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Bureau of Mines Report of Investigations/1988

# Measurement of Incombustible Content of Coal Mine Dust Samples

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**UNITED STATES DEPARTMENT OF THE INTERIOR**



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**UNITED STATES DEPARTMENT OF THE INTERIOR  
Donald Paul Hodel, Secretary**

**BUREAU OF MINES  
David S. Brown, Acting Director**

Library of Congress Cataloging in Publication Data:

**Greninger, Nevin**

Measurement of incombustible content of coal mine dust samples.

(Report of investigations ; 9155)

Bibliography: p. 14

Supt. of Docs. no.: I 28.23:9155.

1. Coal mines and mining--Dust control. 2. Mine dusts. I. Courtney, Welby G. II. Lang, H. (Helen) III. Title. IV. Series: Report of investigations (United States. Bureau of Mines) ; 9155.

TN23.U43

[TN312]

622 s [622'.8]

87-600181

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	μm	micrometer
cm	centimeter	mCi	millicurie
cm <sup>3</sup>	cubic centimeter	min	minute
ft	foot	pct	percent
g	gram	psig	pound per square inch, gauge
g/cm <sup>3</sup>	gram per cubic centimeter	s	second
h	hour	wt pct	weight percent
in	inch	vol pct	volume percent
keV	kilo electron volt	yr	year
kg	kilogram		

# MEASUREMENT OF INCOMBUSTIBLE CONTENT OF COAL MINE DUST SAMPLES

By N. Greninger,<sup>1</sup> W. Courtney,<sup>2</sup> and H. Lang<sup>3</sup>

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## ABSTRACT

The Bureau of Mines conducted a laboratory study of the measurement of the water and rock dust (RD) contents of synthetic mine-type coal dust (CD) mixtures using a commercial unit for measuring water content and several prototype radiometric units for measuring the RD content. The RD content ranged from 50 to 100 wt pct and the added water (AW) ranged from 0 to 15 wt pct.

The total water (TW) content (intrinsic (IW) and AW) of a small dust sample could be readily measured automatically by essentially untrained personnel using a commercial unit. The RD content of a sample containing only IW could readily be measured by moderately trained personnel using several radiometric units. With gamma-ray devices, about 300 g of sample is needed. With the improved beta-ray device, about 25 g of sample is needed. With dry mine dust samples, the estimated RD error range for gamma-ray devices is 0.8 to 2.3 pct and 0.5 to 1.0 pct for beta-ray devices. However, the radiometric measurement of RD content was questionable if the dust mixture also contained AW or if it was uncertain whether the dust mixture contained AW.

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## INTRODUCTION

The blast wave from a methane explosion in a coal mine entry can entrain coal dust that had deposited in the entry. This entrained coal dust could then convert the dangerous methane explosion into a disastrous CD explosion if the CD deposit did not contain sufficient incombustible content such as water, RD,<sup>4</sup> ash, or if the roadway was not otherwise protected, for example, by barriers containing water, RD, or other explosion extinguishing agents.

The Mine Safety and Health Administration (MSHA) requires 80 wt pct incombustible content material in the dust deposit in return airways and 65 wt pct incombustible material elsewhere in the mine, except for the last 40 ft from the face (although dust deposits in closer crosscuts must meet the 65 wt pct requirement). In the presence of methane, the incombustible content must be increased by 0.4 and 1 wt pct, respectively, for each 0.1 vol pct CH<sub>4</sub>; e.g., in the presence of 1 vol pct CH<sub>4</sub>, the incombustible content must be 84 and 75 wt pct in the return and elsewhere.

Enforcement is implemented bimonthly by MSHA inspectors taking samples of the dust deposit every 200 ft in the entry in question. The conventional MSHA sampling zone is a 6-in-wide band across the floor, ribs, and roof to a depth of 1 in wherever possible. If the floor is well rock dusted, but the roof and ribs are considered to be deficient in RD content, the ribs and roof portion of the band sample is kept separate from the floor portion so that separate analysis can be made (1).<sup>5,6</sup> The inspector screens the

sample through a No. 10 sieve (2,000- $\mu$ m pore size) and sends about 200 g of the sieved sample to MSHA's Central Processing Facility at Mount Hope, WV, for analysis.

General analysis involves:

1. Drying the mine dust sample and sieving with a No. 20 screen (841- $\mu$ m pore size).

2. Crushing the mine dust sample until it passes through a No. 60 sieve (250- $\mu$ m pore size).

3. Performing an exploratory analysis for the RD content by measuring the increase in the volume of alcohol in a volumeter. The volumeter technique is sensitive to the difference in the solid densities of CD and RD. If the RD content gives a total inert value sufficiently different from the 65-pct value, in the case of a sample from a nonreturn having no gas or 80 pct from a return having no gas, a compliance decision can be rendered with no further chemical analysis.

4. For marginal samples, a more detailed analysis is performed involving a--

A. Determination of the water content by drying a 1-g sample at 105° C.

B. Determination of the RD content by treating part of the dried sample with dilute HCl and measuring the evolved CO<sub>2</sub>.

C. Determination of ash content by heating part of the dried sample at 750° C for 2 h.<sup>7</sup>

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different incombustible contents can be formed. An explosion can propagate through one of the clouds, rib or roof, should it be deficient in total inert, but not through clouds having a sufficiently high total inert content.

<sup>7</sup>Since the mid-1970's, a low-temperature ashing procedure, (3) similar to that used by the British Mining Inspectorate, has occasionally been used by MSHA.

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<sup>4</sup>Usually limestone [CaCO<sub>3</sub>] or dolomite [CaMg(CO<sub>3</sub>)<sub>2</sub>].

