



Trade &  
Investment  
Mine Safety

NSW CODE OF PRACTICE | WHS (MINES) LEGISLATION

# Roadway dust analysis in underground coal mines



This code of practice has been approved under section 274 of the *Work Health and Safety Act 2011*.

Notice of that approval was published in the NSW Government Gazette referring to this code of practice as Roadway Dust Analysis in Underground Coal Mines on 23 January 2015. This code of practice commenced on 1 February 2015.

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Code of Practice: Roadway dust analysis in underground coal mines

[www.resourcesandenergy.nsw.gov.au/safety](http://www.resourcesandenergy.nsw.gov.au/safety)

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Disclaimer: The information contained in this publication is a code prescribed under the *Work Health Safety Act 2011* ('WHS Act') and WHS (Mines) Act. Users are reminded that a code of practice is admissible in proceedings as evidence of whether or not a duty or obligation under the WHS Act or *Work Health and Safety (Mines) Act 2013* has been complied with. New versions of this code may be issued from time to time. It is the responsibility of users to ensure that the version of the code on which they rely is current by checking the Department of Trade and Investment, Regional Infrastructure and Services website.

## Foreword

The NSW *Code of Practice: Roadway dust analysis in underground coal mines* is an approved code of practice under section 274 of the *Work Health and Safety Act 2011* (the WHS Act).

An approved code of practice is a practical guide to achieving the standards of health, safety and welfare required under the WHS Act, the *Work Health and Safety Regulations 2011* (WHS Regulations), *Work Health and Safety (Mines) Act 2013* (WHS (Mines) Act) and *Work Health and Safety (Mines) Regulations 2014* (WHS(Mines) Regulations)<sup>1</sup>.

A code of practice applies to anyone who has a duty of care in the circumstances described in the code. In most cases, following an approved code of practice would achieve compliance with the health and safety duties in the WHS laws, in relation to the subject matter of the code. Like regulations, codes of practice deal with particular issues and do not cover all hazards or risks that may arise. The health and safety duties require duty holders to consider all risks associated with work, not only those for which regulations and codes of practice exist.

Codes of practice are admissible in court proceedings under the WHS laws. Courts may regard a code of practice as evidence of what is known about a hazard, risk or control and may rely on the code in determining what is reasonably practicable in the circumstances to which the code relates.

Compliance with the WHS laws may be achieved by following another method, such as a technical or an industry standard, if it provides an equivalent or higher standard of work health and safety than the code.

An inspector may refer to an approved code of practice when issuing an improvement or prohibition notice.

### The development of this code of practice

This code of practice has been developed under the 'Inter-Governmental Agreement for Consistency or Uniformity of Mine Safety Legislation and Regulations in NSW, Queensland and Western Australia' and forms part of the mining safety legislative framework for these states. Under this agreement, tri-state model legislation was developed, although designed to be structured and customised differently in each of these states.

The code was developed in consultation with the Non-Core (tri-state) Legislative Working Group representing the following stakeholders from the mining industry in NSW and Queensland:

- NSW Minerals Council
- Construction, Forestry, Mining and Energy Union (CFMEU) – NSW and Queensland
- NSW Trade & Investment (Mine Safety)
- Queensland Resources Council
- Queensland Department of Natural Resources and Mines

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<sup>1</sup> It will sometimes be convenient to refer generally to 'WHS laws', as defined under s5 WHS (Mines) Act, which includes:

- *WHS Act*
- *WHS (Mines) Act*
- *WHS Regulations*
- *WHS (Mines) Regulations*

Accordingly, this code of practice is based on the non-core (tripartite) Legislative Working Group endorsed version on 27 May 2014.

A draft of this code of practice was released for public consultation on 16 June 2014 and was approved by the Minister for Energy and Resources, the Hon Anthony Roberts MP on 19 January 2015. The code will be reviewed as required or when legislation is reviewed.

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## Scope and application

This code of practice provides practical guidance on the appropriate test method to determine whether the explosion suppression content of materials supplied to or within an underground coal mine is suitable, including:

- the content of quartz (free silica) in limestone dust
- size analysis of limestone dust
- the incombustible content of roadway dust in a coal mine.

You should use this code if you are a mine operator of an underground coal mine, or a person conducting a business or undertaking (PCBU), who:

- carries out analysis of limestone dust (stone dust) for use as an explosion suppressant in underground coal mines
- determines the incombustible content of roadway dust in an underground coal mine.

