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Metallic powders — Determination of flow rate by means of a calibrated funnel (Gustavsson flowmeter)

Poudres métalliques — Détermination du temps d'écoulement au moyen d'un entonnoir calibré (cône d'écoulement de Gustavsson)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 2, *Sampling and testing methods for powders (including powders for hardmetals)*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/SS M11, *Powder metallurgy*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 13517:2013), which has been technically revised.

The main changes compared to the previous edition are as follows:

— tolerance for the funnel angle has been added;

— reference grit has been used instead of Chinese emery grit;

— the mandatory Clauses 2 and 3 (Normative references and Terms and definitions) have been added and the subsequent clauses have been renumbered.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Metallic powders — Determination of flow rate by means of a calibrated funnel (Gustavsson flowmeter)

1 Scope

This document specifies a method for determining the flow rate of metallic powders, including powders for hardmetals and mixes of metallic powders and organic additives such as lubricants, by means of a calibrated funnel (Gustavsson flowmeter).

The method is applicable only to powders which flow freely through the specified test orifice.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Measurement of the time required for 50 g of a metallic powder to flow through the orifice of a calibrated funnel of standardized dimensions.

5 Apparatus

5.1 Calibrated funnel, with the dimensions shown in Figure 1 (see Clause 6). The dimensions shown for the flowmeter funnel, including the orifice, are not to be considered controlling factors. Calibration with reference grit, as specified in Clause 6, determines the working flow rate of the funnel.

The funnel shall be made of a non-magnetic, corrosion-resistant metallic material with sufficient wall thickness and hardness to withstand distortion and excessive wear.

5.2 Stand and horizontal vibration-free base, to support the funnel rigidly, e.g. as indicated in Figure 2.

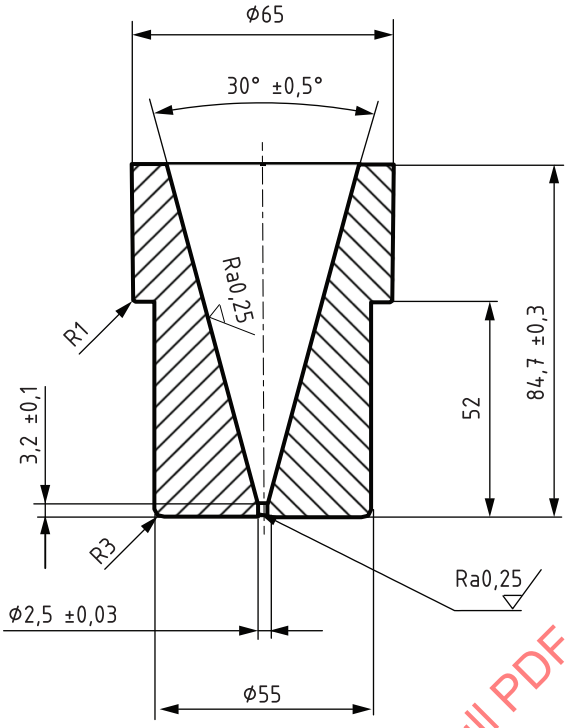
5.3 Balance, of sufficient capacity, capable of weighing the test portion to an accuracy of ± 0,05 g.

5.4 Timing device, capable of measuring the elapsed time to an accuracy of ± 0,1 s.

5.5 Reference grit, a reference powder used for calibration of the funnel[1)].

1) Material complying with 5.5 can be purchased as "Chinese emery grit". This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the company named above. Equivalent products may be used if they can be shown to lead to the same results.

Dimensions in millimetres



NOTE For dimensions with no individual tolerance, refer to ISO 2768-1 and tolerance class medium. Applicable only for new funnels.

Figure 1 — Calibrated funnel (Gustavsson flowmeter)

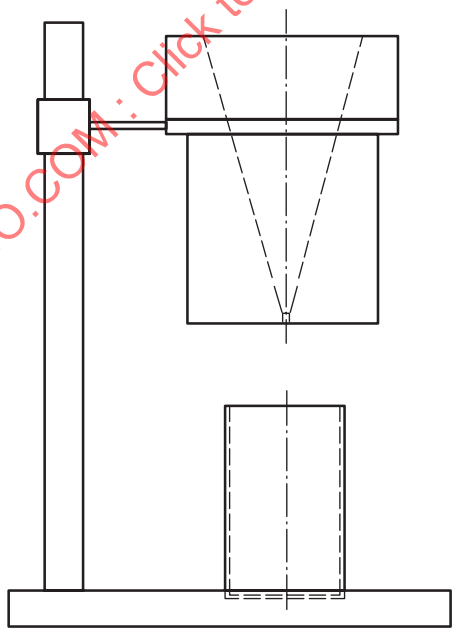


Figure 2 — Arrangement of calibrated funnel and stand

6 Calibration of the funnel

6.1 Calibration by the manufacturer of the funnel

The manufacturer shall supply the flowmeter calibrated as follows.

a) Dry the reference grit (5.5) in an open and clean glass jar at 110 °C for 60 min in air.

b) Cool the reference grit to room temperature in a desiccator.

c) Weigh out 50,0 g ± 0,1 g of the grit.

d) Follow the procedure outlined in Clause 8.

e) Repeat the procedure with the same 50 g mass of the grit, until there are five determinations within 0,4 s;

f) The average of these five determinations is stamped on the bottom of the funnel and shall be within 40,0 s ± 0,5 s.

