铜铍合金化学分析方法

第1部分：铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的测定

电感耦合等离子体原子发射光谱法

编制说明

（送审稿）

西北稀有金属材料研究院宁夏有限公司

2023.9

铜铍合金化学分析方法

第1部分：铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的测定

电感耦合等离子体原子发射光谱法

编制说明

一、工作简况

（一）任务来源

根据2022年4月29日工业和信息化部发布的《工业和信息化部办公厅关于印发2022年第一批行业标准制修订和外文版项目计划的通知》（工信厅科函[2022]94号）的要求，有色金属行业标准《铜铍合金化学分析方法 第1部分：铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的测定 电感耦合等离子体原子发射光谱法》修订项目由全国有色金属标准化技术委员会负责归口，由西北稀有金属材料研究院宁夏有限公司负责起草，该项目计划编号：2022-0224T-YS，项目周期为18个月，完成年限为2023年10月。

（二）项目背景

铜铍合金作为高级精密的弹性元件材料，广泛应用于航空航天、电子通讯、自动化等领域，产品种类较多，根据铍含量分类主要有C17300（含铍量为1.80%～2.10%)，C17500（含铍量为0.40%～0.70%)，及原料铜铍中间合金锭（含铍量为3.50%～4.10%）等。其中铍、钴、镍、钛为其产品添加元素，铁、铝、硅、铅、镁、磷为产品杂质元素，均为影响合金质量的重要指标。精准控制添加元素含量范围，降低杂质元素含量是保障合金产品性能的基本前提，因此精确检测铜铍合金中各元素含量具有重要意义。

目前，铜铍合金相关检测标准主要有GB/T 5121.27-2008（铍测定范围为0.010%～3.00%）、YS/T 470.1-2004（铍测定范围为0.10%～3.00%）、YS/T 470.2-2004（铍测定范围为3.00%～6.00%）、YS/T 470.3-2004（磷测定范围为0.0010%～0.12%），其中YS/T 470.2-2004及YS/T 470.3-2004分别采用滴定法及分光光度法，操作相对繁琐，分析效率较低，难以满足目前快速发展的生产需求。

本次对于YS/T 470.1-2004的修订，充分考虑到标准的适应性，于电感耦合等离子体原子发射光谱法增加磷量的测定，同时根据实际检测需求拓宽部分元素的检测上限及测定下限：铍为0.010％～6.00％，钴、镍为0.010％～3.00％，钛为0.010％～0.50％，铁、铝、硅、镁为0.010％～0.30％，磷为0.0050％～0.20％，铅为0.0020％～0.060％。修订后方法涵盖YS/T 470.1-2004、流程复杂的YS/T 470.2-2004及YS/T 470.3-2004，提高工作效率，利于促进铜铍合金产业的高速发展。

1. 主要参加单位和工作成员及其所做的工作

1.本标准起草单位及其分工

本标准起草单位为：西北稀有金属材料研究院宁夏有限公司、五矿铍业股份有限公司、新疆有色金属研究所、富蕴恒盛铍业有限责任公司、上海有色金属工业技术监测中心有限公司。

其中，西北稀有金属材料研究院宁夏有限公司主要负责试验方案制定，试验样品收集和分发，分析方法研究，试验结果处理，标准文本、试验报告和编制说明撰写等工作；五矿铍业股份有限公司、新疆有色金属研究所为一验单位，主要负责对试验方案中的条件实验进行验证，提供精密度和准确度测试数据，以及对方法提出建议；富蕴恒盛铍业有限责任公司、上海有色金属工业技术监测中心有限公司为二验单位，主要负责提供精密度试验数据，并对标准文本提出修改意见和建议。

标准牵头单位西北稀有金属材料研究院宁夏有限公司是国内唯一的铍材研究和生产基地，国家高新技术企业，建有稀有金属特种材料国家重点实验室。公司分析检测所主持及参与多项铍、铍铝合金、铜铍合金、锑铍芯块等国家军用标准和行业标准制修订工作。五矿铍业股份有限公司系全球从矿石中提炼金属铍及其化合物的三家企业之一，是国内唯一的金属铍和高纯氧化铍生产企业。公司主要从事铍原料的资源控制，铍、锆等系列产品的冶炼、加工及相关材料的研发、制造和销售等。在本次修订中，反馈产品使用情况及方法检测需求信息，同时作为第一验证单位，提供实验样本的准确度验证及精密度数据。新疆有色金属研究所是国内唯一从事轻稀有金属锂铷铯冶炼、分析、新材料开发和推广应用的专业科研生产单位，是新疆维吾尔自治区重点科研院所之一。在本次修订过程中，作为第一验证单位，验证试验条件以及方法的定量下限，提供实验样本的准确度验证及精密度数据。富蕴恒盛铍业有限责任公司，主要经营铍及铍合金产品的加工、销售，矿产品收购、加工、销售，采矿选矿设备的加工，采选矿工艺设计技术咨询，对外贸易等。在本次修订过程中，提供实验样本的精密度数据。上海有色金属工业技术监测中心有限公司主体业务涉及第三方检测服务(含金属材料化学成分检测、性能检测、环保监测、无损检测等)、设备检定、检测培训等方面，是国家质量监督检验检疫总局全国工业产品生产许可证办公室审查部授权的检测机构。在本次修订过程中，提供实验样本的精密度数据，并对标准文稿等提出了相应的修改意见。

2. 本标准起草人员及其工作职责

本标准主要起草人：李晖、王巧、马肖、孙洪涛、温亚勇、王永生、黄华新、朱云、张健康、张新辉、谢奕斌、莫蓉、殷艺丹、李娜、关黎晓、朱新明、郝晶晶。

各起草人在本标准编制过程中的工作职责见表1。

表1 工作成员及所做工作

|  |  |
| --- | --- |
| 起草人 | 所做工作 |
| 李晖、王巧、孙洪涛、  莫蓉、殷艺丹、李娜 | 负责样品搜集、试验方案的确定、条件试验实施、样品测试、方法验证等。 |
| 马肖 | 标准技术内容审核、试验进度组织协调、标准文件和编制说明编写等。 |
| 温亚勇、王永生、黄华新、朱云、谢奕斌、关黎晓、朱新明、郝晶晶 | 对标准文件和编制说明提出修改建议、方法试验验证等。 |
| 张健康、张新辉 | 标准修订工作的整体统筹及指导，技术性修改建议等。 |

（四）主要工作过程

西北稀有金属材料研究院宁夏有限公司在接到标准修订任务后，成立了标准编制组，召开了标准项目编制启动会议，对标准编写工作进行了部署和分工，主要工作过程经历了以下几个阶段。

1.立项阶段

2020年11月，西北稀有金属材料研究院宁夏有限公司向全国有色金属标准化技术委员会重金属分标委提交了YS/T 470.1-2004《铜铍合金化学分析方法 第1部分：电感耦合等离子体原子发射光谱法测定铍、钴、镍、钛、铁、铝、硅、铅、镁量》标准修订的项目建议书、标准草案和立项报告等材料，经全体委员论证同意立项。随后由秘书处组织全体委员网络投票，投票通过后转报给工业和信息化部科技司，并挂网向社会公开征求意见。

2022年4月29日，工业和信息化部发布了《关于印发2022年第一批行业标准制修订和外文版项目计划的通知》（工信厅科函[2022]94号），正式下达该标准的修订任务，标准名称为《铜铍合金化学分析方法 第1部分：铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的测定 电感耦合等离子体原子发射光谱法》，项目计划编号为2022-0224T-YS，项目周期为18个月，完成年限为2023年10月，技术归口单位为全国有色金属标准化技术委员会。

2.起草阶段

2.1任务落实

2022年9月15日至16日，全国有色金属标准化技术委员会重金属分标委在江苏省扬州市召开，并同步召开网络会议。组织召开了《铜铍合金化学分析方法》修订任务落实会。会上确定了由西北稀有金属材料研究院宁夏有限公司牵头负责《铜铍合金化学分析方法 第1部分：铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的测定 电感耦合等离子体原子发射光谱法》的起草工作，由五矿铍业股份有限公司、新疆有色金属研究所、富蕴恒盛铍业有限责任公司、上海有色金属工业技术监测中心有限公司四家单位协助起草；明确了所采用的分析方法及其测定范围；同时确定了样品制备单位、进度安排等事项。

2.2样品收集及试验研究

2022年9月~2022年11月编制组委托五矿铍业股份有限公司、新疆有色金属研究所、富蕴恒盛铍业有限责任公司根据市场上铜铍合金产品的生产和应用情况，结合西北稀有金属材料研究院宁夏有限公司生产及研究情况，开展试验样品的成分设计、选材和制备，充分考虑到试验样品的代表性，共制备了BeCu-1#（含量近似于C17500,TBe0.5-2.5）、BeCu-2#（含量近似于C17720,TBe2）、BeCu-3#（含量近似于铜铍中间合金锭）三种牌号的铜铍合金样品为本标准统一的试验样品，相关信息见表2所示。

表2 本标准试验样品的基本信息

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 序号 | 牌号 | 样品状态 | 铍的含量水平/％ | 钴、镍的含量水平/％ | 钛的含量水平/％ | 铁、铝、硅、镁、磷的含量水平/％ | 铅的含量水平/％ |
| 1 | BeCu-1# | 粉末状 | ~0.40% | ~0.030% | ~0.030% | ~0.010% | ~0.0090% |
| 2 | BeCu-2# | 粉末状 | ~2.50% | ~0.30% | ~0.10% | ~0.10% | ~0.020% |
| 3 | BeCu-3# | 粉末状 | ~4.00% | ~3.00% | ~0.30% | ~0.20% | ~0.050% |

2022年12月~2023年5月编制组开展大量试验研究工作，形成了标准文本和编制说明。试验内容主要包含样分析谱线选择、基体效应的消除等方法影响因素讨论，以及方法相关精密度和准确度验证。

3. 征求意见阶段

编制组通过发函，全国有色金属标准化技术委员会将《铜铍合金化学分析方法 第1部分：铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的测定 电感耦合等离子体原子发射光谱法》征求意见资料在中国有色金属标准质量信息网（www.cnsmq.com）上挂网，向社会公开征求意见。征求意见的单位包括主要生产、经销、使用、科研、第三方检验机构等单位及大专院校，征求意见单位广泛且具有代表性。编制组根据汇总意见，采纳专家意见，对标准讨论稿进行修改和完善，形成预审稿。

2023年7月25日～26日由全国有色金属标准化技术委员会主持，在浙江省宁波市召开标准讨论会，来自中铝洛阳铜加工有限公司、金川集团股份有限公司、国标（北京）检验认证有限公司、广东省科学院工业分析检测中心、五矿铍业股份有限公司等单位的五十多名专家代表参会，对本文件的预审稿、编制说明进行了充分、细致的讨论，并提出修改意见。会后，编制组共发送《征求意见稿》的单位数22个，收到《征求意见稿》后，回函的单位数22个，收到《征求意见稿》后，回函并有建议或意见的单位数10个，详见标准征求意见稿意见汇总处理表。征求意见范围广泛且具代表性，编制组根据汇总意见，采纳以上专家意见，对标准预审稿进行修改和完善，形成送审稿。

二、标准的编制原则

（一）符合性

标准格式严格按照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》、GB/T 20001.4-2015《标准编写规则 第4部分：试验方法标准》等文件的要求编写，并按照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：确定标准测试方法重复性与再现性的基本方法》的要求进行试验数据的统计及重复性限和再现性限的计算。

（二）适用性和先进性

本标准是对YS/T 470.1-2004《铜铍合金化学分析方法 第1部分：电感耦合等离子体发射光谱法测定铍、钴、镍、钛、铁、铝、硅、铅、镁量》的修订起草。在充分调研国内外铜铍合金相关产品标准及行业内铜铍合金分析检测实际需求的基础上，对原标准的测定范围、测定方法、试验操作的技术细节、精密度数据进行修改或补充，进一步规范了铜铍合金中铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的分析方法，同时又体现了行业内对铜铍合金化学成分分析的技术水平，具有先进性、可操作性和广泛的适用性。主要修订内容如下：

1. 增加了电感耦合等离子体原子发射光谱法测定磷量，测定范围：0.0050％～0.20％。
2. 更改了部分元素测定范围，铍测定范围由0.10％～3.00％更改为0.010％～6.00％，钴测定范围由0.10％～3.00％更改为0.010％～3.00％，镍测定范围由0.050％～3.00％更改为0.010％～3.00％，钛测定范围由0.10％～0.50％更改为0.010％～0.50％，铅测定范围由0.0050％～0.060％更改为0.0020％～0.060％。

