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## Iron ores — Determination of phosphorus content — Titrimetric method

*Minerais de fer — Dosage du phosphore — Méthode titrimétrique*

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Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

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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 preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 2599 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This third edition cancels and replaces the second edition (ISO 2599:1983), which has been editorially revised.

# Iron ores — Determination of phosphorus content — Titrimetric method

**WARNING** — This International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

## 1 Scope

This International Standard specifies a titrimetric method for the determination of the phosphorus content of iron ores, using hexaammonium heptamolybdate (ammonium molybdate).

This method is applicable to a concentration range of 0,10 % (*m/m*) to 5,0 % (*m/m*)<sup>1)</sup> of phosphorus in natural iron ores, and iron ore concentrates and agglomerates including sinter products.

This International Standard provides a quality control method for the determination of phosphorus by titration, however, the method cannot be used for referee purposes.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3082:2000, *Iron ores — Sampling and sample preparation procedures*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 7764:1985, *Iron ores — Preparation of predried test samples for chemical analysis*

## 3 Principle

The test portion is decomposed by treatment with hydrochloric, nitric and perchloric acids. The insoluble residue is filtered and the filtrate is reserved as the main solution.

The residue is ignited, treated with sulfuric and hydrofluoric acids to remove silicon dioxide, fused with sodium carbonate and the melt is leached with water. The alkaline solution is filtered and the residue is discarded.

1) This method has been tested internationally on samples containing phosphorus contents ranging from 0,03 % (*m/m*) to 1,50 % (*m/m*).

The filtrate from residue treatment is acidified using hydrochloric acid, iron(III) chloride is added and the phosphorus is collected by precipitation with ammonia solution. The precipitate is dissolved in hydrochloric acid and combined with the main solution. Hydrobromic acid is added and evaporated to fumes of perchloric acid to expel arsenic. The salts are dissolved in water and the solution, or an aliquot, is neutralized with ammonia solution and the acidity is adjusted using nitric acid. The solution is cooled to 20 °C and vanadium is reduced by iron(II) sulfate. Ammonium molybdate is added to the cold solution and the yellow precipitate of ammonium molybdophosphate is collected by filtration.

The precipitate is washed free of acid and dissolved in a slight excess of sodium hydroxide, the excess being titrated with nitric acid.

For test portions containing more than 18 mg of titanium, a modified procedure is used (see 7.4.1.2).

## 4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water that conforms to grade 2 of ISO 3696:1987.

- 4.1 **Ammonium nitrate** ( $\text{NH}_4\text{NO}_3$ ).
- 4.2 **Sodium carbonate** ( $\text{Na}_2\text{CO}_3$ ), anhydrous.
- 4.3 **Hydrochloric acid**,  $\rho$  1,16 g/ml to 1,19 g/ml
- 4.4 **Hydrochloric acid**,  $\rho$  1,16 g/ml to 1,19 g/ml diluted 1 + 1.
- 4.5 **Nitric acid**,  $\rho$  1,42 g/ml.
- 4.6 **Nitric acid**,  $\rho$  1,42 g/ml diluted 1 + 50.
- 4.7 **Nitric acid**,  $\rho$  1,42 g/ml diluted 1 + 5 000.
- 4.8 **Sulfuric acid**,  $\rho$  1,84 g/ml.
- 4.9 **Sulfuric acid**,  $\rho$  1,84 g/ml diluted 1 + 1.
- 4.10 **Perchloric acid**,  $\rho$  1,54 g/ml, 60 % (m/m) solution, or  $\rho$  1,67 g/ml, 70 % (m/m) solution.
- 4.11 **Hydrofluoric acid**,  $\rho$  1,13 g/ml, 40 % (m/m) solution.
- 4.12 **Hydrobromic acid**,  $\rho$  1,38 g/ml, 40 % (m/m) solution or  $\rho$  1,48 g/ml, 47 % (m/m) solution.
- 4.13 **Ammonia solution**,  $\rho$  0,9 g/ml.
- 4.14 **Hydrogen peroxide**, 30 % (m/m) solution, free from phosphate stabilizer.
- 4.15 **Potassium nitrate**, 10 g/l solution.
- 4.16 **Ammonium molybdate**, solution.