It is expected that workers, including laboratory personnel, stone dust suppliers and coal mine dust officers, will also use this code.

## How to use this code of practice

This code includes references to both mandatory and non-mandatory actions. The references to legal requirements contained in the WHS Act and WHS (Mines) Act, WHS Regulations and WHS (Mines) Regulations are not exhaustive and are included for context only.

This code has been prepared to be consistent with the WHS laws as at the date of publication and should be interpreted, to the extent that if there is any ambiguity, in a manner that is consistent with the WHS laws.

To ensure you comply with your legal obligations you must refer to the latest legislation, which is available on the NSW legislation website ([www.legislation.nsw.gov.au](http://www.legislation.nsw.gov.au)).

This publication does not represent a comprehensive statement of the law as it applies to particular problems or to individuals or as a substitute for legal advice. You should seek independent legal advice if you need assistance on the application of the law to your situation.

The words 'must', 'requires' or 'mandatory' indicate that legal requirements exist and must be complied with. The word 'should' indicates a recommended course of action, while 'may' indicates an optional course of action.

Unless otherwise indicated in the text, lists of points in the code should not be read as exhaustive.

## 1 Introduction

Explosion suppressant materials are used in underground coal mines to prevent coal dust becoming an ignition source for explosions and fire.

Limestone dust has been applied in Australian underground coal mines so that the incombustible content of roadway dust remains above the minimum specified in legislation so as to prevent ignitions.

The dust must be of a size and quality so that it is fit for purpose to achieve the intended outcomes of its application.

The use of explosion suppressants must be risk-managed under WHS laws to ensure that the risk to workers:

- is eliminated if this is reasonably practicable and, if not
- that the risk is minimised so far as is reasonably practicable.

Suppressants such as limestone contain constituents that are hazardous to workers who are exposed to it and are identified as not fit for use. This is why it is critical for any explosion suppressant used in underground coal mines to be analysed properly so it is suitable for application.

This code focuses on the quartz (free silica) content in limestone dust, size analysis of stone dust and the methodologies for determining the incombustible content of roadway dust. The mine operator and PCBUs should ensure the most appropriate method of testing is used.

## 2 Content of quartz (free silica) in limestone dust

The WHS (Mines) Regulations makes provision for the quality of stone dust that must be used in underground coal mines as an explosion suppressant:

### WHS (Mines) Regulations

#### 65 Coal dust explosion (cl 657 model WHS Regs)

- (1) In complying with clause 9 in relation to coal dust explosion, the mine operator of an underground coal mine must:
- ...
- (e) ensure that any stone dust used:
- ...
- (iii) does not contain more than 3% by mass of free silica, and
- ...

The following method of analysis is a proven method of testing to determine the free silica content by mass in limestone dust. The use of an alternative method is acceptable provided that there is reliable evidence demonstrating the accuracy and reliability appropriate to the application.

### 2.1 Method for determining quartz in limestone dust

This method covers the determination of quartz (also known as free silica) in limestone dust by a combined chemical and infra-red spectrophotometric technique.

### 2.1.1 Equipment required

For wet chemical stage:

- weighing dish
- spatula
- balance brush, camel hair or similar
- top-loading balance, readable to 0.001 g
- de-ionised water
- hydrochloric acid (HCl), 5M (prepared from hydrochloric acid, AR)
- two beakers, 500 mL, Pyrex
- clock glass
- stirring rod, glass
- wash bottle
- filter paper (ashless, rapid (e.g. Whatman No 41), 12.5 cm)
- filter funnel, glass
- drying oven, with thermostat
- electric muffle furnace capable of 400°C - (if a temperature-programmable muffle furnace is available, a drying oven is not required)
- crucible, fused-silica or platinum, with lid
- analytical balance, readable to 0.0001 g
- desiccator.

Additional for infra-red stage:

- micro-balance, readable to 0.001 mg
- mortar and pestle, agate or aluminium oxide, large
- mortar and pestle, agate or aluminium oxide, small
- ethanol, absolute, AR
- potassium bromide (KBr), spectro grade, dried at 105°C
- grinding mill (shaking type) (sim. Specamill) (optional)
- stainless steel capsule and spheres for mill (optional)
- die assembly, stainless steel, 13-mm, for preparing KBr disks
- bench press, capable of 10 tonnes force
- sample holder, to support 13 mm KBr disks in IR beam
- fourier-transform infra-red spectrophotometer (FTIR).

