的影响，具体数据见表 12、表 13、表 14 和表 15。

### 表 12  检测不同介质校准溶液 2 的强度计数比较

<table>
<thead>
<tr>
<th>介质情况</th>
<th>Li</th>
<th>Be</th>
<th>Cr</th>
<th>Mn</th>
<th>Co</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>标准、内标硝酸介质</td>
<td>163688</td>
<td>38832</td>
<td>335532</td>
<td>410089</td>
<td>317245</td>
<td>69482</td>
<td>141365</td>
<td>37378</td>
</tr>
<tr>
<td>标准盐酸介质，内标硝酸介质</td>
<td>244392</td>
<td>62774</td>
<td>502781</td>
<td>365067</td>
<td>289765</td>
<td>63060</td>
<td>132028</td>
<td>49607</td>
</tr>
<tr>
<td>标准、内标盐酸介质</td>
<td>277165</td>
<td>71575</td>
<td>568588</td>
<td>359670</td>
<td>289712</td>
<td>63506</td>
<td>135546</td>
<td>57855</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>介质情况</th>
<th>Ga</th>
<th>Rb</th>
<th>Sr</th>
<th>Mo</th>
<th>Rh</th>
<th>Cd</th>
<th>In</th>
<th>Cs</th>
</tr>
</thead>
<tbody>
<tr>
<td>标准、内标硝酸介质</td>
<td>141476</td>
<td>304967</td>
<td>412710</td>
<td>85745</td>
<td>341555</td>
<td>42961</td>
<td>561614</td>
<td>518328</td>
</tr>
<tr>
<td>标准盐酸介质，内标硝酸介质</td>
<td>125499</td>
<td>290844</td>
<td>356526</td>
<td>79661</td>
<td>310561</td>
<td>54484</td>
<td>626113</td>
<td>603841</td>
</tr>
<tr>
<td>标准、内标盐酸介质</td>
<td>125146</td>
<td>271145</td>
<td>336695</td>
<td>75990</td>
<td>180167</td>
<td>56137</td>
<td>623767</td>
<td>597075</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>介质情况</th>
<th>Ba</th>
<th>Re</th>
<th>Tl</th>
<th>Pb</th>
<th>Bi</th>
<th>Th</th>
<th>U</th>
</tr>
</thead>
<tbody>
<tr>
<td>标准、内标硝酸介质</td>
<td>413543</td>
<td>190011</td>
<td>319613</td>
<td>225665</td>
<td>328153</td>
<td>331120</td>
<td>398195</td>
</tr>
<tr>
<td>标准盐酸介质，内标硝酸介质</td>
<td>493190</td>
<td>224561</td>
<td>320189</td>
<td>250806</td>
<td>420043</td>
<td>214130</td>
<td>414499</td>
</tr>
<tr>
<td>标准、内标盐酸介质</td>
<td>488573</td>
<td>211895</td>
<td>298632</td>
<td>248017</td>
<td>442269</td>
<td>218592</td>
<td>410295</td>
</tr>
</tbody>
</table>

### 表 13  检测不同介质校准溶液 3 的强度计数比较

<table>
<thead>
<tr>
<th>介质情况</th>
<th>Ti</th>
<th>Zr</th>
<th>Nb</th>
<th>Rh</th>
<th>Sn</th>
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表 14 测定不同介质校准溶液 5 的强度计数比较

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从上述数据可以看出，在盐酸和硝酸介质中各元素的灵敏度是有明显的差异，大多数元素是盐酸介质的强度计数要高于硝酸介质的强度计数，但也有元素是硝酸介质的强度计数高于盐酸介质的强度计数，如：Mn、Co、Ni、Ga、Sr、Mo，特别是内标元素Rh从硝酸介质的30多万的计数降到盐酸介质的不到30万的计数，变化非常大。所以ICP-MS测定时必须严格保证各溶液的介质的一致性。

### 3.6 实验室内验证数据

#### 3.6.1 正确度验证

在本实验室使用本方法分解稀土矿石标准物质，ICP-MS测定GBW07158、GBW07159、GBW07160、GBW07161、GBW07187、GBW07188，对该方法的正确度进行了验证，大部分元素相对误差(RE%) < 15%，正确度良好。结果见表 16。