Dissolve 40 g of finely pulverized crystalline ammonium molybdate tetrahydrate  $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$  in 300 ml of warm water and 80 ml of ammonia solution (4.13). Allow the solution to cool and add this solution in small portions to 600 ml of nitric acid (1 + 1), while stirring. Add a few milligrams of sodium hydrogen phosphate or ammonium hydrogen phosphate and allow to stand for at least 2 d. Filter before use.

**4.17 Iron(III) chloride** solution, containing 3 g of Fe/l.

Dissolve 0,3 g of pure iron, with as small a content of phosphorus as possible, by treatment with hydrochloric acid (4.3), add a few drops of nitric acid (4.5) to oxidize the iron, and boil the mixture to remove chlorine.

Allow the solution to cool and dilute with water to 100 ml.

**4.18 Iron(III) sulfate** solution, 100 g/l.

Dissolve 100 g of iron(II) sulfate heptahydrate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) in 1 l of sulfuric acid (1 + 19).

**4.19 Water**, free of  $\text{CO}_2$ .

Heat distilled or deionized water to boiling point in a conical flask for 5 min to expel carbon dioxide. Cool, protecting the water in an appropriate way against any pickup of carbon dioxide.

**4.20 Sodium hydroxide**, standard volumetric solution,  $c(\text{NaOH}) = 0,2 \text{ mol/l}$  approximately.

Dissolve 8 g of sodium hydroxide in 1 l of freshly boiled and cooled water (4.19). Standardize this solution using either amidosulfuric acid (sulfamic acid) or potassium hydrogen phthalate.

Dry either potassium hydrogen phthalate or sulfamic acid at 105 °C for 1 h and weigh, to the nearest 0,001 g, either approximately 1 g of potassium hydrogen phthalate or approximately 0,5 g of sulfamic acid. Dissolve either material in 50 ml of water free of  $\text{CO}_2$  (4.19) and titrate with sodium hydroxide solution (4.20) using phenolphthalein solution (4.22) as indicator.

**4.21 Nitric acid**, standard volumetric solution  $c(\text{HNO}_3) = 0,2 \text{ mol/l}$  approximately.

Dilute 13 ml of nitric acid (4.5) with water, to 1 l.

Standardize the solution as follows: accurately measure 25 ml of sodium hydroxide standard volumetric solution (4.20) and titrate with nitric acid solution, using phenolphthalein solution (4.22) as indicator.

**4.22 3,3-Bis(4-hydroxyphenyl)phthalide (phenolphthalein)** solution, 0,1 g/100 ml.

Dissolve 0,10 g of powdered phenolphthalein in 90 ml of ethanol, 95 % (V/V), and dilute with water, to 100 ml.

## 5 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks in accordance with ISO 648 and ISO 1042.

## 6 Sampling and samples

### 6.1 Laboratory sample

For analysis, use a laboratory sample of – 100  $\mu\text{m}$  particle size which has been taken and prepared in accordance with ISO 3082. In the case of ores having significant contents of combined water or oxidizable compounds, use a particle size of – 160  $\mu\text{m}$ .

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

## 6.2 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  $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$  as specified in ISO 7764. (This is the predried test sample.)

## 7 Procedure

### 7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with the flowsheet in Annex A, independently, on one predried test sample.

**NOTE** The expression “independently” means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure be carried out either by the same operator at a different time or by a different operator including, in either case, appropriate recalibration.

### 7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, the amount of the predried test sample (6.2) specified in Table 1.

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

**Table 1 — Mass of test portion**

Phosphorus content %	Mass of test portion g	Volume of aliquot portion ml	Volume of ammonium molybdate solution (4.16) ml
0,1 to 0,5	1,0	total	50
0,5 to 2,5	0,5	total	100
2,5 to 5,0	0,5	100/250	100

### 7.3 Blank test and check test

In each run, one blank test and one analysis of a certified 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 6.2.

**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. Where a certified reference material is not available, a reference material may be used (see 8.2.4).

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

Where 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.

## 7.4 Determination

### 7.4.1 Decomposition of the test portion

#### 7.4.1.1 Test portions containing less than 18 mg titanium

##### 7.4.1.1.1 Initial decomposition

Place the test portion (7.2) in a 300 ml beaker, add 25 ml of hydrochloric acid (4.3), cover the beaker with a watch-glass and heat gently to decompose the ore.