3）补充精密度数据，提高了标准的可参照性。

三、标准主要内容的确定依据及主要试验和验证情况分析

（一）测定方法的选择

电感耦合等离子体原子发射光谱法（ICP-AES法）具有分析线性范围宽、低的检出限、元素间干扰小、精密度好、仪器操作简便和分析速度快等优点，适用于铜铍合金中多元素含量的分析。因此本标准采用电感耦合等离子体原子发射光谱仪测定铜铍合金中铍、钴、镍、钛、铁、铝、硅、铅、镁、磷的含量。

（二）测定范围的修订

在制定铜铍合金中铍、钴、镍、钛、铁、铝、硅、铅、镁、磷元素测定范围时，根据YS-T 334-1995《铍青铜棒》，YS/T 260-2016《铜铍中间合金锭》，GB 24459-2009《铍铜合金防爆工具》，GB/T 26313-2010《铍青铜无缝管》等标准文件，以及产品客户的反馈要求，结合日常检测样品实际情况，对产品铍、钴、镍、钛、铁、铝、硅、铅、镁、磷元素范围作了拓展，最终确定出本文件铍、钴、镍、钛、铁、铝、硅、铅、镁、磷元素的测定范围数值，见表3。

表3 产品要求各元素范围与本标准各元素的测定范围的比较

|  |  |  |
| --- | --- | --- |
| 元素 | 产品要求范围/% | 本标准范围/% |
| Be | 0.010～4.10 | 0.010～6.00 |
| Co | 0.010～3.00 | 0.010～3.00 |
| Ni | 0.010～3.00 | 0.010～3.00 |
| Ti | 0.010～0.50 | 0.010～0.50 |
| Fe | 小于0.11 | 0.010～0.30 |
| Al | 0.010～0.20 | 0.010～0.30 |
| Si | 0.010～0.20 | 0.010～0.30 |
| Pb | 小于0.0020 | 0.0020～0.060 |
| Mg | 0.010～0.20 | 0.010～0.30 |
| P | 小于0.0070 | 0.0050～0.20 |

（三）样品预处理

铜基合金易溶于硝酸，但合金中硅量较高的话，仅采用硝酸溶解，溶液浑浊，样品溶解不完全，故需加入一定量的氢氟酸或盐酸助溶。试验对不同硅量的铜铍合金，分别采用硝酸、硝酸-氢氟酸、硝酸-盐酸溶解，于氢氟酸系统进样，测定结果见表4。

表 4 高硅铜铍合金溶解方法测试对比

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 预处理 | 5mL浓硝酸 | 5mL浓硝酸+5滴氢氟酸 | | 5mL浓硝酸+0.5mL盐酸 | | 原标准方法允许差/% |
| 样品编号 | 测定值/% | 测定值/% | 与原方法差值/% | 测定值/% | 与原方法差值/% |
| 1# | 0.101 | 0.102 | 0.001 | 0.102 | 0.001 | 0.005 |
| 2# | 0.158 | 0.155 | 0.003 | 0.157 | 0.001 | 0.008 |
| 3# | 0.170 | 0.172 | 0.002 | 0.173 | 0.003 | 0.008 |
| 4# | 0.202 | 0.205 | 0.003 | 0.205 | 0.003 | 0.008 |
| 5# | 0.243 | 0.257 | 0.014 | 0.259 | 0.016 | 0.008 |
| 6# | 0.302 | 0.316 | 0.014 | 0.320 | 0.018 | 0.008 |

表4可知，在铜铍合金中硅量小于或近似于0.20%时，不同溶解方法数据差距很小，其测定影响可以忽略，即采用5mL浓硝酸即可满足分析要求。当铜铍合金中硅量大于0.20%时，采用5mL浓硝酸溶解所得数据偏低，而硝酸-氢氟酸、硝酸-盐酸溶解数据更高且比较接近。实际操作中，“硝酸-盐酸”溶解比“硝酸-氢氟酸”更简便且易控制，因此，对于高硅铜铍合金（硅的质量分数大于0.20%）样品，采用加入少量盐酸助溶处理。

（四）分析谱线的选择

按照实验方法，对标准系列溶液、各单元素标准溶液（1.0 µg/mL）和样品试液进行谱图扫描分析，分别选择待测元素各2～3条谱线。观察待测元素谱线叠加时的谱图形状、峰强度及元素间的干扰，剔除元素之间有相互干扰的谱线，从中选择“灵敏度高，干扰小、信背比大”的待测元素分析谱线，见表5。

表 5 各元素推荐的分析线波长

|  |  |  |  |
| --- | --- | --- | --- |
| 元素 | 分析线波长/nm | 元素 | 分析线波长/nm |
| Be | 234.861 | Al | 396.152 |
| Co | 228.615 | Si | 288.158 |
| Ni | 231.604 | Mg | 285.213 |
| Ti | 323.658 | Pb | 220.253 |
| Fe | 259.940 | P | 178.222 |

（五）基体效应和干扰试验

实验对比了基体匹配和无基体时的各元素的工作曲线，结果见表6和图1。由表5结果及图1对比工作曲线可以看出，铜基体在本实验选定的样品溶液浓度下，对铍、镍、钛的测定谱线强度基本无显著性影响，但对钴、铁、铝、硅、铅、镁、磷的测定谱线强度有一定影响。

为保证各元素测定的准确性，对各元素标准系列溶液进行基体匹配，以消除干扰。

表6 基体影响实验

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **元素** | **标准系列** | **1#** | **2#** | **3#** | **4#** |
| Be | 纯标曲线强度值 | 192.90 | 25996.39 | 261845.78 | 1274650.14 |
| 铜基体工作曲线强度值 | 2016.03 | 26021.38 | 251144.26 | 1222244.24 |
| Co | 纯标曲线强度值 | 2.29 | 506.49 | 10221.66 | 30287.65 |
| 铜基体工作曲线强度值 | 8.56 | 499.93 | 9695.10 | 29006.24 |
| Ni | 纯标曲线强度值 | 8.65 | 397.49 | 7540.34 | 22294.66 |
| 铜基体工作曲线强度值 | 22.60 | 399.67 | 7317.68 | 21708.97 |
| Ti | 纯标曲线强度值 | 27.94 | 5401.17 | 16505.22 | 54553.78 |
| 铜基体工作曲线强度值 | 42.26 | 5686.71 | 17017.95 | 56428.01 |
| Fe | 纯标曲线强度值 | 60.11 | 1957.44 | 19329.22 | 56362.71 |
| 铜基体工作曲线强度值 | 30.31 | 1716.51 | 17351.44 | 50977.20 |
| Al | 纯标曲线强度值 | 99.24 | 2660.87 | 25375.05 | 74417.94 |
| 铜基体工作曲线强度值 | 74.43 | 2424.56 | 23813.47 | 69328.96 |
| Si | 纯标曲线强度值 | 66.35 | 955.90 | 7205.32 | 20822.20 |
| 铜基体工作曲线强度值 | 148.12 | 768.61 | 6623.97 | 19276.34 |
| Pb | 纯标曲线强度值 | 7.57 | 109.96 | 431.09 | 1215.51 |
| 铜基体工作曲线强度值 | 28.65 | 121.16 | 400.46 | 1116.34 |
| Mg | 纯标曲线强度值 | 48.62 | 3123.60 | 31138.93 | 90196.86 |
| 铜基体工作曲线强度值 | 76.59 | 2774.85 | 27968.40 | 82850.65 |
| P | 纯标曲线强度值 | 7.11 | 63.33 | 572.92 | 1142.34 |
| 铜基体工作曲线强度值 | 3.35 | 61.27 | 563.40 | 1091.61 |

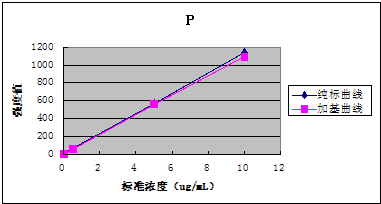
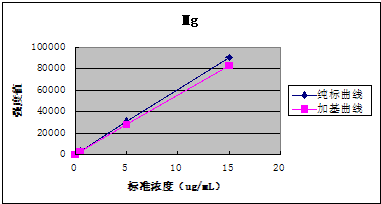
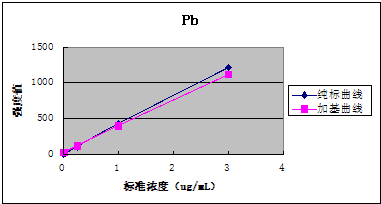
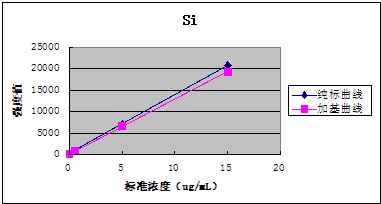
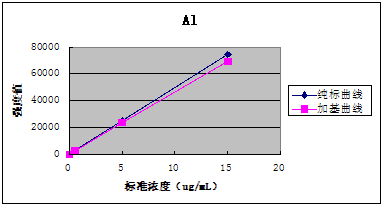
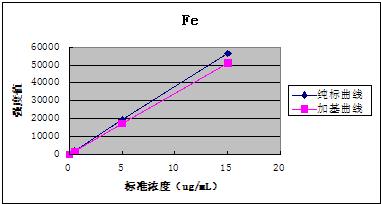
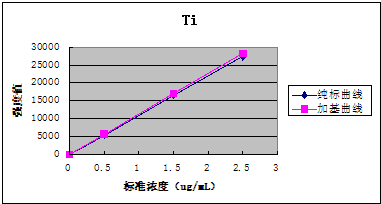
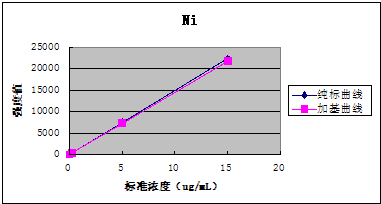
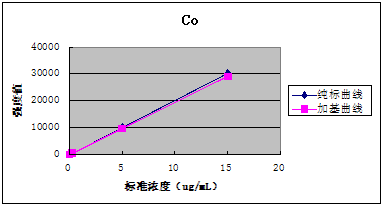
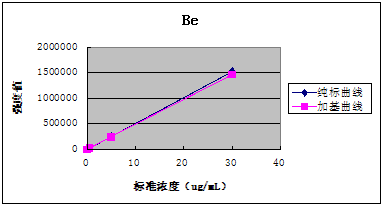


图1 各元素的工作曲线对比

（六）工作曲线与线性回归

按照仪器设定的工作条件对标准溶液系列进行测定，以待测元素质量浓度为横坐标，发射强度为纵坐标，绘制工作曲线。工作曲线的线性方程、相关系数见表7。

表7 工作曲线与线性回归

|  |  |  |  |
| --- | --- | --- | --- |
| 元素 | 分析谱线（nm） | 工作曲线 | 相关系数（r） |
| Be | 234.861 | y=243912.34x+1921.98 | 0.99999 |
| Co | 231.160 | y=9667.41x+9.17 | 1.0000 |
| Ni | 231.604 | y=7270.28x+23.89 | 1.0000 |
| Ti | 337.280 | y=56428.82x+43.64 | 1.0000 |
| Fe | 259.940 | y=171433.01x+29.89 | 0.99997 |
| Al | 396.152 | y=237333.40x+55.63 | 0.99995 |
| Si | 288.158 | y=64220.24x+146.96 | 0.99998 |
| Pb | 220.253 | y=18141.17x+28.77 | 0.99996 |
| Mg | 285.213 | y=275984.34x+53.40 | 0.99999 |
| P | 178.222 | y=5546.59x+5.00 | 0.99989 |

（七）元素检出限和定量下限的确定

按实验方法分别对11份空白溶液进行测定，以3倍的空白溶液标准偏差计算元素的检出限（3s）。以10倍的空白溶液标准偏差对应的元素质量浓度值计算分析方法测定下限，见表8。

