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Ferromolybdenum — Determination of molybdenum content — Gravimetric method

Ferromolybdène — Dosage du molybdène — Méthode gravimétrique

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Foreword

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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.

International Standard ISO 4173 was developed by Technical Committee ISO/TC 132, *Ferroalloys*, and was circulated to the member bodies in September 1978.

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

Australia	France	Poland
Austria	Germany, F.R.	Romania
Brazil	India	South Africa, Rep. of
Bulgaria	Italy	Spain
Canada	Libyan Arab Jamahiriya	Sweden
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Czechoslovakia	Philippines	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

USSR

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Ferromolybdenum — Determination of molybdenum content — Gravimetric method

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of the molybdenum content of ferromolybdenum.

The method is applicable to molybdenum contents in the ranges normally found in ferromolybdenum.

2 Reference

ISO 3713, *Ferroalloys — Sampling and preparation of samples — General rules.*[1)]

3 Principle

Dissolution of a test portion, and separation of the iron with sodium hydroxide. Precipitation of the molybdenum as the hydroxyquinolate, in the presence of EDTA and ammonium oxalate. Filtration, drying and weighing of the anhydrous hydroxyquinolate $[MoO_2(C_9H_6NO)_2]$.

4 Reagents

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

4.1 Sulphuric acid, 50 % (*V*/*V*) solution.

To 400 ml of water, add cautiously 500 ml of sulphuric acid (ϱ 1,84 g/ml). Mix, cool, dilute to 1 000 ml and mix.

4.2 Nitric acid, 25 % (*V*/*V*) solution.

Dilute 250 ml of nitric acid (ϱ 1,42 g/ml) to 1 000 ml and mix.

4.3 Hydrofluoric acid, (ϱ 1,14 g/ml).

4.4 Hydrochloric acid, 50 % (*V*/*V*) solution.

Dilute 500 ml of hydrochloric acid (ϱ 1,16 to 1,18 g/ml) to 1 000 ml and mix.

4.5 Sodium hydroxide, 460 to 480 g/l solution.

Dissolve 460 to 480 g of sodium hydroxide in water, dilute to 1 000 ml and mix. This solution should be prepared in a polyethylene beaker (water-cooled if necessary) and stored in a polyethylene bottle.

4.6 EDTA (*di*sodium salt), 10 g/l solution.

Dissolve 10 g of ethylenediaminetetraacetic acid, *di*sodium salt, dihydrate, in water, dilute to 1 000 ml and mix.

4.7 Ammonia solution, 50 % (*V*/*V*).

Dilute 500 ml of ammonia solution (ϱ 0,91 g/ml) to 1 000 ml and mix.

4.8 8-Hydroxyquinoline, 30 g/l neutralized solution.

Dissolve 3 g of 8-hydroxyquinoline in 12 ml of glacial acetic acid, add 60 ml of water and warm to about 40 °C. Add the ammonia solution (4.7) dropwise until a slight permanent precipitate is formed; just redissolve the precipitate by addition, dropwise, of glacial acetic acid, cool and dilute to 100 ml.

4.9 Nitric acid, ϱ 1,42 g/ml.

4.10 Hydrochloric acid, ϱ 1,16 to 1,18 g/ml.

4.11 Ammonium oxalate.

1) At present at the stage of draft.

5 Apparatus

Usual laboratory equipment.

6 Sample

Use powder which will pass through a sieve with a mesh size of 160 µm, prepared in accordance with ISO 3713.

7 Procedure

7.1 Test portion

Owing to the inhomogeneity of ferromolybdenum, the 160 µm sample should be sieved through a screen of mesh opening 75 to 80 µm and proportionate parts of over- and under-size taken for analysis.

Weigh out 1 g for 75 % ferromolybdenum or 1,25 g for 50 % ferromolybdenum.

7.2 Determination

Place the test portion (7.1) in a 150 ml polytetrafluoroethylene (PTFE) beaker, add 10 ml of the sulphuric acid solution (4.1) and warm without boiling. Add, dropwise, the nitric acid (4.9) until the test portion is dissolved and then add 0,5 ml in excess. Add 5 ml of the hydrofluoric acid (4.3) and evaporate until fumes of sulphur trioxide are evolved. Continue fuming for 15 min, cool, add 5 ml of the hydrochloric acid (4.10) and 5 ml of the nitric acid (4.9), warm for 10 min and dilute to about 50 ml.

