INTERNATIONAL STANDARD 587

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Coal and coke — Determination of chlorine using Eschka mixture

Charbon et coke — Dosage du chlore au moyen du mélange Eschka

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 587 and found it technically suitable for transformation. International Standard ISO 587 therefore replaces ISO Recommendation R 587-1967 to which it is technically identical.

ISO Recommendation R 587 was approved by the Member Bodies of the following countries :

Australia	Egypt, Arab Rep. of	Poland
Austria	France	Romania
Belgium	Germany	South Africa, Rep. of
Brazil	India	Switzerland
Canada	Italy	Turkey
Chile	Japan	United Kingdom
Colombia	Korea, Rep. of	U.S.A.
Czechoslovakia	Netherlands	U.S.S.R.
Denmark	New Zealand	

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 587 into an International Standard :

Czechoslovakia

Printed in Switzerland

Coal and coke — 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 hard coal, brown coal, lignite, and coke, using Eschka mixture. An alternative method, involving combustion at high temperature, is specified in ISO 352.[1)]

2 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.

3 REAGENTS

All reagents shall be of analytical reagent quality and distilled water shall be used throughout.

3.1 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 0,2 mm aperture.

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

Volhard titration

3.3 Nitrobenzene

Store in a dark glass bottle.

3.4 Silver nitrate 0,025 N solution

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 a bottle of amber glass.

3.5 Potassium thiocyanate solution

Dissolve 3,5 g of potassium thiocyanate in water and dilute to 1 000 ml. Standardize by titration against the silver nitrate solution (3.4) and adjust to exact equivalence.

3.6 Saturated solution of iron(III) alum (ammonium iron(III) sulphate) indicator

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

Mohr titration

3.7 Silver nitrate 0,050 N solution

Weigh 8,494 g of silver nitrate, dried as in 3.4, dissolve in water and dilute to 1 000 ml.

3.8 Potassium chromate indicator solution

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

4 APPARATUS

All graduated apparatus shall be of the best analytical quality obtainable.

4.1 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 (see note in 4.4).

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

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

4.4 Balance, sensitive to 0,1 mg.

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.

1) ISO 352, *Hard coal and coke — Determination of chlorine — High temperature combustion method.*

5 PREPARATION OF SAMPLE

The sample used for the determination of chlorine is the analysis sample ground to pass a sieve of 0,2 mm 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.

6 PROCEDURE

6.1 Decomposition of test portion

Weigh accurately a test portion of about 1 g (see note 1) in a scoop and transfer it to a crucible containing 3 g of the Eschka mixture (3.1). Mix intimately, using a small metal spatula, and cover with a further 2 g of the Eschka mixture (3.1).

Place the crucible on the insulating plate, insert both into the muffle 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 under 0,1 %, the mass of the test portion shall be increased to 2 g.

2 Ash and chlorine determinations shall 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 amount of standard chloride solution may be added at this stage and the final calculation adjusted accordingly.

6.2 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 (3.2) and cover the beaker with a clock glass, swirling or stirring the content, if necessary, to help dissolution.

If necessary (see note), filter the solution into a conical beaker through a rapid filtering, hardened, acid-washed filter paper. Wash the paper with a small quantity of hot water (say four lots of 5 to 10 ml) and add 20,0 ml of the silver nitrate solution (3.4) to the beaker. Allow to stand for 15 min, and then, if necessary, cool to room temperature. Add 5 to 10 ml of the nitrobenzene (3.3), shake the solution for 1 min, add 8 to 10 drops of the iron(III) alum indicator solution (3.6) and titrate with the potassium thiocyanate solution (3.5). 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 6.1, note 1) or with high ash coals.

6.3 Mohr titration

Transfer the incinerated mixture to a beaker, wash the crucible with hot water, add the washings to 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, into a conical beaker. Wash the residue with five 5 ml portions of hot water, collecting the washings in the beaker. Neutralize the solution with the nitric acid (3.2), add 10 drops of the potassium chromate indicator (3.8) and titrate with the silver nitrate solution (3.7). The end-point is indicated by the first appearance of a permanent brown coloration.

7 BLANK TEST

Carry out a blank test on 5 g of the Eschka mixture (3.1) incinerated in the muffle 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 should 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

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

where

m is the mass, in grams, of sample taken;

V_1 is the volume, in millilitres, of potassium thiocyanate solution used in the determination;

V_2 is the volume, in millilitres, of potassium thiocyanate solution used in the blank test;

T is the normality of the potassium thiocyanate solution (i.e. 0,025 if the solution is exactly N/40).

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.