表8 元素检出限及测定下限

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 元素 | 测定值  ％ | 平均值  ％ | 标准偏差 | 检出限  ％ | 测定下限  ％ |
| Be | 0.0019,0.0019,0.0015,0.0017,0.0013,0.0019,0.0019,0.0015,0.0017,0.0013,0.0014 | 0.0016 | 0.00023 | 0.00070 | 0.0023 |
| Co | 0.0003,0.0012,0.0010,0.0008,0.0009,0.0003,0.0012,0.0010,0.0008,0.0009,0.0005 | 0.00081 | 0.00030 | 0.00091 | 0.0030 |
| Ni | 0.0009,0.0011,0.0005,0.0001,0.0002,0.0004,0.0009,0.0005,0.0001,0.0002 | 0.00055 | 0.00037 | 0.0011 | 0.0037 |
| Ti | 0.0007,0.0006,0.0006,0.0006,0.0007,0.0008,0.0007,0.0006,0.0006,0.0006,0.0007 | 0.00065 | 0.00007 | 0.00020 | 0.0007 |
| Fe | 0.0001,0.0001,0.0001,0.0002,0.0001,0.0004,0.0001,0.0001,0.0001,0.0002,0.0003 | 0.00016 | 0.00010 | 0.00029 | 0.0010 |
| Al | 0.0003,0.0004,0.0004,0.0003,0.0004,0.0003,0.0004,0.0004,0.0003,0.0004,0.0005 | 0.00037 | 0.00006 | 0.00018 | 0.0006 |
| Si | 0.0005,0.0005,0.0004,0.0004,0.0004,0.0005,0.0005,0.0005,0.0004,0.0005,0.0004 | 0.00045 | 0.00005 | 0.00015 | 0.0005 |
| Pb | 0.0005,0.0004,0.0004,0.0005,0.0006,0.0004,0.0006,0.0002,0.0004,0.0003,0.0004 | 0.00043 | 0.00011 | 0.00034 | 0.0011 |
| Mg | 0.0001,0.0001,0.0002,0.0001,0.0001,0.0001,0.0002,0.0001,0.0002,0.0001,0.0001 | 0.00013 | 0.00004 | 0.00013 | 0.0004 |
| P | 0.0001,0.0012,0.0005,0.0001,0.0003,0.0001,0.0011,0.0005,0.0001,0.0003,0.0008 | 0.00046 | 0.00039 | 0.0012 | 0.0039 |

表8结果表明，该方法的测定下限满足测定范围要求。

第一验证单位（新疆有色金属研究所）的方法测定下限见表9。

表9一验（新疆有色金属研究所）的方法测定下限

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 元素 | 测定值  ％ | 平均值  ％ | 标准偏差 | 检出限  ％ | 测定下限  ％ |
| Be | 0.0011，0.0010，0.0009，0.0005，0.0015，0.0014，0.0014，0.0011，0.0010，0.0007，0.0009 | 0.0010 | 0.00030 | 0.00091 | 0.0031 |
| Co | 0.0007，0.0009，0.0012，0.0007，0.0004，0.0003，0.0009，0.0015，0.0010，0.0011，0.0009 | 0.0009 | 0.00034 | 0.00103 | 0.0035 |
| Ni | 0.0003，0.0002，0.0008，0.0013，0.0007，0.0015，0.0002，0.0021，0.0011，0.0005，0.0003 | 0.0008 | 0.00062 | 0.00186 | 0.0062 |
| Ti | 0.0009，0.0010，0.0005，0.0006，0.0008，0.0009，0.0007，0.0010，0.0007，0.0006，0.0009 | 0.0008 | 0.00017 | 0.00052 | 0.0018 |
| Fe | 0.0004，0.0003，0.0005，0.0004，0.0004，0.0005，0.0006，0.0003，0.0004，0.0005，0.0003 | 0.0004 | 0.00010 | 0.00029 | 0.0010 |
| Al | 0.0005，0.0006，0.0003，0.0004，0.0005，0.0006，0.0004，0.0003，0.0005，0.0006，0.0004 | 0.0005 | 0.00011 | 0.00034 | 0.0012 |
| Si | 0.0007，0.0009，0.0004，0.0005，0.0006，0.0004，0.0004，0.0005，0.0006，0.0003，0.0004 | 0.0005 | 0.00017 | 0.00052 | 0.0018 |
| Pb | 0.0006，0.0004，0.0005，0.0006，0.0004，0.0003，0.0005，0.0007，0.0004，0.0005，0.0005 | 0.0005 | 0.00012 | 0.00035 | 0.0012 |
| Mg | 0.0007，0.0009，0.0007，0.0007，0.0004，0.0003，0.0009，0.0005，0.0010，0.0002，0.0009 | 0.0007 | 0.00027 | 0.00081 | 0.0027 |
| P | 0.0012，0.0009，0.0015，0.0017，0.0015，0.0013，0.001，0.0011，0.0012，0.0016，0.0015 | 0.0013 | 0.00028 | 0.00085 | 0.0029 |

由表9可知，该方法满足测定范围要求，与起草单位结论一致。

（八）准确度试验

1.起草单位加标回收试验

在两个铜铍合金样品中加入不同量的各元素标准溶液，按实验方法进行加标回收试验，结果见表10。

表10 起草单位加标回收试验结果

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 样品编号 | 元素 | 样品值  µg/mL | 加入量  µg/mL | 测得值  µg/mL | 回收率/% |
| BeCu-1# | Be | 2.41 | 2.00 | 4.43 | 101.0 |
| Co | 0.19 | 0.10 | 0.29 | 100.0 |
| Ni | 0.41 | 0.50 | 0.90 | 98.0 |
| Ti | 1.58 | 1.00 | 2.56 | 98.0 |
| Fe | 1.29 | 1.00 | 2.28 | 99.0 |
| Al | 1.02 | 1.00 | 2.03 | 101.0 |
| Si | 1.09 | 1.00 | 2.08 | 99.0 |
| Pb | 0.90 | 1.00 | 1.89 | 99.0 |
| Mg | 1.02 | 1.00 | 2.04 | 102.0 |
| P | 1.99 | 2.00 | 3.97 | 99.0 |
| BeCu-2# | Be | 12.22 | 10.00 | 22.41 | 101.9 |
| Co | 1.53 | 1.00 | 2.52 | 99.0 |
| Ni | 2.03 | 2.00 | 4.01 | 99.0 |
| Ti | 0.60 | 1.00 | 1.60 | 100.0 |
| Fe | 5.90 | 5.00 | 10.99 | 101.8 |
| Al | 5.15 | 5.00 | 10.35 | 104.0 |
| Si | 5.45 | 5.00 | 10.32 | 97.4 |
| Pb | 0.49 | 1.00 | 1.48 | 99.0 |
| Mg | 4.98 | 5.00 | 10.11 | 102.6 |
| P | 7.05 | 5.00 | 11.93 | 97.6 |

由表10数据可知，铜铍合金样品中各元素的加标回收率在97.0％～104.0％之间，方法测定结果准确度满足铜铍合金中各元素含量的分析方法要求。

第一验证单位（新疆有色金属研究所）的加标回收试验结果见表11。

表11 一验（新疆有色金属研究所）加标回收试验结果

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 样品编号 | 元素 | 样品值  µg/mL | 加入量  µg/mL | 测得值  µg/mL | 回收率/% |
| BeCu-1# | Be | 2.38 | 2.00 | 4.371 | 99.6 |
| Co | 0.19 | 0.10 | 0.292 | 102.0 |
| Ni | 0.40 | 0.50 | 0.893 | 98.6 |
| Ti | 1.55 | 1.00 | 2.514 | 96.4 |
| Fe | 1.24 | 1.00 | 2.222 | 98.2 |
| Al | 1.06 | 1.00 | 2.041 | 98.1 |
| Si | 1.05 | 1.00 | 2.063 | 101.3 |
| Pb | 0.86 | 1.00 | 1.881 | 102.1 |
| Mg | 1.00 | 1.00 | 2.020 | 102.0 |
| P | 1.97 | 2.00 | 4.051 | 104.1 |
| BeCu-2# | Be | 12.22 | 10.00 | 22.71 | 104.9 |
| Co | 1.51 | 1.00 | 2.501 | 99.1 |
| Ni | 2.00 | 2.00 | 4.082 | 104.1 |
| Ti | 0.60 | 1.00 | 1.603 | 100.3 |
| Fe | 5.65 | 5.00 | 10.55 | 98.0 |
| Al | 5.55 | 5.00 | 10.63 | 101.6 |
| Si | 5.20 | 5.00 | 10.01 | 96.2 |
| Pb | 0.51 | 1.00 | 1.473 | 96.3 |
| Mg | 4.75 | 5.00 | 9.960 | 104.2 |
| P | 6.95 | 5.00 | 11.93 | 99.6 |

由表11可知，各元素的加标回收率均在96.2％～104.9％之间，说明该方法测定结果准确可靠，与起草单位结论一致。

第一验证单位（五矿铍业股份有限公司）的加标回收试验结果见表12。

表12 一验（五矿铍业股份有限公司）加标回收试验结果

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 样品编号 | 元素 | 样品值 | 加入量 | 测得值 | 回收率/% |
| µg/mL | µg/mL | µg/mL |
| BeCu-1# | Be | 2.37 | 2.00 | 4.327 | 97.9 |
| Co | 0.183 | 0.50 | 0.674 | 98.2 |
| Ni | 0.386 | 0.50 | 0.882 | 99.2 |
| Ti | 1.57 | 5.00 | 6.471 | 98.0 |
| Fe | 1.21 | 1.00 | 2.223 | 101.3 |
| Al | 1.08 | 1.00 | 2.062 | 98.2 |
| Si | 0.965 | 1.00 | 1.983 | 101.8 |
| Pb | 0.825 | 1.00 | 1.804 | 97.9 |
| Mg | 0.935 | 1.00 | 1.972 | 103.7 |
| P | 1.92 | 2.00 | 3.883 | 98.2 |
| BeCu-2# | Be | 12.125 | 10.00 | 21.97 | 98.5 |
| Co | 1.56 | 2.00 | 3.611 | 102.6 |
| Ni | 1.98 | 2.00 | 3.913 | 96.7 |
| Ti | 0.605 | 0.50 | 1.093 | 97.6 |
| Fe | 5.65 | 5.00 | 10.54 | 97.8 |
| Al | 5.3 | 5.00 | 10.53 | 104.6 |
| Si | 5.1 | 5.00 | 10.14 | 100.8 |
| Pb | 0.53 | 0.50 | 1.052 | 104.4 |
| Mg | 4.865 | 5.00 | 9.873 | 100.2 |
| P | 6.8 | 5.00 | 11.74 | 98.8 |

由表12可知，各元素的加标回收率均在96.7％～104.6％之间，说明该方法测定结果准确可靠，与起草单位结论一致。

2.标样测定

以本法测定牌号为99M 301（美国加联公司）、DM 8089（美国加联公司）铜铍合金标准样品，检验方法准确度，结果见表13。

表13 标样测定结果

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 铜铍合金标准样品牌号 | 元素 | 标准值/％ | 不确定度/％ | 测定值/％ | 是否吻合 |
| 99M 301 | Be | 2.00 | 0.02 | 2.01 | 是 |
| Co | 0.010 | 0.001 | 0.011 | 是 |
| Ni | 0.314 | 0.003 | 0.312 | 是 |
| Fe | 0.064 | 0.002 | 0.065 | 是 |
| Al | 0.093 | 0.002 | 0.095 | 是 |
| Si | 0.104 | 0.003 | 0.103 | 是 |
| Pb | 0.0016 | 0.0002 | 0.0016 | 是 |
| Mg | 0.0066 | 0.0004 | 0.0070 | 是 |
| DM 8089 | Be | 0.43 | 0.015 | 0.44 | 是 |
| Co | 2.31 | 0.05 | 2.34 | 是 |
| Ni | 0.095 | 0.003 | 0.092 | 是 |
| Fe | 0.0262 | 0.0012 | 0.0266 | 是 |
| Al | 0.0210 | 0.0007 | 0.0215 | 是 |
| Si | 0.0641 | 0.0025 | 0.0645 | 是 |
| Pb | 0.0005 | 0.00015 | 0.0005 | 是 |
| Mg | 0.0076 | 0.0012 | 0.0086 | 是 |
| P | 0.031 | 0.0006 | 0.0031 | 是 |

由表13数据可知，本法测定两种铜铍合金标准样品与标准值吻合，方法测定结果准确度满足铜铍合金中各元素含量的分析方法要求。

（九）精密度试验

对3个不同铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的铜铍合金样品，采用本方法对铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量分别独立地进行11次测定，测定结果见表14。

