

INTERNATIONAL STANDARD

ISO
8833

First edition
1989-11-15

Magnetite for use in coal preparation — Test methods

Magnétite à utiliser dans la préparation des charbons — Méthodes d'essai



Reference number
ISO 8833 : 1989 (E)

Foreword

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International Standard ISO 8833 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*.

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Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

The basic requirements for a material to be used in dense medium suspension are that it should be inert, have a high relative density and be easy to recover. In coal preparation, the mineral magnetite (FeO , Fe_2O_3) has become the most commonly used material, not only because it fulfils all the above criteria, but also because of its worldwide occurrence either as an iron ore or as a by-product of mining operations for other minerals. Like any mineral, magnetite rarely occurs in the pure state and the presence of gangue minerals may lower the relative density and affect the ferromagnetic properties. In addition, like other minerals in the spinel group, there may be substitution of either or both the divalent and/or trivalent ions by ions of other metals (e.g. Mg^{2+} , Mn^{2+} , Al^{3+} , Cr^{3+} , Mn^{3+}). These variations may seriously affect the efficiency of recovery operations and it is important when designing a coal preparation plant that the manufacturer of the magnetic separators be informed of the source of magnetite to be used and if possible be provided with a sample.

The purpose of this International Standard is to provide a basis for the testing of magnetite for use in coal preparation. It is intended for use by contracting parties in the sale and purchase of magnetite and for coal preparation engineers engaged in the design and quality control fields.

The tests specified in this International Standard will assist the user in the selection of magnetite for use in dense medium suspension and provide a basis for quality control testing. No attempt is made to formulate the requirements for particular plant applications.

The specified tests should ensure that the properties which make magnetite suitable for coal preparation purposes are simply and adequately tested.

These properties are

- a) moisture content;
- b) particle size distribution;
- c) total magnetics content;
- d) relative density;
- e) total iron content;
- f) iron(II) content;
- g) fundamental magnetic properties.

All the above tests may be required for the assessment of magnetite from a new source, but for routine checking of regular supplies, moisture, particle size distribution, total magnetics content and relative density should be sufficient.

Tests for fundamental magnetic properties are not described because the determination of suitable parameters requires very specialized apparatus and techniques and is probably best undertaken by a University or Research Institute whose staff are experienced in making such measurements. There is a lack of consensus as to which

magnetic parameters are relevant in the context of this International Standard. Therefore, it is left open for the interested parties to decide which property to measure. However, there is some evidence to suggest that the coercivity is a guide to the ease of recovery of a magnetite and that the saturation magnetic moment is a measure of the purity of the actual magnetite grains.

This International Standard applies exclusively to the testing of magnetite and no attempt is made to recommend specific tests for the dense medium suspensions of which magnetite forms the solid phase.

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Magnetite for use in coal preparation — Test methods

1 Scope

This International Standard specifies methods for the sampling and testing of magnetite for use in coal preparation.

The test methods are intended primarily for the testing of milled magnetite, the largest particle size of which is usually less than 250 μm . However, the tests are also applicable to unmilled magnetite with an upper particle size limit of about 500 μm .

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 2591 : 1973, *Test sieving*.

ISO 2597 : 1985, *Iron ores — Determination of total iron content — Titrimetric methods*.

ISO 3081 : 1986, *Iron ores — Increment sampling — Manual method*.

ISO 3083 : 1986, *Iron ores — Preparation of samples — Manual method*.

ISO 3310-1 : 1982, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*.

3 Sampling

3.1 General

Sampling shall be carried out in accordance with ISO 3081, which specifies the procedures for taking samples of iron ores loaded onto and discharged from conveyors, railway wagons, bunkers, ships and stockpiles, by the manual increment method, subject to the following provisions.

If possible, carry out sampling during material transfer, using a conventional manual increment shovel conforming to the requirements of ISO 3081.

If sampling during material transfer is impracticable, use sampling spears (see figure 1) for sampling bagged materials or small piles, and screw-type augers (see figure 2) when sampling from existing stockpiles, wagons, ships, etc., or in situations where the material has compacted.

The following procedure shall be used when sampling magnetite contained in sealed bags.

