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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 587

DETERMINATION OF CHLORINE IN COAL AND COKE USING ESCHKA MIXTURE

1st EDITION

July 1967

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BRIEF HISTORY

The ISO Recommendation R 587, *Determination of Chlorine in Coal and Coke using Eschka Mixture*, was drawn up by Technical Committee ISO/TC 27, *Solid Mineral Fuels*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1951 and led, in 1961, to the adoption of a Draft ISO Recommendation.

In March 1964, this Draft ISO Recommendation (No. 677) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in July 1967, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 587 July 1967

DETERMINATION OF CHLORINE IN COAL AND COKE USING ESCHKA MIXTURE

1. SCOPE

This ISO Recommendation describes a method of determining the amount of chlorine in hard coals, cokes, brown coals and lignites, using Eschka mixture. Alternative methods, involving combustion in a calorimetric bomb or at high temperature, are described in separate ISO Recommendations.*

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 should be of analytical reagent quality and distilled water should 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 should entirely pass a test sieve of 0.2 mm aperture.

3.2 **Nitric acid,** chlorine-free, relative density $d = 1.42$.

Volhard titration

3.3 **Nitrobenzene.** Store in a dark glass bottle.

3.4 **Silver nitrate solution 0.025 N.** Heat crushed crystalline silver nitrate at 125 °C for 2 to 3 hours. Dissolve 4.247 g in water and dilute to 1000 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 1000 ml. Standardize by titration against the silver nitrate solution (3.4) and adjust to exact equivalence.

3.6 **Saturated solution of ferric alum (ammonium ferric sulphate) indicator.** Saturate 100 ml of water with ferric 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 solution 0.050 N.** Weigh 8.494 g of silver nitrate, dried as in clause 3.4, dissolve in water and dilute to 1000 ml.

3.8 **Potassium chromate indicator solution.** Dissolve 5 g of potassium chromate in 100 ml of water.

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* See ISO Recommendations R 350, *Determination of Chlorine in Coal by the Bomb-combustion Method,*
R 352, *Determination of Chlorine in Coal by the High Temperature Combustion Method.*

4. APPARATUS

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

4.1 **An 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 below).

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

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.

5. PROCEDURE

5.1 **Decomposition of test portion.** Before commencing the determination, mix the air-dried sample, ground to pass a sieve of 0.2 mm aperture, thoroughly for at least one minute, preferably by mechanical means.

Weigh accurately a test portion of about 1 g (see Note 1, page 7) 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, page 7) and maintain at this temperature for 2 hours (see Note 3, page 7). Check the temperature with a calibrated thermocouple to avoid over-heating. Withdraw the crucible and allow it to cool.

Complete the determination by either the Volhard or the Mohr procedure (see Note 4, page 7).

5.2 **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 contents if necessary, to help dissolution.

If necessary (see Note 5, page 7), 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 4 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 minutes, and then, if necessary, cool to room temperature. Add 5 to 10 ml of the nitrobenzene (3.3), shake the solution for 1 minute, add 8 to 10 drops of the ferric 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.

5.3 **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.