表14 起草单位精密度实验

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 样品 | 元素 | 测定结果/％（n=11） | 平均值/％ | s | RSD/％ |
| BeCu-1# | Be | 0.476,0.495,0.492,0.469,0.493,0.492,0.486,0.472,0.489,0.473,0.494 | 0.485 | 0.010 | 2.07 |
| Co | 0.0378,0.0394,0.0398,0.0384,0.0382,0.0380,0.0394,0.0394,0.0401,0.0384,0.0383 | 0.0388 | 0.00080 | 2.05 |
| Ni | 0.0818,0.0826,0.0828,0.0815,0.0809,0.0820,0.0826,0.0826,0.0828,0.0815,0.0811 | 0.0820 | 0.00070 | 0.85 |
| Ti | 0.313,0.316,0.316,0.315,0.314,0.311,0.316,0.316,0.318,0.315,0.316 | 0.315 | 0.00187 | 0.59 |
| Fe | 0.0248,0.0255,0.0270,0.0262,0.0255,0.0250,0.0255,0.0257,0.0271,0.0262,0.0254 | 0.0258 | 0.00074 | 2.88 |
| Al | 0.0206,0.0201,0.0203,0.0202,0.0203,0.0211,0.0201,0.0201,0.0203,0.0202,0.0208 | 0.0204 | 0.00033 | 1.60 |
| Si | 0.0218,0.0220,0.0218,0.0218,0.0215,0.0220,0.0220,0.0220,0.0218,0.0217,0.0217 | 0.0218 | 0.00016 | 0.74 |
| Pb | 0.0168,0.0185,0.0172,0.0188,0.0184,0.0170,0.0185,0.0182,0.0172,0.0183,0.0184 | 0.0179 | 0.00073 | 4.05 |
| Mg | 0.0211,0.0202,0.0201,0.0201,0.0207,0.0210,0.0202,0.0202,0.0203,0.0201,0.0209 | 0.0204 | 0.00040 | 1.94 |
| P | 0.0386,0.0397,0.0406,0.0407,0.0389,0.0390,0.0397,0.0395,0.0406,0.0405,0.0392 | 0.0397 | 0.00077 | 1.93 |
| BeCu-2# | Be | 2.467,2.471,2.452,2.428,2.463,2.473,2.428,2.466,2.473,2.462,2.432 | 2.456 | 0.018 | 0.74 |
| Co | 0.306,0.301,0.304,0.305,0.309,0.306,0.308,0.304,0.305,0.309,0.302 | 0.305 | 0.00262 | 0.86 |
| Ni | 0.405,0.406,0.405,0.406,0.404,0.405,0.402,0.407,0.406,0.404,0.408 | 0.405 | 0.00162 | 0.40 |
| Ti | 0.120,0.119,0.121,0.121,0.122,0.118,0.119,0.121,0.121,0.123,0.119 | 0.120 | 0.00150 | 1.25 |
| Fe | 0.118,0.117,0.117,0.118,0.118,0.118,0.117,0.119,0.118,0.119,0.117 | 0.118 | 0.00075 | 0.64 |
| Al | 0.104,0.101,0.102,0.103,0.103,0.103,0.103,0.102,0.104,0.103,0.101 | 0.103 | 0.00104 | 1.00 |
| Si | 0.111,0.108,0.109,0.109,0.109,0.110,0.110,0.109,0.108,0.109,0.111 | 0.109 | 0.00103 | 0.94 |
| Pb | 0.0101,0.0102,0.0096,0.0096,0.0098,0.0101,0.0096,0.0099,0.0096,0.0097,0.0095 | 0.0098 | 0.00025 | 2.52 |
| Mg | 0.0995,0.0997,0.0991,0.1000,0.0997,0.0995,0.0997,0.0991,0.1000,0.0997,0.0997 | 0.0996 | 0.00030 | 0.30 |
| P | 0.141,0.140,0.142,0.141,0.142,0.141,0.140,0.139,0.141,0.142,0.143 | 0.141 | 0.00114 | 0.81 |
| BeCu-3# | Be | 3.908,3.829,3.832,3.877,3.861,3.894,3.922,3.834,3.834,3.817,3.911 | 3.865 | 0.0039 | 1.00 |
| Co | 2.794,2.767,2.792,2.766,2.795,2.794,2.766,2.795,2.771,2.795,2.764 | 2.781 | 0.014 | 0.52 |
| Ni | 2.824,2.789,2.821,2.789,2.816,2.811,2.787,2.802,2.789,2.816,2.785 | 2.803 | 0.015 | 0.55 |
| Ti | 0.0319,0.0311,0.0311,0.0312,0.0315,0.0319,0.0311,0.0314,0.0312,0.0315,0.0312 | 0.0314 | 0.00030 | 0.96 |
| Fe | 0.265,0.262,0.263,0.261,0.263,0.265,0.262,0.263,0.265,0.263,0.261 | 0.263 | 0.00148 | 0.56 |
| Al | 0.261,0.254,0.253,0.253,0.258,0.261,0.254,0.258,0.253,0.255,0.254 | 0.256 | 0.00312 | 1.22 |
| Si | 0.0629,0.0622,0.0581,0.0606,0.0599,0.0621,0.0617,0.0599,0.0606,0.0583,0.0618 | 0.0607 | 0.00158 | 2.61 |
| Pb | 0.0489,0.0463,0.0482,0.0487,0.0488,0.0489,0.0463,0.0481,0.0487,0.0487,0.0468 | 0.0480 | 0.00105 | 2.18 |
| Mg | 0.264,0.252,0.256,0.254,0.254,0.264,0.251,0.256,0.254,0.257,0.254 | 0.256 | 0.00431 | 1.68 |
| P | 0.0161,0.0165,0.0157,0.0172,0.0165,0.0163,0.0165,0.0166,0.0172,0.0165,0.0165 | 0.0165 | 0.00043 | 2.59 |

由表14结果可知，本法的RSD％在0.30％～4.05％之间，精密度较好，能满足方法分析要求。

第一验证单位（五矿铍业股份有限公司）的精密度试验结果见表15。

表15 一验（五矿铍业股份有限公司）精密度实验

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 样品 | 元素 | 测定结果/％（n=11） | 平均值/％ | s | RSD/％ |
| BeCu-1# | Be | 0.462,0.466,0.484,0.462,0.482,0.463,0.485,0.467,0.469,0.483,0.461 | 0.471 | 0.010 | 2.12 |
| Co | 0.0366,0.0377,0.0365,0.0355,0.0376,0.0355,0.0366,0.0375,0.0364,0.0367,0.0359 | 0.0366 | 0.00077 | 2.11 |
| Ni | 0.0757,0.0776,0.0785,0.0777,0.0765,0.0784,0.0774,0.0767,0.0766,0.0775,0.0768 | 0.0772 | 0.00085 | 1.10 |
| Ti | 0.318,0.314,0.319,0.315,0.313,0.316,0.312,0.311,0.314,0.311,0.313 | 0.314 | 0.00264 | 0.84 |
| Fe | 0.0231,0.0247,0.0241,0.0247,0.0244,0.0238,0.0236,0.0233,0.0247,0.0244,0.0249 | 0.0242 | 0.00062 | 2.57 |
| Al | 0.0213,0.0212,0.0215,0.0222,0.0221,0.0211,0.0224,0.0211,0.0212,0.0221,0.0219 | 0.0216 | 0.00050 | 2.30 |
| Si | 0.0197,0.0194,0.0193,0.0192,0.0197,0.0184,0.0195,0.0193,0.0184,0.0193,0.0199 | 0.0193 | 0.00048 | 2.52 |
| Pb | 0.0161,0.0159,0.0165,0.0171,0.0155,0.0164,0.0163,0.0169,0.0176,0.0153,0.0178 | 0.0165 | 0.00080 | 4.86 |
| Mg | 0.0203,0.0187,0.0173,0.0198,0.0186,0.0181,0.0183,0.0183,0.0196,0.0184,0.0182 | 0.0187 | 0.00087 | 4.65 |
| P | 0.0375,0.0384,0.0392,0.0377,0.0376,0.0391,0.0377,0.0395,0.0394,0.0388,0.0372 | 0.0384 | 0.00086 | 2.24 |
| BeCu-2# | Be | 2.407,2.401,2.428,2.407,2.433,2.422,2.411, 2.439,2.442,2.417,2.435 | 2.422 | 0.014 | 0.59 |
| Co | 0.318,0.314,0.309,0.305,0.313,0.316,0.307,0.307,0.315,0.315,0.312 | 0.312 | 0.0428 | 1.37 |
| Ni | 0.396,0.399,0.395,0.392,0.396,0.395,0.396,0.395,0.394,0.393,0.401 | 0.396 | 0.00254 | 0.64 |
| Ti | 0.117,0.124,0.123,0.122,0.123,0.117,0.123,0.117,0.124,0.118,0.122 | 0.121 | 0.00298 | 2.47 |
| Fe | 0.111,0.114,0.113,0.110,0.111,0.114,0.115,0.113,0.110,0.113,0.115 | 0.113 | 0.00186 | 1.65 |
| Al | 0.107,0.106,0.103,0.108,0.117,0.108,0.108,0.103,0.104,0.107,0.106 | 0.106 | 0.00192 | 1.81 |
| Si | 0.103,0.103,0.102,0.103,0.0992,0.102 0.0992,0.102,0.0997,0.101,0.103 | 0.102 | 0.00154 | 1.52 |
| Pb | 0.0101,0.0114,0.0103,0.0112,0.0100,0.0104,0.0113,0.0103,0.0104,0.0109,0.0106 | 0.0106 | 0.00049 | 4.65 |
| Mg | 0.0964,0.0962,0.0979,0.0965,0.0986,0.0985,0.0961,0.0962,0.0965,0.0989,0.0982 | 0.0973 | 0.00113 | 1.16 |
| P | 0.138,0.134,0.139,0.135,0.134,0.134,0.137,0.135,0.135,0.135,0.138 | 0.136 | 0.00183 | 1.35 |
| BeCu-3# | Be | 3.821,3.817,3.866,3.824,3.874,3.817,3.846,3.816,3.837,3.796,3.847 | 3.832 | 0.024 | 0.61 |
| Co | 2.753,2.741,2.752,2.749,2.759,2.745,2.748,2.754,2.734,2.747,2.739 | 2.747 | 0.00728 | 0.27 |
| Ni | 2.773,2.771,2.768,2.779,2.789,2.787,2.768,2.766,2.789,2.767,2.795 | 2.777 | 0.0107 | 0.39 |
| Ti | 0.0319,0.0311,0.0311,0.0312,0.0315,0.0319,0.0311,0.0314,0.0312,0.0315,0.0312 | 0.0314 | 0.00030 | 0.96 |
| Fe | 0.257,0.258,0.254,0.256,0.252,0.251,0.259,0.252,0.259,0.255,0.259 | 0.256 | 0.00304 | 1.19 |
| Al | 0.255,0.257,0.259,0.251,0.254,0.251,0.255,0.253,0.255,0.258,0.252 | 0.255 | 0.00270 | 1.06 |
| Si | 0.0579,0.0593,0.0585,0.0598,0.0592,0.0586,0.0587,0.0603,0.0587,0.0589,0.0591 | 0.0590 | 0.00065 | 1.11 |
| Pb | 0.0457,0.0428,0.0444,0.0437,0.0453,0.0457,0.0448,0.0432,0.0459,0.0447,0.0431 | 0.0445 | 0.00113 | 2.54 |
| Mg | 0.256,0.255,0.254,0.249,0.261,0.256,0.265,0.254,0.249,0.247,0.259 | 0.255 | 0.00054 | 2.12 |
| P | 0.0153,0.0147,0.0155,0.0161,0.0165,0.0154,0.0149,0.0162,0.0158,0.0149,0.0161 | 0.0156 | 0.00060 | 3.87 |