Into a tall-form beaker (of capacity 650 to 800 ml), place 50 ml of the sodium hydroxide solution (4.5) and 50 ml of water. Heat to boiling and place a funnel in the mouth of the beaker.

NOTE — A suitable funnel may be made by drawing out the stem of a 75 mm funnel to produce a jet size of diameter approximately 1 mm.

Slowly transfer the test solution, via the funnel, into the beaker, ensuring that the sodium hydroxide solution continues to boil; rinse the PTFE beaker with hot water (85 to 90 °C) and transfer the washings to the funnel. Rinse the funnel and upper surface of the tall-form beaker, boil the solution for 2 to 3 min, dilute to about 450 ml and cool.

Transfer to a 500 ml volumetric flask, dilute to the mark and mix well. Dry the original tall-form beaker, transfer into it the contents of the volumetric flask and allow to stand for 15 to 20 min.

Filter a portion of the solution through a dry, rapid-hardened filter paper and transfer a 100 ml aliquot to a 400 ml beaker. Dilute to about 200 ml, and add 10 ml of the EDTA solution (4.6) and 3 g of the ammonium oxalate (4.11). Warm gently to dissolve the ammonium oxalate, cool to room temperature and adjust to pH 4,0 (using a pH meter) by addition of the hydrochloric acid solution (4.4) and the ammonia solution (4.7).

Heat the solution to boiling and add 20 ml of the hydroxyquinoline solution (4.8), ensuring that the solution continues to boil throughout the addition.

NOTE — It is essential that the precipitation be carried out in a boiling solution. If precipitation is carried out in a solution below boiling point, the precipitate formed is more difficult to wash and initially gives high recoveries; if dried to constant mass (perhaps 2 to 3 days), low recoveries may sometimes be obtained.

Allow to stand for 5 to 10 min at 80 to 90 °C with occasional stirring and filter through a tared sintered glass crucible, of porosity 5 to 10 µm, using gentle suction. Completely remove the precipitate from the walls of the beaker using a rubber-tipped glass rod, transfer to the crucible and wash the precipitate in the crucible with about 100 ml of hot water (80 to 90 °C).

NOTE — During filtration, the solution should be added at such a rate that not more than half the crucible is filled at any one time. This procedure facilitates the efficient washing of the crucible and provides a precipitate free from soluble salts.

Dry overnight at 125 °C, cool and weigh. Re-dry to constant mass and weigh as anhydrous molybdenum hydroxyquinolate.

NOTE — The following procedures are recommended for handling and cooling sintered glass crucibles.

a) After suction washing, the outer crucible wall should be washed with hot water, dried on a filter paper and, from that stage, should not be touched with bare hands. The crucibles should preferably be handled with tweezers or for a minimum period in a gloved hand.

b) After oven drying, both the empty crucibles and those containing the hydroxyquinolate precipitate should be placed in an efficient desiccator and allowed to stand at room temperature for 1 h before weighing.

c) Before use, crucibles should be washed several times with hot water, the nitric acid solution (4.2) and finally with water and dried at 125 °C.

After use, remove the bulk of the precipitate with a jet of water, place the crucible in a small beaker containing 5 to 10 ml of the sulphuric acid solution (4.1), and fill the crucible with the nitric acid (4.9). Heat the beaker until fumes of sulphur trioxide are evolved, cool, remove the crucible from its beaker and pass about 100 ml of hot water through the sinter under gentle suction. Treat the washed crucibles as in a) and b) above.

8 Expression of results

The molybdenum content, expressed as a percentage by mass of the sample, is given by the formula

$$\frac{m_1 \times 23{,}05}{m_0}$$

where

m_1 is the mass, in grams, of the anhydrous molybdenum hydroxyquinolate;

m_0 is the mass, in grams, of the test portion in the final aliquot.

9 Reproducibility

A planned trial of this method was carried out by seven analysts each from a different laboratory. Five tests were carried out by each analyst on each of two samples. From the results obtained, the 95 % confidence limits ($2s$) given in the table have been calculated.

Table — Reproducibility according to alloy type

Alloy type	Molybdenum % (*m/m*)	Components of standard deviation		Reproducibility index $2s$
		s_b	s_w	
50 % Ferromolybdenum	50,33	0,081	0,090	0,24
75 % Ferromolybdenum	75,34	0,085	0,100	0,26

10 Test report

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.