

中华人民共和国黑色冶金行业标准
Ferrous Metallurgy Industry Standard of P. R. China
YB/T 4145-2006

Industry Standards: Ceramic Crucible for Carbon and Sulfur Analysis

Brand :CSCERAMIC Factory :CS CERAMIC CO.,LTD

YB/T 4145-2006 (China)

Dedicated Carbon Sulfur Analyzing Crucible



1. Scope

This standard defines terminologies, structure, technical requirements, usage requirements, package, storage and transportation, marks, and quality certificates of dedicated **carbon sulfur analyzing crucible**.

This standard is applicable to dedicated carbon sulfur analyzing crucibles (hereinafter referred as to carbon sulfur crucibles) those made from natural mineral rock, by process of pressing and sintering.

2. Normative Reference Documents

Items in following documents are quoted so become items of this standard. Reference documents those with date noted, their later modification list (exclude corrigendum content) and revised edition are applicable to this standard. However, for application of latest edition of the documents, discussion and agreement according to this standard will be encouraged. Documents those without noted date, their latest edition is applicable to this standard.

GB/T 3299 Method for determination of water absorption of daily ceramics

GB/T 3301 Method for determination of volume deviation, diameter deviation, height deviation, weight deviation and defect size of daily ceramics

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3. Terminologies and definitions

The following terminologies and definitions are applicable to this standard.

3.1 Edge-lack

Breakage in rim of crucible

3.2 Fissure

Tiny crack in surface of crucible

3.3 Crackle

Striped fault due to crack of the crucible body

3.4 Fusion hole

Cavity formed due to fusion of fusible content during the process of sintering.

3.5 Speaks or Iron spots

Colored stain in product surface, also called iron spots.

3.6 Heat-resistance

The capability that when put the crucible in high temperature furnace and raise the temperature to 1500℃, it can still keep unbroken.

3.7 Ratio of crack

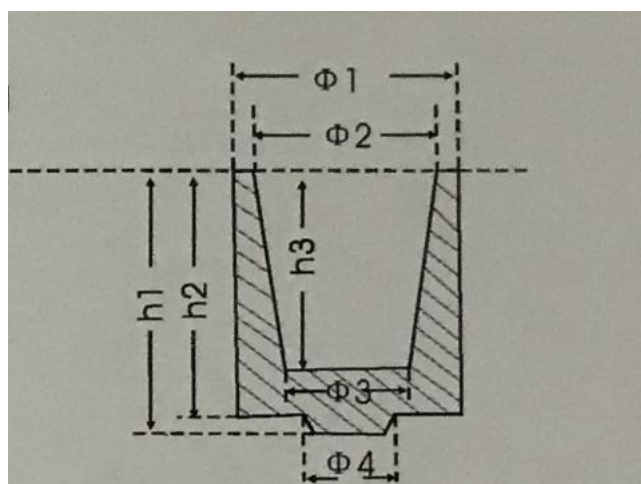
When put crucibles in high temperature furnace and raised the temperature to 1500℃, the ratio of crucible that crack appears.

3.8 Percent of water absorption

When the open pores of crucible body are saturated with water, the weight percentage of absorbed water to constant weight after drying the crucible at 110℃.

4、 Structure and codes

4.1 Main structure of crucible, see figure 1



4.2 Codes of crucibles are denoted by capital phonetic alphabet, PT for normal carbon sulfur

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crucible and CD for super low carbon sulfur crucible, the followed Arabic numerals are height and external diameter.

For example, PT 23*23 means normal carbon sulfur crucible with height of 23mm and external diameter of 23mm.

5、 Technical requirements

5.1 Raw material of carbon sulfur crucibles

5.1.1 Usually mixture of Kaolin (Al_2O_3) and quartzite(SiO_2) with certain ratio are used as raw material, main technical are as table 1.

ram material name	Kaolin	quartzite
main chemical component		
(mass fraction) %	Al_2O_3 : 70-80	$\text{SiO}_2 \geq 90$
mixing ratio %	40-60	40-60

5.1.2 Usually granularity of raw material is 0.055mm~0.08mm

5.1.3 Free carbon and impurity oxide in raw material should be removed before molding.

5.1.4 Before molding, carbon content in raw material should be less than 0.001% and sulfur content should be less than 0.001%.

5.2 Appearance and specification of crucible

5.2.1 Any crackle or defect like edge lack, fusion hole, fissure, iron sport that may affect normal use is not acceptable.