For preparation of standards:

- dish, micro, aluminium
- spatula, micro
- quartz, primary standard, A9950.

## 2.1.2 Procedure

### Wet chemical stage

Thoroughly mix the sample supplied. Sampling systematically from the whole sample, weigh on the top-loading balance to the nearest 0.01 g, about 1 g of the stone dust to be tested in a suitable weighing dish. If the percentage of acid-insoluble matter in the sample is approximately known, weigh enough sample to produce about 0.05 g of residue. In this case, it may be necessary to use a different size beaker and a different quantity of hydrochloric acid (HCl) in the chemical dissolution.

Transfer the weighed sample quantitatively to a 500 mL beaker. Moisten with de-ionised water. Add 100 mL of hydrochloric acid (HCl), 5M, while holding a clock glass over the beaker to minimise spattering. Cover and stand for one hour with occasional stirring. When all effervescence has stopped, rinse any splashing from the underside of the clock glass and the sides of the beaker into the solution. Filter the solution through an ashless filter paper, and quantitatively transfer all the insoluble residue to the filter paper. Rinse the residue on the filter paper with de-ionised water at least three times to eliminate all traces of acid.

Remove the filter paper from the funnel, fold and put in a crucible. Put the open crucible in a drying oven (or temperature-programmable muffle furnace) at 105°C for one hour. Ignite in a muffle furnace (or temperature-programmable muffle furnace) at 400°C until free of carbon.

Transfer the crucible to a desiccator to cool.

When the crucible is cool (30 minutes), weigh the crucible with its lid and the residue, to 0.0001 g on the analytical balance. Then transfer the residue to a suitable specimen tube, brushing all material from the crucible and its lid. Cap the tube. Weigh the empty crucible and lid to 0.0001 g on the analytical balance.

*Concentration of insoluble residue (percent)*

$$E = \frac{A - B}{D} \times 100$$

where:

- E = Concentration of insoluble residue, expressed as a percentage
- A = Mass of crucible + lid + residue, expressed in grams
- B = Mass of crucible + lid, empty, expressed in grams
- D = Mass of original sample, expressed in grams

*Note:* if the percentage of insoluble residue in the stone dust is less than the allowable percentage of free silica in stone dust for underground use, the infra-red determination of quartz may be omitted from the procedure.

### Preparation of KBr disk

Transfer the insoluble material to a large mortar. Grind thoroughly until all gritty particles are reduced to a smooth, uniform powder.

Transfer about 10 mg of the powder to a small mortar. (Note: for a large quantity of residue, it may be necessary to sample in two stages to ensure the material analysed is truly representative of the whole residue.) Moisten with just sufficient absolute ethanol. Grind thoroughly for at least 10 minutes, remoistening if necessary.

Transfer the ground powder to a drying oven at 80°C for 15 minutes. Allow to cool in a desiccator.

Weigh (on micro-balance) 5 mg of the ground up powder. Weigh about 500 mg of spectro-grade potassium bromide (KBr). Blend in stages with the ground powder, only adding sufficient KBr at each stage to double or triple the quantity being mixed. At each stage, mix thoroughly in the mortar and pestle, but do not grind. Alternatively blend in a shaking-type mill, using a stainless steel capsule with stainless steel spheres, for 10 minutes.

Weigh 200 mg of the mixture. Transfer the mixture quantitatively into the die assembly. Tap to settle the mixture. Gently insert the plunger and twist to flatten the top of the powder in the die. Remove the plunger, insert the top stainless steel pellet, and reinsert the plunger.

Insert the die assembly into the press. Continue applying vacuum to the die assembly and let stand for 5 minutes. Steadily apply the pressure of the press to the die up to 10 tonnes. Leave the die under pressure for 2 minutes. Release the pressure and vacuum.

Remove the disk from the die, using the force of the press if necessary. Weigh the disk on the analytical balance. If the disk is not to be scanned immediately, store in a desiccator.

*Note:* the technique of preparing the KBr disk may vary according to the equipment in use. However, fine grinding of the portion for analysis is crucial. No more than 10 mg of sample should be ground at a time in the final grind before analysis, and it must be ground thoroughly as described.

#### Preparation of standards

A series of working standards is prepared from primary standard quartz that has been finely ground by the same two steps (dry and wet) as described above for the unknown. Weigh the quartz on a micro balance and mix in spectro grade KBr weighed on the analytical balance, as follows:

No. of Standard	Quartz (mg)	KBr (mg)	Concentration (mg/200mg)
1	2.5	5000	0.1
2	5.0	5000	0.2
3	5.0	2000	0.5
4	5.0	1000	1.0
5	5.0	500	2.0

Mixing is carried out as for the sample. Then weigh 200 mg of the mixture, transfer to the die assembly and prepare the disk as for the sample.