- Select a number of bags in accordance with the requirements of table 1.
- Open the bags and incline them so that the sampling spear can be inserted at an angle close to the horizontal. Insert the spear fully with the slot underneath and rotate the spear through two complete revolutions.
- Rotate again through 180° so that the open slot is uppermost and withdraw the spear containing the increment.
- Place the increment in a container fitted with an airtight lid.
- Repeat this procedure until all the requisite bags have been sampled and a total sample mass of approximately 1 kg is obtained.

Table 1 — Minimum number of bags to be sampled

Number of bags in batch	Minimum number of bags to be sampled
< 5	All bags
5 to 250	5
> 250	1 per 50 (to the nearest 50 bags)

When screw-type augers are used, the stockpile or loaded material shall be systematically sampled at various depths, including the bottom of the pile if practicable, to ensure representative sampling.

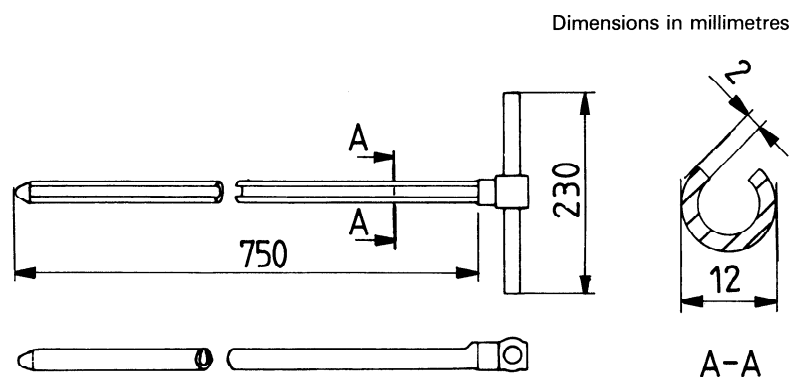
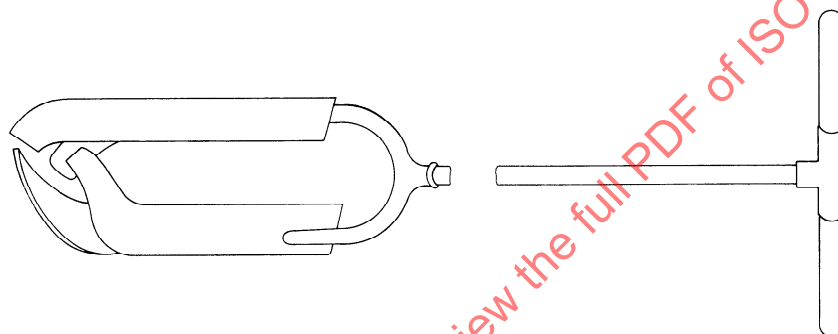


Figure 1 — Sampling spear (typical dimensions)



NOTE — Various diameters and lengths will be needed for specific pile sizes.

Figure 2 — Sampling auger

3.2 Preparation of samples

The gross sample, subsamples or increments obtained in accordance with ISO 3081 shall be prepared in accordance with ISO 3083 subject to the following provisions.

- a) If it proves difficult to carry out sample division because the sample is too wet, pre-dry the sample sufficiently to overcome this difficulty. If necessary, determine the moisture content before drying.
- b) If the maximum particle size of the magnetite is such that grinding of the material is necessary in order to comply with the preparation procedures, then take separate samples for moisture determination, size analysis and physical/chemical analysis. The mass of sample for a particular test shall be that specified in ISO 3083.

Magnetite used in coal preparation is usually in a finely divided form. Therefore, it will generally not be necessary to grind the material and it will be sufficient to provide a single sample for all analytical purposes. This sample shall have a mass of not less than 1 kg and be stored in an airtight container to ensure that it is protected from any alteration.

- c) If sampling is for comparative purposes between contracting parties, at least four samples shall be prepared. Three of the samples are intended for the seller, the purchaser and arbitration, and one is held in reserve.

- d) Further sample division to obtain test portions for a particular analytical test shall preferably be carried out using the increment division method specified in ISO 3083. Sample division using riffle dividers, or other similar equipment, or random spot sampling may be used provided that these methods can be shown to be free of significant bias.

4 Determination of moisture content

4.1 General

The total moisture content of a consignment may be required for both commercial and safe shipment purposes. A knowledge of the total moisture content of samples prepared for laboratory analysis may also be required for subsequent conversion of the analytical results to the dry basis.

