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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 13221-2026

Replacing GB/T 13220-1991

**Determination of powder particle size distribution - Sonic
sieving and small angle X-ray scattering methods**

粉末粒度分布的测定 声波筛分法和X射线小角散射法

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Table of Contents

- 4 Acoustic sieving method
- 4.2 Instruments and Equipment
- 4.4 Test Procedure
- 4.4.3 Weigh the powder to an accuracy of
- 4.5 Experimental Data Processing
- 5 Small-angle X-ray scattering method
- 5.2 Instruments and Equipment
- 5.3 Sampling and Sample Preparation
- 5.4 Test Procedure
- 5.4.1 Test Preparation
- 5.4.2 Strength Measurement
- 5.5 Processing of Test Results
- 5.5.1 Calculation of Results

Foreword

This document conforms to GB/T 1.1-2020 "Standardization Work Guidelines Part

1. Structure and Drafting Rules of Standardization Documents". Drafting is scheduled. This document supersedes GB/T 13220-1991 "Determination of Particle Size Distribution of Fine Powders - Sonic Sieving Method" and GB/T 13221-2004. "Determination of Particle Size Distribution of Nanopowders by X-ray Small Angle Scattering". This document is based on GB/T 13221-2004 and integrates GB/T 13220- The content of.1991. Compared with GB/T 13221-2004 and GB/T 13220-1991, apart from structural adjustments and editorial changes, the main differences are... The technological changes are as follows:

- a) The scope of application has been changed (see Chapter 1, Chapter 1 of GB/T 13221-2004);
- b) The test environment requirements have been removed (see 5.2.4 of GB/T 13221-2004);
- c) The collodion was changed to cellulose acetate (see 5.3.2 and 5.3.3, 5.1 of GB/T 13221-2004);

d) The experimental procedure for the small-angle X-ray scattering method has been modified, and the conversion relationship has been incorporated into Appendix A (see 5.4, Appendix A, GB/T 13221-). Chapter 7 (2004)

e) The "cumulative distribution curve" (see 8.2.2.2 of GB/T 13221-2004) has been deleted.

f) The test report was amended (see Chapter 6, Chapter 10 of GB/T 13221-2004). Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents. This document was proposed by the China Nonferrous Metals Industry Association. This document is under the jurisdiction of the National Technical Committee on Standardization of Nonferrous Metals (SAC/TC 243). This document was drafted by: China Iron & Steel Research Institute Co., Ltd., Antai Tianlong Tungsten & Molybdenum Technology Co., Ltd., Jiangsu University of Science and Technology, and Beijing Iron & Steel Research Institute. Gaona Technology Co., Ltd., Kunming University of Science and Technology, Engineering and Technology Branch of CNOOC Energy Development Co., Ltd., Hebei Construction Engineering The college, Xi'an Ouzhong Materials Technology Co., Ltd., and Xiamen Tungsten Co., Ltd. The main drafters of this document are. Zhang Xiaodan, Huang Xin, Huo Jing, Ren Shugui, Zhang Chao, Qu Jinglong, Liang Feng, Liu Jingchao, Luo Zhiqiang, Ren Zhiguo, and Xie Zhipeng. Deng Han, Zhang Shulan, Shang Chuanbao, Zhu Zhen, Fan Chaoying, Li Ming. The release history of this document and the document it replaces is as follows: --First published in.1991 as GB/T 13221-1991, and revised for the first time in.2004; --This is the second revision, incorporating GB/T 13220-1991 "Determination of Particle Size Distribution of Fine Powders - Acoustic Sieving Method" content. Determination of powder particle size distribution Acoustic sieving method and X-ray small angle scattering method

1.Scope This document describes a method for determining powder particle size distribution using acoustic sieving and small-angle X-ray scattering. This document applies to the determination of powder particle size distribution. The range of test results is as follows: particle size not less than 20 μm by ultrasonic sieving method, X-ray... The particle size in line small-angle scattering is no greater than 300 nm. This document does not apply to powders such as flakes or needles with obviously unequal axonal shapes.

3.Terms and Definitions This document does not contain any terms or definitions that need to be defined.