#### Infra-red stage

The KBr disk should be uniform and almost clear. Insert into the sample holder. Run the FTIR spectrogram using resolution  $4\text{ cm}^{-1}$ , 16 scans, range  $4400 - 450\text{ cm}^{-1}$ , strong apodization.

When the spectrogram has run, inspect for satisfactoriness. Particularly note unusually shaped or asymmetric peaks. These may indicate insufficiently ground sample. In this case a new KBr disk must be prepared.

Because of the presence of interfering constituents, it may be impossible to use the measurement and calculation methods described below. It may be possible to use

alternative absorption peaks, or in some cases an X-ray diffractometric (XRD) method may be used.

Display in absorption mode. Measure the absorption at the two characteristic quartz peaks (799 and 779  $\text{cm}^{-1}$ ) if present, and at the outer backgrounds.

Run spectrograms for standards exactly as for the sample, and as nearly as possible at the same time.

Measure the peak and background absorptions just as for the sample.

#### Calculations

For each peak reading (at 799 and 779  $\text{cm}^{-1}$ ), for each standard and sample, calculate by interpolation the equivalent background reading, and subtract this from the peak reading as follows.

*Equivalent background reading at  $x \text{ cm}^{-1}$ ,  $A_B$ :*

$$A_B = (A_L - A_R) \frac{N_L - x}{N_L - N_R}$$

where:

- $A_L$  = background absorption measured at left of peaks
- $A_R$  = background absorption measured at right of peaks
- $N_L$  =  $\text{cm}^{-1}$  of background absorption measured at left of peaks
- $N_R$  =  $\text{cm}^{-1}$  of background absorption measured at right of peaks
- $x$  =  $\text{cm}^{-1}$  of peak (799 or 779)

*Net peak reading at  $x \text{ cm}^{-1}$*

$$A'_x = A_x - A_B$$

Where:

- $A'_x$  = net absorption measured at  $x \text{ cm}^{-1}$
- $A_x$  = absorption measured at  $x \text{ cm}^{-1}$
- $x$  =  $\text{cm}^{-1}$  of peak (799 or 779)

For the working standards, for each wavenumber, plot net absorption as ordinate against milligrams of quartz in the standard, or carry out an equivalent least-squares regression.

If reasonably linear plots are obtained, these may be used as the calibration graphs.

The mass of quartz in an unknown sample is determined from  $A'_x$ , the net peak absorption at each wavenumber, by reading off the appropriate calibration graph. These should agree within  $\pm 5\%$ . Take the mean.

*Concentration of quartz in the insoluble residue (percent)*

$$G = 100M_S \frac{m_K + m_R}{m_R m_P}$$

Where:

- $G$  = concentration of quartz in the insoluble residue, expressed as percentage
- $M_S$  = mean milligrams of quartz obtained from the calibration graphs

- $m_R$  = mass of sample of insoluble residue mixed with KBr to make the disk, expressed in milligrams
- $m_K$  = mass of KBr used to mix with the sample, expressed in milligrams
- $m_P$  = mass of finished disk, expressed in milligrams

*Concentration of quartz in the original sample of stone dust (percent)*

$$H = \frac{GE}{100}$$

where:

- H = concentration of quartz in the original sample of stone dust, expressed in percent
- G = concentration of quartz in the insoluble residue, expressed in percent, as calculated above
- E = concentration of insoluble residue in the original sample, expressed in percent, as calculated in section 3.2 below.

### 3 Size analysis of stone dust

The WHS Mines Regulations sets a minimum standard for the quality of stone dust that must be used in underground coal mines as an explosion suppressant:

#### WHS (Mines) Regulations

##### 65 Coal dust explosion (cl 657 model WHS Regs)

- (1) In complying with clause 9 in relation to coal dust explosion, the mine operator of an underground coal mine must: ...
- (e) ensure that any stone dust used:
- ...
- (iv) has a composition such that at least 95% by mass of the stone dust is finer than 250 micrometres and of that stone dust that is finer than 250 micrometers, at least 60% by mass (and not more than 80% by mass) is finer than 75 micrometres, and ...