The total moisture content of a consignment is often required in order to comply with various maritime regulations as, when moisture is present in excess, it may result in stability problems during shipping.

The total moisture content shall be determined either in one stage or in two stages, where the free moisture and air-dried moisture contents are separately determined. This latter method is used when large quantities are involved or if predrying is necessary in order to carry out the sample preparation (see 3.2).

In both methods the aim is to determine the total moisture content only, but it has been found that loosely-bound sulfur compounds which may be present in the magnetite matrix may also be released. In general, errors from this source will be insignificant.

The determination shall be carried out on a mass of approximately 1 kg for a consignment sample and a mass of 100 g for a sample prepared for laboratory analysis.

When a gross sample is taken from a consignment, at least four moisture samples shall be prepared and tested. Where possible, duplicate determinations should be carried out on laboratory analysis samples.

4.2 Apparatus

4.2.1 Drying trays of non-corrodible material

NOTE — A minimum loading area of 9 dm² is recommended for 1 kg test portions and of 1,5 dm² for 100 g test portions.

4.2.2 Balances

- a) A balance capable of weighing up to 5 kg to an accuracy of $\pm 0,1$ g.
- b) A balance capable of weighing up to 200 g to an accuracy of $\pm 0,01$ g.

4.2.3 Laboratory air-oven, capable of being maintained at a temperature in the range 105 °C to 110 °C and of accommodating the drying trays.

4.2.4 Desiccator

4.3 Weighings

All weighings described in 4.4 and 4.5 shall be carried out using the appropriate balance (4.2.2) to an accuracy of $\pm 0,1$ g if a 1 kg test portion is taken or to an accuracy of $\pm 0,01$ g if a 100 g test portion is taken.

4.4 Method 1: One-stage method

4.4.1 Test portion

Take a test portion of approximately 1 kg or approximately 100 g, as appropriate (see 4.1 and 4.3), from the sample, in accordance with 3.2.

4.4.2 Procedure

Weigh a clean, dry drying tray (4.2.1) (m_1), spread the test portion uniformly onto the tray and reweigh (m_2).

Place the uncovered tray in the air oven (4.2.3) maintained at a temperature in the range 105 °C to 110 °C, and dry to constant mass (m_3). Weigh it after minimum cooling in the case of a 1 kg test portion or after allowing the tray and sample to cool in the desiccator (4.2.4) in the case of a 100 g test portion.

4.4.3 Expression of results

The total moisture content of the sample, expressed as a percentage by mass, is calculated from the formula

$$\frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the drying tray;

m_2 is the initial mass, in grams, of the drying tray with the test portion;

m_3 is the final mass, in grams, of the drying tray with the test portion after oven drying.

Report the result to one decimal place.

4.5 Method 2: Two-stage method

4.5.1 Free moisture

The mass of the test portion and the test procedure are essentially as laid down for the one-stage method, except that the test portion is allowed to attain constant mass by exposure to air at ambient temperature rather than by drying in the heated air oven.

In this determination it is only necessary for the test portion to reach an approximate equilibrium state, as any remaining moisture will be included in the second stage determination of air-dried moisture content.

The free moisture content of the sample, expressed as a percentage by mass, is calculated from the equation

$$FM = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the drying tray;

m_2 is the initial mass, in grams, of the drying tray with the test portion;

m_3 is the final mass, in grams, of the drying tray with the test portion after air drying.

4.5.2 Air-dried moisture content

Take a test portion of approximately 100 g using the material air-dried in the free moisture content determination (4.5.1), sub-sampling if necessary.

Carry out the procedure as described in 4.4.2.

Calculate the air-dried moisture content of the sample, expressed as a percentage by mass, from the equation

$$ADM = \frac{m_5 - m_6}{m_5 - m_4} \times 100$$

where

m_4 is the mass, in grams, of the drying tray;

m_5 is the initial mass, in grams, of the drying tray with the test portion;

m_6 is the final mass, in grams, of the drying tray with the test portion after drying.

4.5.3 Expression of results

The total moisture content of the sample, expressed as a percentage by mass, is calculated from the formula

$$FM + ADM \times \frac{100 - FM}{100}$$

where

FM is the free moisture content, expressed as a percentage by mass;

ADM is the air-dried moisture content, expressed as a percentage by mass.

Report the result to one decimal place.

