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ISO RECOMMENDATION

R 1812

CHEMICAL ANALYSIS OF COPPER AND COPPER ALLOYS

SPECTROPHOTOMETRIC DETERMINATION OF IRON IN COPPER ALLOYS

1st EDITION

August 1971

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BRIEF HISTORY

The ISO Recommendation R 1812, *Chemical analysis of copper and copper alloys – Spectrophotometric determination of iron in copper alloys*, was drawn up by Technical Committee ISO/TC 26, *Copper and copper alloys*, the Secretariat of which is held by the Deutscher Normenausschuss (DNA).

Work on this question led to the adoption of Draft ISO Recommendation No. 1812, which was circulated to all the ISO Member Bodies for enquiry in March 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Poland
Belgium	India	South Africa, Rep. of
Brazil	Iran	Spain
Canada	Israel	Sweden
Chile	Italy	Switzerland
Czechoslovakia	Japan	Turkey
Finland	Netherlands	U.A.R.
France	New Zealand	United Kingdom
Germany	Norway	U.S.A.
Greece	Peru	Yugoslavia

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

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ISO Recommendation | R 1812 | August 1971

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1. SCOPE

This ISO Recommendation describes a spectrophotometric method for the determination of iron in copper alloys.

The method is applicable to the determination of iron contents up to 0.4 % in any of the copper alloys listed in ISO Recommendations.

2. PRINCIPLE

Extraction of the iron as the iron (III)-chloro-complex with methylisobutyl ketone, and spectrophotometric determination as the iron (II)-*o*-phenanthroline complex.

3. REAGENTS

All the reagents should be of the analytical grade. Use distilled or deionized water. For the determination of iron contents lower than 0.01 % use double distilled water.

3.1 *Hydrochloric acid* 7 + 3.

Mix 70 ml of hydrochloric acid (d = 1.19) with 30 ml of water.

3.2 *Hydrogen peroxide*, 30 %.

3.3 *Hydrochloric acid* 1 + 1.

Mix 50 ml of hydrochloric acid (d = 1.19) with 50 ml of water.

3.4 *Methylisobutyl ketone.*

3.5 *Ascorbic acid* solution.

Dissolve 5 g of ascorbic acid in water and dilute to 500 ml (this solution is stable for 3 to 4 days).

3.6 *Buffered o-phenanthroline solution*.

Mix 1 g of *o*-phenanthroline hydrochloride with 215 ml of glacial acetic acid in a 500 ml volumetric flask and add, while cooling, 265 ml of ammonia solution (d = 0.91). This mixture should have a pH of 6.5 ± 0.1. Adjust, if necessary, by adding either ammonia solution or acetic acid, then dilute to 500 ml. This solution is stable.

3.7 *Iron* stock solution, (1 ml ≙ 0.1 mg of Fe).

Dissolve 0.1 ± 0.01 g of high purity iron in 20 ml of HCl (d = 1.19) and dilute to 1 litre.

3.8 *Iron* standard solution, (1 ml ≙ 10 µg of Fe).

Dilute 50 ml of iron stock solution (3.7) to 500 ml.

4. APPARATUS

4.1 Ordinary laboratory apparatus.

NOTE. – All glassware should be rinsed with hot hydrochloric acid (3.3) until the surface is free of iron.

4.2 *Spectrophotometer.*

5. SAMPLING

Follow the procedure given in ISO Recommendation R . . .*.

6. PROCEDURE

6.1 Test portion

$m_0 = 5 \pm 0.001$ g of the test sample.

6.2 Blank test

Carry out a blank test using the same procedure and quantities of all reagents employed in the test.

6.3 Determination

6.3.1 Dissolve the test portion in 40 ml of hydrochloric acid (3.1) and 40 ml of hydrogen peroxide (3.2) added in small portions. Cool until the violent reaction has ceased. When the test portion is completely dissolved, heat to boiling and continue boiling for approximately 2 minutes** to remove excess hydrogen peroxide and cool.

(*a*) With iron contents less than 0.004 %, transfer the solution to a 250 ml separating funnel and wash the beaker with hydrochloric acid (3.3).

(*b*) With iron contents between 0.003 and 0.04 %, dilute the solution to 250 ml with hydrochloric acid (3.3) and transfer 25.0 ml to the separating funnel.

(*c*) With iron contents between 0.03 and 0.4 %, dilute the solution to 500 ml with water, transfer 5.0 ml to the separating funnel and add 20 ml of hydrochloric acid (3.3). Allow to stand until any turbidity disappears.

6.3.2 Add 20 ml of methylisobutyl ketone (3.4) to the separating funnel and shake for 15 seconds. Allow the phases to separate, discard the aqueous phase and wash the organic phase three times with 20 ml of hydrochloric acid (3.3) until free of copper. If it should happen that the phases will separate only with difficulty, then the separation can be accelerated by adding about 2 ml of gasoline (boiling point range approximately 40 to 100 °C) to the emulsified phase mixture without any further shaking. Re-extract the iron from the organic phase by shaking vigorously for 20 seconds, with two successive 10 ml portions of ascorbic acid solution (3.5).

6.3.3 Transfer the aqueous extracts to a 50 ml volumetric flask and mix with 5.0 ml of buffered *o*-phenanthroline solution (3.6). Dilute to the mark and within 30 minutes take the spectrophotometric reading against water at the wavelength of maximum absorption (usually 510 nm, but variations may occur).

6.4 Preparation of calibration curve

By means of a pipette transfer amounts of iron standard solution (3.8), from 0 to 20 ml corresponding to 0 to 200 µg of Fe, to a series of 50 ml volumetric flasks. To each add 20 ml of ascorbic acid solution (3.5) and mix. Allow to stand for 1 minute, then add 5 ml of buffered *o*-phenanthroline solution (3.6). Dilute to the mark and take the spectrophotometric readings against water as described in clause 6.3.3 using 2 cm cells, and prepare a calibration curve.

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* Under study.

** For dissolving silicon-containing alloys, especially silicon rich, such as copper-silicon alloy, add 50 drops of hydrofluoric acid (40 %); in this case use a beaker made of teflon or similar material, or a platinum dish. If dissolution is complete, the solution can be transferred to glassware and all the following steps can be handled in this.

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