For decomposition of the test portion, place the beaker in a low temperature zone (60 °C to 100 °C) of the hot-plate to digest the sample for about 1 h, then transfer to a higher temperature zone and heat for about 10 min just below boiling.

Add 5 ml of nitric acid (4.5) and 15 ml of perchloric acid (4.10), cover the beaker and heat to dense white fumes of perchloric acid. Maintain a steady refluxing of the acid on the walls of the beaker for about 10 min. Allow the beaker to cool, add about 50 ml of warm water and heat to dissolve soluble salts.

Filter the solution through a close-texture paper and wash the residue three to five times with nitric acid (4.6) and finally with hot water until free from acid, receiving the filtrate and washings in a 300 ml beaker. Evaporate the filtrate by gently heating and reserve as the main solution.

**NOTE** If a sample obviously contains organic matter, carbonates or sulfides, and decomposition becomes easier by roasting, place the test portion, accurately weighed, in a porcelain crucible. Cover the crucible and roast at 700 °C to 750 °C for about 30 min. Allow the crucible to cool to room temperature in a desiccator, wash out the contents of the crucible into a 300 ml beaker, and proceed as directed in 7.4.1.1.

##### 7.4.1.1.2 Treatment of the residue

While the main solution is evaporating, place the filter paper and residue in a platinum crucible, dry, char the paper and finally ignite at 750 °C to 800 °C. Cool the crucible, moisten the residue with a few drops of sulfuric acid (4.9), add 5 ml of hydrofluoric acid (4.11) and evaporate to remove silicon dioxide and sulfuric acid. Cool the crucible, add 3 g of sodium carbonate (4.2) and fuse until a clear melt is obtained.

Cool the crucible, place it in a 300 ml beaker, add 100 ml of water and heat to disintegrate the melt and to dissolve all soluble salts. Remove and rinse the crucible. Filter the alkaline solution through a medium-texture paper and wash with warm water, collecting the filtrate and washings in a 300 ml beaker. Discard the residue.

Add hydrochloric acid (4.3) to the filtrate until it is acidic, boil to remove carbon dioxide, cool and add 10,0 ml of iron(III) chloride solution (4.17). Add ammonia solution (4.13) in small portions while stirring, until the solution is slightly alkaline (indicator paper), in order to precipitate iron hydroxide and iron phosphate. Boil the solution for about 2 min, then remove from the source of heat. Allow the precipitate to settle, filter through a rapid filter paper and wash with hot water. Discard the filtrate.

Place the beaker containing the main solution from 7.4.1.1.1 under the funnel. Dissolve the precipitate by pouring on to the paper about 10 ml of hydrochloric acid (4.4). Wash with warm water.

#### 7.4.1.2 Test portions containing more than 18 mg titanium

If the test portion contains more than 18 mg of titanium, the following procedure should be applied.

Place the test portion (7.2) in a platinum crucible lined with 1 g of fused sodium carbonate. Mix the test portion with ten times its mass of fusion mixture (sodium carbonate:sodium nitrate 10:1) and heat gently at first, then at a higher temperature to complete fusion of the test portion. After cooling, place the crucible together with its contents in a 300 ml beaker, add about 100 ml of warm water and stir gently. Remove the melt from the crucible, rinse the crucible with water and remove.

Heat the contents of the beaker to boiling in order to dissolve soluble salts and filter off any insoluble residue through a medium-texture filter paper. Wash the residue thoroughly with sodium carbonate solution (2 %) and discard the residue. Transfer the filtrate and washings to a 500 ml beaker, add hydrochloric acid (4.3) until the solution is acidic and boil it to remove carbon dioxide.

After cooling, add 10,0 ml of iron(III) chloride solution (4.17) and add ammonia solution (4.13) in small portions while stirring, until the solution is slightly alkaline, in order to precipitate iron hydroxide and iron phosphate. Boil the solution for about 2 min, remove from the source of heat, allow the precipitate to settle, filter through a rapid filter paper, wash with hot water and discard the filtrate and washings.

Pour about 10 ml of warm hydrochloric acid (4.4) on to the filter paper to dissolve the precipitate then wash with hot water. Collect the solution and washings in a 300 ml beaker, add 15 ml of perchloric acid (4.10), evaporate the solution to about 30 ml and proceed to 7.4.2.