<sup>5</sup>Underlined numbers in parentheses refer to items in the list of references at the end of this report.

<sup>6</sup>Large-scale explosion tests have shown that insufficiently rock dusted ribs and roof areas cannot be compensated (2) for by putting extra amounts of RD on the floor, because separate clouds of dust of

The test results for the total incombustible content are reported by the Mount Hope facility to the local MSHA office, which advises the mine of the results.

This procedure of collecting the sample, transporting it out of the mine, and forwarding it to the Mount Hope facility for an exploratory-type and/or complete analysis is not only expensive but also protracted, taking at least several weeks. If the incombustible content of the mine dust sample were too low, the mine would have been in an unsafe condition during the several weeks before the mine was advised of the analysis results.

For the past 15 yr, the Bureau has funded the development of several radiometric hardware units, which theoretically would afford faster and simpler analysis and thus would minimize the time and expense involved in the analysis of coal mine dust samples. The radiometric backscatter technique is required to determine incombustible content to within  $\pm 3$  wt pct for 80 pct of 25 actual mine samples and 44 assorted standard samples containing 55 to 80 wt pct incombustible content and also including water, pyrite,  $MgCO_3$ , ash, sand, and hydraulic oil. The wet samples were to contain 5 and 10 wt pct AW (Code of Federal Regulations, section 29.61).

This mine dust analysis program was originally conceived to involve a screening-type in situ measurement by the MSHA inspector of an undisturbed mine dust deposit with a portable meter containing radioactive material.<sup>8</sup>

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<sup>8</sup>It was felt that an in situ screening-type radiometric device could easily detect a very low or very high amount of RD in a floor sample. The intended objective was to hasten and facilitate the compliance determination process relevant to inert content. For non-marginal samples it was thought that a rapid compliance decision could be

The measurement process would utilize a radiometric backscatter technique, a fairly common approach used in industry in materials processing, to monitor parameters such as concentration, thickness, and density. However, since beta and gamma rays interact strongly with RD and ash and interact only very weakly with water, a radiometric approach is limited to measuring the RD content and/or ash content of the mine dust sample and will not measure the water content of the sample.

A radiometric meter could probably not be designed for safe use in the rugged mine environment. Making it strong enough to keep it from being crushed by a massive piece of mining equipment would greatly increase its size, weight, and cost. However, an analysis at the local MSHA field office should be reasonably satisfactory from a time viewpoint. The personnel would have to receive special training in the use of radiometric devices.

In 1982, the general objective of this program therefore was changed to the measurement of the total incombustible content (water, RD, and ash) of conventional MSHA band samples by moderately trained personnel at the local MSHA office.

The specific objectives of the present project were to investigate in the laboratory the measurement of water content of mine-type dust mixtures using several techniques and the measurement of the RD content using several hardware units that utilize radiometric techniques.

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rendered underground and rock dusting could be rapidly implemented, if needed. Reducing the number of nonmarginal conventional band samples that otherwise would be collected and forwarded to the MSHA Mount Hope facility for conventional analysis would greatly simplify and speed up the inspection process.

## EXPERIMENTAL APPROACH AND RESULTS

## SAMPLE PREPARATION

Synthetic mine dust samples were prepared from commercial RD and pulverized Pittsburgh-seam CD. The as-received RD contained 0.26 wt pct IW, while the coal contained 2.0 wt pct IW, but these dusts will be termed "dry." The coal dust also contained 6.0 wt pct ash. Dry RD and CD were weighed on a laboratory balance and mixed by tumbling in a plastic bag and in a 6-in-diam steel coffee can. Main emphasis was given to dust mixtures having RD:CD ratios of 100:0, 80:20, 65:35, and 50:50; e.g., 80 g of RD was mixed with 20 g of CD to give the 80:20 dry mix, while pure rock gives the 100:0 mix.

Dust samples to simulate damp field samples containing AW were prepared by adding tap water to the dry dust mixtures. The portion of TW in a damp mix is calculated from the portion of IW in the dry components and the AW.

Nominal and actual compositions of the principal dust mixtures used here are given in table 1. The values for CD and RD are based on the weights used in preparing the mixtures. For example, an 80:20 mix containing about 15 wt pct AW was prepared by adding 17.6 g of water to 100 g of a dry 80:20 mix and will be referred to as an 80:20:15 mix, but actually contained 67.7 wt pct RD, 16.9 wt pct CD, and 15.4 wt pct TW.

To evaluate whether adding water to a dry mixture of coal and limestone dust causes a change in the amount of RD present in the mixture, samples with and without AW were prepared and analyzed for moisture, carbon dioxide, and carbon-to-hydrogen ratio.

A dry mixture of 65 wt pct RD and 35 wt pct CD was split into two portions. To one portion, water was added to give 10 wt pct AW. Analysis results from Geochemical Testing Co. gave for the dry sample 1.15 wt pct moisture, 14.8 wt pct carbon dioxide, and a carbon-to-hydrogen ratio of 15.5:1. The analysis results for the wet sample gave an air dry loss of 9.98 wt pct water, 14.8 wt pct carbon dioxide, and a carbon-to-hydrogen ratio of 16.0:1. Adding water to the dry mixture did not change the amount of RD present in the mixture.

## WATER ANALYSIS

Main emphasis was given to analyzing the TW content of the above samples with a commercial moisture analyzer, COMPUTRAC Model MA-2A.<sup>9</sup> The instrument automatically weighs the cold, empty sample pan with a force cell. A 3- to 4-g dust sample is then placed on the sample

<sup>9</sup>Reference to specific equipment does not imply endorsement by the Bureau of Mines.

