

# Brown coals and lignites – Method of extraction for the determination of sodium and potassium soluble in dilute hydrochloric acid

Charbons bruns et lignites – Méthode d'extraction en vue des dosages du sodium et du potassium solubles dans l'acide chlorhydrique dilué iTeh STANDARD PREVIEW

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Descriptors: coal, lignite, chemical analysis, extraction, sodium, potassium.

# 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 1952 and found it technically suitable for transformation. International Standard ISO 1952 therefore replaces ISO Recommendation R 1952-1971 to which it is technically identical.

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ISO Recommendation R 1952 was approved dby 7 fined Member 9 Bodies 6 of the following countries :

Canada	India	Sweden
Chile	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Denmark	New Zealand	Turkey
Egypt. Arab Rep. of	Poland	United Kingdom
France	Portugal	U.S.A.
Germany	Romania	U.S.S.R.
Greece	South Africa, Rep. of	Yugoslavia

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1952 into an International Standard.

○ International Organization for Standardization, 1976 •

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# Brown coals and lignites – Method of extraction for the determination of sodium and potassium soluble in dilute hydrochloric acid

# **1 SCOPE AND FIELD OF APPLICATION**

This International Standard specifies a method of extraction for the determination, in brown coals and lignites, of sodium and potassium occurring as inorganic salts or as alkali humates which are soluble in dilute hydrochloric acid. These forms of alkali may cause difficulties in combustion of certain brown coals and lignites (salt coals).

This International Standard standardizes the extraction procedure only; the determination of sodium and potassium in the extract may be carried out by any accurate method.

# iTeh STANDARD

# 2 REFERENCE

ISO 1015, Brown coals and lignites **Standards** moisture content – Direct volumetric method.

# ISO 1952:1976

#### 3 PRINCIPLE

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The sample of brown coal or lignite is extracted with boiling 0,005 N hydrochloric acid<sup>1</sup>) and the extract centrifuged. The sodium and potassium in solution are determined by any precise analytical method (for example, gravimetric method or flame photometric method).

#### **4 REAGENTS**

During the analysis, use only reagents of recognized analytical grade and only distilled or water of at least equivalent purity.

4.1 Hydrochloric acid, 0,005 N solution.

**4.2** Ethanol, 95 % (V/V).

# 5 APPARATUS

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

5.1 One-mark volumetric flask, capacity 250 ml.

**5.2 Laboratory centrifuge**, capable of operation at about 2 000 rev/min.

5.3 Polyethylene bottle, minimum capacity 250 ml.

5.4 Balance, accurate to 0,1 mg.

# 6 SAMPLE

Spread the laboratory sample on a tray and allow it to attain approximate moisture equilibrium with the atmosphere. Crush the sample so that it passes a 212  $\mu$ m square mesh sieve. The analysis sample thus prepared shall be stored in a stoppered container filled to more than 80 % of its capacity.

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Weigh accurately  $1.5 \pm 0.05$  g of the sample and transfer to a 250 ml beaker. Wet the sample with 3 ml of the ethanol (4.2), add 100 ml of the hydrochloric acid (4.1) and boil slowly for 15 min. The beaker shall be uncovered, to allow the alcohol to evaporate. Cool the suspension, transfer quantitatively into centrifuge containers and centrifuge for 5 min at 2 000 rev/min. Decant the solution into the one-mark volumetric flask (5.1) (see note), transfer the residue into the original beaker using 100 ml of the hydrochloric acid, boil again for 15 min and centrifuge as before. Decant the solution into the same volumetric flask, wash the residue with warm hydrochloric acid (4.1), using the centrifuge for separation, and transfer all the washings to the volumetric flask. Cool the contents of the flask to room temperature and make up to the mark with the hydrochloric acid. Store the solution in the polyethylene bottle (5.3).

Determine the sodium and potassium in the solution by any accurate method (for example gravimetric method or flame photometric method).

Determine the moisture content on a separate portion of the sample by the method specified in ISO 1015.

<sup>1)</sup> See A. Lissner and W. Goebel, Freiberger Forschungshefte A 203, 1961.

Calculate the sodium and potassium contents on a moisture-free basis, using formulae suitable for the methods adopted for the determinations.

NOTE – The solution may be light yellow in colour, because of the presence of organic acids; however, this does not affect the determination significantly.

# 8 PRECISION OF THE METHOD

Element	Maximum acceptable differences between results		
determined	Repeatability	Reproducibility	
Sodium	0,04 % absolute	0,05 % absolute	
Potassium	0,02 % absolute	0,03 % absolute	

### 8.1 Repeatability

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator with the same apparatus and analytical procedure on two representative portions taken from the same analysis sample, should not differ by more than the adjacent value.

### 8.2 Reproducibility

The means of the results of duplicate determinations, carried out in each of two laboratories on representative portions taken from the same sample after the last stage of sample preparation, should not differ by more than the adjacent value.

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