5 Treatment of samples prior to laboratory analysis

Drying of the magnetite, either during the sample preparation stage or subsequently, may cause the magnetite to form agglomerates or, less usually a hard cake. Caking only occurs when oven drying is employed with magnetite that is extremely wet.

If agglomerates are observed in the dried material, it will be necessary to restore the material to its original consistency of discrete particles. This is best achieved using a rubber-covered roller to break down the agglomerates; it may also be helpful to use a test sieve of 106 μm nominal size of openings beforehand to separate larger agglomerates from already discrete particles. If the sample is required for particle size analysis, take care to ensure that the sample is not altered in respect of particle size.

If the agglomerates are strongly bonded or the material is caked to such an extent that it cannot be restored to its original con-

sistency, discard the sample. Prepare a further sample in the air-dried condition and use this for subsequent analysis.

It may be helpful to demagnetize the prepared material prior to analysis. However, in no circumstances shall a demagnetized sample be used in the determination of magnetic content or fundamental magnetic properties.

Unless otherwise specified, dry all prepared laboratory analysis samples to constant mass as described in 4.4.2 and cool in a desiccator prior to sub-sampling to obtain the required test portion. If analyses are carried out on air-dried material, it will be necessary to determine the air-dried moisture content (4.5.2) and calculate the dry mass of the test portion in order to convert the analysis result to the dry basis. The dry mass of the test portion is calculated from the equation

$$m_d = m_{ad} \times \frac{100 - ADM}{100}$$

where

m_d is the dry mass, in grams, of the test portion;

m_{ad} is the air-dried mass, in grams, of the test portion;

ADM is the air-dried moisture content, expressed as a percentage by mass.

6 Determination of particle size distribution

6.1 General

The particle size distribution shall be determined using test sieves and a sub-sieve classifier.

NOTE — In the context of this International Standard, the term "sub-sieve" is taken to mean particle sizes less than 38 μm .

As some magnetites have a strong tendency to form agglomerates, it is recommended that when sizing magnetite, particularly from a new source, a microscopic examination of both the test sample and sized fractions be carried out to check for the presence of agglomerates. However, the microscope shall not be used for sizing purposes, because the results are too subjective.

The sample used for size determination shall not have been used for other tests or purposes which in any way modify the mass or particle size.

6.2 Sieve analysis

The test sieves shall have square holes and shall comply with ISO 3310-1. The number of sieves used and the interval between successive aperture sizes shall adequately cover the size range of the material to a lower limit of 38 μm .

NOTE — The number of sieves used should not normally be less than four.

The mass of the test portion shall not be less than 25 g and not more than 100 g.

Carry out sieving using a wet sieving method in accordance with ISO 2591.

Express the results as the percentage by mass passing a sieve of given size of openings.

6.3 Sub-sieve analysis

The sub-sieve analysis of magnetite is made difficult by the agglomeration of individual particles due to the earth's or other magnetic fields; this precludes the use of both wet sedimentation and wet elutriation methods and the analysis is best carried out using a centrifugal classifier. The fundamental requirement is that any agglomerates are broken down without degradation of individual particles.

The mass of the test portion will depend on the particular method adopted but should not be less than 10 g.

Express the results as the percentage by mass which is smaller than a given particle size.

7 Determination of total magnetics content

7.1 General

Magnetic concentrates are usually prepared on permanent magnet separators. On coal preparation plants the recovery and densification of magnetite medium is carried out using similar equipment. The total magnetics content of a material is a measure of the ultimate recovery that can be achieved. It does not give any indication of the likely efficiency of a recovery system but provides both an ideal to which plant separator performance can be referred, and a useful parameter for routine quality control purposes.

NOTE — A test involving a single pass through a Davis tube or similar device may, by suitable choice of magnetic field strength and fluid flow, give an approximation to plant separator performance. While this test provides a useful parameter for engineers, it will not determine the total magnetics content. If a multiple pass procedure is used with such a device the results obtained will approach those obtained by the standard method as described below.

7.2 Apparatus

The apparatus is shown in figure 3 and consists essentially of a vertical glass tube of 8 mm nominal bore and about 500 mm long fitted with magnets to give a field strength of not less than 60 mT at the centre of the tube. The glass tube and magnets are supported by a laboratory retort stand assembly.