TABLE 1. - Actual RD:CD:TW contents of synthetic coal mine dust mixtures, weight percent

Nominal RD:CD mixture	0 wt pct water			5 wt pct water			10 wt pct water			15 wt pct water		
	RD	CD	TW	RD	CD	TW	RD	CD	TW	RD	CD	TW
100:0.....	99.7	0	0.3	94.6	0	5.4	89.7	0	10.3	84.9	0	15.1
80:20.....	79.6	19.9	.5	75.6	18.9	5.5	71.6	17.9	10.5	67.7	6.9	15.4
65:35.....	64.6	34.6	.8	61.3	33.0	5.7	58.1	31.3	10.6	54.6	29.7	15.7
50:50.....	49.4	49.4	1.2	46.8	46.8	6.4	44.6	44.6	10.8	42.1	42.1	15.8
0:100.....	0	98.0	2.0	0	93.3	6.7	0	88.4	11.6	0	83.5	16.5

RD Rock dust. CD Coal dust. TW Total water.

pan and is automatically preweighed and periodically postweighed as the pan is heated at 105° C<sup>10</sup> by a small, Nichrome furnace.

Analysis takes about 7 min when TW is about 5 wt pct and 20 min when TW is about 20 wt pct. The MA-2A unit weighs 12 kg, uses line power, and costs \$4,000.<sup>11</sup>

Typical moisture value levels are given in table 1. A sample prepared by adding 5 pct moisture to a dry 80 pct RD mixture gave a moisture value of 4.45 pct. Samples prepared by adding 10 wt pct moisture gave values of 9.77 and 9.56 pct, respectively, for 65 and 80 pct RD mixtures. The reported value of TW is the average of three measurements. The 0.1 standard deviation of the triplicate measurement of TW was considered to be excellent.

Conventional analysis for TW by drying a damp sample overnight at 105° C in an oven according to ASTM procedures (Geochemical Testing Co.) gave results about 10 pct higher than those obtained with the COMPUTRAC MA-2A; e.g., 0.93 versus 0.83 wt pct TW for a 65:35:0 mixture, 5.3 versus 5.1 wt pct TW for a 65:35:5 mixture, and 10.9 versus 10.3 wt pct for a 65:35:10 mixture. This disparity is considered to be reasonably satisfactory, since most mine dust samples should not be overly damp.

Several other techniques can be used to measure water content or dry a large sample for subsequent RD analysis. A simple home-type microwave oven is a convenient way to dry a large dust sample, but requires preweighing and postweighing for

TW analysis. A microwave drying oven coupled to a load cell for TW analysis is commercially available, but is more expensive than the COMPUTRAC device. Shaking damp mine dust with a coarse molecular-sieve material for a few minutes removes all of the AW, but the degree of removal of the inherent water is uncertain.

#### ROCK DUST ANALYSIS

Three different units, which utilized radioactivity techniques for analyzing the RD content of conventional mine dust samples, were evaluated here. One unit was a prototype gamma-ray meter developed by Envirotech under Bureau contract. Another unit was a beta-ray meter developed by and currently in use in Poland and loaned to the Bureau by the CSE Corp. The third unit was a commercial gamma-ray meter originally developed for use by customs officials by Radiation Monitoring Devices (RMD). A fourth unit was a breadboard-type gamma-ray meter developed by GCA Corp. under Bureau contract and was received after this project was ended.

Both the Envirotech and GCA units were constrained to use similar strength sources and radiation geometries by contract specification requirements. Differences in detectors and signal processing were used.

#### Envirotech Meter

The Envirotech instrument uses a 100-mCi americium-241 source, which emits 60-keV gamma rays. The gamma rays interact strongly with the RD and ash components of the mine dust sample (but not as strongly with the water component) to produce 49-keV X-rays, which are detected by an ion chamber located in the meter. The prototype unit weighs 825 g, is battery powered, 5 by 4 by 2 in, and estimated to eventually cost about \$5,000.

<sup>10</sup>The temperature settings can range from 75° to 165° C. The low and high settings are made at the factory. On the Bureau's unit, the fixed settings were 105° and 165° C.

<sup>11</sup>Water analysis with the COMPUTRAC Model MA-5A operating at 165° C gave results that agreed with those obtained with the less expensive MA-2A unit.

The unit was designed for in situ use by the MSHA inspector placing the meter directly on a several-inch-thick undisturbed mine deposit. The radiation source in the present prototype version is covered by a protective shutter, which is manually opened by the operator to expose the radiation source when a measurement is being taken.

Such an approach is now considered to be ill-advised because of--

1. The possibility of losing an instrument having a relatively large amount of radioactive material (Am-241) with a 458-yr half-life. Such a device could also be crushed by a massive piece of mining equipment, resulting in a radioactive contamination problem.
2. The shutter accidentally opening and exposing nearby personnel to strong radiation.
3. The device not measuring the RD content of a thin dust deposit because of interference by the substrate.
4. Possible interference by particles larger than 841  $\mu\text{m}$  diam.<sup>12</sup>

However, laboratory tests were done here with the synthetic dust samples to determine the operating characteristics of the unit and to assess the general applicability of the gamma-ray approach for measuring the RD content of a conventional band sample.<sup>13</sup> In these

<sup>12</sup>Inclusion of coal particles between 841- and 2,000- $\mu\text{m}$  diam gave (4) an ashing value for the total incombustible content, which was about 6 wt pct below that for a sample excluding the 841- $\mu\text{m}$  and larger particles.