表 16 敞开酸溶稀土矿石标准物质检测结果

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### 3.6.2 精密度验证

本实验室使用本方法分别独立分解 12 份稀土矿石国家一级标准物质 GBW07160、GBW07187，ICP-MS 测定，对该方法的精密度进行了验证，大部分元素相对标准偏差（RSD%）< 5%，精密度良好。检测结果见表 17。

表 17 敞开酸溶稀土矿石标准物质的精密度检测结果
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上述准确度和精密度结果表明，混合酸分解稀土矿石，ICP-MS 测定的分析方法能够比较好的满足一般稀土矿石对稀土元素等元素的分析要求。
### 3.7 分析方法参数

本方法的测量条件实验是在 PE 公司的光谱仪 PerkinElmer NexION 300D 上进行的，由于为多元素分析，要兼顾各元素的结果，经过仪器测量条件实验优化得到仪器的参考工作参数见表 18，不同的厂家的仪器，工作参数各不相同，需要根据所测样品做仪器测量条件实验来确定。

表 18 电感耦合等离子体质谱仪工作参考条件

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### 3.8 方法质量参数确定（方法准确度协作试验）

#### 3.8.1 方法精密度协作试验样品的选择

根据 GB/T 6379.1-2004 测量方法与结果的准确度（正确度与精密度）第一部分：总则与定义，用于方法准确度试验的物料必须满足代表性、均匀性和较大的水平变化范围。选择五个稀土矿石的国家一级标准物质为方法精密度试验样品，见表 19。验证样品各稀土元素的含量见表 20，所测元素力尽量覆盖涵盖较大的水平范围，但因为是多元素分析，难免有顾此失彼的情况。

表 19 精密度协作试验样品编号表

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3.8.2 方法精密度协作试验数据统计分析

按照 GB/T 6379.1-2004 的要求，邀请了 13 个实验室参加方法准确度协作试验（在数据统计之时，收回 12 家实验室数据），5 个精密度协作试验样品发放到各家实验室（实验室代码和名称见表 2），每个实验室的实验人员依据提供的分析方法（草案）。12 家实验数据，按照 GB/T6379.2-2004 的要求对所接受的精密试验样品要求测试的元素提供 4 个独立分析数据，给每个参加试验的实验室只提供测定元素的含量范围。

将检测数据汇总，按 GB/T6379.2-2004 统计计算方法的重复性限与再现性限及偏倚，各元素分析准确度协作试验数据汇总统计分析见表 21～表 92。结果表明，给出的水平范围内，其绝对差值超过重复性限 (r) 和再现性限 (R) 的情况均不超过 5%。

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注：**为离群值，*为岐离值，离群值剔除，岐离值仍参与计算。
表 23  Be 准确度协作试验数据汇总表

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### 表 26  Sc 准确度协作试验数据统计表

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### 表 28  Mn 准确度协作试验数据统计表

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### 表 31  Ni 准确度协作试验数据汇总表

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### 表 32  Ni 准确度协作试验数据统计表

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### 表 33  Cu 准确度协作试验数据汇总表

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### 表 34  Cu 准确度协作试验数据统计表

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### 表 36  Zn 准确度协作试验数据统计表

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### 表 37  Ga 准确度协作试验数据汇总表

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### 表 38  Ga 准确度协作试验数据统计表

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### 表 39  Rb 准确度协作试验数据汇总表

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### 表40  Rb准确度协作试验数据统计表

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<th>GBW07188</th>
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<td>12</td>
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<td>总平均值（μg/g）</td>
<td>655</td>
<td>647</td>
<td>109</td>
<td>1079</td>
<td>389</td>
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<td>613</td>
<td>101</td>
<td>1100</td>
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<td>8.14</td>
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<td>-3.64</td>
</tr>
<tr>
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<td>9.716</td>
<td>3.004</td>
<td>25.551</td>
<td>7.016</td>
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表 4 1  Y 准确度协作试验数据汇总表