由表15可知，样品中各元素的相对标准偏差在0.27％～4.86％之间，说明该方法的精密度良好，与起草单位结论一致。

第一验证单位（新疆有色金属研究所）的精密度试验结果见表16。

表16 一验（新疆有色金属研究所）精密度实验

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 样品 | 元素 | 测定结果/％（n=11） | 平均值/％ | s | RSD/％ |
| BeCu-1# | Be | 0.466,0.469,0.459,0.471,0.455,0.464,  0.468,0.471,0.463,0.462,0.451 | 0.464 | 0.0065 | 1.40 |
| Co | 0.0376,0.0398,0.0380,0.0390,0.0389,0.0370,0.0378,0.0378,0.0371,0.0387,0.0377 | 0.0381 | 0.00088 | 2.30 |
| Ni | 0.0805,0.0799,0.0792,0.0820,0.0810,0.0799,0.0802,0.0795,0.0812,0.0813,0.0820 | 0.0806 | 0.00097 | 1.20 |
| Ti | 0.306,0.312,0.310,0.310,0.308,0.312,0.306,0.306,0.308,0.309,0.308 | 0.309 | 0.0023 | 0.75 |
| Fe | 0.0249，0.0241，0.0244，0.0243，0.0248，0.0250，0.0249，0.0250，0.0247，0.0248，0.0253 | 0.0248 | 0.00035 | 1.42 |
| Al | 0.0212，0.0210，0.0205，0.0210，0.0202，0.0217，0.0200，0.0213，0.0219，0.0216，0.0221 | 0.0211 | 0.00069 | 3.25 |
| Si | 0.0209,0.0211,0.0208,0.0208,0.0202,0.0211,0.0202,0.0213,0.0208,0.0216,0.0210 | 0.0209 | 0.00042 | 2.00 |
| Pb | 0.0171,0.0167,0.0174,0.0171,0.0177,0.0169,0.0169,0.0173,0.0168,0.0168,0.0170 | 0.0171 | 0.00031 | 1.80 |
| Mg | 0.0203,0.0198,0.0196,0.0200,0.0198,0.0201,0.0200,0.0202,0.0196,0.0200,0.0206 | 0.0200 | 0.00029 | 1.45 |
| P | 0.0393,0.0396,0.0390,0.0387,0.0394,0.0391,0.0396,0.0395,0.0395,0.0391,0.0391 | 0.0393 | 0.00030 | 0.77 |
| BeCu-2# | Be | 2.411,2.428,2.417,2.431,2.433,2.414,2.435,2.404,2.452,2.451,2.423 | 2.427 | 0.015 | 0.64 |
| Co | 0.299,0.302,0.300,0.299,0.297,0.300,0.299,0.305,0.306,0.304,0.296 | 0.301 | 0.0031 | 1.03 |
| Ni | 0.397,0.398,0.400,0.399,0.403,0.401,0.402,0.397,0.395,0.399,0.407 | 0.400 | 0.0034 | 0.84 |
| Ti | 0.123,0.123,0.120,0.120,0.120,0.116,0.116,0.117,0.116,0.117,0.118 | 0.119 | 0.0024 | 2.04 |
| Fe | 0.112,0.113,0.112,0.113,0.113,0.112,0.112,0.113,0.112,0.113,0.114 | 0.113 | 0.00050 | 0.45 |
| Al | 0.110,0.109,0.111,0.109,0.111,0.113,0.109,0.111,0.112,0.111,0.115 | 0.111 | 0.0018 | 1.66 |
| Si | 0.106,0.105,0.104,0.104,0.104,0.104,0.104,0.104,0.102,0.103,0.104 | 0.104 | 0.0010 | 0.95 |
| Pb | 0.0103,0.0102,0.0101,0.0096,0.0105,0.0100,0.0100,0.0102,0.0104,0.0098,0.0105 | 0.0101 | 0.00028 | 2.80 |
| Mg | 0.0963,0.0938,0.0947,0.0939,0.0962,0.0937,0.0950,0.0957,0.0940,0.0955,0.0961 | 0.0950 | 0.0010 | 1.08 |
| P | 0.135,0.140,0.139,0.138,0.140,0.138,0.137,0.138,0.139,0.139,0.142 | 0.139 | 0.0018 | 1.30 |
| BeCu-3# | Be | 3.789,3.811,3.812,3.835,3.811,3.807,3.786,3.814,3.831,3.822,3.821 | 3.833 | 0.016 | 0.42 |
| Co | 2.751,2.756,2.728,2.746,2.736,2.741,2.756,2.735,2.741,2.725,2.757 | 2.743 | 0.011 | 0.41 |
| Ni | 2.813,2.787,2.797,2.780,2.807,2.789,2.803,2.787,2.794,2.814,2.803 | 2.798 | 0.011 | 0.40 |
| Ti | 0.0314,0.0326,0.0302,0.0316,0.0299,0.0310,0.0308,0.0320,0.0302,0.0298,0.0308 | 0.0309 | 0.00086 | 2.76 |
| Fe | 0.267,0.265,0.263,0.264,0.262,0.260,0.261,0.259,0.263,0.264,0.267 | 0.263 | 0.0027 | 1.01 |
| Al | 0.257,0.260,0.262,0.258,0.259,0.261,0.261,0.259,0.259,0.264,0.264 | 0.261 | 0.0023 | 0.89 |
| Si | 0.0593,0.0590,0.0591,0.0597,0.0595,0.0593,0.0599,0.0595,0.0592,0.0600,0.0597 | 0.0595 | 0.00031 | 0.52 |
| Pb | 0.0437,0.0441,0.0434,0.0450,0.0444,0.0458,0.0466,0.0447,0.0442,0.0451,0.0465 | 0.0451 | 0.0014 | 3.02 |
| Mg | 0.244,0.248,0.242,0.247,0.245,0.249,0.244,0.248,0.247,0.248,0.248 | 0.246 | 0.0024 | 0.96 |
| P | 0.0169,0.0170,0.0172,0.0175,0.0168,0.0178,0.0169,0.0167,0.0166,0.0166，0.0176 | 0.0170 | 0.00040 | 2.34 |

由表16结果可知，本法的RSD％在0.40％～3.25％之间，说明该方法的精密度良好，与起草单位结论一致。

第二验证单位（富蕴恒盛铍业有限责任公司）的精密度试验结果见表17。

表17 二验（富蕴恒盛铍业有限责任公司）精密度实验

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 样品 | 元素 | 测定结果/％（n=11） | 平均值/％ | s | RSD/％ |
| BeCu-1# | Be | 0.473,0.466,0.473,0.455,0.468,0.478,0.482,0.467,0.473,,0.466,0.467 | 0.470 | 0.0072 | 1.53 |
| Co | 0.0377,0.0379,0.0395,0.0377,0.0393,0.0390,0.0393,0.0385,0.0378,0.0387,0.0390 | 0.0386 | 0.00067 | 1.74 |
| Ni | 0.0801,0.0810,0.0811,0.0813,0.0815,0.0794,0.0778,0.0791,0.0786,0.0800,0.0803 | 0.0800 | 0.0012 | 1.44 |
| Ti | 0.306,0.305,0.303,0.308,0.309,0.304,0.308,0.302,0.303,0.310,0.308, | 0.306 | 0.0025 | 0.83 |
| Fe | 0.0265,0.0262,0.0261,0.0264,0.0261,0.0263,0.0260,0.0260,0.0259,0.0268,0.0258, | 0.0262 | 0.00027 | 1.04 |
| Al | 0.0212,0.0209,0.0213,0.0214,0.0207,0.0208,0.0210,0.0204,0.0206,0.0205,0.0212 | 0.0209 | 0.00031 | 1.46 |
| Si | 0.0220,0.0216,0.0215,0.0199,0.0206,0.0211,0.0219,0.0205,0.0202,0.0217,0.0200 | 0.0210 | 0.00075 | 3.57 |
| Pb | 0.0171,0.0168,0.0168,0.0173,0.0167,0.0169,0.0170,0.0173,0.0165,0.0164,0.0167 | 0.0169 | 0.00029 | 1.70 |
| Mg | 0.0199,0.0198,0.0195,0.0195,0.0196,0.0194,0.0196,0.0198,0.0198,0.0199,0.0195 | 0.0197 | 0.00018 | 0.89 |
| P | 0.0401,0.0398,0.0389,0.0391,0.0395,0.0399,0.0395,0.0395,0.0386,0.0393,0.0388 | 0.0394 | 0.00044 | 1.12 |
| BeCu-2# | Be | 2.423,2.437,2.424,2.455,2.436,2.444,  2.454,2.434,2.416,2.465,2.438 | 2.439 | 0.015 | 0.61 |
| Co | 0.300,0.296,0.294,0.301,0.301,0.304,0.303,0.297,0.293,0.298,0.299 | 0.299 | 0.0034 | 1.15 |
| Ni | 0.407,0.396,0.399,0.394,0.397,0.399,0.407,0.406,0.409,0.403,0.410 | 0.402 | 0.0053 | 1.32 |
| Ti | 0.117,0.117,0.117,0.120,0.118,0.119,0.118,0.118,0.116,0.116,0.117 | 0.118 | 0.0011 | 1.01 |
| Fe | 0.114,0.109,0.109,0.114,0.113,0.110,0.111,0.110,0.110,0.112,0.112 | 0.111 | 0.0017 | 1.52 |
| Al | 0.108,0.106,0.107,0.107,0.107,0.106,0.107,0.107,0.107,0.108,0.106 | 0.107 | 0.00050 | 0.46 |
| Si | 0.104,0.103,0.104,0.107,0.107,0.106,0.105,0.104,0.104,0.104,0.103 | 0.105 | 0.0011 | 1.09 |
| Pb | 0.0097,0.0097,0.0092,0.0086,0.0083,0.0087,0.0091,0.0093,0.0097,0.0096,0.0096 | 0.0092 | 0.00046 | 5.03 |
| Mg | 0.0958,0.0961,0.0977,0.0967,0.0969,0.0963,0.0964，0.0977,0.0967,0.0973,0.0972 | 0.0968 | 0.00059 | 0.61 |
| P | 0.139,0.139,0.140,0.139,0.137,0.136,0.136,0.135,0.137,0.136,0.136 | 0.137 | 0.0016 | 1.16 |
| BeCu-3# | Be | 3.893,3.833,3.855,3.911,3.877,3.877,3.821,3.846,3.875,3.904,3.841 | 3.867 | 0.030 | 0.61 |
| Co | 2.776,2.804,2.776,2.770,2.802,2.790,2.771,2.783,2.769,2.777,2.789 | 2.782 | 0.012 | 0.42 |
| Ni | 2.792,2.784,2.786,2.813,2.817,2.804,2.821,2.801,2.799,2.821,2.805 | 2.804 | 0.013 | 0.47 |
| Ti | 0.0289,0.0316,0.0298,0.0312,0.0310,0.0291,0.0294,0.0291,0.0315,0.0294,0.0309 | 0.0302 | 0.0010 | 3.36 |
| Fe | 0.255,0.256,0.253,0.251,0.255,0.250,0.258,0.256,0.254,0.257,0.260 | 0.255 | 0.0029 | 1.13 |
| Al | 0.250,0.246,0.249,0.249,0.255,0.254,0.256,0.252,0.258,0.258,0.257 | 0.253 | 0.0040 | 1.59 |
| Si | 0.0591,0.0584,0.0593,0.0585,0.0588,0.0612,0.0604,0.0594,0.0595,0.0612,0.0611 | 0.0597 | 0.00107 | 1.80 |
| Pb | 0.0457,0.0469,0.0470,0.0470,0.0479,0.0457,0.0479,0.0491,0.0477,0.0486,0.0470 | 0.0473 | 0.0010 | 2.14 |
| Mg | 0.253,0.254,0.253,0.254,0.254,0.254,0.254,0.255,0.255,0.254,0.254 | 0.254 | 0.00066 | 0.26 |
| P | 0.0162,0.0156,0.0161,0.0161,0.0158,0.0160,0.0164,0.0157,0.0160,0.0157,0.0160 | 0.0160 | 0.00022 | 1.39 |