#### 7.4.2 Perchloric acid fuming

Evaporate the combined solutions to about 30 ml, add 5 ml of hydrobromic acid (4.12) and continue to evaporate, in the uncovered beaker, to dense white fumes of perchloric acid. Cool, add 20 ml of water and again evaporate to dense white fumes of perchloric acid. Cover the beaker and maintain a steady refluxing of the acid on the wall of the beaker for 10 min. Cool, add about 50 ml of water and warm to dissolve the salts. If a precipitate of silica appears here, filter the solution into a 500 ml Erlenmeyer flask and wash eight times with warm nitric acid (4.6).

If the entire solution is to be used for the analysis, leave the filtrate in the 500 ml Erlenmeyer flask. If an aliquot is to be used, transfer to a 250 ml volumetric flask, dilute to volume and mix. Transfer an aliquot, as listed in Table 1, to a 500 ml Erlenmeyer flask.

#### 7.4.3 Precipitation and treatment of precipitate

Evaporate by heating or dilute with water to about 60 ml and cool to room temperature. Add ammonia solution (4.13) until a small precipitate of iron(III) hydroxide persists after shaking or stirring. Add nitric acid (4.5) until the precipitate just dissolves and then 5 ml in excess.

**NOTE 1** If a precipitate of manganese dioxide remains here, add a small amount of ammonium iron(II) sulfate crystals or a few drops of hydrogen peroxide (4.14), to reduce and dissolve manganese, then boil the solution to oxidize excess of iron(II) or to remove the excess of hydrogen peroxide and cool.

Add 3 g of ammonium nitrate (4.1), shake to dissolve and dilute to about 100 ml. Cool to 20 °C, then add 5 ml of iron(III) sulfate solution (4.18) to reduce the vanadium.

Precipitate the phosphorus by the addition of the amount of ammonium molybdate solution (4.16) specified in Table 1. Stopper and shake the flask vigorously for 10 min and allow to stand for 1 h or until the ammonium molybdate precipitate has settled completely.

**NOTE 2** If the phosphorus content is small, it may be necessary to allow the solution to stand for 4 h or overnight for complete settling.

Collect the precipitate on a small, close-texture paper or on a filtering tube containing paper pulp and wash the flask and the precipitate with nitric acid (4.6) until iron ions are no longer detected in the washings. Wash the flask three times and the precipitate five times with dilute nitric acid (4.7).

**NOTE 3** The yellow precipitate tends to climb, hence, when washing the precipitate, the jet of the wash solution should be directed to the top of the paper and spirally downward.

Wash the flask and the precipitate three times with potassium nitrate wash solution (4.15) until the washings are free from acid as shown by an indicator test.

#### 7.4.4 Titration

Return the precipitate with the filter paper to the original flask, add 50 ml of cold, freshly boiled water (4.19) and shake to disintegrate the paper. Add a slight measured excess of sodium hydroxide standard volumetric solution (4.20) and shake the mixture to dissolve the yellow precipitate. Add a few drops of phenolphthalein indicator (4.22), dilute to about 150 ml with water (4.19) and titrate the excess sodium hydroxide with nitric acid standard volumetric solution (4.21), until the red colour of the solution disappears with a final drop of the titrant.

### 8 Expression of results

#### 8.1 Calculation of phosphorus content

The analytical values obtained shall first be corrected with the blank value and then the phosphorus content  $w_P$  of each analysis shall be calculated as a percentage by mass to four decimal places using the following equation:

$$w_P(\%) = \frac{c_1 V_1 - c_2 V_2}{m} \times 0,001347 \times 100 \quad (1)$$

where

- $c_1$  is the concentration, in moles per litre, of sodium hydroxide standard volumetric solution (4.20);
- $V_1$  is the volume, in millilitres, of sodium hydroxide standard volumetric solution (4.20), taken in 7.4.4;
- $c_2$  is the concentration, in moles per litre, of nitric acid standard volumetric solution (4.21);
- $V_2$  is the volume, in millilitres, of nitric acid standard volumetric solution (4.21), used for the titration in 7.4.4;
- $m$  is the mass, in grams, of the test portion;

0,001 347 is the mass, in grams, of phosphorus corresponding to 1,00 ml of sodium hydroxide solution,  $c(\text{NaOH}) = 1,000 \text{ mol/l}$ .