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<th>ug/g</th>
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<td>GBW07161</td>
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<td>1008</td>
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<td>2483</td>
<td>1003</td>
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<td>1005</td>
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### 表 42 Y 准确度协作试验数据统计表

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<th>样品/水平</th>
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<th>GBW07161</th>
<th>GBW07187</th>
<th>GBW07188</th>
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<tr>
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<td>2386</td>
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### 表 43 Nb 准确度协作试验数据汇总表

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分析元素: Nb  计量单位: ug/g

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<th>GBW07161</th>
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表44 Nb 准确度协作试验数据统计表

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<th>GBW07161</th>
<th>GBW07187</th>
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</tr>
</thead>
<tbody>
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<tr>
<td>相对误差(RE%)</td>
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<td>-</td>
<td>-</td>
<td>-</td>
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表45 Mo 准确度协作试验数据汇总表

| 分析元素: Mo  计量单位: ug/g |
|----------|-----------|-----------|-----------|-----------|-----------|
| 外发样品 | GBW07159 | GBW07160 | GBW07161 | GBW07187 | GBW07188 |
| 1_1 | 0.44 | 0.48 | 0.64 | 1.12 | 1.01 |
| 1_2 | 0.42 | 0.44 | 0.60 | 1.16 | 1.01 |
| 1_3 | 0.41 | 0.44 | 0.68 | 1.13 | 1.05 |
| 1_4 | 0.41 | 0.45 | 0.62 | 1.16 | 1.05 |
| 2_1 | 0.50 | 0.50 | 0.75 | 1.15 | 0.98 |
| 2_2 | 0.52 | 0.48 | 0.80 | 1.20 | 0.95 |
| 2_3 | 0.51 | 0.50 | 0.70 | 1.18 | 0.93 |
| 2_4 | 0.53 | 0.51 | 0.79 | 1.25 | 0.97 |
| 3_1 | 0.28 | 0.26 | 0.52 | 0.93** | 1.14 |
| 3_2 | 0.31 | 0.20 | 0.57 | 1.01** | 1.01 |
| 3_3 | 0.21 | 0.29 | 0.45 | 0.95** | 1.01 |
| 3_4 | 0.28 | 0.29 | 0.63 | 1.34** | 1.04 |
| 4_1 | 0.43 | 0.47 | 0.67 | 1.09 | 1.07 |
| 4_2 | 0.42 | 0.48 | 0.62 | 1.12 | 1.06 |
| 4_3 | 0.44 | 0.44 | 0.68 | 1.14 | 1.09 |
| 4_4 | 0.41 | 0.45 | 0.63 | 1.08 | 1.11 |
| 5_1 | 0.41 | 0.41 | 0.59 | 0.95 | 1.14 |
### 分析元素: Mo

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<th>GBW07161</th>
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<th>GBW07188</th>
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<tr>
<td>7_2</td>
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表 46  Mo 准确度协作试验数据统计表

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<th>GBW07187</th>
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<td>-</td>
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<td>-</td>
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<tr>
<td>相对误差（RE%）</td>
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<td>-</td>
<td>-</td>
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## 表 48  In 准确度协作试验数据统计表

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## 表 49  Cs 准确度协作试验数据汇总表

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<td>外发样品</td>
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### 表 50  Cs 准确度协作试验数据统计表

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### 表 51  La 准确度协作试验数据汇总表

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重复性标准差（$S_r$） | 0.399 | 1.912 | 41.930 | 42.680 | 35.498 |
| 再现性标准差（$S_R$） | 0.956 | 5.189 | 148.005 | 113.996 | 83.234 |
| 重复性限（$r$） | 1.118 | 5.355 | 117.404 | 119.504 | 99.394 |
| 再现性限（$R$） | 2.676 | 14.530 | 414.415 | 319.188 | 233.055 |

$γ=S_R/S_r$ | 2.39 | 2.71 | 3.53 | 2.67 | 2.34 |

$A=1.96f$ | 0.55 | 0.56 | 0.55 | 0.54 | 0.55 |

测量方法偏倚 $δ$ | -0.37 | -2.07 | 130.50 | 98.79 | 17.86 |
| $δ-AS_r$ | -0.89 | -4.97 | 49.32 | 37.78 | -27.85 |
| $δ+AS_r$ | 0.16 | 0.84 | 211.68 | 159.81 | 63.57 |