5.2.2 Surface defect of crucible should conform to the standard in table 2

surface defect assessing standard of **carbon sulfur crucible**

Defect item	range	criterion	defect item	range	criterion
Edge lack	length>2.0mm	not acceptable	fusion hole	diameter $\leq 1.5\text{mm}$	no more than 3
	width>1.5mm	not acceptable		depth $\leq 1.0\text{mm}$	no more than 3
	length 1.0mm~2mm	no more than 3	fissure	internal surface	not acceptable
	width 1.0mm~1.5mm	no more than 3		external surface	no limit
	length <1.0mm	no limit	crackle	internal surface	not acceptable
	width <1.0mm	no limit		external surface	not acceptable

● 5.2.3

Surface color and luster should be even, mottle or spot in internal surface are not acceptable. In external surface, total mottle area should be less than 10% of column superficial area.

5.2.4 dimension and allowable deviation of crucible should conform to the standard in table 3

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Table 3 dimension and allowable deviation of CS crucible

	h1		h2		h3		Ø 1		Ø 2		Ø 3		Ø 4	
Cs ceramic crucible code	basic dimension	Allowable deviation	basic dimension	Allowable deviation	basic dimension	Allowable deviation	basic dimension	Allowable deviation	basic dimension	Allowable deviation	basic dimension	Allowable deviation	basic dimension	Allowable deviation
PT 23*23	24	±1 ±0	23	±1 ±0	15	±0	23	±0.5	18	±0.5	14	±0.5	11	0-1
PT 25*25	26	±1	25	±1	18	±0.5	25	±0.5	19	±0.5	16	±1	11	0-1
CT 25*25	26	±1	25	±1	18	±0.5	25	±0.5	19	±0.5	18	±1	11	0-1

5.2.5 Thickness of crucible wall thickness should be uniform.

5.3 chemical performance

5.3.1 Baseline value of crucible: for normal crucible, carbon baseline value should be less than 0.0005%, and sulfur less than 0.0005%; for super low carbon sulfur crucible, carbon baseline value should be less than 0.0003%, and sulfur less than 0.0002%.

5.4 Physical performance

5.4.1 Thermal endurance of crucible: usually no higher than 150℃

5.4.2 Wracking ratio of crucible: less than 3%

5.4.3 Water absorption: larger than 3%

5.5 Inspection items

5.5.1 Carbon and sulfur element content in raw material

Inspection method of carbon and sulfur element content should conform to regulation in appendix A

5.5.2 Appearance inspection

Usually self-inspection is implemented under natural light, conform to requirements in table 2 (the measuring implement should with qualified precision)

5.5.3 Specification inspection

Main dimension inspection of crucible should conform to standard GB/T3301

5.5.4 Baseline value test

Baseline value test should conform to appendix A, carbon and sulfur content test with at least 40 samples continuously, and the arithmetic average will be regarded as carbon and sulfur baseline value of crucible.

5.5.5 Cracking ratio inspection

When test baseline value, cracking ratio can be inspected simultaneously, which is the number of crucibles that with crackle divided by total quantity of crucible.

5.5.6 thermal endurance test

Put crucibles in high temperature furnace, raise the temperature to 1500℃ and keep for 5 minutes. Then turn of the power, wait until the furnace drop to room temperature and take out

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the crucibles, deformation should not occur, and the dimension should conform to standard in table 3.

5.5.7 water absorption test

Water absorption test should be implemented as per standard GB/T3299.

5.5.8 Final inspection before delivery and judgement

Factory should implement sample inspection for every batch (kiln) of product, and sample quantity should be 0.1% of total product quantity, or no less than 100 pieces. If sample inspection is qualified, then the current batch (kiln) of products are judged as being qualified, and delivery is allowable. If sample inspection is not qualified, then extra tightened sample inspection can be implemented, sample 0.2% of total quantity or no less than 200 pieces and inspect, if still not qualified, then the current batch (kiln) of products are judged as being unqualified and delivery is now allowable.

6 Use requirements

Besides raw material and fabrication process, crucible baseline may also from absorption. Before using, influence of water, SO₂ and CO₂ to analyzing result should be avoided. For this purpose, two methods below are recommended.

Method 1, put **carbon sulfur ceramic crucible** in high temperature furnace, raise the temperature to 1200℃ and burn it for 2 hours, then take it out and get cooled, put into desiccator for demand (preservation time no longer than 4 hours)

Method 2, put **carbon-sulfuric analysis crucible** in tube-type crucible furnace, induce pure oxygen (purity 99.% or higher), and burn it no less than 1 hour at 1200℃.

