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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 350

DETERMINATION OF CHLORINE IN COAL BY THE BOMB-COMBUSTION METHOD

1st EDITION

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For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 350, *Determination of Chlorine in Coal by the Bomb-Combustion Method*, was drawn up by Technical Committee ISO/TC 27, *Solid Mineral Fuels*, the Secretariat of which is held by the British Standards Institution (B.S.I.).

Work on this question by the Technical Committee began in 1953 and led, in 1957, to the adoption of a Draft ISO Recommendation.

In May 1958, this Draft ISO Recommendation (No. 240) 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:

Austria
Belgium
Burma
Chile
Czechoslovakia
Denmark
Germany
Greece
India
Israel
Italy
Japan
Mexico
Netherlands
New Zealand
Poland
Portugal
Republic
of South Africa
Romania
Spain
United Kingdom
U.S.S.R.
Yugoslavia

One Member Body opposed the approval of the Draft: France.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in December 1963, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 350 December 1963

DETERMINATION OF CHLORINE IN COAL BY THE BOMB-COMBUSTION METHOD

1. PRINCIPLE

During combustion of the coal in the calorimetric bomb in the determination of calorific value, all the chlorine present is released and is found as chlorides in the bomb washings. It can, therefore, be readily determined titrimetrically (see note below).

If the determination is not associated with a determination of calorific value, precautions are taken to minimize ejection of ash from the crucible, by mixing the coal with Eschka mixture, and to promote rapid removal of all the acid gases from the vapour phase, by using ammonium carbonate solution instead of water in the bomb.

NOTE. — The chlorine may also be determined potentiometrically.

2. APPARATUS

All graduated apparatus should be of the best analytical quality obtainable, and the balance used should be sensitive to 0.1 mg.

2.1 **Calorimetric bomb,** which is not attacked by the acid gases produced, e.g. one of chromium-nickel-molybdenum austenitic steel, with the usual fittings for ignition as in the determination of calorific value, and of 300 ± 50 ml capacity. A slow-release valve is unnecessary.

2.2 **Crucible of platinum, nickel-chromium or silica,** approximately 25 mm in diameter and approximately 20 mm high.

2.3 **Cylindrical metal vessel,** such that the bomb will be fully immersed when approximately two litres of water are added.

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 by mass 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.

3.3 **Ammonium carbonate solution,** 10 per cent (weight/volume). Dissolve 10 g of ammonium carbonate in 100 ml of water. The solution should be freshly prepared as required.

3.4 Volhard titration

3.4.1 *Silver nitrate solution,* 0.025 N. Heat crushed crystalline silver nitrate at 125 °C for 2 to 3 hours. Dissolve 4.247 g of the dried silver nitrate in water and dilute to 1000 ml. Store in a bottle of amber glass.

3.4.2 *Potassium thiocyanate solution.* Dissolve approximately 3.5 g of potassium thiocyanate in water and dilute to 1000 ml. Standardize by titration against the silver nitrate solution (3.4.1) and adjust to exact equivalence.

3.4.3 *Nitrobenzene*, light in colour.

3.4.4 *Ferric alum (ammonium ferric sulphate) indicator solution.* Add just sufficient of the nitric acid (3.2) to a cold saturated solution of ferric alum to remove the brown colour.

3.5 Mohr titration

3.5.1 *Silver nitrate solution*, 0.050 N. Dissolve 8.494 g of dried silver nitrate, prepared as in clause 3.4.1 in water, and dilute to 1000 ml.

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

4. PROCEDURE

4.1 After a determination of calorific value

At the end of the determination of calorific value, remove the bomb from the cylindrical vessel, release the pressure, open the bomb and wash all the internal parts, including the crucible, with 50 ml of hot distilled water, collecting the washings in a filtration beaker. Add about 3 ml of the nitric acid (3.2), mix and filter, using a filter paper pad or a hardened rapid-filtering paper, into a conical filtration beaker. Using a 10 ml portion of hot water each time, wash the beaker and the filter four times.

Add 20.0 ml of the silver nitrate solution (3.4.1), mix and leave to stand for 15 minutes. Cool to room temperature. Add 5 to 10 ml of the nitrobenzene (3.4.3), shake for 1 minute, add 8 to 10 drops of the ferric alum solution (3.4.4) and titrate with the potassium thiocyanate solution (3.4.2). The end point of the determination is reached when the solution becomes faintly orange-pink in colour.

4.2 As a separate determination

4.2.1 *Preparation of solution.* Before commencing the determination, mix the air-dried sample of coal, ground to pass a sieve of 0.2 mm aperture, thoroughly for at least 1 minute, preferably by mechanical means. Weigh to the nearest 0.1 mg, about 1 g of the sample, mix intimately with 1 g of the Eschka mixture * (3.1) in a porcelain dish, and transfer to the crucible. Add 10 ml of the ammonium carbonate solution (3.3) to the calorimetric bomb.**

Stretch a fine platinum or nickel-chromium wire between the poles of the bomb and tie to it a piece of cotton thread, dropping the loose ends into the crucible. Assemble the bomb, fill with oxygen to 25 atm. pressure, completely immerse the bomb in water in the cylindrical vessel and fire the charge as in the determination of calorific value.

Ten minutes after firing, remove the bomb from the cylindrical vessel, release the pressure and open the bomb (see Note 1 below). Transfer the contents of the bomb to a filtration beaker, and rinse the crucible and all internal parts of the bomb with 50 ml of hot distilled water, collecting the washings in the filtration beaker.

The determination may be completed by either the Volhard or the Mohr titration (see Note 2 below).

4.2.2 *Volhard titration.* Add 5 ml of the nitric acid (3.2). A clear solution will usually be obtained (see Note 3 below) and can be used directly for titration. Proceed as stated in the second paragraph of clause 4.1.

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* A procedure devised by E. Strambi (see ISO Recommendation R 159, *Determination of Total Sulphur in Coal by the Strambi Method).*

** A procedure devised by Z. K. A. Moszynski (J. Appl. Chem., London, 1955, 5, 467).