

GRAVIMETRIC METHOD FOR DETERMINING INCOMBUSTIBLE CONTENT FOR DUST SAMPLES

ANALYTE:	INCOMBUSTIBLE CONTENT
MATRIX:	COAL MINE DUST
PROCEDURE:	GRAVIMETRIC
DATE ISSUED:	08/29/1976
DATE REVISED:	03/24/2003
METHOD NUMBER:	MH-102
RANGE:	0 – 100 PERCENT
PRECISION:	± 0.2 PERCENT
CLASSIFICATION:	OPERATIONAL

1. Principle of the Method

1.1 Dust samples are collected as outlined in the Coal Mine Health and Safety Inspection Manual.

1.2 Samples are analyzed utilizing Slow-Ashing Techniques.

2. Range and Sensitivity

The Range of the method is 0 – 100 percent and the sensitivity is 0.01 percent.

3. Interferences

Coals of high iron content may present some difficulty in ignition to constant weight because of the oxidation and reduction of iron oxides.

4. Precision and Accuracy

The precision of the method is ± 0.2% and the accuracy is ± 0.5%.

5. Advantages and Disadvantages of the Method

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The major advantage is the simplicity of the method and the primary disadvantage, the time required to completely ash the sample.

6. Apparatus

6.1 Sampling Equipment

The sampling equipment for collection of coal mine dust samples consists of the following:

6.1.1 Sampling scoop – approximately 6 inches wide and 4-1/2 inches long.

6.1.2 Receiving pan with separate 10-mesh screen – approximately 7 inches by 7 inches.

6.1.3 A piece of oilcloth or rubberized cloth about 2 feet square.

6.1.4 Containers for samples (plastic bags) and tags for identification.

6.2 Analytical Balance with 0.1-milligram sensitivity.

6.3 Moisture Oven capable of being controlled at 110 degrees Centigrade.

6.4 Furnace with adequate air circulation and capable of having its temperature regulated at 515 degrees Centigrade.

6.5 Porcelain Crucibles

A capsule type, 22.5 millimeter in depth and 42.5 millimeter in diameter is recommended.

7. Quality Assurance Standards

7.1 The proper operation of the furnace should be checked periodically with a coal sample of known ash content and a sample of pure sodium or calcium carbonate. A one gram sample of coal should be completely ashed in approximately 2.5 hours at 515°C, and there should be no significant loss in weight of the pure carbonate sample in 2.5 hours at 515°C.

7.2 To document analytical precision, a replicate analysis should be made on at least one randomly selected sample, for each batch of mine dust samples analyzed.

8. Procedure

8.1 Collection and Shipping of Samples

Samples are collected and shipped as outlined in the Coal Mine Health and Safety Inspection Manual.

8.2 Analysis of Samples

8.2.1 Prepare the sample for analysis by passing through a No. 20 sieve. In sieving, the entire sample should be shaken until it is evident that no more of the coarse particles will pass through into the pan beneath. Transfer screened material to a storage can.

8.2.2 Wet samples that must be dried before they will pass through the sieve are processed in the following manner. The entire sample must be weighed carefully before and after air drying; the moisture lost in drying should be included with the incombustible content of the gravimetric determination.

8.2.3 Moisture

Place approximately 0.5 gram in a pre-weighed porcelain crucible. Heat for 1 hour at 110°C ($\pm 2^\circ\text{C}$). Remove from oven, cool and weigh rapidly on analytical balance.

8.2.4 Ash + Carbonates

Place the crucible containing the residue from the moisture determination in a cold furnace. Adjust the temperature to 515°C. Hold at this temperature for 2.5 hours (total time with warm-up is 4.0 hours). Remove the crucible from the furnace, cool and weigh.

9. Calculations

9.1 Determine the percent moisture

$$\text{Percent Moisture} = (\text{Weight Loss} / \text{Sample Weight}) \times 100$$

9.2 Determine the percent residue

$$\text{Percent Residue} = (\text{Weight Ash} / \text{Sample Weight}) \times 100$$

9.3 Determine total incombustible

$$\text{Total Incombustible} = \% \text{ Moisture} + \% \text{ Residue}$$

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to perform electrical work, to main-tain electrical equipment, and to con-duct examinations and tests of all elec-trical equipment.

Respirable dust. Dust collected with a sampling device approved by the Sec-retary and the Secretary of Health and Human Services in accordance with part 74—Coal Mine Dust Personal Sam-pler Units of this title. Sampling de-vice approvals issued by the Secretary of the Interior and Secretary of Health, Education, and Welfare are continued in effect.

Rock dust. Pulverized limestone, do-lomite, gypsum, anhydrite, shale, adobe, or other inert material, pref-erably light colored, 100 percent of which will pass through a sieve having 20 meshes per linear inch and 70 per-cent or more of which will pass through a sieve having 200 meshes per linear inch; the particles of which when wetted and dried will not cohere to form a cake which will not be dispersed into separate particles by a light blast of air; and which does not contain more than 5 percent combustible matter or more than a total of 4 percent free and combined silica (SiO_2), or, where the Secretary finds that such silica con-centrations are not available, which does not contain more than 5 percent of free and combined silica.

Secretary. The Secretary of Labor or the Secretary's delegate.

Self-Contained Self-Rescuer (SCSR). A type of closed-circuit, self-contained breathing apparatus approved by MSHA and NIOSH under 42 CFR part 84 for escape only from underground mines.

Short circuit. An abnormal connection of relatively low impedance, whether made accidentally or intentionally, be-tween two points of different potential.

Working face. Any place in a coal mine in which work of extracting coal from its natural deposit in the earth is performed during the mining cycle.

Working place. The area of a coal mine in by the last open crosscut.

Working section. All areas of the coal mine from the loading point of the sec-tion to and including the working faces.

[57 FR 20913, May 15, 1992, as amended at 60 FR 30401, June 8, 1995; 67 FR 11001, Mar. 11, 2002]

Subpart B—Qualified and Certified Persons

§ 75.100 Certified person.

(a) The provisions of Subpart D—Ventilation of this part 75 require that certain examinations and tests be made by a certified person. A certified person within the meaning of those provisions is a person who has been certified as a mine foreman (mine man-ager), an assistant mine foreman (sec-tion foreman), or a preshift examiner (mine examiner). A person who has been so certified is also a qualified per-son within the meaning of those provi-sions of subpart D of this part which require that certain tests be made by a qualified person and within the mean-ing of § 75.1106.

(b) A person who is certified as a mine foreman, an assistant mine fore-man, or a preshift examiner by the State in which the coal mine is located is, to the extent of the State's certi-fication, a certified person within the meaning of the provisions of subpart D of this part and § 75.1106 referred to in paragraph (a) of this section.

(c)(1) The Secretary may certify per-sons in the categories of mine foreman, assistant mine foreman, and preshift examiner whenever the State in which persons are presently employed in these categories does not provide for such certification. A person's initial certification by MSHA is valid for as long as the person continues to satisfy the requirements necessary to obtain the certification and is employed at the same coal mine or by the same independent contractor. The mine op-erator or independent contractor shall make an application which satisfac-torily shows that each such person has had at least 2 years underground expe-rience in a coal mine, and has held the position of mine foreman, assistant mine foreman, or preshift examiner for a period of 6 months immediately pre-ceding the filing of the application, and is qualified to test for methane and for oxygen deficiency. Applications for Secretarial certification should be sub-mitted in writing to the Health and Safety Activity, Mine Safety and Health Administration, Certification and Qualification Center, P.O. Box