7. Packaging, Storage and Transportation, Marking and Quality Certificate

7.1 Packing

7.1.1 Crucible shall be packed with tin foil or pure aluminum white paper, and the minimum packing unit shall be 10 pieces for each roll. Rolls should be put into paper packing cases with plastic film as inner liner, and should be arranged in sequence with corrugated paper as the interval between layers, for each case, total quantity should be 1000 pieces.

7.1.2 If the buyer has special requirements for packaging, then buyer and seller can have a further discussion.

7.2. Storage and transportation: The crucible shall be stored in a well-ventilated warehouse, and the air shall not contain any corrosive gas. Moisture should be kept away. Packed crucibles shall be suitable for any means of transportation, collision rain hit should be avoided during transportation.

7.3. Marking: Surface of the packing box shall be marked with non-fading marking, including product name, quantity, specification, crucible carbon and sulfur baseline value, production date and manufacturer name, etc.

7.4. Quality Certificate: Quality certificate that verify products comfort to requirements of

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the contract or relative standard should be enclosed in the packing box. Quality certificate should indicate the items below:

A) Name of manufacturer; B) Product name, specification, model, carbon and sulfur baseline value of crucible; C) Production date; D) Code number of inspector; E) Standard number or contract number

Appendix A (information appendix)

Baseline Determination of Crucible (Material)

A.1 reagents

A.1.1 pure iron flux: the particle size is less than 1.25mm, and mass fraction of both carbon and broken iron are less than 0.0005%.

A.1.2 vanadium pentoxide: purely analyzed, put it into a muffle furnace at 600 °C for 2 hours before using, and cool it in a dryer for reserve.

A.1.3 tungsten flux: the particle size is 0.4~0.8mm, and mass fraction of sulfur and sulfur are less than 0.001% and 0.0005% respectively.

A.1.4 ceramic crucible: use a ceramic crucible of good quality, burn it 4 hours at 1200 °C in a muffle furnace before using, then cool it overnight in the furnace and take it out after cooling down to room temperature in the dryer for reserve.

A.1.5 oxygen: high purity (higher than 99.5%)

A.2 instruments

A2.1 high frequency infrared sulfur analyzer: sensitivity 0.00001%

A.3 steps of analysis

A.3.1 Sample: Grind the "dedicated crucible for sulfur analysis" to less than 0.15mm with an agate mortar and roast it at 105-110 °C for 2h, then take it out and cool down to room temperature in a dryer for reserve.

A.3.2 render crucible (secondary crucible): Add 1 gram pure iron flux (A.1.1) and 1 gram tungsten flux (A.1.3) to a ceramic crucible treated as step A.1.4. Put the crucible in high-frequency induction furnace of infrared carbon crucible analyzer, aerate oxygen and heat it with analysis program. Take it down after the analysis is completed, after slightly cooled, then put it in a dryer (check and confirm no cracks) to cool it down to room temperature for reserve.

A.3.3 instrument calibration

Weigh standard sample 1.000 gram (C:0.00xx%, S:0.000xx% or C:0.000xx%, S: 0.0000xx%) and put it in render crucible. Add 1 gram pure iron flux (A.1.1) and 0.6 gram vanadium pentoxide (A.1.2) and input the mass value of weighed standard sample into the instrument mass compensator. Start analysis, repeat measurement for three times, and take the average value for instrument calibration.

A.3.4 measurement (baseline value of crucible material)

Accurately weigh 1.000 gram standard sample and put it in the bottom of render crucible

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and add 1 gram pure iron flux (A.1.1) and 0.6 gram vanadium pentoxide (A.1.2), and input the mass value of weighed standard sample into the instrument mass compensator. Complete the analysis, carbon and sulfur value given by the instrument is baseline value of the crucible material.

A.3.5 measurement (baseline value of crucible)

Take a pretreated ceramic crucible (or ceramic crucible without any pretreatment) and place it on an electronic balance. After getting the net weight, add 1 gram pure iron flux (A.1.1) and 0.6 gram vanadium pentoxide (A.1.2), and input mass value of 1.000 gram into the instrument mass compensator. Complete the analysis, carbon and sulfur value given by the instrument is baseline value of the crucible.