<sup>13</sup>Theory suggests a relative photoelectric absorption of 2.8 for  $\text{H}_2\text{O}$  and 51.8 for  $\text{CaCO}_3$  taking 1 for C. (The photoelectric mass absorption coefficient varies with the effective atomic number raised to the fifth power.) Because FE has such a high atomic number, the relative absorption of iron compounds is quite large. Gamma-ray devices can be used to analyze CD-RD dust mixtures because of absorption differences.

laboratory tests, the unit was used only by a skilled operator wearing a radiation badge. To protect the operator from intense radiation, a protective lead shield was placed around the dust sample, which was enclosed in a 6-in-diam plastic container. The lead shield served as an inner liner between two nested stainless steel buckets into which the sample was placed prior to irradiation.

The Envirotech meter was calibrated using dry uncompactd 50:50 and 100:0 mixtures and adjusting lower and upper calibration potentiometers so that the meter reads out the appropriate RD values for the two samples. The meter was found to be linear for samples having intermediate RD values.

Initial work with small dust samples of about 100 g and a sample depth of about 0.5 in indicated that such a sample depth was too shallow; i.e., the measurement depended on the nature of the support material below the dust sample. Subsequent work used a 1,000-g sample, giving a sample depth of about 5 in, which was just sufficient to eliminate the effect of the substrate.

The dust sample was "hand-fluffed" in its plastic bag to minimize compaction. The meter was placed on the fluffed sample. The radiation shutter was opened, and the sample was irradiated for 22 s. (This time period provides an acceptable standard deviation value for the counting of the radiation events by the detector.) The sample was then re-fluffed and remeasured several times to determine the statistical scatter associated with the measurement. The fluffed dust sample was compacted with an estimated 0.5 psig pressure (meter weight and minimal hand pressure), but will be termed "uncompactd."

The meter reading was linearly dependent upon the RD content of the dry uncompactd dust sample (fig. 1) and was satisfactorily reproducible; i.e., the standard deviation of the measured percent RD content ranged from 0.8 to 2.3 and was typically about 1.5.

However, the value of the RD measured by the Envirotech meter was sensitive to the AW content of the dust mixture. Figure 2 shows a plot of the RD content measured with the Envirotech meter versus AW content and indicates that the meter reading initially increased and then decreased as the AW content increased.

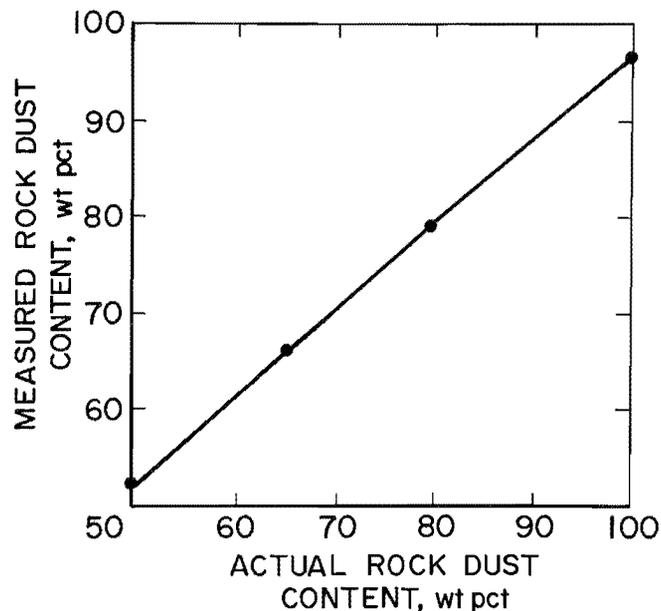


FIGURE 1.—Envirotech measurement of RD content of dry uncompact dust mixtures.

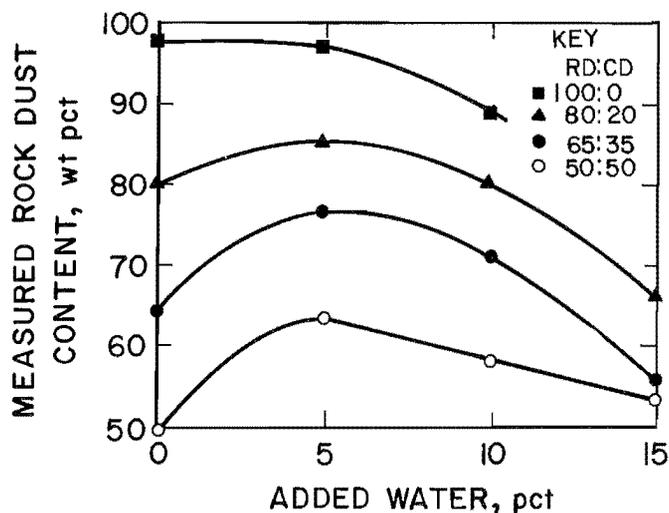


FIGURE 2.—Envirotech measurement of RD content of dust mixtures containing AW.

In figure 2, RD:CD ratios of 100:0, 80:20, etc. denote the ratios of the wt pct RD to the wt pct CD in the dry mixtures. For example, with a 65:35 mix, the meter reading was 64, 76, 71, and 56 for 0, 5, 10, and 15 wt pct AW, respectively, while the RD actually was 65, 61, 58, and 55 wt pct for these AW levels (table 1). This parabolic effect and the disparity between the measured and actual RD values decreased as the RD content of the mix increased, but was very appreciable in the RD range of main interest here; i.e., the 65 to 80 wt pct range.