由表17结果可知，本法的RSD％在0.26％～5.03％之间，精密度较好，能满足方法分析要求。与起草单位结论一致。

第二验证单位（上海有色金属工业技术监测中心有限公司）的精密度试验结果见表18。

表18 二验（上海有色金属工业技术监测中心有限公司）精密度实验

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 样品 | 元素 | 测定结果/％（n=11） | 平均值/％ | s | RSD/％ |
| BeCu-1# | Be | 0.489/0.485/0.487/0.485/0.481/0.481/0.480/0.478/0.488/0.482/0.482 | 0.483 | 0.0036 | 0.74 |
| Co | 0.0385/0.0387/0.0388/0.0384/0.0387/0.0384/0.0385/0.0390/0.0385/0.0379/0.0383 | 0.0385 | 0.00029 | 0.75 |
| Ni | 0.0839/0.0856/0.0877/0.0855/0.0861/0.0815/0.0820/0.0826/0.0818/0.0812/0.0833 | 0.0837 | 0.0022 | 2.60 |
| Ti | 0.314/0.304/0.324/0.320/0.329/0.321/0.309/0.314/0.325/0.322/0.317 | 0.318 | 0.00738 | 2.32 |
| Fe | 0.0251/0.0254/0.0250/0.0254/0.0250/0.0261/0.0266/0.0259/0.0264/0.0266/0.0257 | 0.0257 | 0.00062 | 2.40 |
| Al | 0.0198/0.0205/0.0203/0.0198/0.0192/0.0209/0.0210/0.0199/0.0208/0.0204/0.0214 | 0.0203 | 0.00058 | 2.85 |
| Si | 0.0203/0.0205/0.0220/0.0204/0.0193/0.0216/0.0211/0.0207/0.0203/0.0214/0.0199 | 0.0206 | 0.00078 | 3.79 |
| Pb | 0.0176/0.0172/0.0171/0.0174/0.0168/0.0175/0.0179/0.0169/0.0173/0.0174/0.0169 | 0.0173 | 0.00034 | 1.94 |
| Mg | 0.0207/0.0202/0.0192/0.0186/0.0187/0.0204/0.0206/0.0194/0.0206/0.0199/0.0203 | 0.0199 | 0.00077 | 3.89 |
| P | 0.0380/0.0385/0.0388/0.0389/0.0399/0.0394/0.0396/0.0393/0.0387/0.0395/0.0389 | 0.0390 | 0.00055 | 1.41 |
| BeCu-2# | Be | 2.437/2.429/2.418/2.417/2.431/2.430/2.413/2.424/2.424/2.434/2.433 | 2.426 | 0.00778 | 0.32 |
| Co | 0.293/0.294/0.297/0.290/0.299/0.304/0.306/0.297/0.311/0.295/0.316 | 0.305 | 0.00262 | 0.86 |
| Ni | 0.403/0.403/0.404/0.405/0.402/0.400/0.396/0.401/0.404/0.411/0.406 | 0.403 | 0.00376 | 0.93 |
| Ti | 0.121/0.119/0.118/0.120/0.121/0.120/0.119/0.121/0.120/0.121/0.119 | 0.120 | 0.00104 | 0.87 |
| Fe | 0.114/0.115/0.113/0.113/0.112/0.112/0.112/0.116/0.111/0.114/0.117 | 0.114 | 0.00186 | 1.64 |
| Al | 0.102/0.102/0.103/0.103/0.103/0.101/0.102/0.101/0.101/0.103/0.103 | 0.102 | 0.00087 | 0.86 |
| Si | 0.110/0.111/0.109/0.108/0.108/0.111/0.102/0.101/0.119/0.114/0.103 | 0.109 | 0.00533 | 4.90 |
| Pb | 0.0093/0.0097/0.0094/0.0095/0.0096/0.0095/0.0089/0.0093/0.0094/0.0089/0.0096 | 0.00937 | 0.00027 | 2.83 |
| Mg | 0.0993/0.0998/0.0996/0.0987/0.0995/0.0998/0.0997/0.0989/0.0984/0.0983/0.0992 | 0.0992 | 0.00055 | 0.55 |
| P | 0.139/0.138/0.138/0.136/0.139/0.144/0.139/0.141/0.142/0.137/0.141 | 0.139 | 0.00234 | 1.68 |
| BeCu-3# | Be | 3.815/3.813/3.824/3.831/3.814/3.822/3.816/3.824/3.819/3.825/3.821 | 3.820 | 0.00555 | 0.15 |
| Co | 2.795/2.779/2.786/2.794/2.797/2.791/2.783/2.774/2.776/2.766/2.782 | 2.784 | 0.00980 | 0.35 |
| Ni | 2.818/2.818/2.824/2.819/2.835/2.834/2.817/2.812/2.812/2.825/2.806 | 2.820 | 0.00897 | 0.32 |
| Ti | 0.0311/0.0289/0.0304/0.0310/0.0318/0.0309/0.0304/0.0311/0.0319/0.0299/0.0304 | 0.0307 | 0.00085 | 2.77 |
| Fe | 0.255/0.254/0.256/0.254/0.260/0.255/0.256/0.259/0.254/0.254/0.257 | 0.256 | 0.0021 | 0.82 |
| Al | 0.257/0.251/0.254/0.256/0.259/0.253/0.258/0.255/0.254/0.257/0.250 | 0.255 | 0.0028 | 1.12 |
| Si | 0.0602/0.0595/0.0611/0.0598/0.0602/0.0607/0.0612/0.0597/0.0607/0.0603/0.0605 | 0.0604 | 0.00055 | 0.92 |
| Pb | 0.0440/0.0422/0.0485/0.0436/0.0476/0.0466/0.0459/0.0431/0.0436/0.0447/0.0429 | 0.0448 | 0.00209 | 4.62 |
| Mg | 0.249/0.244/0.245/0.250/0.251/0.250/0.244/0.245/0.251/0.244/0.253 | 0.249 | 0.0034 | 1.38 |
| P | 0.0169/0.0163/0.0161/0.0165/0.0155/0.0160/0.0164/0.0159/0.0168/0.0170/0.0161 | 0.0163 | 0.00046 | 2.82 |

由表18结果可知，本法的RSD％在0.15％～4.90％之间，精密度较好，能满足方法分析要求。与起草单位结论一致。

（十）主要实验（或验证）的分析

按照GB/T 6379.2-2004《测量方法与结果的准确度》，通过对西北稀有金属材料研究院宁夏有限公司、五矿铍业股份有限公司、新疆有色金属研究所、富蕴恒盛铍业有限责任公司、上海有色金属工业技术监测中心有限公司五家单位的试验数据和验证数据分别按照重复性和再现性的公式进行计算，得到了不同含量的重复性限和再现性限。

1.样品数据对比

起草单位与验证单位的试验结果统计对比见表19～表28。

表19 铍的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.485 | 0.010 | 2.456 | 0.018 | 3.865 | 0.039 |
| 五矿铍业股份有限公司 | 一验 | 0.471 | 0.010 | 2.422 | 0.014 | 3.832 | 0.024 |
| 新疆有色金属研究所 | 一验 | 0.464 | 0.0065 | 2.427 | 0.015 | 3.813 | 0.015 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.470 | 0.0072 | 2.439 | 0.015 | 3.867 | 0.030 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.483 | 0.0036 | 2.426 | 0.0078 | 3.820 | 0.0056 |

表20 钴的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.0388 | 0.00080 | 0.305 | 0.00262 | 2.781 | 0.014 |
| 五矿铍业股份有限公司 | 一验 | 0.0366 | 0.00077 | 0.312 | 0.00428 | 2.747 | 0.0073 |
| 新疆有色金属研究所 | 一验 | 0.0381 | 0.00088 | 0.301 | 0.0031 | 2.743 | 0.011 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.0386 | 0.00067 | 0.299 | 0.0034 | 2.782 | 0.012 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.0385 | 0.00029 | 0.305 | 0.00262 | 2.784 | 0.0098 |

表21 镍的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.0820 | 0.00070 | 0.405 | 0.00162 | 2.803 | 0.015 |
| 五矿铍业股份有限公司 | 一验 | 0.0772 | 0.00085 | 0.396 | 0.00254 | 2.777 | 0.0107 |
| 新疆有色金属研究所 | 一验 | 0.0806 | 0.00097 | 0.400 | 0.0034 | 2.798 | 0.011 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.0800 | 0.0012 | 0.402 | 0.0053 | 2.804 | 0.013 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.0837 | 0.00218 | 0.403 | 0.00376 | 2.820 | 0.00897 |

表22 钛的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.0314 | 0.00030 | 0.120 | 0.00150 | 0.315 | 0.00187 |
| 五矿铍业股份有限公司 | 一验 | 0.0314 | 0.00030 | 0.121 | 0.00298 | 0.314 | 0.00264 |
| 新疆有色金属研究所 | 一验 | 0.0309 | 0.00086 | 0.119 | 0.0024 | 0.309 | 0.0023 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.0302 | 0.0010 | 0.118 | 0.0011 | 0.306 | 0.0025 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.0308 | 0.00085 | 0.120 | 0.00104 | 0.318 | 0.00738 |

表23 铁的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.0258 | 0.00074 | 0.118 | 0.00075 | 0.263 | 0.00148 |
| 五矿铍业股份有限公司 | 一验 | 0.0242 | 0.00062 | 0.113 | 0.00186 | 0.256 | 0.00304 |
| 新疆有色金属研究所 | 一验 | 0.0248 | 0.00035 | 0.113 | 0.00050 | 0.263 | 0.0027 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.0258 | 0.00027 | 0.111 | 0.0017 | 0.255 | 0.0029 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.0257 | 0.00062 | 0.114 | 0.00186 | 0.256 | 0.0021 |

表24 铝的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.0204 | 0.00033 | 0.103 | 0.00103 | 0.256 | 0.00313 |
| 五矿铍业股份有限公司 | 一验 | 0.0216 | 0.00050 | 0.106 | 0.00192 | 0.255 | 0.00270 |
| 新疆有色金属研究所 | 一验 | 0.0211 | 0.00069 | 0.111 | 0.0019 | 0.261 | 0.0023 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.0209 | 0.00031 | 0.107 | 0.00050 | 0.253 | 0.0040 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.0203 | 0.00058 | 0.102 | 0.00087 | 0.255 | 0.0028 |

表25 硅的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.0218 | 0.00033 | 0.0607 | 0.00158 | 0.109 | 0.00103 |
| 五矿铍业股份有限公司 | 一验 | 0.0193 | 0.00048 | 0.0590 | 0.00065 | 0.102 | 0.00152 |
| 新疆有色金属研究所 | 一验 | 0.0209 | 0.00042 | 0.0595 | 0.00031 | 0.104 | 0.0010 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.0210 | 0.00075 | 0.0597 | 0.00107 | 0.105 | 0.0011 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.0207 | 0.00078 | 0.0603 | 0.00055 | 0.109 | 0.00533 |

表26 铅的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.00979 | 0.00025 | 0.0179 | 0.00073 | 0.0480 | 0.00105 |
| 五矿铍业股份有限公司 | 一验 | 0.0106 | 0.00049 | 0.0165 | 0.00080 | 0.0445 | 0.00113 |
| 新疆有色金属研究所 | 一验 | 0.0101 | 0.00028 | 0.0171 | 0.00031 | 0.0451 | 0.0014 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.00920 | 0.00046 | 0.0169 | 0.00029 | 0.0473 | 0.0010 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.00937 | 0.00027 | 0.0173 | 0.00034 | 0.0448 | 0.00207 |

表27 镁的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.0204 | 0.00040 | 0.0996 | 0.00030 | 0.256 | 0.00431 |
| 五矿铍业股份有限公司 | 一验 | 0.0187 | 0.00087 | 0.0973 | 0.00113 | 0.255 | 0.00540 |
| 新疆有色金属研究所 | 一验 | 0.0200 | 0.00029 | 0.0950 | 0.0010 | 0.246 | 0.0024 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.0197 | 0.00018 | 0.0968 | 0.00059 | 0.254 | 0.00066 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.0199 | 0.00077 | 0.0992 | 0.00055 | 0.249 | 0.0034 |

表28 磷的试验结果对比

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 试验单位 | | 水平1 | | 水平2 | | 水平3 | |
| ,% | s | ,% | s | ,% | s |
| 西北稀有金属材料研究院宁夏有限公司 | 起草 | 0.0165 | 0.00043 | 0.0397 | 0.00077 | 0.141 | 0.00134 |
| 五矿铍业股份有限公司 | 一验 | 0.0156 | 0.00060 | 0.0384 | 0.00086 | 0.136 | 0.00183 |
| 新疆有色金属研究所 | 一验 | 0.0170 | 0.00040 | 0.0393 | 0.00030 | 0.139 | 0.0018 |
| 富蕴恒盛铍业有限责任公司 | 二验 | 0.0160 | 0.00022 | 0.0394 | 0.00044 | 0.137 | 0.0016 |
| 上海有色金属工业技术监测中心有限公司 | 二验 | 0.0163 | 0.00046 | 0.0390 | 0.00055 | 0.139 | 0.00234 |

采用格拉布斯检验方法，分别对五家单位中铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的检测数据进行异常值情况分析，结果见表29～表78。

, 

表29 不同铍含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.485 | 0.010 | 1.593 | 0.996 | 2.234 | 无异常值 |
| BeCu-2# | 2.456 | 0.018 | 1.549 | 0.940 | 2.234 | 无异常值 |
| BeCu-3# | 3.865 | 0.039 | 1.247 | 1.480 | 2.234 | 无异常值 |

表30 不同钴含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0388 | 0.00080 | 1.250 | 1.625 | 2.234 | 无异常值 |
| BeCu-2# | 0.305 | 0.00262 | 1.527 | 1.527 | 2.234 | 无异常值 |
| BeCu-3# | 2.781 | 0.014 | 1.180 | 1.972 | 2.234 | 无异常值 |

表31 不同镍含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0820 | 0.00070 | 1.571 | 1.143 | 2.234 | 无异常值 |
| BeCu-2# | 0.405 | 0.00162 | 1.852 | 1.852 | 2.234 | 无异常值 |
| BeCu-3# | 2.803 | 0.015 | 1.113 | 1.440 | 2.234 | 无异常值 |

表32 不同钛含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.315 | 0.00187 | 2.139 | 1.604 | 2.234 | 无异常值 |
| BeCu-2# | 0.120 | 0.00150 | 1.333 | 2.000 | 2.234 | 无异常值 |
| BeCu-3# | 0.0314 | 0.00030 | 1.000 | 1.667 | 2.234 | 无异常值 |

表33 不同铁含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0258 | 0.00074 | 1.351 | 1.757 | 2.234 | 无异常值 |
| BeCu-2# | 0.118 | 0.00075 | 1.333 | 1.333 | 2.234 | 无异常值 |
| BeCu-3# | 0.263 | 0.00148 | 1.351 | 1.351 | 2.234 | 无异常值 |

表34 不同铝含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0204 | 0.00033 | 0.909 | 2.121 | 2.234 | 无异常值 |
| BeCu-2# | 0.103 | 0.00104 | 1.941 | 0.971 | 2.234 | 无异常值 |
| BeCu-3# | 0.256 | 0.00312 | 0.962 | 1.603 | 2.234 | 无异常值 |

表35 不同硅含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0218 | 0.00016 | 1.875 | 1.250 | 2.234 | 无异常值 |
| BeCu-2# | 0.109 | 0.00103 | 0.971 | 1.942 | 2.234 | 无异常值 |
| BeCu-3# | 0.0607 | 0.00158 | 1.642 | 1.390 | 2.234 | 无异常值 |