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碳硫分析专用坩埚

Special Oven for Carbon and Sulphur Analysis



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碳硫分析专用坩埚

1、范围

本标准规定了碳硫分析专用坩埚的术语、结构、技术要求、使用要求以及包装、储运、标志和质量证明书。

本标准适用于以天然矿物岩石为原料，经加压、烧结成型的碳硫分析专用坩埚（以下简称坩埚）。

2、规范性引用文件

下列文件中的条款通过本标准的引用而成为本标准的条款。凡是注日期的引用文件，其随后所有的修改单（不包括勘误的内容）或修订版均不适用于本标准。然而，鼓励根据本标准达成协议的各方研究是否可使用这些文件的最新版本。凡是不注日期的引用文件，其最新版本适用于本标准。

GB/T 3299 日用陶瓷器吸水率测定方法

GB/T 3301 日用陶瓷器的容积、口径误差、高度误差、重量误差、缺陷尺寸的测量方法。

3、术语和定义

下列术语和定义适用于本标准。

3.1 缺棱 Edge-lack

坩埚口边缘的破损现象。

3.2 龟纹 Fissure

坩埚表面不规则的微小裂纹。

3.3 裂纹 Crackle

指坯开裂而形成的网状缺陷。

3.4 熔洞 Fusion hole

易熔物在烧成过程中熔融而产生的孔洞。

3.5 斑点 Specks or iron spots

制品表面呈现的有色污点，又称铁点。

3.6 耐温性 Heat-resistance

坩埚在高温炉中，随温度升至1500℃时而不被损坏的能力。

3.7 开裂率 Ratio of crack

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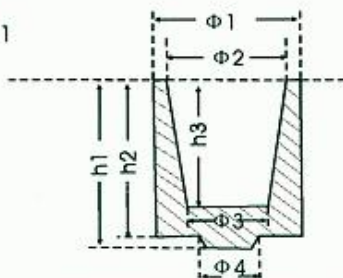
坩埚在高温炉中，由炉温升至1500℃左右时，坩埚开裂的比率。

3.8 吸水率 Percent of water absorption

陶瓷胎体开口气孔吸饱水后，所吸入水的重量对试样经110℃干燥至恒重后的重量百分比。

4、结构及代号

4.1 坩埚主要结构，见图1



4.2 坩埚的代号用大写普通和超低两字的汉语拼音字首表示，即PT表示普通碳硫坩埚，CD表示超低碳硫坩埚，随后的阿拉伯数字表示坩埚的高度和外径尺寸。

例如：PT 23×23 表示普通型坩埚，高度为23mm，外径为23mm。

5、技术要求

5.1 原料

5.1.1 原料一般选择高岭土(Al_2O_3)、石英石(SiO_2)按一定比例混合，主要技术指标见表1

原料名称	高岭土	石英石
主要化学成分 (质量分数)，%	Al_2O_3 : 70-80	$SiO_2 \geq 90$
混合比，%	40-60	40-60

5.1.2 原料的粒度一般为0.055mm~0.800mm。

5.1.3 在成型前应除掉原料中存在的游离碳，以及影响产品质量的杂质氧化物。

5.1.4 成型前原料碳含量应小于0.0010%；硫应小于0.0010%。

5.2 坩埚的外观及规格：

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5.4.3 吸水率：吸水率大于3%。

5.5 检验内容：

5.5.1 原料中碳、硫元素的测定

原料中碳、硫元素的检验方法参照附录A的规定。

5.5.2 外观检验：

通常在自然光条件下进行自测，并应符合表2的要求（用符合精度要求的量具检查）。

5.5.3 规格检验：

坩埚的主要规格尺寸检验，按GB/T3301的规定。

5.5.4 空白值测定：

坩埚的空白值检验参照附录A的规定，一般连续测量不少于40个坩埚的碳硫含量，用其算术平均值作为坩埚的碳硫空白值。

5.5.5 开裂率的检验：

在测量碳、硫空白值的同时，观察坩埚的开裂情况，用已开裂坩埚除以总坩埚数计算出开裂率。

5.5.6 耐温性检验：

坩埚置于高温炉中，随室温升至1500℃，保温5min。关闭电源，随炉温降至室温后，取出检验坩埚无变形，尺寸符合表3中规定。

5.5.7 吸水率检验：

坩埚的吸水率测定按GB/T3299规定。

5.5.8 出厂检验及判定：

生产厂家应对每批（窑）产品进行随机抽样检验。检验抽样数为成品的1%或不少于100只，如果抽样检验合格，即本批（窑）产品合格，允许出厂。如果检验不合格可再进行加严抽样检验，加严检验抽样数为成品的2%或不少于200只，加严检验仍不合格，则视为本批（窑）产品不合格，不予出厂。

