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**Metallic Powders - Determination of Acid-insoluble Content
in Iron, Copper, Tin and Bronze Powders**

金属粉末 铁、铜、锡和青铜粉末中酸不溶物含量的测定

(ISO 4496:2017, IDT)

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Metallic Powders - Determination of Acid-insoluble Content in Iron, Copper, Tin and Bronze Powders

1 Scope

This document describes a method for the determination of the content of non-metallic materials insoluble in common mineral acids in metallic powders of iron, copper, tin and bronze.

This document applies to the determination of acid-insoluble silica and silicates, carbides, alumina, clays or other poorly soluble oxides introduced in raw materials or during the production process.

2 Normative References

This document does not have normative references.

3 Terms and Definitions

This document does not have terms or definitions that need to be defined.

4 Field of Application

This Method can be applied to iron powder, copper powder, tin powder, bronze alloy powder and copper-tin mixed powder without added lubricant.

5 Reagents

During the analysis, only analytically pure reagents, distilled water or water of equivalent purity are used. The required reagents are shown in Table 1.

min. After the solution cools down, let it stand for 5 min.

8.1.4 Use medium-speed filter paper to filter the solution and use hot water or hot hydrochloric acid (5.2) to clean the residue. Repeat cleaning, until iron salts cannot be detected in the water. Potassium thiocyanide (5.3) can be selected as the detection reagent.

8.1.5 Weigh-take the crucible (the mass is m_2), accurate to 0.0001 g. Place the filter paper with residue in the crucible, place the crucible on the electric stove to dry and carbonize the filter paper. In a muffle furnace with a temperature of 900 °C ~ 1,000 °C, burn the crucible, until the difference in continuous weighing does not exceed 0.0001 g after the crucible containing the residue cools down. Completely cool the crucible in a desiccator.

8.1.6 Weigh the mass of the crucible containing the residue (the mass is m_3), accurate to 0.0001 g.

8.2 Copper, Tin and Bronze Powders

8.2.1 Weigh-take 5 g (accurate to 0.0001 g) of sample as the test portion (the mass is m_1). Place the test portion in a glass beaker.

8.2.2 Add 50 mL of hydrochloric acid (5.5) and use a watch glass to cover the glass beaker. Place the glass beaker on the edge of the electric stove, at a low temperature, decompose it for at least 30 min.

8.2.3 Remove the glass beaker, after it slightly cools down, add 50 mL of nitric acid (5.6) and wait for the initial reaction. The initial reaction usually occurs 10 min after adding nitric acid. After the reaction is completed, add another 50 mL of nitric acid (5.6).

8.2.4 Place the glass beaker on the electric stove, heat the solution to boiling, and maintain the boiling, until the volume of the solution is reduced by half. If the residue is black, remove the glass beaker from the electric stove, add a few milliliters of hydrogen peroxide (5.7) and maintain the boiling for 2 min. Repeatedly add hydrogen peroxide (5.7), until there is no black residue.

8.2.5 Slowly add 50 mL of hot water, then, heat it to boiling. Maintain the boiling for 1 min, after the solution cools down, let it stand for 5 min.

8.2.6 Use medium-speed filter paper to filter the solution. Firstly, use hot hydrochloric acid (5.5) to clean the residue, and finally, use hot water to wash it. Repeatedly use hot water to clean it, until:

---For copper powder and bronze powder, copper salts cannot be detected in the water using sodium diethyldithiocarbamate (5.9), or

---For tin powder, tin salts cannot be detected using sodium sulfide (5.10) or hydrogen sulfide (5.11).

If it is possible that lead sulfate is present, firstly, use hot ammonium acetate (5.8) to clean it

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