表36 不同铅含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0179 | 0.00016 | 1.507 | 1.233 | 2.234 | 无异常值 |
| BeCu-2# | 0.0098 | 0.00025 | 1.200 | 1.600 | 2.234 | 无异常值 |
| BeCu-3# | 0.0480 | 0.00105 | 1.619 | 0.857 | 2.234 | 无异常值 |

表37 不同镁含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0204 | 0.00040 | 0.750 | 1.750 | 2.234 | 无异常值 |
| BeCu-2# | 0.0996 | 0.00030 | 1.667 | 1.333 | 2.234 | 无异常值 |
| BeCu-3# | 0.256 | 0.00431 | 1.160 | 1.856 | 2.234 | 无异常值 |

表38 不同磷含量水平样品分析结果异常值分析（西材院）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0397 | 0.00077 | 1.428 | 1.299 | 2.234 | 无异常值 |
| BeCu-2# | 0.141 | 0.00114 | 1.754 | 1.754 | 2.234 | 无异常值 |
| BeCu-3# | 0.0165 | 0.00043 | 1.860 | 1.628 | 2.234 | 无异常值 |

表39 不同铍含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.471 | 0.010 | 1.000 | 1.400 | 2.234 | 无异常值 |
| BeCu-2# | 2.422 | 0.014 | 1.466 | 1.396 | 2.234 | 无异常值 |
| BeCu-3# | 3.832 | 0.024 | 1.529 | 1.784 | 2.234 | 无异常值 |

表40 不同钴含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0366 | 0.00077 | 1.421 | 1.421 | 2.234 | 无异常值 |
| BeCu-2# | 0.312 | 0.00423 | 1.637 | 1.403 | 2.234 | 无异常值 |
| BeCu-3# | 2.747 | 0.00728 | 1.785 | 1.647 | 2.234 | 无异常值 |

表41 不同镍含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0820 | 0.00070 | 1.571 | 1.143 | 2.234 | 无异常值 |
| BeCu-2# | 0.396 | 0.00254 | 1.574 | 1.968 | 2.234 | 无异常值 |
| BeCu-3# | 2.777 | 0.0107 | 1.120 | 1.586 | 2.234 | 无异常值 |

表42 不同钛含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.314 | 0.00264 | 1.137 | 1.895 | 2.234 | 无异常值 |
| BeCu-2# | 0.121 | 0.00298 | 1.341 | 1.006 | 2.234 | 无异常值 |
| BeCu-3# | 0.0314 | 0.00030 | 0.999 | 1.665 | 2.234 | 无异常值 |

表43 不同铁含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0242 | 0.00062 | 1.773 | 1.129 | 2.234 | 无异常值 |
| BeCu-2# | 0.113 | 0.00186 | 1.614 | 1.076 | 2.234 | 无异常值 |
| BeCu-3# | 0.256 | 0.00304 | 1.644 | 0.986 | 2.234 | 无异常值 |

表44 不同铝含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0216 | 0.00050 | 1.003 | 1.604 | 2.234 | 无异常值 |
| BeCu-2# | 0.106 | 0.00192 | 1.562 | 1.041 | 2.234 | 无异常值 |
| BeCu-3# | 0.255 | 0.00270 | 1.483 | 1.483 | 2.234 | 无异常值 |

表45 不同硅含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0193 | 0.00048 | 1.854 | 1.236 | 2.234 | 无异常值 |
| BeCu-2# | 0.102 | 0.00154 | 1.815 | 0.648 | 2.234 | 无异常值 |
| BeCu-3# | 0.0590 | 0.00065 | 1.681 | 1.987 | 2.234 | 无异常值 |

表46 不同铅含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0165 | 0.00080 | 1.497 | 1.621 | 2.234 | 无异常值 |
| BeCu-2# | 0.0106 | 0.00049 | 1.012 | 1.417 | 2.234 | 无异常值 |
| BeCu-3# | 0.0445 | 0.00113 | 1.503 | 1.238 | 2.234 | 无异常值 |

表47 不同镁含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0187 | 0.00087 | 1.609 | 1.839 | 2.234 | 无异常值 |
| BeCu-2# | 0.0973 | 0.00113 | 1.061 | 1.414 | 2.234 | 无异常值 |
| BeCu-3# | 0.255 | 0.00540 | 1.480 | 1.851 | 2.234 | 无异常值 |

表48 不同磷含量水平样品分析结果异常值分析（五矿铍业）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0384 | 0.00086 | 1.399 | 1.282 | 2.234 | 无异常值 |
| BeCu-2# | 0.136 | 0.00183 | 1.090 | 1.636 | 2.234 | 无异常值 |
| BeCu-3# | 0.0156 | 0.00060 | 1.492 | 1.492 | 2.234 | 无异常值 |

表49 不同铍含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.464 | 0.0065 | 2.004 | 1.079 | 2.234 | 无异常值 |
| BeCu-2# | 2.427 | 0.015 | 1.488 | 1.618 | 2.234 | 无异常值 |
| BeCu-3# | 3.813 | 0.015 | 1.779 | 1.450 | 2.234 | 无异常值 |

表50 不同钴含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0381 | 0.00088 | 1.250 | 1.932 | 2.234 | 无异常值 |
| BeCu-2# | 0.301 | 0.0031 | 1.613 | 1.613 | 2.234 | 无异常值 |
| BeCu-3# | 2.743 | 0.011 | 1.585 | 1.247 | 2.234 | 无异常值 |

表51 不同镍含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0806 | 0.00097 | 1.443 | 1.443 | 2.234 | 无异常值 |
| BeCu-2# | 0.400 | 0.0034 | 1.471 | 2.059 | 2.234 | 无异常值 |
| BeCu-3# | 2.798 | 0.011 | 1.562 | 1.449 | 2.234 | 无异常值 |

表52 不同钛含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.309 | 0.0023 | 1.304 | 1.3043 | 2.234 | 无异常值 |
| BeCu-2# | 0.119 | 0.0024 | 1.250 | 1.667 | 2.234 | 无异常值 |
| BeCu-3# | 0.0309 | 0.00086 | 1.279 | 1.977 | 2.234 | 无异常值 |

表53 不同铁含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0248 | 0.00035 | 2.000 | 1.429 | 2.234 | 无异常值 |
| BeCu-2# | 0.113 | 0.0005 | 2.000 | 2.000 | 2.234 | 无异常值 |
| BeCu-3# | 0.263 | 0.0027 | 1.482 | 1.482 | 2.234 | 无异常值 |

表54 不同铝含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0211 | 0.00069 | 1.594 | 1.449 | 2.234 | 无异常值 |
| BeCu-2# | 0.111 | 0.0018 | 1.111 | 2.222 | 2.234 | 无异常值 |
| BeCu-3# | 0.261 | 0.0023 | 1.739 | 1.304 | 2.234 | 无异常值 |

表55 不同硅含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0209 | 0.00042 | 1.667 | 1.667 | 2.234 | 无异常值 |
| BeCu-2# | 0.104 | 0.0010 | 2.000 | 2.000 | 2.234 | 无异常值 |
| BeCu-3# | 0.0595 | 0.00031 | 1.613 | 1.613 | 2.234 | 无异常值 |

表56 不同铅含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0171 | 0.00031 | 1.290 | 1.936 | 2.234 | 无异常值 |
| BeCu-2# | 0.0101 | 0.00028 | 1.786 | 1.429 | 2.234 | 无异常值 |
| BeCu-3# | 0.0451 | 0.0014 | 1.214 | 1.071 | 2.234 | 无异常值 |

表57 不同镁含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0200 | 0.00029 | 1.379 | 2.069 | 2.234 | 无异常值 |
| BeCu-2# | 0.0950 | 0.0010 | 1.300 | 1.300 | 2.234 | 无异常值 |
| BeCu-3# | 0.246 | 0.0024 | 1.667 | 1.250 | 2.234 | 无异常值 |

表58 不同磷含量水平样品分析结果异常值分析（新疆有色）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0393 | 0.00030 | 2.000 | 1.0000 | 2.234 | 无异常值 |
| BeCu-2# | 0.139 | 0.0018 | 2.222 | 1.667 | 2.234 | 无异常值 |
| BeCu-3# | 0.0170 | 0.0004 | 1.000 | 1.500 | 2.234 | 无异常值 |

表59 不同铍含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.470 | 0.0072 | 2.093 | 1.674 | 2.234 | 无异常值 |
| BeCu-2# | 2.439 | 0.015 | 1.540 | 1.741 | 2.234 | 无异常值 |
| BeCu-3# | 3.867 | 0.030 | 1.553 | 1.485 | 2.234 | 无异常值 |

表60 不同钴含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0386 | 0.00067 | 1.343 | 1.343 | 2.234 | 无异常值 |
| BeCu-2# | 0.299 | 0.0034 | 1.765 | 1.471 | 2.234 | 无异常值 |
| BeCu-3# | 2.782 | 0.012 | 1.000 | 1.833 | 2.234 | 无异常值 |

表61 不同镍含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0800 | 0.0012 | 1.833 | 1.250 | 2.234 | 无异常值 |
| BeCu-2# | 0.402 | 0.0053 | 1.509 | 1.321 | 2.234 | 无异常值 |
| BeCu-3# | 2.804 | 0.013 | 1.513 | 1.299 | 2.234 | 无异常值 |

表62 不同钛含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.306 | 0.0025 | 1.6000 | 1.600 | 2.234 | 无异常值 |
| BeCu-2# | 0.118 | 0.0011 | 1.818 | 1.818 | 2.234 | 无异常值 |
| BeCu-3# | 0.0302 | 0.0010 | 1.300 | 1.400 | 2.234 | 无异常值 |

表63 不同铁含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0258 | 0.00027 | 1.482 | 2.222 | 2.234 | 无异常值 |
| BeCu-2# | 0.111 | 0.0017 | 1.177 | 1.765 | 2.234 | 无异常值 |
| BeCu-3# | 0.255 | 0.0029 | 1.724 | 1.724 | 2.234 | 无异常值 |

表64 不同铝含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0209 | 0.00031 | 1.613 | 1.613 | 2.234 | 无异常值 |
| BeCu-2# | 0.107 | 0.0005 | 2.000 | 2.000 | 2.234 | 无异常值 |
| BeCu-3# | 0.253 | 0.0040 | 1.750 | 1.250 | 2.234 | 无异常值 |

表65 不同硅含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0210 | 0.00075 | 1.467 | 1.333 | 2.234 | 无异常值 |
| BeCu-2# | 0.105 | 0.0011 | 1.818 | 1.818 | 2.234 | 无异常值 |
| BeCu-3# | 0.0597 | 0.00107 | 1.227 | 1.380 | 2.234 | 无异常值 |

表66 不同铅含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0169 | 0.00029 | 1.724 | 1.379 | 2.234 | 无异常值 |
| BeCu-2# | 0.0092 | 0.00046 | 1.956 | 1.087 | 2.234 | 无异常值 |
| BeCu-3# | 0.0473 | 0.0010 | 1.600 | 1.800 | 2.234 | 无异常值 |

表67 不同镁含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0197 | 0.00018 | 1.667 | 1.111 | 2.234 | 无异常值 |
| BeCu-2# | 0.0968 | 0.00059 | 1.695 | 1.525 | 2.234 | 无异常值 |
| BeCu-3# | 0.254 | 0.00066 | 1.515 | 1.515 | 2.234 | 无异常值 |

表68 不同磷含量水平样品分析结果异常值分析（富蕴恒盛）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0394 | 0.00044 | 1.818 | 1.591 | 2.234 | 无异常值 |
| BeCu-2# | 0.137 | 0.0016 | 1.250 | 1.875 | 2.234 | 无异常值 |
| BeCu-3# | 0.0160 | 0.00022 | 1.818 | 1.818 | 2.234 | 无异常值 |

表69 不同铍含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.483 | 0.00356 | 1.405 | 1.685 | 2.234 | 无异常值 |
| BeCu-2# | 2.426 | 0.00778 | 1.672 | 1.415 | 2.234 | 无异常值 |
| BeCu-3# | 3.820 | 0.00556 | 1.260 | 1.980 | 2.234 | 无异常值 |

表70 不同钴含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0385 | 0.00029 | 2.075 | 1.729 | 2.234 | 无异常值 |
| BeCu-2# | 0.305 | 0.00262 | 1.528 | 1.528 | 2.234 | 无异常值 |
| BeCu-3# | 2.784 | 0.00980 | 1.836 | 1.326 | 2.234 | 无异常值 |