NOTE — The fields of the magnets should not be opposing.

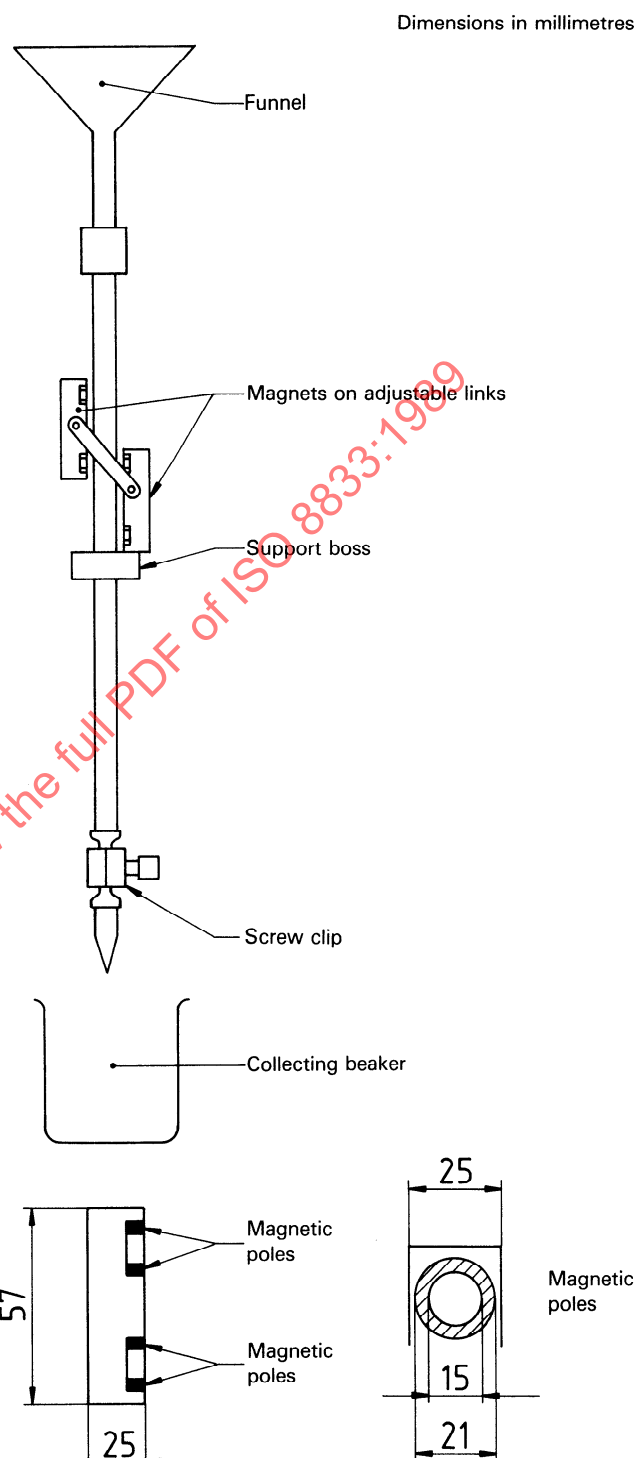


Figure 3 — Apparatus for determination of magnetics content

7.3 Procedure

7.3.1 Take a test portion of approximately 2 g in accordance with 3.2; place it in a previously tared beaker of 100 ml capacity and weigh to an accuracy of ± 2 mg. Add about 5 drops of a suitable wetting agent and about 50 ml of water. Mix by swirling the solids and liquids in the beaker and then allow the contents to stand for about 5 min. This should be sufficient to ensure that the solids are adequately wetted.

7.3.2 Fill the apparatus with water up to the funnel and slowly transfer the contents of the 100 ml beaker into the funnel. Open the screw clip to give a nominal flow of about 5 ml/min (about 1 drop per second) into a 600 ml collecting beaker and clean the material retained by the magnets by moving them up and down over a short length of tube so as to agitate the material and release trapped impurities. Add water to the funnel as necessary to keep the retained magnetic material immersed at all times. If choking occurs which causes a cessation of flow and prevents efficient cleaning of the material, release sufficient material into the collecting beaker to re-establish flow before further cleaning is attempted.

During the cleaning operation, observe the impurities as they are released and continue the washing and agitation until perfectly clear water is discharged.

