GBW07511 硒化锌人工合成晶体矿物电子探针成分分析标准物质

1. Foreword

This certified reference material, is a sort of synthetic semi-conductor compound provided by the state-run Factory NO.739. The grain size of the sample: 20-60 mesh.

The major X-ray diffution intensity:

	d(Å)	3.27	2.00	1.71
	I/I$_0$	100	70	44

Cubic System $T_d^2-F_{43}^-m$ a$_0$=5.667 Z=4

Calculating Formula

$$Zn_{1.006}Se_{1.00}$$

The sample is characterized by the homogeneous distribution of elements, the purity of material used and the stability under the bombardment of electron beam. Therefore, it can be used as the certified refcrence material in electron probe for quantitative analysis.

2. Form of Sale

The minimum selling unit of the materiac is granule. Each sample granule may be made into electron probe analytic sample stage or be sold as single granule according to the requirement of consumers.

3. Brief Description of Sample Preparation

The sample, previously in form of lump, is firstly cleaned with distilled water and ground to a grain size of less than 20 mesh; Secondly, the ground material is sieved after cleaning and drying and the inclusion in the part of 20-60mesh is eliminated; Finally, the backscatting graph checking with electron probe is carried out by means of random sampling, and the result indicates that the purity of the sample meets the requirement.

4. Measurement of Characteristic Value

Characteristic values are measured by classical chemical analysis. Two analytic specimens of each about 200mg are made from the sample randomly and then are analyzed by two laboratories respectively. The average values of the two results obtaincd are used as standard values. The analysis method is as follows:

Element	Method(1)	Method(2)
Zn	Volumetry	Volumetry
Se	Volumetry	Volumetry
Analytic Unit	Institute of Multipurpose Utilization of Mineral Resources	Laboratory of Bureau of Geology and Mineral Resources of Jiangsu Province

Analyzer: Cheng Huashi Yang Wensi

5. Standard Value and Uncertainty

Element	Standard Value (wt%)	Uncertainty (%)
Zn	45.38	0.35
Se	54.44	0.55

The uncertainty is mainly caused by analytic error, inhomogeneity of material, and the drift due to instrumetal instability and deviation dueto counting statistics when using electron probe.

6. Homogeneity Test

Sampling Method: Take 30 granules of sample randomly from about 1000 granules and make them into sample stages.

Measuring Instrument: French-made CAMEBAX-MICRO electron probe

Working Condition: Accelerating Voltage: 20kv; Beam Current: 1.25×10^{-8}A

Beam Diameter: 2μm

Measuring Method: Measure 3 points randomly on each sample granule and three times for each point (counting after 10 seconds).

Testing Method: Test with variance analysis F and inhomogeneitsy value, and the results are satisfactory if inhomogenity value vc<1.0%.

Testing Result:

Zn Vc=0.23% ; Se Vc=0.29%

7. Stability Test

Testing method is the same as the homogeneity test. Take 3 granules of sample randomly, one point for each granule and one time for each point (10 seconds for each time). The Stability value SI is the average value of the results of above three points. The stability is satisfactory under the bombardment of electron beam if SI<3.

Result:

Zn SI=1.36; Se SI=1.34

8. Proper Using and Storing Method

Using Method: Mosaic sample granules into sample stages made of conductive powder resin, or glue them with glue bonds such as epoxy resin. Then they are ready to use after a plane has been made and been polished on them (Carbon film should be plated or conductive bond should be used if the glue bond is not conductive). The sample granules should be pure, and the temperature and pressure should not be too high in the preparation to avoid the break of samples and possi-

ble changes. The prepared planes should be smooth and un-polluted. The carbon film should be plated soundly. When measuring, use the certified reference material and determinand samples under the same conditions. The elemental contents of determinand samples can be obtained by rectifying the results from measurement. To reduce the error caused by the inhomogeneity of the certified reference material to the minimum extent, it is better to measuring 3—5 points each time and to take the average value as the final result. The certified reference matrial should be re-polished and re-plated after using for a period of time.

Storing Method: Store the certified reference materal in sample rooms or vacuum containers or desicators.