表71 不同镍含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0837 | 0.00218 | 1.571 | 1.143 | 2.234 | 无异常值 |
| BeCu-2# | 0.403 | 0.00376 | 1.860 | 2.126 | 2.234 | 无异常值 |
| BeCu-3# | 2.820 | 0.00897 | 1.146 | 1.834 | 2.234 | 无异常值 |

表72 不同钛含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.318 | 0.00738 | 1.897 | 1.490 | 2.234 | 无异常值 |
| BeCu-2# | 0.120 | 0.00104 | 1.915 | 0.957 | 2.234 | 无异常值 |
| BeCu-3# | 0.0307 | 0.00085 | 2.114 | 1.409 | 2.234 | 无异常值 |

表73 不同铁含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0257 | 0.00062 | 1.134 | 1.459 | 2.234 | 无异常值 |
| BeCu-2# | 0.114 | 0.00186 | 1.610 | 1.610 | 2.234 | 无异常值 |
| BeCu-3# | 0.256 | 0.0021 | 0.957 | 1.915 | 2.234 | 无异常值 |

表74 不同铝含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0203 | 0.00058 | 1.855 | 1.546 | 2.234 | 无异常值 |
| BeCu-2# | 0.102 | 0.00087 | 1.144 | 1.144 | 2.234 | 无异常值 |
| BeCu-3# | 0.255 | 0.0021 | 1.758 | 1.406 | 2.234 | 无异常值 |

表75 不同硅含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0207 | 0.00078 | 1.779 | 1.651 | 2.234 | 无异常值 |
| BeCu-2# | 0.109 | 0.00533 | 1.501 | 1.876 | 2.234 | 无异常值 |
| BeCu-3# | 0.0604 | 0.00055 | 1.630 | 1.449 | 2.234 | 无异常值 |

表76 不同铅含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0173 | 0.00034 | 1.493 | 1.791 | 2.234 | 无异常值 |
| BeCu-2# | 0.00937 | 0.00027 | 1.774 | 1.246 | 2.234 | 无异常值 |
| BeCu-3# | 0.0448 | 0.00207 | 1.257 | 1.189 | 2.234 | 无异常值 |

表77 不同镁含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0199 | 0.00077 | 1.681 | 1.034 | 2.234 | 无异常值 |
| BeCu-2# | 0.0992 | 0.00055 | 1.638 | 1.092 | 2.234 | 无异常值 |
| BeCu-3# | 0.249 | 0.0034 | 1.166 | 1.458 | 2.234 | 无异常值 |

表78 不同磷含量水平样品分析结果异常值分析（上海监测）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 样品 | /％ | s | G1/％ | Gn/％ | 舍弃界限值/n=11,a=0.05 | 结论 |
| BeCu-1# | 0.0390 | 0.00055 | 1.811 | 1.630 | 2.234 | 无异常值 |
| BeCu-2# | 0.139 | 0.00234 | 1.282 | 2.137 | 2.234 | 无异常值 |
| BeCu-3# | 0.0163 | 0.00046 | 1.739 | 1.522 | 2.234 | 无异常值 |

根据格拉布斯检验方法，查表得：n=11,a=0.05时，舍弃界限值为2.234。表29～表78数据结果显示铍、钴、镍、钛、铁、铝、硅、铅、镁、磷不同含量样品的各单位11次测定数据均无异常值。

2.方法的重复性限和再现性限

1.重复性

在重复性条件下获得的两次独立测试结果的测定值，在表79给出的平均值范围内，这两个测试结果的绝对差值不超过重复性限（*r*），超过重复性限（*r*）情况不超过5％。重复性限（*r*）按表79数据采用线性内插法或外延法求得。精密度试验原始数据见附录A。

表79 重复性限

|  |  |  |  |
| --- | --- | --- | --- |
| *w*Be /％ | 0.48 | 2.43 | 3.84 |
| *r*/％ | 0.03 | 0.05 | 0.08 |
| *w*Co /％ | 0.038 | 0.30 | 2.77 |
| *r*/％ | 0.002 | 0.02 | 0.04 |
| *w*Ni /％ | 0.081 | 0.40 | 2.80 |
| *r*/％ | 0.004 | 0.01 | 0.04 |
| *w*Ti /％ | 0.031 | 0.12 | 0.31 |
| *r*/％ | 0.003 | 0.01 | 0.02 |
| *w*Fe /％ | 0.025 | 0.11 | 0.26 |
| *r*/％ | 0.002 | 0.01 | 0.01 |
| *w*Al /％ | 0.021 | 0.11 | 0.26 |
| *r*/％ | 0.002 | 0.01 | 0.01 |
| *w*Si /％ | 0.021 | 0.060 | 0.11 |
| *r*/％ | 0.002 | 0.003 | 0.01 |
| *w*Pb /％ | 0.0098 | 0.017 | 0.046 |
| *r*/％ | 0.0011 | 0.002 | 0.004 |
| *w*Mg /％ | 0.020 | 0.10 | 0.25 |
| *r*/％ | 0.002 | 0.01 | 0.01 |
| *w*P /％ | 0.016 | 0.039 | 0.14 |
| *r*/％ | 0.002 | 0.003 | 0.01 |

2. 再现性

在再现性条件下获得的两次独立测试结果的测定值，在表80给出的平均值范围内，两个测试结果的绝对差值不应超过再现性限（*R*），超过再现性限（*R*）的情况不超过5%，再现性限（*R*）按表80数据采用线性内插法或外延法求得。精密度试验数据见附录A。

表80 再现性限

|  |  |  |  |
| --- | --- | --- | --- |
| *w*Be /％ | 0.48 | 2.43 | 3.84 |
| *R*/％ | 0.04 | 0.06 | 0.10 |
| *w*Co /％ | 0.038 | 0.30 | 2.77 |
| *R*/％ | 0.004 | 0.02 | 0.07 |
| *w*Ni /％ | 0.081 | 0.40 | 2.80 |
| *R*/％ | 0.008 | 0.02 | 0.06 |
| *w*Ti /％ | 0.031 | 0.12 | 0.31 |
| *R*/％ | 0.003 | 0.01 | 0.02 |
| *w*Fe /％ | 0.025 | 0.11 | 0.26 |
| *R*/％ | 0.003 | 0.01 | 0.02 |
| *w*Al /％ | 0.021 | 0.11 | 0.26 |
| *R*/％ | 0.003 | 0.02 | 0.02 |
| *w*Si /％ | 0.021 | 0.060 | 0.11 |
| *R*/％ | 0.003 | 0.004 | 0.02 |
| *w*Pb /％ | 0.0098 | 0.017 | 0.046 |
| *R*/％ | 0.0019 | 0.003 | 0.006 |
| *w*Mg /％ | 0.020 | 0.10 | 0.25 |
| *R*/％ | 0.003 | 0.01 | 0.02 |
| *w*P /％ | 0.016 | 0.039 | 0.14 |
| *R*/％ | 0.002 | 0.003 | 0.01 |

四、标准中涉及专利的情况

本文件不涉及专利问题。

五、预期达到的社会效益等情况

本文件充分考虑了国内外铜铍合金研制及生产企业和用户单位的生产工艺技术水平。本文件颁布执行后，将进一步规范铜铍合金中铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的分析检验工作，更好地指导相关行业铜铍合金的分析检测和应用水平；有利于生产采用统一的分析方法开展产品质量检验工作，有利于市场公平交易环境的形成，具有较大的社会效益。

六、采用国际标准和国外先进标准的情况

无。

七、与现行法律、法规、强制性国家标准及相关标准协调配套情况

本标准的技术内容与现行相关法律、法规和强制性国家标准没有冲突。在标准修订过程中充分考虑到了国内外铜铍合金相关产品标准的技术内容，修订后铍、钴、镍、钛、铁、铝、硅、铅、镁、磷元素的测定范围完全覆盖了目前所有的铜铍合金品种，能够与国内现行的铜铍合金产品标准配套使用。本标准内容全面、条款详细、格式规范，符合GB/T 1.1-2020的相关要求。

八、重大分歧意见的处理经过和依据

无。

九、标准作为强制性或推荐性标准的建议

根据标准化法和有关文件规定，建议本标准性质为推荐性行业标准。

十、贯彻标准的要求和措施建议

（一）在标准实施前应保证标准文本在铜铍合金相关生产和应用单位及检测机构中有充足的供应，这是保证新标准贯彻实施的基础。

（二）针对标准使用的不同对象，有侧重地开展标准的宣贯培训，以保证标准的贯彻实施。

（三）对于标准使用过程中出现的疑问，起草单位有义务进行必要的解释。

（四）建议本标准批准发布6个月后实施。

十一、废止现行有关标准的建议

建议废止 YS/T 470.1-2004《铜铍合金化学分析方法 电感耦合等离子体发射光谱法测定铍、钴、镍、钛、铁、铝、硅、铅、镁量》。

十二、其他应予以说明的事项

无。

《铜铍合金化学分析方法》标准修订编制组

2023-9

附件：

标准征求意见稿意见汇总处理表

标准项目名称：铜铍合金化学分析方法 第1部分：铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的测定 电感耦合等离子体原子发射光谱法

承办人：马肖 共 2 页 第 1 页

标准项目负责起草单位：西北稀有金属材料研究院宁夏有限公司 电话:0952-2098318

2023年9月13 日填写

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 序号 | 标准章条编号 | 意见内容 | 提出单位 | 处理意见 | 备注 |
| 1 | 2 | 删除“2 规范性引用文件”中“GB/T 8170 数值修约规则与极限数值的表示和判定”。 | 有色金属技术经济研究院有限责任公司 | 采纳 |  |
| 2 | 4 | 更改“4 原理”部分，增加硅的质量分数大于0.20%时，溶解描述。 | 中铝洛阳铜加工有限公司 | 采纳 |  |
| 3 | 5 | 更改“5 试剂和材料”中部分试剂和材料的表述内容。 | 国标（北京）检验认证有限公司 | 采纳 |  |
| 4 | 5.3 | 增加“5.3 盐酸”，以及修改后续对应试剂序号。 | 绍兴市质量监督检测院 | 采纳 |  |
| 5 | 8.3 | 删除“8.3 空白试验”中“称取与试料对等的金属铜，”的表述及要求。 | 中色奥博特铜铝业有限公司 | 采纳 |  |
| 6 | 8.4.4 | 增加“8.4.4”，当硅的检测结果大于0.20％时，优化样品溶解方法。 | 贵溪金信金属有限公司 | 采纳 |  |
| 7 | 8.5 | 更改“8.5 工作曲线的绘制”中关于“铜基体溶液”的具体量的表述。 | 广东省科学院工业分析检测中心 | 采纳 |  |
| 8 | 9 | 于公式中增加“f--稀释因子”及相关描述 | 金川集团股份有限公司 | 采纳 |  |
| 9 | 9 | 删除“9 试验数据处理”中“数值修约按GB/T 8170执行。”。 | 常州工学院 | 采纳 |  |
| 10 | 10 | 更改“10 精密度”中关于铍的重复性限及再现性限数据，采用更多设备测试以取得更有代表性水平测试数据。 | 有研亿金新材料有限公司 | 采纳 |  |
| 11 |  | 没有意见 | 北京时代蔽连科技有限公司 |  |  |

标准征求意见稿意见汇总处理表

标准项目名称：铜铍合金化学分析方法 第1部分：铍、钴、镍、钛、铁、铝、硅、铅、镁、磷含量的测定 电感耦合等离子体原子发射光谱法

承办人：马肖 共 2 页 第 2 页

标准项目负责起草单位：西北稀有金属材料研究院宁夏有限公司 电话:0952-2098318

2023年9月13 日填写

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 序号 | 标准章条编号 | 意见内容 | 提出单位 | 处理意见 | 备注 |
| 12 |  | 没有意见 | 国合通用测试评价认证股份公司 |  |  |
| 13 |  | 没有意见 | 江西铜业股份有限公司 |  |  |
| 14 |  | 没有意见 | 江苏理工学院 |  |  |
| 15 |  | 没有意见 | 钢铁研究总院有限公司 |  |  |
| 16 |  | 没有意见 | 株洲硬质合金集团有限公司 |  |  |
| 17 |  | 没有意见 | 自贡硬质合金有限责任公司成都  分公司 |  |  |
| 18 |  | 没有意见 | 河南科技大学 |  |  |
| 19 |  | 没有意见 | 国合通用（青岛）测试评价有限  公司 |  |  |
| 20 |  | 没有意见 | 昆明冶金研究院有限公司 |  |  |
| 21 |  | 没有意见 | 广东省科学院新材料研究所 |  |  |
| 22 |  | 没有意见 | 北矿检测技术股份有限公司 |  |  |

说明（1）发送《征求意见稿》的单位数：22 个；

（2）收到《征求意见稿》后，回函的单位数： 22个；

（3）收到《征求意见稿》后，回函并有建议或意见的单位数：10个；

（4）没有回函的单位数：0 个。