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表 54  Ce 准确度协作试验数据统计表

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表 55  Pr 准确度协作试验数据汇总表

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### 表 57 Nd 准确度协作试验数据汇总表

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表 58  Nd 准确度协作试验数据统计表

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γ=SR/Sr

A=1.96f

表 59  Sm 准确度协作试验数据汇总表

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### 表 60 Sm 准确度协作试验数据统计表

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### 表 62  Eu 准确度协作试验数据统计表

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表 64  Gd 准确度协作试验数据统计表

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Tb 准确度协作试验数据统计表

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表 67  
Dy 准确度协作试验数据汇总表

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### 表 68 Dy 准确度协作试验数据统计表

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### 表 69 Ho 准确度协作试验数据统计表

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表 70  Ho 准确度协作试验数据统计表

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表 71  Er 准确度协作试验数据汇总表
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计量单位: μg/g

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表 72 Er 准确度协作试验数据统计表

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表 73 Tm 准确度协作试验数据汇总表
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### 表 74 Tm 准确度协作试验数据统计表

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表 76 Yb 准确度协作试验数据统计表
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表 77  
Lu 准确度协作试验数据汇总表

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表 78 Lu 准确度协作试验数据统计表

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表 82  W 准确度协作试验数据统计表
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表 84 Tl 准确度协作试验数据统计表

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### 表 87  Bi 准确度协作试验数据汇总表

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表 88 Bi 准确度协作试验数据统计表
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表 90 Th 准确度协作试验数据统计表

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<td>-0.13</td>
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表 91 U 准确度协作试验数据汇总表

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<th>GBW07161</th>
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表92  U 准确度协作试验数据统计表

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<td>相对误差（%）</td>
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3.9 主要试验的分析综述报告及技术经济论证

稀土矿石分析可分为稀土元素总量的测定、稀土元素分量的测定以及稀土矿石其它元素含量的测定。稀土元素总量一般采用经典化学方法测定，是根据各个稀土元素在化学性质上的相似性质，采用重量法、光度法和容量法进行测定。稀土元素分量的测定，主要有发射（电弧、火花等为光源）光谱法（像板采集强度信号的摄谱法）、化学光谱法、波长色散x射线荧光光谱法、离子交换-薄膜制样波长色散x射线荧光光谱法、极谱法、石墨炉原子吸收光谱法和中子活化法等。随着电感耦合等离子体发射光谱仪、电感耦合等离子体质谱仪等大型仪器的发展，相继报道了许多利用该仪器测定稀土元素的方法，目前已经成为稀土元素分量测定的主要手段，特别是电感耦合等离子体质谱仪法更是痕量或超痕量元素分析的重要手段。

在已颁布的稀土矿石标准分析方法中，只测定稀土各元素的量和主量元素，并且主量元素的测定采用化学分析方法，其分析方法的缺点是：分析流程长，步骤复杂、速度慢、测定元素单一、劳动强度大。

稀土元素采用过氧化钠碱熔，经过阳离子交换树脂分离富集，ICP-AES法测定15个稀土元素，分析流程长，步骤复杂、速度慢、劳动强度大。

本部分采用ICP-MS分析技术，结合新的混合酸分解样品方法建立的稀土矿石化学分析方法，其特点是：测定元素多、分析速度快，各元素的动态线性范围宽，能够测定不同品位的稀土矿石样品的次痕量元素及稀土元素，分析方法简单，易于掌握和推广。ICP-MS现已在各个行业的检测实验室，各个地区的科研院所、工厂矿山等大量装备使用，在分析测试过程中被广泛使用的分析手段，也是地质行业多元素分析的重要手段之一。
第四章 采用国际标准和国外先进标准的程度以及与国际、国内同类标准水平的对比情况