The parabolic and disparity effects were thought to be possibly due to a variation in the bulk density of the dust mixture; i.e., the bulk density of a 65:35:0 mix was  $0.8 \text{ g/cm}^3$  and that of a 65:35:5 mix was  $0.6 \text{ g/cm}^3$ . However, compaction of a damp mix did not eliminate the parabolic and disparity effects. Figure 3 shows a plot of the meter

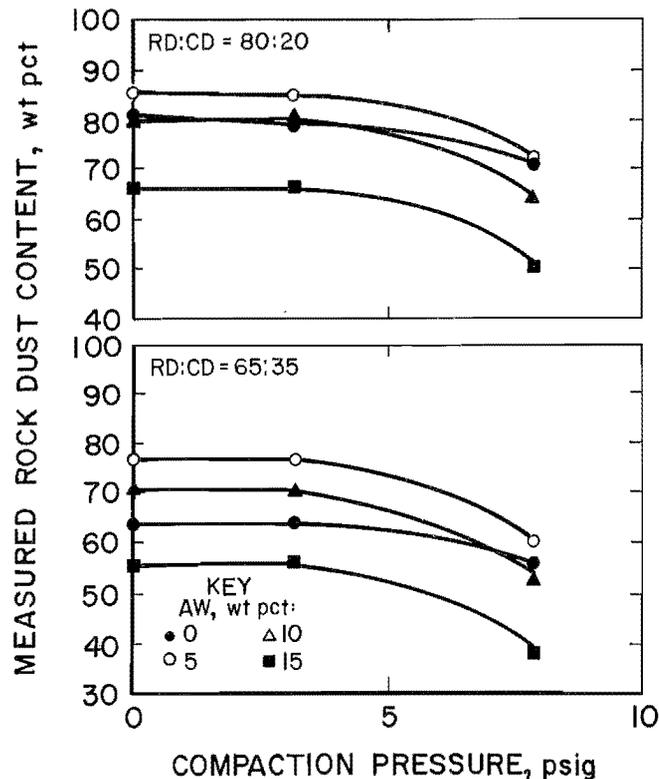


FIGURE 3.—Effect of sample compaction on Envirotech measurement of RD content of mixtures containing AW.

reading versus the compaction pressure for the 80:20 dry and damp samples and similarly for the 65:35 dry and damp samples. In figure 3, RD:CD ratios of 80:20 and 65:35 denote the ratios of the wt pct RD to the wt pct CD in the dry mixtures. The meter readings were unchanged when the mixtures were compacted at 3.5 psig, and compaction at 7.9 psig did not eliminate the parabolic and disparity effects. For example, when the 80:20 mixes were compacted at 7.9 psig, the meter read 71, 72, 65, and 51 wt pct RD for the 0, 5, 10, and 15 wt pct AW mixes, while the actual RD was 80, 76, 72, and 68 wt pct. The meter thus read 9 to 18 pct low. These two pressure values were computed from the weights of lead bricks placed on a 6-in-diam piston of known weight in a vertical compaction chamber.

This disparity between the Envirotech-measured and the actual RD values is excessive and remains unexplained. Therefore, the Envirotech unit cannot presently be used to measure the RD content of a mine dust sample to the desired accuracy if the dust sample contains a small amount of AW or if it is uncertain that the sample contains AW.

#### CSE Meter

The Polish CSE meter (5) uses a 5-mCi strontium-90 source that emits 540-keV beta rays. The beta rays are scattered strongly by the RD and the ash components of a mine dust sample (but not as strongly by any water in the sample), and the backscattered rays are measured by the meter. This prototype device has been used in Poland for research purposes. The instrument weighs about 6 kg, is battery powered, 11 by 7 by 4 in, and probably would cost under \$10,000. The unit has a 220-cm<sup>3</sup> sample chamber that provides a dust sample height of 6 cm. The beta rays are weak and penetrate less than 1 cm into the dust sample. The unit is designed so that the radiation source is normally only exposed when the sample

chamber is filled with dust; therefore, it is reasonably safe to be used by a moderately trained operator.

As with the Envirotech unit, the CSE meter is designed to provide a linear relation between the meter reading and the RD content and use a two-point calibration employing dusts with known RD contents.

The CSE meter was calibrated using uncompact dry 50:50 and 100:0 dust mixtures. The meter reading was linearly dependent upon the RD content of the uncompact dry samples (fig. 4) with replicate readings having a standard deviation of 0.5, or about one-third that of the Envirotech meter.

As with the Envirotech unit, the CSE reading was also very significantly affected by the presence of AW (fig. 5). For example, with uncompact 80:20 mixtures, the meter read 81, 62, and 60 wt pct RD when the sample contained 0, 5, and 10 wt pct AW, while the actual RD content was 80, 76, and 72 wt pct. With 65:35 mixtures, the meter read 74 and 54 wt pct RD with mixtures containing

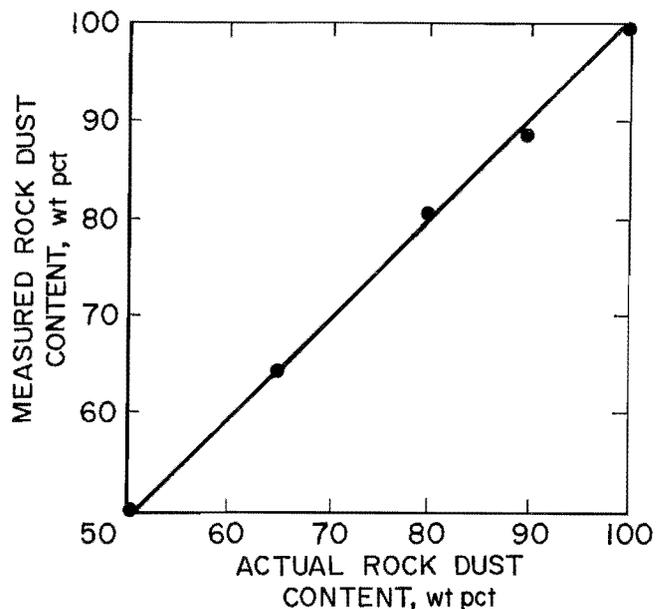


FIGURE 4.—CSE measurement of RD content of dry uncompact dust mixtures.

