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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

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FOREWORD

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It has been approved by the member bodies of the following countries :

Australia	Germany	Sweden
Austria	Italy	Turkey
Bulgaria	Mexico	United Kingdom
Canada	Poland	U.S.A.
Chile	Portugal	U.S.S.R.
Czechoslovakia	Romania	Yugoslavia
Egypt, Arab Rep. of	South Africa, Rep. of	
France	Spain	

No member body expressed disapproval of the document.

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

1 SCOPE

This International Standard 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 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.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

See the table for the reagents required.

4 APPARATUS

Ordinary laboratory apparatus and

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

4.2 Glass filter funnel, diameter approximately 70 mm.

4.3 Filter paper for medium precipitates, diameter approximately 110 mm.

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

4.5 Fused silica or **porcelain crucibles,** pretreated to constant mass at 900 to 1 000 °C and stored in a desiccator.

5 SAMPLING

5.1 Determine the content of insoluble matter on two test portions.

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

Type of powder	Reagent	Density, ρ g/ml	Concentration of solution
Iron	Hydrochloric acid (3.1)	1,19	1 + 1
	Hydrochloric acid (3.2)	1,19	1 + 25
	Potassium thiocyanate (3.3)		5 %
	Nitric acid (3.4)	1,42	concentrated
Copper Tin Bronze	Hydrochloric acid (3.5)	1,19	concentrated
	Nitric acid (3.6)	1,42	1 + 1
	Hydrogen peroxide (3.7)		30 %
	Ammonium acetate (3.8)		200 g/l
Copper Bronze	Sodium diethyldithiocarbamate (3.9)		4 %
Tin	Sodium sulphide (3.10)		
	Hydrogen sulphide (3.11)		

6 PROCEDURE

6.1 Iron powder

6.1.1 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.

6.1.2 Carefully add 100 ml of hydrochloric acid (3.1) 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).

NOTE – If it is desired to exclude carbides as a part of the insoluble matter, add 20 ml of nitric acid (3.4) to the hydrochloric acid (3.1) used according to 6.1.2. Then proceed according to 6.1.3 to 6.1.6.

6.1.3 Place the glass beaker on a hot-plate and heat the solution to boiling. Maintain boiling for about 1 min. Then add 150 ml of water, reheat to boiling and maintain for about 1 min. Allow the solution to cool and settle for 5 min.

6.1.4 Filter the solution through the medium filter paper and wash the residue alternately with hot water and hot hydrochloric acid (3.2). Repeat the washing until iron salts cannot be detected in the washing water, for example by means of potassium thiocyanate (3.3).

6.1.5 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 hot-plate to dry and char the paper. Heat in the furnace at a temperature between 900 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.

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

6.2 Tin, copper and bronze powders

6.2.1 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.

6.2.2 Carefully add 50 ml of hydrochloric acid (3.5), cover with a watch glass, place on the edge of a hot-plate and digest at low temperature for a minimum of 30 min.

6.2.3 Remove the beaker, cool slightly, cautiously add 50 ml of nitric acid (3.6) 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 (3.6).

6.2.4 Place the beaker on a hot-plate and heat the solution to boiling. Maintain boiling until the volume is reduced to one-half.

NOTE – If the residue is black, remove the beaker from the hot-plate, cautiously add a few millilitres of hydrogen peroxide (3.7), and boil for 2 min. Repeat the treatment with hydrogen peroxide (3.7) until no black residue is left.

6.2.5 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.

6.2.6 Filter the solution through a medium filter paper and wash the residue first with hot hydrochloric acid (3.5) 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 (3.9);

– in the case of tin powder, tin salts in the washing water cannot be detected, for example by sodium sulphide (3.10) or hydrogen sulphide (3.11).

NOTE – If the presence of lead sulphate is suspected, wash once or twice with a hot solution of ammonium acetate (3.8) and then with water.

6.2.7 Weigh a crucible to the nearest 0,000 1 g (mass m_2) and in it place the filter paper with the residue. Place the crucible on a hot-plate to dry and char the paper. Heat in the furnace at a temperature between 900 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.

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

7 EXPRESSION OF RESULTS

7.1 The acid-insoluble content, AIC, expressed as a percentage by mass, is given by the formula

$$AIC = \frac{m_3 - m_2}{m_1} \times 100$$

where

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

m_2 is the mass, in grams, of the dry, pretreated empty crucible;

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

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

7.3 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.

7.4 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 %.

8 TEST REPORT

The test report shall include the following information :

a) reference to this International Standard;

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

c) the result obtained;

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

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

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