International Standard 587

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Solid mineral fuels — Determination of chlorine using Eschka mixture

Combustibles minéraux solides — Dosage du chlore au moyen du mélange Eschka

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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 587 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels.*

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 587-1974), which had been approved by the member bodies of the following countries :

Australia
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No member body had expressed disapproval of the document.

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INTERNATIONAL STANDARD **ISO 587-1981 (E)**

Solid mineral fuels — Determination of chlorine using Eschka mixture

1 Scope and field of application

This International Standard specifies a method of determining the amount of chlorine in all solid mineral fuels, using Eschka mixture. An alternative method, involving combustion at high temperature, is specified in ISO 352.

2 References

ISO 352, *Solid mineral fuels — Determination of chlorine — High temperature combustion method.*

ISO 1170, *Coal and coke — Calculation of analyses to different bases.*

3 Principle

The sample is ignited in intimate contact with Eschka mixture in an oxidizing atmosphere to remove combustible matter and to convert the chlorine to alkali chlorides. These are extracted with nitric acid or water and determined by titration by either the Volhard or the Mohr procedure.

4 Reagents

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

4.1 Eschka mixture.

Mix two parts by mass of light calcined magnesium oxide with one part of anhydrous sodium or potassium carbonate. The mixture shall entirely pass a test sieve of 200 µm aperture.

4.2 Nitric acid, chlorine-free, ϱ 1,42 g/ml.

For the Volhard titration

4.3 Nitrobenzene.

Store this reagent in a dark glass bottle.

4.4 Silver nitrate, standard reference solution, $c(AgNO_3) = 0{,}025$ mol/l.

Heat crushed crystalline silver nitrate at 125 °C for 2 to 3 h. Dissolve 4,247 g in water and dilute to 1 000 ml. Store in an amber glass bottle.

4.5 Potassium thiocyanate, standard volumetric solution, $c(KSCN) = 0{,}025$ mol/l.

Dissolve 3,5 g of potassium thiocyanate in water and dilute to 1 000 ml. Standardize by titration against the silver nitrate solution (4.4) and adjust to exact equivalence.

4.6 Saturated solution of iron(III) alum [ammonium iron(III) sulphate] indicator.

Saturate 100 ml of water with approximately 125 g of iron(III) alum $[(NH_4)_2SO_4.\ Fe_2(SO_4)_3.\ 24H_2O]$ and add sufficient nitric acid (3.2) to remove the brown colour.

For the Mohr titration

4.7 Silver nitrate, standard volumetric solution, $c(AgNO_3) = 0{,}050$ mol/l.

Weigh 8,494 g of silver nitrate, dried as in 4.4, dissolve in water and dilute to 1 000 ml.

4.8 Potassium chromate, indicator solution.

Dissolve 5 g of potassium chromate in 100 ml of water.

5 Apparatus

Ordinary laboratory apparatus : Graduated glassware shall conform to the relevant International Standards drawn up by ISO/TC 48, *Laboratory glassware and related apparatus.*

5.1 Electrically heated muffle furnace, with a zone of substantially uniform temperature at 675 ± 25 °C and a ventilation rate of 4 to 6 air changes per minute.

NOTE — The necessary rate of air change can be obtained by using a suitably designed muffle furnace and may be checked by means of a pitot-static tube.

5.2 Crucibles, of porcelain or silica, capacity approximately 25 ml.

5.3 Insulating plate, 6 mm thick, of silica or other suitable material, which fits easily in the muffle.

5.4 Balance, accurate to 0,1 mg.

6 Preparation of sample

The coal or coke used for the determination of chlorine is the analysis sample ground to pass a sieve of 200 µm aperture. If necessary, expose the sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere.

Before commencing the determination, mix the air-dried sample thoroughly for at least 1 min, preferably by mechanical means.