6.2 Calibration by the user of the funnel

The flow rate of the reference sample shall be determined by the above method. If the flow rate has changed to be outside 40,0 s/50 g ± 0,5 s/50 g, a correction factor shall be used when measuring different powders. This correction factor is obtained by dividing 40,0 by this new value for the Reference grit.

It is recommended that the users periodically verify whether a correction is needed or not.

It is recommended that, before a correction factor is adopted, the cause of the change be investigated. If the flow rate has decreased, it is probable that repeated use has burnished the orifice and a (new) correction factor is justified. An increase in flow rate may indicate a coating of soft powder on the orifice. This coating should be carefully removed and the calibration test repeated.

It is recommended that the use of a funnel be discontinued after the duration of flow of the reference sample has decreased to less than 37 s.

7 Sampling

7.1 The mass of the test sample shall be at least 200 g.

7.2 In general, the powder shall be tested in the as-received condition. In certain cases, and after agreement between the supplier and user, the powder may be dried. However, if the powder is susceptible to oxidation, the drying shall take place in a vacuum or in inert gas. If the powder contains volatile substances, it shall not be dried.

7.3 Immediately before the test, weigh out a 50,0 g ± 0,1 g test portion.

7.4 Alternatively, a test portion of 90 g to 110 g can be sampled and weighed to a precision of ± 0,1 g or better.

NOTE The intention of the alternative execution is to facilitate full automation of measurement of flow and apparent density of powders.

7.5 The determination shall be carried out on three test portions.

8 Procedure

Transfer the test portion to the funnel, keeping the discharge orifice closed by a dry finger. Take care that the stem of the funnel is filled with powder. Start the timing device (5.4) when the orifice is opened and stop it at the instant when the last of the powder leaves the orifice. Record the elapsed time measured to the nearest 0,1 s.

Alternatively, the orifice can be kept open, when the test portion is transferred to the funnel with the rest of the procedure being the same.

If the powder does not begin to flow when the orifice is opened, one slight tap on the funnel to start the flow is permitted. If this has no effect, or if the flow stops during the test, the powder is considered to possess no flowability according to the test method described in this document.

9 Expression of results

Calculate the arithmetic mean of the results of the three determinations and report the value in seconds per 50 g, rounded to the nearest second.

If the alternative size of the test portion according to 7.4 is applied, the result of each determination shall, before the calculation of the arithmetic mean of the results, be divided by the mass of the sample and then be multiplied by 50 g. The result is thus recalculated in seconds per 50 g.

If a correction factor (see 6.2) should be used, the average shall be multiplied by this correction factor.

10 Precision

Two plain iron powders and four iron or bronze powder mixes were included in the inter-laboratory study to develop this precision statement. Compositions of the mixes are presented in Table 1.

Table 1 — Mix compositions of powder included in the inter-laboratory study

Designation	Mix composition
Plain iron powder 1	Plain atomized iron powder
Plain iron powder 2	Plain sponge iron powder
Bronze powder mix	Bronze powder + 0,375 % Stearic acid + 0,375 % Zinc stearate
Iron powder mix 1	Atomized iron powder + 0,8 % Graphite + 0,8 % Amide wax
Iron powder mix 2	Atomized iron powder + 2 % Ni powder + 0,8 % Graphite + 0,8 % Amide wax
Iron powder mix 3	Atomized iron powder + 0,8 % Graphite + 0,8 % Zinc stearate

In Table 2, the repeatability and reproducibility are presented as one standard deviation.

Table 2 — Repeatability and reproducibility as standard deviations

Tested powder	Level (average flow time)	Repeatability standard deviation s_r	Reproducibility standard deviation s_R
Plain iron powder 1	25 s	0,3 s	0,6 s
Plain iron powder 2	32 s	0,5 s	0,7 s
Bronze powder mix	45 s	2,6 s	3,2 s
Iron powder mix 1	48 s	1,1 s	2,2 s
Iron powder mix 2	56 s	1,0 s	2,0 s
Iron powder mix 3	60 s	0,8 s	4,7 s

The difference between two test results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit (r), see Table 3, on average not more than once in 20 cases in the normal and correct operation of the method.

Test results on identical test material reported by two laboratories will differ by more than the reproducibility limit (R), see Table 3, on average not more than once in 20 cases in the normal and correct operation of the method.

Table 3 — Repeatability and reproducibility, difference between two tests at 95 % probability level

Tested powder	Level (average flow time)	Repeatability limit r	Reproducibility limit R
Plain iron powder 1	25 s	0,9 s	1,8 s
Plain iron powder 2	32 s	1,3 s	2,0 s
Bronze powder mix	45 s	7,3 s	9,0 s
Iron powder mix 1	48 s	3,0 s	6,2 s
Iron powder mix 2	56 s	2,7 s	5,7 s
Iron powder mix 3	60 s	2,2 s	13,1 s

The accuracy data were determined from an experiment organized and analysed in accordance with ISO 5725-2 in 2011, involving 17 laboratories and 6 levels. Data from none of the laboratories contained outliers.

11 Test report

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 13517:2020;

b) all details for identification of the test sample;

c) the result obtained expressed in s/50 g;

d) the use of an open orifice;

e) all operations not specified by this document, or regarded as optional (e.g. the drying procedure applied and whether flow has been induced by tapping the funnel);

f) details of any occurrence which may have affected the result.