9. Units and Personnel of Research and Cooperation

Unit in Charge: Inatitute of Multipurpose Utilization of mineral Resoures

Wang Shugen, Cheng Huashi, Yang Youfu, Mao Zhichao, Zhou Kaixun

Cooperative Unit: Nanjing Institute of Geolgy and Mineral Resources

Wang Guanhua, Chao Fuwei

Laboratory of Bureau of Geology Resources of Jiangsu Province

Yang Wensi, Zhang Shujun

一、序

本标准物质**硒化锌**是人工合成半导体化合物材料，由国营七三九厂提供，样品粒度：20—60目。主要X射线粉晶衍射线及强度是：

d(Å)	3.27	2.00	1.71
I/I。	100	70	44

立方晶系　$T_d^2-F\bar{4}3m$　　$a_。=5.667$　　$Z=4$

计算化学式：　$Zn_{1.008}Se_{1.00}$

元素分布均匀，材料纯净，在电子束轰击下稳定，可作为电子探针定量分析标准物质使用。

二、发售形式

发售的最小包装单元为颗粒。每个颗粒根据用户需要可制成电子探针分析样座出售或单个颗粒出售。

三、制备方法简述

所提供的样品呈块状。首先用蒸馏水洗净，再破碎至小于20目，清洗、烘干，随后将样品筛分为数级，去掉20—60目部份的夹杂物，随机抽样进行电子探针背散射图象检查，其纯度符合要求。

四、特性量值测量方法

特性量值是用经典化学分析方法进行测量，在样品中随机取200毫克左右样品两份，分别由两个实验室进行分析，两份分析结果的平均值作为标准值，各元素分析方法如下：

元素	方法(1)	方法(2)
Zn	容量法	容量法
Se	容量法	容量法
分析单位	地矿部矿产综合利用研究所	江苏省地矿局实验室
分析者：	陈华实	杨文思

五、标准值及其确定度

元素	标准值（wt%）	不确定度（%）
Zn	45.38	0.35
Se	54.44	0.55

不确定度主要来自化学分析误差、材料不均匀性以及电子探针检验时仪器漂移和计数统计偏差。

六、均匀性检验

取样方法：在约1000粒左右的**硒化锌**中，随机取30粒，制成电子探针分析样品座。

测试仪器：法国产CAMEBAX—MICRO电子探针

工作条件：加速电压20KV，束流1.25×10^{-8}A

电子束直径：2μm

测试方法：在30个颗粒上，每粒随机测3点，每点测3次，每次测10秒计数。

检验方法：用方差分析F检验和不匀率值来检验，当其不匀率$Vc<1.0\%$时，认为符合要求。

均匀性检验结果：

Zn　　Vc=0.23%；　　Se　　Vc=0.29%

七、稳定性检验

检验方法与均匀度检验相同。测试方法是在样品上随机取3粒，每粒测1点，每点测1次，每次10秒计数。计算每点的稳定度，3点平均值为样品稳定度（SI），当SI＜3时，认为样品在电子束轰击下稳定。

稳定性检验结果：Zn　　SI=1.36；　　Se　　SI=1.34

八、正确使用及储存方法

使用方法：将样品颗粒镶嵌在导电树脂粉的样座中，或胶结在环氧树脂等胶结物中，将样品磨出一个平面，抛光（若胶结物不导电应引以导电胶或镀上碳膜）后即可使用。制备时必须注意，颗粒应纯净，制样时温度压力不可过大，以免样品碎裂或变化。所制成的表面，必须尽可能平整、光滑，没有污染。镀膜牢固。使用时，将此标准物质与试样在相同条件下测定，所得结果经校正计算即可得到试样相应元素的含量。为减少标准物质不均匀性引起的误差，最好在标准物质上一次测3—5个点，取其平均计数作为标准物质计数。使用一段时间后，可重新抛光、镀膜。

储存方法：将标准物质置于仪器样品室或其它真空容器、干燥器中。

九、研制、协作单位和个人

负责单位：地质矿产部矿产综合利用研究所

王树根、陈华实、杨有富、毛治超、邹开勋

合作单位：地质矿产部南京地质矿产研究所

王关华、晁福为

江苏省地矿局实验室

杨文思、张淑君