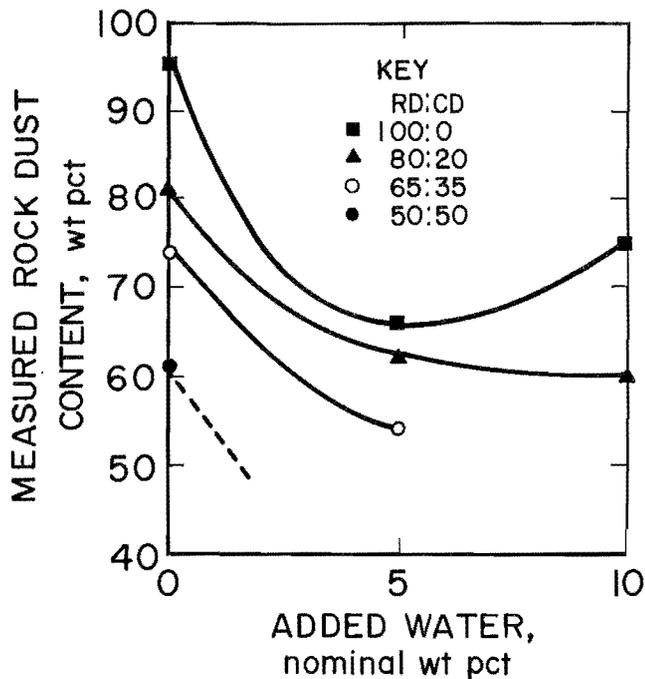


FIGURE 5.—CSE measurement of RD content of uncompactd dust mixtures containing AW.

0 and 5 wt pct AW, while the actual RD content was 65 and 61 wt pct. The dashed line for the 50:50 mixture (fig. 5) indicates an extrapolation.

Most European and British mines are very deep. In many of these deep mines, the added moisture level is fairly constant. The majority of American mines are not deep. The moisture content in mine dust for the typical American "wet" mine can vary widely. For example, dust samples taken in the winter season near the portal tend to be drier than those from the face area as a result of the dust being conditioned by the entering air having a low humidity. By the time the entering air has reached the face, it has lost some of its drying ability as a result of having picked up moisture along the way. During the winter season in mines having deep levels and an uppermost level near the surface, samples taken from the deepest level tend to be more moist than those from the uppermost level. Furthermore, "wet" mines tend to be wetter in the spring and summer than in the fall and winter.

Therefore, for most American mines, the CSE meter also cannot be used to measure the RD content if the sample contains even a small amount of AW or if the AW content is uncertain.

The Polish investigators often calibrate their beta-ray meters for a fixed level of AW. Thus, for samples having a constant added moisture level, a beta-ray meter could then be used to analyze for the RD content. When the samples have widely different added moisture levels, it is a different matter. The Polish investigators have found it useful to dry these samples with silica gel and then sieve to remove the spent silica gel before irradiating.

#### RMD Meter

The RMD Portable Contraband Detector was originally developed for customs officials by RMD to detect narcotics such as cocaine hidden (for example) in a metal car door.

The RMD meter uses a 10-mCi cobalt-57 source, which emits 122-keV gamma rays and measures the backscattered gamma rays. The commercial unit weighs 1,500 g, is battery powered, 10 by 9 by 4 in, and costs about \$10,000. The emitted radiation is purposely unshielded, so the operator must be careful to avoid directing the radiation at individuals and should wear a radiation monitoring badge.

With Bureau funding, RMD conducted a brief feasibility study of the usefulness of its meter for measuring the RD content of coal mine dust samples. Figure 6 shows a plot of the measured count rate versus RD content for uncompactd dry and damp samples and indicates that the measurement of RD content is apparently insensitive to the water content of the dust sample when the steel container and its contents were irradiated. The upper line in figure 6 pertains to measurements taken when the can containing the sample was irradiated through the plastic lid. The lower line in figure 6 pertains to measurements taken when the can

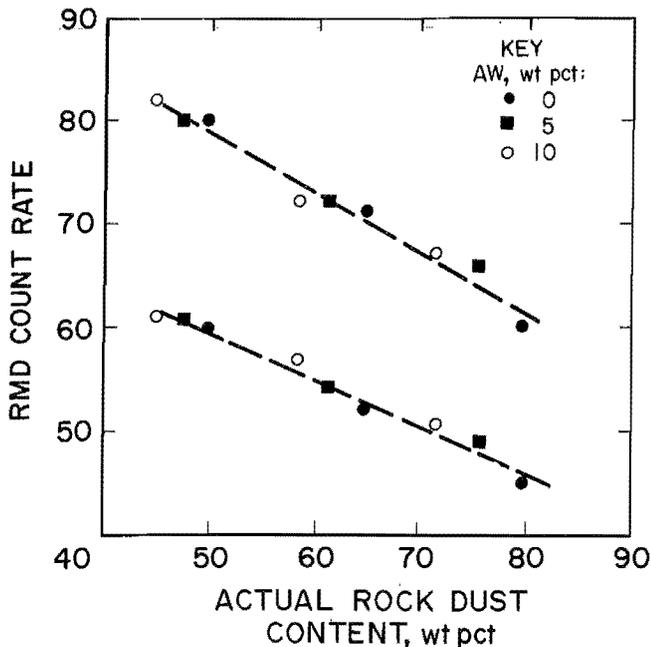


FIGURE 6.—RMD count rate versus RD content for dust mixtures containing AW.

containing the sample was irradiated through the steel bottom.

