

# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

## ISO RECOMMENDATION R 348

DETERMINATION OF MOISTURE  
IN THE ANALYSIS SAMPLE OF COAL  
BY THE DIRECT VOLUMETRIC METHOD

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## Amendment 1 – November 1971 – to ISO Recommendation R 348-1963

**Amendment 1  
to ISO Recommendation R 348-1963**

**DETERMINATION OF MOISTURE  
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BY THE DIRECT VOLUMETRIC METHOD**

**Section 2, PRINCIPLE**

Delete “or xylene” in the 1st, 2nd and 4th lines.  
Delete the 2nd paragraph.

**Section 3, APPARATUS**

Delete “or xylene” in the 4th line of clause 3.3.

**Section 4, REAGENTS**

Replace “, or” in the 2nd line by a full stop.  
Delete the 3rd line (4.2).  
Delete “either” and “or xylene” in the 1st line of the Note.

**Section 5, PROCEDURE**

Delete “or of the xylene (4.2)” in the 2nd line of the 2nd paragraph.  
Delete “employed” in the 3rd line of the 3rd paragraph.

**Section 6, CALCULATION AND EXPRESSION OF RESULTS**

Replace “The entraining . . . stated” by “The condition of saturation of the entraining reagent should be stated”.

**BRIEF HISTORY**

Amendment 1 to ISO Recommendation R 348-1963, *Determination of moisture in the analysis sample of coal by the direct volumetric method*, was drawn up by Technical Committee ISO/TC 27, *Solid mineral fuels*, the Secretariat of which is held by the British Standards Institution (BSI).

Draft ISO Recommendation No. 1931 was drawn up on this subject, and was circulated to all the ISO Member Bodies for enquiry in April 1970. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	India	Switzerland
Belgium	Iran	Thailand
Canada	Korea, Rep. of	Turkey
Chile	Netherlands	U.A.R.
Czechoslovakia	New Zealand	United Kingdom
Denmark	Poland	U.S.A.
France	Portugal	U.S.S.R.
Germany	South Africa, Rep. of	Yugoslavia
Greece	Sweden	

The following Member Body opposed the approval of the Draft :

Italy

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as AMENDMENT 1 to ISO Recommendation R 348-1963.

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## BRIEF HISTORY

The ISO Recommendation R 348, *Determination of Moisture in the Analysis Sample of Coal by the Direct Volumetric 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 1950 and led, in 1957, to the adoption of a Draft ISO Recommendation.

In February 1958, this Draft ISO Recommendation (No. 212) 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	Germany	Romania
Bulgaria	India	Spain
Burma	Italy	Turkey
Chile	Japan	United Kingdom
Czechoslovakia	Mexico	U.S.S.R.
Denmark	Poland	Yugoslavia
France	Portugal	

Six Member Bodies opposed the approval of the Draft:

Belgium, Canada, Netherlands,  
New Zealand, Republic of South Africa, U.S.A.

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.

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**DETERMINATION OF MOISTURE  
IN THE ANALYSIS SAMPLE OF COAL  
BY THE DIRECT VOLUMETRIC METHOD**

**1. INTRODUCTION**

Two direct methods are available for the determination of moisture in the analysis sample of coal, namely

- (1) a gravimetric method,\* in which the moisture is determined in an atmosphere of nitrogen, and
- (2) a volumetric method, in which the moisture is determined by distillation.

This ISO Recommendation describes the direct volumetric method.

Since coal is hygroscopic, its moisture will vary with change of humidity of the atmosphere, and the moisture in the analysis sample is, therefore, determined whenever portions are weighed out for other analytical determinations, e.g. volatile matter, calorific value, carbon and hydrogen, etc. If all the portions taken for analysis are weighed out on the same day and at about the same time, and if the analyses are proceeded with without delay, one determination of moisture will suffice.

**2. PRINCIPLE**

Coal is heated under reflux conditions with boiling toluene or xylene. The moisture from the coal is entrained by the toluene or xylene vapour and carried to a condenser fitted with a graduated receiver. The water then separates in the receiver, to form the lower layer, whilst the excess toluene or xylene is returned to the distillation flask by means of an overflow. The moisture in the coal is calculated from the mass of the sample and the amount of water collected.

The results obtained using toluene and xylene may not be identical for all coals, but any differences should be within the tolerances of the method (see section 7).

**3. APPARATUS**

All graduated apparatus should be of the best analytical quality obtainable and the balance used should be sensitive to 10 mg.

- 3.1 **Distillation flask**, of 500 ml minimum capacity.
- 3.2 **Condenser**, of 200 mm minimum length, fitted at its lower end with an extended lip to direct the distillate into the receiver along its axis without touching the sides.

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\* See ISO Recommendation R 331, *Determination of Moisture in the Analysis Sample of Coal by the Direct Gravimetric Method*.

**3.3 Receiver**, for the condensed water, graduated in tenths of a millilitre.

The condenser, the receiver and the distillation flask are fitted together by means of ground glass joints (see note below). An overflow tube connected to the receiver or to the lower portion of the condenser permits the return of condensed toluene or xylene to the distillation flask. The condenser may be fitted to condense either an upward or a downward vapour stream.

NOTE. — It is important that the receiver and the condenser should be clean. To ensure this, they should be treated with a cleansing reagent, such as a strong solution of potassium dichromate in sulphuric acid.

**3.4 Glass tubing**. Pieces about 5 mm in diameter and 5 mm long, with sharp edges.

**3.5 Spray tube**. Glass tube through which reagent can be supplied to wash down the inner surface of the condenser (this precaution is required only when an upward-flow condenser is employed).

#### 4. REAGENTS

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

**4.1 Toluene** (see note below). Boiling point 110 °C, or

**4.2 Xylene** (see note below). Boiling point range from 135 to 140 °C.

NOTE. — In view of the low solubility of water in either toluene or xylene, it can be shown that only a very small possibility of error in the determination may arise from variation in the condition of saturation of the entraining reagent. In order to reduce this error to insignificance, however, it is recommended as a precaution that the reagent should be used in the same condition during use as during calibration of the apparatus.

#### 5. PROCEDURE

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 accurately about 100 g (see Note 1 below) of the sample and transfer to the distillation flask. Add 200 ml of the toluene (4.1) or of the xylene (4.2) in such a way that any coal adhering to the neck or sides of the flask is washed down by the reagent. Place two or three pieces of glass tubing in the flask to prevent violent ebullition and assemble the apparatus. Heat sufficiently rapidly to keep the contents of the flask boiling briskly (see Note 2 below).

Continue the distillation until no further water collects in the graduated receiver. If an upward condenser is used, wash down any drops of water adhering to the inner surface of the condenser or to the upper walls of the graduated receiver with the reagent employed, using the spray tube. Continue the distillation for a sufficient time to ensure that any water washed back into the distillation flask has been entrained and carried over into the graduated receiver.

Standardize each apparatus by distilling a series of accurately measured amounts of water, covering the range of moisture contents in the fuels likely to be encountered. Plot a graph, showing the millilitres of distilled water added against the millilitres of water recovered and use it to correct the volume of water obtained from each sample.