INTERNATIONAL STANDARD

ISO 4496

Second edition
2017-07

Metallic powders — Determination of acid-insoluble content in iron, copper, tin and bronze powders

Poudres métalliques — Détermination de la teneur en insolubles dans les acides pour les poudres de fer, de cuivre, d'étain et de bronze

Reference number
ISO 4496:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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 on 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 the following URL: 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)*.

This second edition cancels and replaces the first edition (ISO 4496:1978), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— "ashless grade (less than 0,01 % residual ash [1])" has been added in 6.3;

— general formatting of the structure.

Metallic powders — Determination of acid-insoluble content in iron, copper, tin and bronze powders

1 Scope

This document specifies methods for determining, in iron, copper, tin and bronze powders, the approximate content of non-metallic materials which are insoluble in the ordinary mineral acids.

The insoluble matter referred to is generally considered to be acid-insoluble silica and silicates, carbides, alumina, clays or other refractory oxides which are either present in the raw material from which the powders are manufactured or introduced during the manufacturing process.

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:

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

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

4 Field of application

The methods are applicable to lubricant-free metallic powders of iron, copper, tin, alloy bronze and elemental mixtures of copper and tin.

5 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity. See Table 1 for the required reagents.

Table 1 — Required reagents

Type of powder	Reagent	Density, ρ g/ml	Concentration of solution
Iron	Hydrochloric acid (5.1)	1,19	1 + 1
	Hydrochloric acid (5.2)	1,19	1 + 25
	Potassium thiocyanate (5.3)	—	5 %
	Nitric acid (5.4)	1,42	concentrated
Copper Tin Bronze	Hydrochloric acid (5.5)	1,19	concentrated
	Nitric acid (5.6)	1,42	1 + 1
	Hydrogen peroxide (5.7)	—	30 %
	Ammonium acetate (5.8)	—	200 g/l
Copper Bronze	Sodium diethyldithiocarbamate (5.9)	—	4 %
Tin	Sodium sulfide (5.10) Hydrogen sulfide (5.11)	—	—

6 Equipment

Ordinary laboratory equipment and the following.

6.1 Laboratory balance, of sufficient capacity and capable of weighing to an accuracy of ± 0,000 1 g.

6.2 Glass filter funnel, diameter approximately 70 mm.

6.3 Filter paper, ashless grade (less than 0,01 % residual ash [1]) for medium precipitates, diameter approximately 110 mm.

6.4 Furnace, capable of operating between 900 °C and 1 000 °C.

6.5 Fused silica or porcelain crucibles, pre-treated to constant mass at 900 °C to 1 000 °C and stored in a desiccator.

7 Sampling

7.1 Number of test portions

Determine the content of insoluble matter on two test portions.

7.2 Mass of test portion

The mass of the test portion shall be approximately 5 g.

8 Procedure

8.1 Iron powder

8.1.1 Weigh, to the nearest 0,000 1 g, a test portion of approximately 5 g of the test sample (mass m_1) and transfer it to a glass beaker.

8.1.2 Carefully add 100 ml of hydrochloric acid (5.1) and cover the beaker with a watch glass. Allow the solution to stand at room temperature until the reaction is completed (no further evolution of hydrogen).

If it is desired to exclude carbides as a part of the insoluble matter, add 20 ml of nitric acid (5.4) to the hydrochloric acid (5.1) used according to 8.1.2. Then proceed according to 8.1.3 to 8.1.6.

8.1.3 Place the glass beaker on a hotplate and heat the solution to boiling. Maintain boiling for about 1 min. Add 150 ml of water, reheat to boiling and maintain for about 1 min. Allow the solution to cool and settle for 5 min.

8.1.4 Filter the solution through the medium filter paper and wash the residue alternately with hot water and hot hydrochloric acid (5.2). Repeat the washing until iron salts cannot be detected in the washing water, for example, by means of potassium thiocyanate (5.3).

8.1.5 Weigh a crucible to the nearest 0,000 1 g (mass m_2) and place in it the filter paper with the residue. Place the crucible on a hotplate to dry and char the paper. Heat in the furnace at a temperature between 900 °C and 1 000 °C until the difference between two consecutive weighings of the crucible with the residue after cooling is not greater than 0,000 1 g. Allow the crucible to cool completely in a desiccator.

8.1.6 Determine the mass of the crucible with the residue to the nearest 0,000 1 g (mass m_3).

8.2 Copper, tin and bronze powders

8.2.1 Weigh, to the nearest 0,000 1 g, a test portion of approximately 5 g of the test sample (mass m_1) and transfer it to a glass beaker.

8.2.2 Carefully add 50 ml of hydrochloric acid (5.5), cover with a watch glass, place on the edge of a hotplate and digest at low temperature for a minimum of 30 min.

8.2.3 Remove the beaker, cool slightly, cautiously add 50 ml of nitric acid (5.6) and wait for the initial reaction, which starts after about 10 min. After the reaction is completed, add a further 50 ml of nitric acid (5.6).

8.2.4 Place the beaker on a hotplate and heat the solution to boiling. Maintain boiling until the volume is reduced to one-half.

If the residue is black, remove the beaker from the hotplate, cautiously add a few millilitres of hydrogen peroxide (5.7), and boil for 2 min. Repeat the treatment with hydrogen peroxide (5.7) until no black residue is left.

8.2.5 Slowly add 50 ml of hot water and reheat to boiling. Maintain boiling for about 1 min. Allow the solution to cool and settle for 5 min.

8.2.6 Filter the solution through a medium filter paper and wash the residue first with hot hydrochloric acid (5.5) and finally with hot water. Repeat the washing with water until

— in the case of copper and bronze powders, copper salts in the washing water cannot be detected, for example, by sodium diethyldithiocarbamate (5.9), or

— in the case of tin powder, tin salts in the washing water cannot be detected, for example, by sodium sulfide (5.10) or hydrogen sulfide (5.11).

If the presence of lead sulfate is suspected, wash once or twice with a hot solution of ammonium acetate (5.8) and then with water.

8.2.7 Weigh a crucible to the nearest 0,000 1 g (mass m_2) and place in it the filter paper with the residue. Place the crucible on a hotplate to dry and char the paper. Heat in the furnace at a temperature between 900 °C and 1 000 °C until the difference between two consecutive weighings of the crucible with the residue after cooling is not greater than 0,000 1 g. Allow the crucible to cool completely in a desiccator.

8.2.8 Determine the mass of the crucible with the residue to the nearest 0,000 1 g (mass m_3).

9 Expression of results

9.1 Calculation of acid-insoluble content

The acid-insoluble content (AIC), expressed as a percentage by mass, is given by Formula (1):

$$AIC = \frac{m_3 - m_2}{m_1} \times 100 \quad (1)$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the dry, pre-treated empty crucible;

m_3 is the mass, in grams, of the crucible with the residue.

9.2 Precision

Calculate the result of each determination to the nearest 0,01 %.

9.3 Permissible difference

The maximum permissible difference between the two determinations shall not exceed 10 % of the mean value or 0,02 % in absolute value, whichever is greater.

9.4 Mean value

Report the arithmetical mean of the two determinations rounded to the nearest 0,02 % for contents up to and including 0,25 % and to the nearest 0,05 % for contents greater than 0,25 %.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 4496;

b) all details necessary for identification of the test sample;

c) the result obtained;

d) all operations not specified by this document, or regarded as optional;

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