However, exploratory tests with a prototype sample holder containing 300 g of a compacted dust mixture were unsatisfactory in that the count rate now significantly depended upon AW. This project was ended before the effect of compaction could be clarified.

#### GCA Meter

The GCA instrument uses a 100-mCi americium-241 source that emits 60-keV

gamma rays and measures the backscattered gamma rays with a doped Cd-Te crystal. The eventual unit should weigh about 1,000 g, be battery powered, 5 by 4 by 2 in, and cost about \$5,000.

The present breadboard unit shielded the powerful radiation source with a spring-loaded shutter, which is manually opened when a measurement is made. Care must be taken to avoid exposure of personnel to the emitted and scattered gamma rays and also the x-rays formed by the interaction of the gamma rays with the sample.

The GCA unit was originally intended for in situ use by the MSHA inspector and was purposefully designed to be mainly sensitive to the dust volume situated about 0.5 in below the surface of the dust deposit. The unit was designed to be calibrated with 100:0 and 65:35 dust mixtures.

The GCA contract scope did not include an investigation of the effect of AW on the measurement of RD content. Although GCA considered that the measurement was expected to be sensitive to the bulk density of the dust mixture, this aspect was not investigated.

The unit was received by the Bureau after this project was ended, and no Bureau tests of the GCA unit were done. The Envirotech and GCA units would probably give similar results, since both had essentially similar design specifications and used the same strength americium-241 sources.

#### DISCUSSION

The original objective of a screening-type in situ measurement of the RD content of a dust sample to decide whether to take the conventional band sample had considerable merit. However, the use of a radiation-type meter was decidedly injudicious because of the potential safety problems associated with the use of radiation devices in the rugged mine environment.

The effect of AW on the intensity of the backscatter radiation signal in the case of gamma rays and beta rays is complex. At least two scattering factors

are involved: mass density and effective atomic number. Adding water to mixtures of dry Pittsburgh pulverized CD and RD causes a decrease in bulk density, which is especially significant at the 5 wt pct AW level (about 30 pct). After reaching a minimum value of bulk density, as more and more water is added, the bulk density increases. The porous structure of the coal dust can absorb some moisture, at least a few percent, once surface tension forces are overcome and wetting takes place. When a small amount of water is added, it may not effectively enter the

micropores at first. After some of the water enters the macropores, the balance may reside between the particles. This might promote a slight separation of the particles, leading to a decrease in the bulk density. Specifically, how the attraction or repulsion forces behave is not clear. Once the macropores and micropores become filled, which might take up to about 5 pct, the additional AW must reside between the particles in the mixture, or ooze out to the upper surface of the mixture. Clearly, radiation scattering is dependent on the mass density of the dust. The presence of water either in the micropores or macropores, or between close coal particles, changes the effective atomic number for the coal particles in the CD-RD mixture. The radiation scattering processes show a more regular pattern after effective wetting has occurred and most of the pore spaces filled. Compaction of the swelled dust removes much of the mass density effect, but it does not alter the change in effective atomic number.

The dust sample can be dried and its RD content measured. From the RD content of the dry mixture and the total moisture content of the wet mixture, a mass balance can be used to compute the added moisture pct in the mixture. Using a mass balance and values for the RD content in the dry dust, the water and ash content of the pure coal and inherent moisture content of the pure coal, enables one to compute the total incombustible content in wet coal mine dust.

The TW content of a mine dust sample can be readily measured in about 10 min with the commercial COMPUTRAC MA-2A unit. Sample size is small (about 3 g), and negligible training is required. Hardware cost is about \$4,000.

Further tests with the RMD meter using the prototype sample holder probably would be necessary to quantitatively characterize the severe effect of AW, which was also observed with the Envirotech and CSE meters. In practice, any effect of AW on a radiation measurement

of RD content can be readily eliminated by first drying the dust sample, even in an ordinary microwave oven, for example. However, a larger sample (about 300 g) is required along with moderate training and safety precautions, and a radiation-type meter is somewhat expensive (\$5,000 to \$10,000).

Since the experimental portion of this study was completed, the Central Mining Institute of Poland has refined (6) the design of its beta-ray meter. The new version can be used to analyze mine dust samples as small as 25 g in mass. The new unit, however, is still sensitive to added moisture. The Polish investigators recommend drying moist samples with coarse silica gel before measuring the RD content with the beta-ray meter. The improved digital version has been tested with actual mine samples and reportedly is being used underground in operating mines. It meets Polish mine permissibility requirements. The results of a comparison between total inert values determined by ashing and by the new beta-ray meter are quite good (6). Table 2 shows comparisons between total inert content values obtained by ashing in a furnace and by using a beta-ray-backscatter device. The agreement is quite good for the low, moderate, and high RD content ranges. The standard deviation is less than 1 wt pct.

TABLE 2.- Comparisons of total inert values determined by ashing and by the Central Mining Institute of Poland's beta-ray meter, weight percent

Mine dust type	RD content	Total inert value	
		Furnace	Mean
Coarse <sup>1</sup> ..	Low.....	54.5	55.5±0.56
	Moderate...	71.6	72.8± .49
	High.....	90.8	92.9± .61
Fine <sup>2</sup> ....	Low.....	54.5	55.8± .53
	Moderate...	71.8	70.7± .69
	High.....	90.6	89.8± .95

<sup>1</sup>25 wt pct <75 μm.