NOTE — Any excessive frothing can be controlled by the judicious addition of methylated spirits.

7.3.3 Close the screw clip, remove the magnets so that the magnetic material (magnetics) is released. Open the screw clip and wash the magnetics into a 100 ml beaker. After allowing the magnetics to settle, decant the water while holding a strong magnet on the base of the beaker to prevent any loss of magnetics from the beaker.

7.3.4 Reposition the magnet, refill the apparatus with water and open the screw clip to start the flow. Slowly transfer the liquid and solids from the first 600 ml beaker into the funnel and allow the mixture to run slowly through the apparatus into a second 600 ml beaker while collecting magnetics on the magnets.

7.3.5 Examine the contents of the 600 ml collecting beaker to ensure that no magnetics are present by placing it on a strong magnet and observing the solids while the beaker is slowly moved. If there are no magnetics in the beaker, discard the contents. If magnetics are present, return the contents to the funnel, run them through the apparatus again into an empty 600 ml beaker, examine the contents as before and repeat this process until no magnetics appear in the collecting beaker.

7.3.6 Place an empty 600 ml beaker under the tube and clean the material retained by the magnets by adding water to the funnel and moving the magnets up and down, continuing until clear water is discharged. Recover the magnetics as described in 7.3.3, collecting them in the same 100 ml beaker.

7.3.7 Repeat steps 7.3.4, 7.3.5 and 7.3.6, using the liquid and solids collected in the 600 ml breaker during step 7.3.6 each time, until no material is retained by the magnets during step 7.3.4.

NOTE — Two further cycles are usually sufficient to complete the process.

7.3.8 Dry all the recovered magnetics to constant mass as described in 4.4.2, cooling in a desiccator and weighing to an accuracy of ± 1 mg.

7.4 Expression of results

The total magnetics content, expressed as a percentage by mass, is calculated from the formula

$$\frac{m_2}{m_1} \times 100$$

where

m_1 is the dry mass, in grams, of the prepared test portion;

m_2 is the dry mass, in grams, of the recovered magnetics.

Report the result to one decimal place.

8 Determination of relative density

8.1 Principle

Determination of the volume occupied by a known mass of sample using a density bottle and a suitable test liquid.

8.2 Test liquid

Any liquid with which the magnetite can be shown to have insignificant reaction or solubility may be used in the test.

NOTE — The preferred liquid is distilled water as this results in a more direct method less liable to measurement error. The use of industrial ethanol or denatured ethanol is recommended if distilled water is unsuitable.

8.3 Apparatus

8.3.1 Density bottles (two), of capacity 50 ml.

8.3.2 Balance, capable of weighing up to 200 g to an accuracy of ± 1 mg.

8.3.3 De-aeration vessel, desiccator or bell jar on a steel plate.

8.3.4 Thermostatically controlled water bath, capable of being maintained at a temperature of $25 \text{ }^{\circ}\text{C} \pm 0,1 \text{ }^{\circ}\text{C}$.

8.3.5 Laboratory air-oven, capable of being maintained at a temperature in the range $105 \text{ }^{\circ}\text{C}$ to $110 \text{ }^{\circ}\text{C}$.

8.3.6 Vacuum pump.

8.4 Sample

The determination shall be carried out on material dried to constant mass at a temperature in the range $105 \text{ }^{\circ}\text{C}$ to $110 \text{ }^{\circ}\text{C}$.

NOTE — Any moisture present in the sample will result in large errors in the determined relative density.

8.5 Temperature

The determination shall normally be carried out at a temperature of $25\text{ }^{\circ}\text{C} \pm 0,1\text{ }^{\circ}\text{C}$.

NOTES

- 1 If the ambient temperature is greater than $25\text{ }^{\circ}\text{C}$, an appropriate temperature may be selected and the result reported at that temperature.
- 2 If temperature control facilities are not available, the determination may be carried out at ambient temperature and the result reported at that temperature. Under these circumstances the accuracy of the method is much reduced.

8.6 Number of determinations

Duplicate determinations shall be made and the test shall be repeated if the difference between the determinations is greater than 0,02.

8.7 Test portion

Take a test portion of not less than 15 g from the dried sample by sub-sampling in accordance with 3.2.

