

International Standard



4687

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**Iron ores — Determination of phosphorus content —
Molybdenum blue spectrophotometric method***Minerais de fer — Dosage du phosphore — Méthode spectrophotométrique au bleu de molybdène***First edition — 1984-06-01****UDC 553.31 : 543.42 : 546.18****Ref. No. ISO 4687-1984 (E)****Descriptors** : metalliferous minerals, iron ores, chemical analysis, determination of content, phosphorus, spectrophotometric analysis**Documents**
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (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 authorized 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.

International Standard ISO 4687 was developed by Technical Committee ISO/TC 102, *Iron ores*, and was circulated to the member bodies in June 1982.

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

Australia	Germany, F. R.	Romania
Austria	India	South Africa, Rep. of
Canada	Italy	Spain
China	Japan	Sweden
Czechoslovakia	Korea, Dem. P. Rep. of	United Kingdom
Egypt, Arab Rep. of	Poland	USA
France	Portugal	Venezuela

No member body expressed disapproval of the document.

Iron ores — Determination of phosphorus content — Molybdenum blue spectrophotometric method

1 Scope and field of application

This International Standard specifies a molybdenum blue spectrophotometric method for the determination of phosphorus in iron ores.

This method is applicable to a concentration range of 0,005 to 0,2 % (m/m) of phosphorus in natural iron ores, and iron ore concentrates and agglomerates including sinter products.

The arsenic content of the sample does not affect the determination.

2 References

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3081, *Iron ores — Increment sampling — Manual method*.

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method*.¹⁾

ISO 3083, *Iron ores — Preparation of samples — Manual method*.

ISO 7764, *Iron ores — Preparation of predried test sample for chemical analysis*.¹⁾

3 Principle

Decomposition of the test portion, either

- by sintering with sodium peroxide, followed by treatment with hydrochloric and perchloric acids (this method is recommended for lower-grade ores with a high content of amphoteric elements), or
- by treatment with hydrochloric, nitric and perchloric acids (this method is recommended for high-grade ores with a low content of gangue).

Dehydration of silica, dilution and filtration, and ignition of the residue. Removal of the silica by evaporation with hydrofluoric and sulfuric acids and fusion with sodium carbonate. Dissolution with hydrochloric acid, and combination with the main solution.

Addition of molybdate to convert the phosphate into molybdophosphate complex and reduction to molybdenum blue with ascorbic acid.

Spectrophotometric measurement of the absorbance of the molybdenum blue complex at a wavelength of approximately 725 nm.

4 Reagents

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

4.1 Sodium peroxide (Na_2O_2), powder.

Sodium peroxide should be kept away from humidity and should not be used once it has begun to agglomerate.

4.2 Sodium carbonate (Na_2CO_3), anhydrous.

4.3 Hydrochloric acid, ρ 1,16 to 1,19 g/ml.

4.4 Hydrochloric acid, ρ 1,16 to 1,19 g/ml, diluted 1 + 1.

4.5 Hydrochloric acid, ρ 1,16 to 1,19 g/ml, diluted 1 + 9.

4.6 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1.

4.7 Perchloric acid, ρ 1,54 g/ml, 60 % (m/m) solution, or ρ 1,67 g/ml, 70 % (m/m) solution.

4.8 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (m/m) solution, or ρ 1,185 g/ml, 48 % (m/m) solution.

4.9 Hydrobromic acid, ρ 1,38 g/ml, 40 % (m/m) solution, or ρ 1,48 g/ml, 47 % (m/m) solution.

4.10 Nitric acid, ρ 1,4 g/ml.

4.11 Hydrogen peroxide, 3 % (V/V), free from phosphate stabilizer.

Dilute hydrogen peroxide 30 % (m/m), (100 volumes) in the volume ratio of 1 + 9.

1) At present at the stage of draft.

4.12 Ascorbic acid ($C_6H_8O_6$), 2 g/l solution.

Prepare this solution fresh on the day of use.

4.13 Sodium disulfite (sodium pyrosulfite), ($Na_2S_2O_5$), 150 g/l solution.

Prepare this solution on the day of use.

4.14 Sodium molybdate, dihydrate ($Na_2MoO_4 \cdot 2H_2O$), 20 g/l solution.

4.15 Bismuth sulfate [$Bi_2(SO_4)_3$] solution.

Dissolve 4,25 g of bismuth sulfate in 280 ml of the sulfuric acid (4.6) and heat until the salt is dissolved completely. Cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water, and mix.

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

This solution can also be prepared using metallic bismuth instead of bismuth sulfate.

4.16 Phosphorus standard solution, containing 0,010 g of P per litre.

Dry potassium dihydrogenorthophosphate (KH_2PO_4) at 110 °C to constant mass and cool in a desiccator. Dissolve 0,220 g of the dried salt in water, transfer to a 500 ml one-mark volumetric flask, dilute to the mark and mix. Transfer 25,0 ml of this solution to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,010 mg of P.

5 Apparatus

Ordinary laboratory equipment and

5.1 One-mark volumetric flasks of capacity 100 ml, made of borosilicate glass.

5.2 Muffle furnaces, adjustable to 400 ± 20 °C and 800 to 1 000 °C.

5.3 Spectrophotometer, suitable for measurement of absorption in the region of 725 nm.

6 Sampling and samples

For analysis, use a laboratory sample of minus 100 μm particle size which has been taken in accordance with ISO 3081 or ISO 3082 and prepared in accordance with ISO 3082 or ISO 3083. In the case of ores with significant contents of combined water or oxidizable compounds use a particle size of minus 160 μm .

NOTE — A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

6.1 Preparation of predried test samples

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. Dry the test sample at 105 ± 2 °C as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

Carry out the analysis generally in duplicate, independently, on one predried test sample.

NOTE — The expression "independently" implies that the repetition of the procedure shall be carried out at a different time and include appropriate recalibration.

7.1 Blank test and check test

In each run, one blank test and one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A predried test sample of the certified reference material shall be prepared as specified in 6.1.

NOTE — The certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar to ensure that in either case no significant changes in the analytical procedure would become necessary.

When the analysis is carried out on several samples at the same time, the blank value may be represented by one test, providing the procedure is the same and the reagents used are from the same reagent bottles.

When the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately 1 g of the predried test sample obtained in accordance with 6.1.

NOTE — The test portion should be taken and weighed quickly in order to avoid reabsorption of moisture.

7.3 Determination

7.3.1 Decomposition of the test portion

If the decomposition is to be based on alkali sintering, proceed as instructed in 7.3.1.1. If the decomposition is to be based on acid attack, proceed as instructed in 7.3.1.2.

7.3.1.1 Alkali sinter attack

CAUTION — Protective goggles should be used during the following operation.

Place the test portion (7.2) in a 40 ml nickel crucible, add 3 g of the sodium peroxide (4.1), mix well using a nickel spatula, and tamp the mixture. Place the crucible for 1 or 2 min at the entrance of a muffle furnace (5.2), the temperature of which is regulated at 400 ± 20 °C, and then transfer into the closed furnace for about 1 h to effect sintering.