<sup>2</sup>85 wt pct <75 μm.

Table 3 summarizes the properties of the gamma and beta radiation meters mentioned in this report, including features of the improved beta-ray meter. Possibly, the CMI meter might be further refined to accurately analyze for RD content in less than 10-g samples, which would facilitate the analysis of rib and roof samples. Small beta-ray devices are commonly used in printed circuit board manufacturing to monitor coating thickness. These are readily available and cost about \$3,000.

The several-week time lag associated with analysis of the band sample at MSHA's Mount Hope facility would be largely avoided if the analysis could be performed at the local MSHA office. In MSHA field offices, radiometric devices such as the improved beta-ray device could easily, rapidly, and safely be used to measure the RD content. The use of the improved beta-ray unit, along with a rapid moisture analyzer and values of the coal's ash and IW content, would provide a measurement of the total incombustible content to within about  $\pm 1$  wt pct for a 30-g mine dust sample.

An optical meter recently developed by M. Sapko (7), appears to be a very satisfactory in situ screening tool for estimating the RD content of a mine dust sample. Drying the dust sample with a

molecular sieve material or a microwave oven probably could be performed routinely to improve the accuracy of the RD measurement. Only a small sample (about 5 g) is required, negligible training and safety precautions are required, and cost would be low (under \$1,000). The meter does require calibration with CD and RD, but the technique otherwise appears simple. The meter should become commercially available in the near future. It could be used both underground and in an MSHA field office.

The use of a optical meter as an in situ screening-type device would enable the measurement of RD content to within  $\pm 3$  wt pct. For the majority of instances, a quick compliance decision could be rendered, often within 10 to 30 min. Fortunately, most of the sampling sites underground are relatively dry. For difficult and marginal cases, the samples should be taken to the surface for subsequent analysis. Sample processing at the MSHA field office should not be too burdensome. If these marginal and/or difficult samples were processed at a nearby MSHA field office using a rapid moisture analyzer, and if the CMI (improved) beta-ray meter were used, and if the ash content and intrinsic moisture of the pure coal were known, the total incombustible content could be

TABLE 3. - Comparison of rock dust meter properties<sup>1</sup>

Parameter	Envirotech	RMD	CGA	CSE	CMI
Size.....in..	5 by 4 by 2	10 by 9 by 4	5 by 4 by 2	11 by 7 by 4	8 by 3 by 7
Weight.....g..	825	1,500	1,000	6,000	3,000
Estimated cost....	\$5,000	\$10,000	\$5,000	<\$10,000	<\$10,000
Radioactive source	Am-241	Co-57	Am-241	Sr-90	Sr-90
Rock dust content range....wt pct..	50-100	0-100	50-100	50-100	50-100
Estimated RD error range, $\pm$ wt pct..	0.8-2.3	<2.0	<2.5	0.5-1.0	0.5-1.0
Sample mass....g..	>300	>300	>300	100-160	25
Radiation type....	Gamma	Gamma	Gamma	Beta	Beta
Source strength mCi..	100	10	100	5	5
Energy emitted keV..	60	122	60	540	540

<sup>1</sup>All meters battery powered; approximate measurement time, 30 s.

determined easily to within  $\pm 1$  wt pct in 30 min. Information on the test results could be conveyed back to mine company officials by the morning of the next day, if not sooner. The difficult or marginal samples could still be sent to the MSHA Mount Hope facility for a subsequent confirmation-type analysis.

Instituting a combined in situ, field office, and Mount Hope facility effort would simplify and speed up the procedure for compliance determination, without sacrificing accuracy. Doing more of the analyses in situ and/or at the field office should enable a significant reduction in the turnaround time for samples sent to the Mount Hope facility. This would also permit an expansion of the in situ sampling efforts by Government inspectors.

At the field office, either the beta-ray or optical-reflectivity meter could be used to determine the RD content. Although the optical device would be simpler, easier, and cheaper to use than the beta-ray device, it would have a larger standard deviation. For analysis with the optical system, about 5 or 10 g of sample would be needed. If the wet dust for the RD determination is first dried by molecular sieve, then 5 g is needed for total moisture determination and 5 g for RD content determination with the optical meter. If the sample processed in the rapid moisture analyzer is subsequently used with the optical-reflectivity meter, then only about 5 g of sample is needed. For analysis with the improved beta-ray system, about 30 g of sample would be needed: 5 g for the total moisture determination and 25 g for the RD content determination.

Many of the rapid techniques for RD content determination and moisture analysis are also of interest to mine company personnel who monitor their rock dusting program.

Lastly, analysis of the ash content of the mine dust sample is required to fully characterize the inert content of the dust sample. The present techniques for determining ash content require moderately trained personnel and moderately expensive equipment (about \$11,000). An automated ashing-weighing technique could probably be devised and should not be too expensive, but such equipment does not appear to be commercially available at present.

Briefly summarizing the major points:

1. The optical meter now under development by the Bureau appears to provide a reasonably satisfactory screening-type in situ tool to decide whether to take a conventional band sample.

2. Relevant to processing a 10-g band sample at the local MSHA office--

- A. Analysis for TW and RD can easily and inexpensively be done by modestly trained personnel using a moisture analyzer, such as the COMPUTRAC MA-2A unit and the prototype optical RD meter.

- B. Analysis for ash requires moderately trained personnel at present, but an automated technique could probably be devised.

3. The optical meter and rapid moisture analyzer would also be of significant help to the mine company safety personnel involved in monitoring their rock dusting operations. The RD meter could be used both underground and in the local mine office. The rapid moisture analyzer could be used in the local mine office to refine the estimate of total inert content in the mine dust sample.

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