8.8 Procedure

Weigh a density bottle (8.3.1) complete with stopper; then transfer the test portion to the bottle, re-insert the stopper and re-weigh. Both weighings shall be to an accuracy of $\pm 1\text{ mg}$.

Add liquid to half fill the density bottle and transfer to the de-aeration vessel (8.3.3). Gradually reduce the pressure within the vessel to less than 2,5 kPa (25 mbar) and maintain at reduced pressure until de-aeration is complete. This process usually takes about 15 min. Take care, particularly in the early stages of evacuation, to prevent the loss of contents as the liquid boils. It is useful, near completion of the evacuation, to tap the de-aeration vessel lightly to release trapped air from the magnetite. On completion, allow air to re-enter the vessel gradually.

Remove the density bottle from the de-aeration vessel and add de-aerated liquid until it is almost full.

NOTE — The bottle is not completely filled, to allow for subsequent expansion during temperature equalization.

Support the density bottle in the water bath (8.3.4), controlled at $25\text{ }^{\circ}\text{C} \pm 0,1\text{ }^{\circ}\text{C}$, for at least 45 min. While the bottle is still in the bath, insert the stopper into the bottle, exercising care not to trap any air bubbles; then remove any excess liquid from the top of the stopper using filter paper. Remove the bottle from the water bath and remove the water from the exterior of the bottle, taking great care not to force liquid from the bottle either by external pressure or by heating the bottle in the hand. Weigh the bottle complete with stopper and its contents to an accuracy of $\pm 1\text{ mg}$.

8.9 Calibration

Calibrate the density bottle with distilled water, and with the liquid medium used in the test if other than distilled water. The calibration procedure is essentially that of 8.8 except that

the bottle is filled with the relevant liquid only. Carry out the calibration with the utmost care, following as exactly as possible the method used in 8.8 with regard to weighings, de-aeration, temperature equalization and drying of the bottle. The time between removing the bottle from the bath and the final weighing shall be as near as possible the same in both cases, to minimize possible errors due to convection effects from a warm bottle and evaporation of the liquid contents of the bottle and of any remaining surface moisture on the bottle.

8.10 Expression of results

The relative density of magnetite at $25\text{ }^{\circ}\text{C}$ with respect to water at $25\text{ }^{\circ}\text{C}$ is calculated from the equation

$$d_{25\text{ }^{\circ}\text{C}}^{25\text{ }^{\circ}\text{C}} = \frac{m_2 - m_1}{(m_2 - m_1) - (m_3 - m_5)} \times \frac{m_5 - m_1}{m_4 - m_1}$$

where

m_1 is the mass, in grams, of the density bottle and stopper;

m_2 is the mass, in grams, of the density bottle and stopper with the test portion;

m_3 is the mass, in grams, of the density bottle and stopper with the test portion and test liquid;

m_4 is the mass, in grams, of the density bottle and stopper with distilled water;

m_5 is the mass, in grams, of the density bottle and stopper with the test liquid.

If distilled water only is used in the determination, the above equation reduces to

$$d_{25\text{ }^{\circ}\text{C}}^{25\text{ }^{\circ}\text{C}} = \frac{m_2 - m_1}{(m_2 - m_1) - (m_3 - m_4)}$$

Calculate the mean of duplicate determinations and report the result to two decimal places.

9 Determination of total iron content

Determine the total iron content by the titrimetric method specified in ISO 2597 (Method 1) and report the result as a percentage by mass to an accuracy of $\pm 0,5\%$ absolute.

The principle of the method is as follows.

Fusion with alkali and dissolution of the melt with water. Filtration, with the filtrate being discarded. Dissolution of the residue in hydrochloric acid. Reduction of trivalent iron in the solution, using tin(II) chloride. Oxidation of excess reductant with mercury(II) chloride. Titration of the reduced iron with potassium dichromate solution, using sodium diphenylaminesulfonate as indicator.

10 Determination of iron(II) content

10.1 Principle

Determination of the iron(II) content by dissolving the test portion in hydrochloric acid under an atmosphere of nitrogen; titration of the iron(II) taken into solution with standard potassium dichromate solution.

10.2 Reagents

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

10.2.1 Hydrochloric acid, dilute solution.

Dilute hydrochloric acid solution (ρ 1,18 g/ml) 1 + 1 with water.

10.2.2 Orthophosphoric acid, dilute solution.

Dilute orthophosphoric acid solution (ρ 1,75 g/ml) 1 + 9 with water.

