INTERNATIONAL STANDARD 1054

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Zinc — Determination of cadmium content — Polarographic method

Zincs de fonderie — Dosage du cadmium — Méthode polarographique

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 18 has reviewed ISO Recommendation R 1054 and found it technically suitable for transformation. International Standard ISO 1054 therefore replaces ISO Recommendation R 1054-1969 to which it is technically identical.

ISO Recommendation R 1054 was approved by the Member Bodies of the following countries :

Australia	Germany	Poland
Belgium	Greece	South Africa, Rep. of
Brazil	India	Spain
Bulgaria	Iran	Sweden
Canada	Israel	Turkey
Chile	Italy	United Kingdom
Czechoslovakia	Korea, Dem. P. Rep. of	U.S.A.
Egypt, Arab Rep. of	Korea, Rep. of	U.S.S.R.
France	Norway	Yugoslavia

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

Japan

The Member Body of the following country disapproved the transformation of ISO/R 1054 into an International Standard :

Spain

Zinc — Determination of cadmium content — Polarographic method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a polarographic method for the determination of the cadmium content of zinc.

The method is applicable to the following types of zinc : Zn 99,95, Zn 99,5, Zn 98,5 and Zn 98, defined in ISO/R 752, with cadmium contents between 0,01 and 0,5 %.

This method is not applicable when the copper content is more than four times higher than the cadmium content.

2 REFERENCES

ISO/R 752, *Zinc ingots.*

ISO 3751, *Zinc ingots — Selection and preparation of samples for chemical analysis.* [1)]

3 PRINCIPLE

Polarographic determination of the cadmium content in ammoniacal solution.

4 REAGENTS

During the analysis, use only reagents of analytical reagent grade and distilled or demineralized water.

4.1 Sodium sulphite, anhydrous.

4.2 Granulated zinc at least 99,99 % pure.

4.3 Hydrochloric acid, ρ 1,19 g/ml.

4.4 Nitric acid, ρ 1,3 to 1,4 g/ml.

4.5 Ammonia solution, ρ 0,91 g/ml.

4.6 Pure gelatin, 5 g/l solution.

Add 1 g of salicylic acid per litre of solution as preservative.

4.7 Cadmium standard solution No. 1.

Dissolve 1,250 g of pure cadmium in a minimum amount of nitric acid (4.4). Make up the volume to 500 ml with water.

1 ml of this solution contains 2,5 mg of cadmium.

4.8 Cadmium standard solution No. 2.

Dilute the standard cadmium solution No. 1 (4.7) five times.

1 ml of this solution contains 0,5 mg of cadmium.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Polarograph.

5.2 Thermostatically controlled water bath.

6 SAMPLING

Sampling shall be carried out in accordance with the requirements of ISO 3751.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,01 g, 5 g of the test sample.

7.2 Plotting of the calibration curve

Assuming that a calibration curve is to be established by six steps corresponding to cadmium contents of 0 — 0,02 — 0,05 — 0,1 — 0,25 and 0,50 % then

7.2.1 Weigh six 5 g samples of pure zinc (4.2) to the nearest 0,01 g.

7.2.2 Continue as outlined in 7.3.1 to 7.3.3.

1) At present at the stage of draft.

7.2.3 Add respectively 0, 2, 5 and 10 ml of standard cadmium solution No. 2 (4.8) and also 5 and 10 ml of standard cadmium solution No. 1 (4.7).

7.2.4 For each flask, proceed as outlined in 7.3.4 to 7.3.6.

7.3 Determination

7.3.1 Transfer the test portion to a 100 ml beaker covered with a watch-glass and attack with 20 ml of hydrochloric acid (4.3) and 5 ml of nitric acid (4.4), added carefully in small portions.

7.3.2 After complete dissolution, add 5 ml of water and boil for a short time. Allow to cool.

7.3.3 Transfer quantitatively to a 100 ml volumetric flask.

7.3.4 Add successively

- 40 ml of ammonia solution (4.5);
- 0,5 g of sodium sulphite (4.1);
- 2,5 ml of gelatin solution (4.6).

Allow to cool.

7.3.5 Make up the volume to 100 ml with water. Mix.

7.3.6 Allow the precipitate of hydroxide to settle sufficiently or, if necessary, filter the solution.

7.4 Polarographic measurement

Polarograph between 0 and − 1 V.

The step for cadmium occurs at about − 0,6 V relative to a mercury electrode or at − 0,85 V relative to a saturated calomel electrode.

8 EXPRESSION OF RESULTS

Read from the calibration curve the values corresponding to the step heights obtained.

9 TEST REPORT

The test report shall mention the method used and the results obtained. It shall also mention all operational details not provided for in this International Standard, or any optional details, as well as any circumstances which could have influenced the results.

The test report shall include all details required for complete identification of the sample.