4.1 国际稀土矿石标准方法查新

经过国际 ISO 和美国 ASTM 查新，未发现有关稀土矿石相关成分分析标准方法。

4.2 国内稀土矿石标准方法查新

目前，查到的标准主要有稀土矿石中稀土分量的测定；稀土矿石中氧化钪量的测定；以及稀土精矿中稀土总量、钍量、氧化钙、氧化钯、氧化锆、氧化铌量、氧化硅量、氧化铝量、氧化铁量、稀土配分量、五氧化二磷量等项目的测定。已颁布的稀土矿石标准分析方法中，只测定稀土各元素的量和主量元素，并且主量元素的测定采用化学分析方法，其分析方法的缺点是：分析流程长，步骤复杂、速度慢，测定元素单一，劳动强度大。稀土元素的测定采用过氧化钠碱熔，经过阳离子交换树脂分离富集，ICP-AES 法测定 15 个稀土元素，分析流程长，步骤复杂、速度慢，劳动强度大。

本标准采用 ICP-MS 分析技术，结合新的混合酸分解样品方法建立的稀土矿石化学分析方法，其特点是：测定元素多，分析速度快，各元素的动态线性范围宽，能够测定不同品位的稀土矿石样品的次痕量元素及稀土元素，分析方法简单，易于掌握和推广。ICP-MS 现已在各个行业的检测实验室，各个地区的科研院所、工厂矿山等大量装备使用，在分析测试过程中被广泛使用的分析手段，也是地质行业多元素分析的重要手段之一。
第五章 与有关的现行法律、法规和标准的关系

本标准在起草时遵循了《中华人民共和国标准化法》等法律规定，按照 GB/T 1.1-2020 《标准化工作导则 第 1 部分：标准化文件的结构和起草规则》和 GB/T 20001.4-2015 《标准编写规则 第 4 部分：试验方法标准》的要求进行编写，该标准为新研制的稀土矿石化学分析方法。

本标准可实现稀土矿石次痕量多元素同时测定，是 GB/T 17417-2010 系列稀土矿石化学分析方法和 GB/T 18114-2010 系列稀土精矿化学分析方法的补充。

第六章 重大分歧意见的处理经过和依据

标准制定过程中无重大分歧意见，对一些不准确之处、与专业名词进行了进一步规范，起草小组根据专家意见进行了认真修改，通过了充分的研究与讨论，
第七章 标准作为强制性和推荐性标准的建议

我国标准化法规定：保障人体健康、人身财产安全的标准和法律、行政法规规定强制执行的标准属于强制性标准。
由于本标准不涉及以下几方面的技术要求：
1、有关国家安全的技术要求；
2、保障人体健康和人身、财产安全的要求；
3、产品及产品生产、储运和使用中的安全、卫生、环境保护要求及国家需要控制的工程建设的其他要求；
4、工程建设的质量、安全、卫生、环境保护按要求及国家需要控制的工程建设的其他要求；
5、污染物排放限值和环境质量要求；
6、保护动植物生命安全和健康要求；
7、防止欺骗、保护消费者利益的要求；
8、国家需要控制的重要产品的技术要求。
因此，建议本标准为推荐性标准。

第八章 贯彻标准的要求和措施建议

本标准发布后，建议由全国自然资源与国土空间规划标准化技术委员会制定
标准贯彻实施计划。有条件的实验室，可根据需要选择采用本标准开展稀土矿石样品中相关成分分析，以加强对本标准的推广应用。

建议标准发布后 3 个月内实施。
第九章 废止现行有关标准的建议

本标准为首次发布，无现行标准和本标准类同，不涉及废止现行标准问题。
第十章 其他应予说明的事项

10.1 关于修改标准名称的说明

标准计划名称为：《稀土矿石化学分析方法 第 3 部分：锂、铍、钪、锰等36 个元素量的测定 混合酸分解-电感耦合等离子体质谱法》，报批标准名称为：《稀土矿石化学分析方法 第 3 部分 锂、铍、钪、锰、钴、镍、铜、锌、镓、铷、铌、钼、铟、铯、钽、钨、铊、铅、铋、钍、铀及 15 个稀土元素含量的测定 混合酸分解—电感耦合等离子体质谱法》，更改的原因为：进一步规范标准名称，并与相关标准相一致，此为编辑性修改，不涉及标准范围改动。