4 Acoustic sieving method

4.1 Principle Sound waves of a certain frequency and amplitude generated by a sound wave generator force the air inside the sieve frame to vibrate, and the powder flows with the vibrating air onto the sieve surface. The upward motion causes particles smaller than the sieve openings to pass through and enter the next stage of the sieve, thus achieving the

purpose of sieving. After sieving, the powders at each stage are weighed. By measuring the mass, the particle size distribution of the powder can be determined.

4.2 Instruments and Equipment

4.2.1 Test sieves The test sieve consists of a sieve surface and a sieve frame. The selection of the sieve surface should conform to the requirements of GB/T 6003.1, and the sieve aperture size should conform to GB/T 6005. The regulations stipulate that the fit between the sieve frame and the sieve surface should be airtight to ensure that no powder is trapped or leaked during sieving. (Diagram of a test sieve device) See Figure 1.

4.2.2 Acoustic Screening Machine Any device that can achieve the purpose of acoustic sieving can be used. The recommended frequency range is

50 Hz to 100 Hz. A schematic diagram is shown in Figure 2.

4.2.3 Balance The graduation value is

0.001 g.

4.3 Sampling and Sample Preparation Sampling methods should be carried out in accordance with GB/T 5314. The powder should be tested according to the received condition, and the powder amount should preferably be 3 g to 7 g, or... The determination shall be made through negotiation between the supplier and the buyer. If requested by the buyer, the powder may be dried and weighed. If the powder is easily oxidized, it should be dried under a vacuum or inert atmosphere. Drying should be carried out under the following conditions. If the powder contains volatile substances, it should not be dried.

4.4 Test Procedure

4.4.1 Setting test parameters. The acoustic vibration frequency should be

50 Hz~100 Hz, and the recommended time for a single acoustic sieving test is 5 min~15 min.

4.4.2 Assemble the cleaned test sieves on the sieve base in order of larger sieve aperture size at the top and smaller sieve aperture size at the bottom, and weigh the empty sieves.

4.4.3 Weigh the powder to an accuracy of

0.01 g.

4.4.4 Pour the weighed powder into the top test sieve, then start the test sieve (4.2.1) to conduct the sieving test.

4.4.5 After sieving, weigh each test sieve. The mass difference between each test sieve before and after sieving is the powder mass of the corresponding particle size. The weighing process should maintain an accuracy of

0.01 g. The loss of powder after sieving should not exceed 5%.

4.4.6 After the sieving test, the test sieve should be carefully cleaned, but the sieve surface should not be damaged.

5 Small-angle X-ray scattering method

5.1 Principle When an extremely fine X-ray beam passes through a layer of nanoparticle powder, it is scattered by electrons within the particles, resulting in a very small angular region near the original beam. The particles disperse and undergo small-angle X-ray scattering. The intensity distribution of this scattering is closely related to the particle size and distribution of the powder, from which the scattering intensity can be calculated. The average particle size, median diameter, and dispersion of the powder. The conversion relationship between X-ray small-angle scattering intensity and powder particle size should conform to Appendix A. According to the regulations.

5.2 Instruments and Equipment

5.2.1 Balance The scale division is

0.1 mg.

5.2.2 Small-Angle X-ray Scattering Analyzer An X-ray small-angle scattering (SAS) instrument includes an X-ray source, an optical system, a small-angle goniometer or a wide-angle goniometer with a collimation system, a sample holder, and... Detector. The linear range of the detector's count rate should meet the requirements for determining small-angle scattering particle size distribution.

5.3 Sampling and Sample Preparation

5.3.1 The prepared nanopowder small-angle scattering specimens should meet the following requirements.

- a) The volume fraction of the powder to be tested in the sample is less than 3%;
- b) Place the test piece on the sample holder and control the thickness by measuring the absorption attenuation rate of X-rays on the test piece. A recommended absorption attenuation rate is... Between 50% and 70%;
- c) The powder particles are dispersed evenly within the effective size range of the test piece;
- d) The test piece should be flat and free of cracks. The size should be determined according to the size of the instrument, with a recommended size of 20 mm x 10 mm.

5.3.2 Prepare cellulose acetate solutions with a mass concentration of 50 g/L to 100 g/L using cellulose acetate without small-angle scattering effect and analytical grade acetone.