7 Procedure

7.1 Decomposition of test portion

Weigh, to the nearest 0,1 mg, a test portion of about 1 g (see note 1) in a scoop and transfer it to a crucible (5.2) containing 3 g of the Eschka mixture (4.1). Mix intimately, using a small metal spatula, and cover with a further 2 g of the Eschka mixture.

Place the crucible on the insulating plate (5.3), insert both into the muffle furnace (5.1) at 675 ± 25 °C (see note 2) and maintain at this temperature for 2 h (see note 3). Withdraw the crucible and allow it to cool.

Complete the determination by either the Volhard or the Mohr procedure (see note 4).

NOTES

1 When the chlorine content is less than 0,1 % (m/m), the mass of the test portion should be increased to 2 g.

2 Ash and chlorine determination should not be carried out in the same muffle at the same time.

3 If desired, the sample may be placed in a cold muffle, which is then heated to 675 ± 25 °C and maintained at this temperature for 2 h.

4 If the expected chlorine content is very small, a known volume of standard chloride solution may be added at this stage and the final calculation adjusted accordingly.

7.2 Volhard titration

Transfer the incinerated mixture to a beaker, wash the crucible with about 125 ml of hot water and add the washings to the beaker. Cautiously add 20 ml of the nitric acid (4.2) and cover the beaker with a clock glass, swirling or stirring the contents, if necessary, to help dissolution.

If necessary (see the note), filter the solution through a rapid filtering, hardened, acid-washed filter paper, collecting the filtrate in a conical flask. Wash the paper with a small quantity of hot water (for example four lots of 5 to 10 ml) and add 20,0 ml of the silver nitrate solution (4.4) to the flask. Allow to stand for 15 min, and then, if necessary, cool to ambient temperature. Add 5 to 10 ml of the nitrobenzene (4.3), shake the solution for 1 min, add 8 to 10 drops of the iron(III) alum indicator solution (4.6) and titrate with the potassium thiocyanate solution (4.5). The end-point is reached when the solution becomes faintly orange-pink in colour.

NOTE — Filtration is usually unnecessary when using 1 g test portions of low ash fuels, but is required when dealing with test portions larger than 1 g (see 7.1, note 1) or with high ash coals.

7.3 Mohr titration

Transfer the incinerated mixture to a beaker, wash the crucible with hot water, collect the washings in the beaker, and crush the residue in the beaker with a flat-ended glass rod. Heat the solution to boiling point and filter, using a filter paper pad or rapid filtering paper, collecting the filtrate in a conical flask. Wash the residue with five 5 ml portions of hot water, collecting the washings in the flask. Neutralize the solution with the nitric acid (4.2), add 10 drops of the potassium chromate indicator (4.8) and titrate with the silver nitrate solution (4.7). The end-point is indicated by the first appearance of a permanent brown coloration.

7.4 Blank test

Carry out a blank test on 5 g of the Eschka mixture (4.1) incinerated in the muffle furnace at the same time as the test portions, the blank being subsequently treated in the same manner and titrated to the same end-point as an actual sample.[1] This assesses both the chlorine in the reagents and any contamination from the laboratory atmosphere. The latter shall not be quantitatively significant.

8 Expression of results

8.1 Volhard titration

The chlorine (Cl) content of the sample as analysed[2], expressed as a percentage by mass, is given by the formula

$$\mathrm{Cl} = \frac{3{,}545\, c\, (V_2 - V_1)}{m}$$

where

V_1 is the volume, in millilitres, of the potassium thiocyanate solution (4.5) used in the determination (7.2);

V_2 is the volume, in millilitres, of the potassium thiocyanate solution (4.5) used in the blank test (7.4);

c is the true concentration, expressed in moles per litre, of the potassium thiocyanate solution (4.5);

m is the mass, in grams, of the test portion.

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1) Recognition of the end-point colour is facilitated by comparison with a previously titrated blank solution.

2) Calculation of the results to other bases is dealt with in ISO 1170.