10.2.3 Potassium dichromate solution, standard volumetric solution, $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0,017 \text{ mol/l}$.

Pulverize 6 g of potassium dichromate in an agate mortar and dry at a temperature of approximately 105 °C; cool in a desiccator. Weigh 4,904 g, dissolve in water and dilute to 1 000 ml in a one-mark volumetric flask.

10.2.4 Sodium diphenylaminesulfonate indicator, 20 g/l solution.

10.3 Apparatus

Ordinary laboratory apparatus, and

10.3.1 Dissolution/titration apparatus (see figure 4), comprising the following items.

10.3.1.1 Round bottom, two-neck flask, of capacity 250 ml.

10.3.1.2 Dropping funnel, of capacity 25 ml.

10.3.1.3 Water cooled condenser.

10.3.1.4 Gas inlet tube.

10.3.2 Micro-Bunsen burner.

10.3.3 Gas flow meter, calibrated for 500 ml/min gas flow.

10.3.4 Nitrogen supply.

10.3.5 One-mark pipettes, of capacity 25 ml and 100 ml, complying with ISO 648.

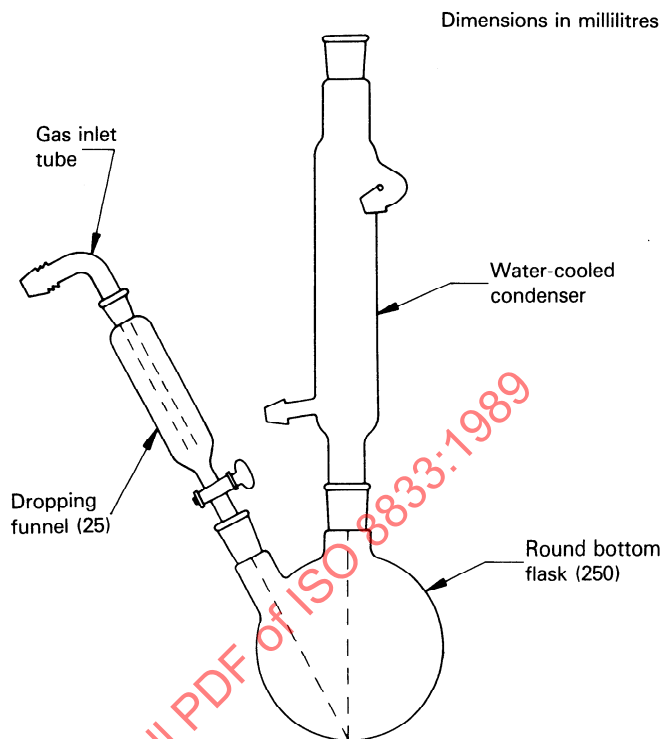


Figure 4 — Dissolution/titration apparatus

10.4 Procedure

Weigh, to an accuracy of $\pm 1 \text{ mg}$, approximately 0,25 g of the air-dried sample and transfer to the reaction flask (10.3.1.1). Concurrently carry out an air-dried moisture determination as specified in 4.5.2 to allow correction of the iron(II) result to the dry basis.

Attach the flask to the condenser (10.3.1.3) and place the dropping funnel (10.3.1.2) in the side neck. Attach the gas inlet tube (10.3.1.4) to the dropping funnel and connect to the nitrogen supply (10.3.4). Purge the apparatus for at least 5 min with a nitrogen flow of 500 ml/min.

Close the dropping funnel stopcock, remove the gas inlet tube and add 25 ml of the hydrochloric acid solution (10.2.1) to the dropping funnel.

Reconnect the nitrogen supply, open the stopcock and allow the nitrogen flow to push the acid into the flask. Continue to purge with nitrogen at 500 ml/min.

Boil gently for 10 min with the nitrogen flowing. Remove from the burner (10.3.2), disconnect the flask from the condenser and, while still purging with nitrogen, cool the flask in a water bath.

When cool, add 100 ml of the orthophosphoric acid solution (10.2.2) and five drops of the sodium diphenylaminesulfonate indicator solution (10.2.4). Continue purging with nitrogen additions.

Titrate immediately with standard potassium dichromate solution (10.2.3) until one drop changes the solution colour from green to purple. Continue purging with nitrogen during the titration.