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GB/T 1425-2021

Replacing GB/T 1425-1996

Determination of melting temperature range for precious metals and their alloys - Testing method of thermal analysis

贵金属及其合金熔化温度范围的测定 热分析试验方法

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1 Scope

GB/T 1425-2021 is the Chinese national standard on determination of melting temperature range for precious metals and their alloys - testing method of thermal analysis, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 20 August 2021 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 March 2022. Classification: ICS 77.040.99, CCS H21. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This document specifies a method for determining the melting temperature range of precious metals and their alloys by using differential thermal analyzers or differential scanning calorimeters. This document is applicable to the test of the melting temperature range of precious metals and their alloys, and the test range is 25 °C~2000 °C.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable

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to this standard.

GB/T 6425 Nomenclature for thermal analysis

GB/T 8170 Rules of rounding off for numerical values and expression and judgement of limiting values

3 Terms and definitions

The terms and definitions defined in GB/T 6425 and the following are applicable to this document.

3.1 Melting temperature range The interval between the initial melting temperature Ti and the final melting temperature Tf is expressed as Ti ~ Tf

5.2 Reference material The reference material shall have no enthalpy change in the test temperature range and shall be stored in a desiccator for later use. Typical reference materials include calcined alpha-Al2O3, platinum sheets, glass beads, silicone oil, sample vessels, etc.

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6.1 Thermal Analysis Instruments

6.1.1 Differential thermal analyzer: the maximum heating rate shall be not less than 10 °C/min, with sufficient accuracy and adjustment ability, so that the melting peak generated by the temperature difference between the sample and the reference material when the sample is being melted can be automatically recorded in the 10%~90% indexing range of the recorder. Reading accuracy of the temperature curve: the deviation shall not exceed ±0.5 °C when the temperature is lower than 300 °C, ±1 °C when the temperature is 300 °C~700 °C, ±2 °C when the temperature is >700 °C~1600 °C, and ±3 °C when the temperature is >1600 °C~2000 °C.

6.1.2 Differential scanning calorimeter: the maximum heating rate shall be not less than 10 °C/min, with sufficient accuracy and adjustment ability, so that the melting peak generated by the energy difference input between the sample and the reference material when the sample is being melted can be automatically recorded within the 10%~90% indexing range of the recorder. Reading accuracy of the temperature curve: the deviation shall not exceed ±0.5 °C when the temperature is lower than 300 °C, ±1 °C when the temperature is 300 °C~700 °C, ±2 °C when the temperature is >700 °C~1600 °C.

6.2 Sample vessel Made of aluminum or other high thermal conductivity metals, borosilicate glass, fused alumina, fused silica and other materials, it (or the crucible) shall not occur any phase changes within the test temperature range and not react with the reference materials or the samples.

6.3 Pressure Conditions If the test needs to be carried out under pressure conditions, then: - the test pressure of the compressed air source shall be maintained at

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0.1 MPa~

1.27 MPa; - the pressure regulating converter shall be able to measure and adjust the test pressure to the specified value, and the error shall be less than 5%.

7 Samples

7.1 For liquid or slurry samples, take samples after mixing; for sheet metal samples, take samples after shredding.

7.2 On the premise of not affecting the sample, vibration filling, molding, extrusion and other methods can be used to improve the heat conduction between the sample and the sample vessel.

7.3 Unless otherwise stated, the samples will be measured as received without any pretreatment.

7.4 If it is required to perform mechanical processing or heat treatment on the sample before analysis, the corresponding processing method, process and conditions shall be indicated in the report; For heat treatment, the mass loss before and after treatment shall also be recorded.

7.5 Samples shall not be diluted. If the sensitivity of the instrument and the stability of the baseline allow, it is recommended to use a smaller amount of sample for the test. The recommended sample volume should be 2 mg~20 mg, or less than 1/3 of the volume of the sample vessel. If the sample has the potential to suddenly release a large amount of latent heat, the amount of sample shall be appropriately reduced.

8 Calibration

8.1 In the calibrated temperature range, select the calibration material in Appendix A to calibrate the temperature indication value of the instrument.

8.2 The instrument sensitivity, heating rate, ambient atmosphere, gas flow rate and other conditions selected for calibration shall be consistent with the corresponding conditions during the test.

8.3 Under the fixed-point temperature of a certain calibration material, the correction value of the instrument temperature indication is determined by the difference between the corresponding melting temperature in Appendix A and the extrapolated initial point temperature of the melting peak of the calibration curve.

8.4 The instrument calibration cycle shall be carried out according to the requirements of the selected equipment.

9 Test steps

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9.1 Turn on the power supply of the thermal analysis instrument, and the preheating time of the instrument shall be not less than 20 min.

9.2 Use an analytical balance to weigh the sample with a mass of 2 mg~20 mg, and place it in the corresponding sample vessel; the weight shall be accurate to

0.01 mg.

9.3 According to the situation of the sample, select an appropriate reference material. In order to ensure the thermal neutrality of the reference material, the reference material used for the first time shall be carried out a calcination treatment at a temperature no lower than 1000 °C for 2 h.

9.4 Place the crucibles containing the sample and the reference material respectively in the corresponding positions on the clean support, and record the filling of the sample in detail.

9.5 During the whole testing process, the ambient atmosphere shall be consistent. If it is an atmospheric environment, it shall be in an isobaric spontaneous convection state; if using an inert protective atmosphere, after flushing the sample chamber, the flow rate of the supplied gas shall not be greater than 50 mL/min (under 1 atmosphere). For higher precision measurements, the humidity of the atmosphere shall be controlled. If it is in the atmosphere, magnesium perchlorate and other substances shall be used for drying; if it is in other atmospheres, a similar drying treatment shall also be done.

9.6 Select the appropriate instrument sensitivity, so that the corresponding melting peak generated during the melting process of the sample can be recorded in the 10%~90% indexing range of the recorder.

9.7 Using the rapid heating method, first heat the sample to a temperature of about 50°C lower than the initial melting temperature, and keep it for 5 minutes; then, heat the sample at the rate of 10°C/min until the sample is completely melted. For unknown samples, a heating rate of 10 °C/min is recommended. If the melting temperature range of the sample is less than 300 °C, the heating rate shall be reduced, and the recommended heating rate shall be not more than 5 °C/min. If other heating rates are used, it shall be noted in the report.

9.8 Each sample shall be tested at least twice; take the average value as the result.

9.9 In order to avoid the influence of supercooling on the measurement results, under normal circumstances, it is specified that the test shall be only carried out under heating conditions.

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