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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 352

DETERMINATION OF CHLORINE IN COAL BY THE HIGH TEMPERATURE COMBUSTION METHOD

1st EDITION

December 1963

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Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 352, *Determination of Chlorine in Coal by the High Temperature 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. 242) 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	India	Portugal
Belgium	Israel	Republic
Burma	Italy	of South Africa
Chile	Japan	Romania
Czechoslovakia	Mexico	Spain
Denmark	Netherlands	United Kingdom
Germany	New Zealand	U.S.S.R.
Greece	Poland	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.

DETERMINATION OF CHLORINE IN COAL BY THE HIGH TEMPERATURE COMBUSTION METHOD

1. PRINCIPLE

Coal is burnt in a stream of oxygen, in a tube furnace at a temperature of 1250 or 1350 °C, and the acid gases (chlorine and oxides of sulphur) formed are absorbed in hydrogen peroxide. The acid solution is neutralized with sodium borate and the sodium chloride formed is converted by reaction with mercuric oxycyanide to sodium hydroxide, which is determined volumetrically.

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 **Furnace**, capable of heating a tube of approximately 28 mm external diameter over a length of approximately 15 cm to a maximum temperature of 1250 or 1350 °C. The furnace may, conveniently, be heated electrically, using either silicon carbide resistance rods (controlled by a variable transformer) or a resistance wire (controlled by a variable resistance).
- 2.2 **Aluminous porcelain tube**, of approximately 28 mm external diameter, 3 mm wall thickness and 65 cm length, which is gas-tight at the working temperature. A straight tube is most convenient and is used in conjunction with an adapter of fused silica having a bell-shaped end, which gives a narrow clearance with the inner wall of the heated tube, and a heat-resistant stopper (acrylonitrile or chloroprene is suitable). Alternatively, the tube may have, at the exit, a beak end, with a tubulure to enable condensation products to be washed out after a test; or a straight tube of aluminous porcelain may be used in conjunction with a borosilicate glass adapter, having a cap-shaped end which fits on the outer wall of the tube.
- 2.3 **Oxygen cylinder**, fitted with a needle valve to control the rate of flow of oxygen, and a flow meter to measure up to 500 ml/min. The oxygen should, as a precautionary measure, be passed through a U-tube packed with soda-asbestos.
- 2.4 **Combustion boats**, of iron-free, unglazed porcelain, 62.5 mm long, 12.5 mm wide and 10 mm deep.
- 2.5 **Heat-resistant wire**, about 1.5 mm thick and having a bent end to remove the boats from the tube.
- 2.6 **Silica pusher**, with a disk end for pushing the boat into the hot zone. The pusher passes through a T-piece fitted into the bung at the inlet end of the tube and is held in a rubber sleeve (see note below) which fits over the free arm of the T-piece. The sleeve prevents the escape of oxygen (which enters at the stem of the T) although permitting movement of the pusher.

NOTE. — The rubber sleeve should be changed periodically to avoid leakage.