#### 8.2 General treatment of results

##### 8.2.1 Repeatability and permissible tolerance

The precision of this analytical method is expressed by the following regression equations:

See Annexes B and C.

$$R_d = 0,019\ 8X + 0,009\ 4 \quad (2)$$

$$P = 0,089\ 1X + 0,007\ 4 \quad (3)$$

$$\sigma_d = 0,007\ 0X + 0,003\ 4 \quad (4)$$

$$\sigma_L = 0,0315X + 0,0019 \quad (5)$$

where

- $R_d$  is the independent duplicate limit;

- $X$  is the phosphorus content, as a percentage by mass, of the test sample.

NOTE For the within-laboratory Equations (2) and (4), the arithmetic mean of the duplicate values is taken; for the between-laboratories Equations (3) and (5), the arithmetic mean of the final results (8.2.5) of the two laboratories is taken.

$P$  is the permissible tolerance between laboratories;

$\sigma_d$  is the independent duplicate standard deviation;

$\sigma_L$  is the between-laboratories standard deviation.

### 8.2.2 Determination of analytical result

Having computed the independent duplicate results using Equation (1), compare them with the independent duplicate limit,  $R_d$ , using the procedure given in Annex A, and obtain the final laboratory result  $\mu$  (see 8.2.5).

### 8.2.3 Between-laboratories precision

Between-laboratories precision is used to determine the agreement between the final results reported by two laboratories. The assumption is that both laboratories followed the procedure described in 8.2.2.

Compute the following quantity:

$$\mu_{1,2} = \frac{\mu_1 + \mu_2}{2} \quad (6)$$

where

$\mu_{1,2}$  is the mean of the final results;

$\mu_1$  is the final result reported by laboratory 1;

$\mu_2$  is the final result reported by laboratory 2.

Substitute  $\mu_{1,2}$  for  $X$  in Equation (3) and calculate  $P$ .

If  $|\mu_1 - \mu_2| \leq P$ , the final results are in agreement.

### 8.2.4 Check for trueness

The trueness of the analytical method shall be checked by applying it to a certified reference material (CRM) or a reference material (RM) (see Note in 7.3). Calculate the analytical result ( $\mu$ ) for the CRM/RM using the procedures described in 8.1 and 8.2, and compare it with the reference or certified value  $A_c$ . There are two possibilities:

a)  $|\mu_c - A_c| \leq C$  in which case the difference between the reported result and the certified/reference value is statistically insignificant;

b)  $|\mu_c - A_c| > C$  in which case the difference between the reported result and the certified/reference value is statistically significant.

where

$\mu_c$  is the final result for the certified reference material;

$A_c$  is the certified/reference value for the CRM/RM;

$C$  is a value dependent on the type of CRM/RM used.

NOTE 1 Certified reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35:1989, *Certification of reference materials — General and statistical principles*.

For a CRM certified by an interlaboratory test programme:

$$C = \sqrt{\sigma_L^2 + \frac{\sigma_d^2}{n} + V(A_c)}$$

where

$V(A_c)$  is the variance of the certified value  $A_c$  ( $= 0$  for a CRM certified by only one laboratory);

$n$  is the number of replicate determinations carried out on the CRM/RM.

NOTE 2 This type of CRM should be avoided unless it is known to have an unbiased certified value.

### 8.2.5 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operations specified in Annex A, calculated to four decimal places and rounded off to the second decimal place as follows:

- where the figure in the third decimal place is less than 5, it is discarded and the figure in the second decimal place is kept unchanged;
- where the figure in the third decimal place is 5 and there is a figure other than 0 in the second decimal place, or where the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one;
- where the figure in the third decimal place is 5 and there is no figure other than 0 in the fourth decimal place, the 5 is discarded and the figure in the second decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.

### 8.3 Oxide factor

$$w_{P_2O_5} (\%) = 2,2914 w_P (\%)$$

## 9 Test report

The test report shall include the following information:

- name and address of the testing laboratory;
- date of issue of the test report;
- reference to this International Standard, i.e. ISO 2599;
- details necessary for the identification of the sample;
- result of the analysis;
- reference number of the result;
- any characteristics noticed during the determination, and any operations not specified in this International Standard that may have had an influence on the result, either for the test sample or the certified reference material(s).

## Annex A

(normative)

### Flowsheet of the procedure for the acceptance of analytical values for test samples

Start with independent duplicate results