6 使用要求：

坩埚除原料和制作过程中带来的空白外，还具有一定的吸附性，使用前应避免水分、SO₂和CO₂对分析结果的影响。推荐采用以下两种方法。

方法1，将坩埚置于高温炉中，在温度1200℃下灼烧不少于2h，取出稍冷，放置于干燥器备用（不宜超过4h）。

方法2，将坩埚置于管式坩埚处理炉中，通入纯氧气（质量分数99.5%），在温度1200℃下灼烧不少于1h。

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7 包装、储运、标识和质量证明书

7.1 包装

7.1.1 坩埚采用锡箔或纯铝白纸包装，最小包装单元为每10只一卷筒。放入衬有塑料薄膜的纸质包装箱内，依次排列好，层与层之间隔以瓦楞纸，每箱装1000只。

7.1.2 需方对包装如有特殊要求，可由供需双方协议商定。

7.2 储运：坩埚应储存在通风良好的库房中，空气中不应含有腐蚀气体，并注意防潮。包装好的坩埚应适应任何运输工具的运输，运输途中应避免碰撞和雨、雪的直接淋袭。

7.3 标识：包装箱的表面需注有不掉色的标志，包括产品名称、数量、规格型号、坩埚的碳硫空白值、出厂日期以及生产厂名称等。

7.4 质量证明书：包装箱内须附有证明产品符合订货合同或标准要求的质量证明书，质量证明书上应注明。

A)生产厂名称；B)产品名称、规格型号及坩埚的碳硫空白值；C)出厂或生产日期；D)检验人员代号；E)标准号或合同号。

附录A：（资料附录）

坩埚（材质）空白的测定

A.1 试剂

A.1.1 纯铁助熔剂：粒度小于1.25mm，含碳、硫的质量分数分别小于0.0005%。

A.1.2 五氧化二钒：分析纯，使用前在马弗炉中600℃烘2h，放入干燥器中冷却备用。

A.1.3 钨助熔剂：粒度为0.4mm-0.8mm，含碳、硫的质量分数分别小于0.001%、0.0005%。

A.1.4 瓷坩埚：用质量较好的瓷坩埚，使用前在马弗炉中1200℃灼烧4h，在炉中随炉冷却过夜，取出于干燥器中冷至室温备用。

A.1.5 氧气：高纯（纯度99.5%以上）。

A.2 仪器：

A.2.1 高频红外碳硫测定仪：灵敏度0.00001%。

A.3 分析步骤

A.3.1 试样：将予分析的“碳硫分析专用坩埚”用玛瑙研钵研碎，研磨至0.15mm以下，在烘箱中105℃-110℃烘2h，取出在干燥器中冷至室温备用。

A.3.2 打底坩埚（二次坩埚）：将A.1.4处理过的瓷坩埚，加1g纯铁助熔剂[A.1.1]，1g钨助熔剂[A.1.3]，置于红外碳硫测定仪的高频感应加热炉内，通气。用分析程序加热，分析完毕后取下，稍冷放入干燥器内（检查无裂开）冷至室温备用。

A.3.3 仪器校准

称取标准样品1.000g（C：0.00xx%，S：0.000xx%或C：0.000xx%，S：0.000xx%，置于打底坩埚中，加入1g纯铁助熔剂[A.1.1]，0.6g五氧化二钒[A.1.2]；将所称取的标准样品的质量值输入仪器质量补偿器，开始分析，重复测定三次，取其平均值进行仪器校准。

A.3.4 测量（坩埚材质的空白）

准确称取试样1.000g，置于打底坩埚中，再加入1g纯铁助熔剂[A.1.1]，0.6g五氧化二钒[A.1.2]，将称取的试样的质量值输入仪器质量补偿器，开始分析完毕后，仪器所给出的碳硫值即为坩埚材质的空白值。

A.3.5 测量（坩埚的空白）

取经过前处理的瓷坩埚（或未经过任何预处理的瓷坩埚）放在电子天平上，去皮后加入1g纯铁助熔剂[A.1.1]，0.6g五氧化二钒[A.1.2]，将1.000g的质量值输入仪器质量补偿器，开始分析，分析完毕后，仪器所给出的碳、硫值即为坩埚的空白值。

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