The following method of analysis is a proven method of testing to determine the size distribution by mass in stone dust. The use of an alternate method is acceptable provided that there is reliable evidence demonstrating the accuracy and reliability appropriate to this application.

#### 3.1 Method for size analysis of stone dust

##### 3.1.1 Scope

This method covers the quantitative determination of the particle size distribution of stone dust finer than 250 micrometres and 75 micrometres by sieve analysis. The determination is to ensure that stone dust complies with the requirements for use in coal mines.

##### 3.1.2 Equipment required

- standard sieves of aperture 250 micrometres and of aperture 75 micrometres, complying with AS 1152:1993, BS 410-1:2000, or ISO 3310-1:2000
- top-loading balance which weighs to 0.01 g

- laboratory air oven maintained at 105°C - 110°C
- desiccator
- clock glasses or dishes.

## 3.2 Procedure

### 3.2.1 Preparation of the sample

The total contents of a standard bag of stone dust (or a sample of approximately 10kg if the stone dust is supplied in a bulk container) shall be coned and quartered and/or riffled to provide a minimum representative sample of 500 g.

### 3.2.2 Drying of sample

Weigh about 110 g of air-dried stone dust on the top-loading balance to 0.1 g on to a weighed dish or dishes. Dishes should be of sufficient size and number that the dust is spread to a depth of no more than 1 cm. Place the dishes in an air oven at 105°C - 110°C to dry for at least 1 hour. Remove from air oven and place in desiccator. Allow to cool and then weigh.

Return to air oven for further drying for 1 hour. Remove from air oven and put in desiccator. Allow to cool and then weigh. If the second weight of the sample is within 0.1 g of the first weight then the sample is considered dry. If not, repeat the drying, cooling and weighing until no further loss is observed on weighing.

Determination of dust content that passes 250 micrometres and 75 micrometres

100 g of the sample dried as above is to be sieved by hand or mechanical means to determine the percentage of dust passing nested 250 micrometre and 75 micrometre sieves. The endpoint is considered to have been reached if, during a further 2 minutes sieving, not more than 0.1 g passes into the receiver. Weigh the dust collected in the receiver, and the dust collected on the 250 micrometre test sieve.

Calculations

*Fraction of dry dust passing the 250 micrometre test sieve:*

$$F_{250} = \frac{M_D - M_{250}}{M_D} \times 100$$

Where:

$F_{250}$  = fraction of dry dust which passes the 250 micrometre sieve, expressed as a percentage of the dust taken for sieving

$M_D$  = mass of dried dust taken for sieving, expressed in grams

$M_{250}$  = mass of dust retained on the 250 micrometre sieve, expressed in grams

*Fraction of dry dust passing the 75 micrometre test sieve, as a percentage of dust passing the 250 micrometre test sieve:*

$$F_{75} = \frac{M_P}{M_D - M_{250}} \times 100$$

Where:

$F_{75}$  = fraction of dry dust which passes the 75 micrometre sieve, expressed as a percentage of the dust which passes the 250 micrometre sieve

$M_D$  = mass of dried dust taken for sieving, expressed in grams

$M_P$  = mass of dust collected in the receiver after sieving, expressed in grams

$M_{250}$  = mass of dust retained on the 250 micrometre sieve, expressed in grams

### Report

The report of the test must include the following results, each of which may be rounded to the nearest whole number:

- (a) the percentage by weight of the dry dust which passes the 250 micrometre test sieve
- (b) the percentage by weight of the dry dust that passes the 75 micrometre test sieve, expressed as a percentage of the dust that passes the 250 micrometre test sieve.

## 4 Analysis of incombustible content

The WHS (Mines) Regulations sets the minimum mandatory requirements for the incombustible content of roadway dust at an underground mine:

### WHS (Mines) Regulations

#### 65 Coal dust explosion (cl 657 model WHS Regs)

(1) In complying with clause 9 in relation to coal dust explosion, the mine operator of an underground coal mine must:

...

- (c) limit or remove coal dust accumulation on roadways and other surfaces in mine roadways to ensure that the amount of incombustible material contained in roadway dust at the mine is kept at or above the following concentration levels:
  - (i) for dust in a panel roadway within 200 metres outbye the last completed line of cut-throughs in the panel—85%,
  - (ii) for dust in any 200 metre section of panel roadway within 400 metres of a longwall face—85%,
  - (iii) for dust in a return roadway to which subparagraphs (i) and (ii) do not apply—80%,
  - (v) for dust in an intake roadway to which subparagraphs (i) and (ii) do not apply—70%, and

...

The following methods of analysis are proven methods of testing to determine the incombustible content of roadway dust. The three methods described are:

1. **Chemical method for roadway dust** – used for preparing the colour samples used for the colorimetric method.
2. **Colorimetric method for roadway dust** – used by the roadway dust sampler appointed at the mine for the routine testing required under clause 66 WHS (Mines) Regulations. This method gives an instant result so that any remedial action can be taken in the appropriate time.
3. **Density method for roadway dust** – used by government officials to determine the approximate level of incombustible material in a roadway dust sample they have collected and to select which samples require re-testing by the chemical method (i.e. which samples are close to the statutory limit).

Each of these methods of testing are set out in 4.1, 4.2 and 4.3 below.

In the event of any dispute or uncertainty regarding the incombustible content of dust, the chemical method is to be regarded as authoritative on the subject.

## 4.1 Analysis method – chemical

### 4.1.1 Equipment required:

- an analytical balance with a sensitivity of 0.1 milligram or better
- a 250 micrometre aperture size sieve. The sieve must comply with AS 1152:1993, BS 410-1:2000, or ISO 3310-1:2000 and should be equipped with lid and receiver
- a hot air oven regulated to maintain a temperature of  $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$
- a furnace equipped with a temperature controller that will maintain its temperature at  $515^{\circ}\text{C} \pm 15^{\circ}\text{C}$
- dishes made of silica or other suitable material
- a desiccator.

### 4.1.2 Standards

- limestone
- mixture of coal dust and limestone of known composition (incombustible content approximately 75%-80%).

### 4.1.3 Method

1. Air dry (or microwave for a nominal 2 minutes), if necessary, sufficiently to allow the sample to be sieved successfully. A correction may be made for moisture lost. If it is desired to make this correction, weigh the dust before drying, and designate this weight as A. Weigh after drying, and designate this weight as B. Calculate the percentage loss M as:

$$M = \frac{A - B}{A} \times 100$$

2. Sieve the sample through the 250 micrometre sieve.
3. Weigh about 1 gram of the material which passed through the 250 micrometre sieve to the nearest 0.1 milligram in an open dish and place in the air oven for one hour at  $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Designate the weight of material taken as W.
4. Cool the sample in a desiccator and reweigh. The loss in weight is the moisture in the sample. Designate this loss in weight as D.
5. Place the sample in the furnace at  $500^{\circ}\text{C}$  to  $530^{\circ}\text{C}$  for 2 hours to burn off combustible material.

**Note:** quality control samples of limestone dust and a mixture of coal dust and limestone of known composition should be processed with each batch of samples. These samples should be weighed before and after ignition. The limestone sample is to check that the temperature of the furnace does not exceed the decomposition temperature of the limestone. The coal/limestone mixture is to confirm complete combustion.

6. Cool the residue in a desiccator and weigh it. Designate the weight of residue as R.
7. Calculate the percentage total incombustible content of the roadway dust.

Calculation of percentage total incombustible content of the roadway dust:

$$I = \frac{D + R}{W} \times 100$$

Where:

- I = Percentage total incombustible content of the roadway dust which passes through a standard 250 micrometre sieve
- D = Weight of moisture
- R = Weight of residue after ignition
- W = Weight of sample taken

Moisture correction

If it is necessary for a dust sample to be air dried before being sieved through a 250 micrometre sieve, a correction may be made to the incombustible content. The corrected total incombustible content of the dust is equal to:

$$I_{\text{Corr}} = \frac{I(100 - M)}{100}$$

Where:

- $I_{\text{Corr}}$  = Corrected total incombustible content
- M = Percentage loss of weight during air drying
- I = Percentage total incombustible content of the air dried dust as determined above.

This analysis method may be carried out by manual or automatic methods.

## 4.2 Analysis method – colorimetric

### 4.2.1 Equipment required:

- sieve of nominal aperture 250 micrometres, complying with AS 1152:1993, BS 410-1:2000, or ISO 3310-1:2000
- supply of clean white paper
- spatula (capable of dispensing as much dust as can be heaped on a five cent coin)
- standard colour sample, prepared as below.

### 4.2.3 Method

1. Air dry (or microwave for a nominal 2 minutes), if necessary, sufficiently to allow the sample to be sieved successfully. Note: the water content of the sample is not calculated for this test method. Pass the sample through the 250 micrometre sieve and mix the sieved sample thoroughly, but do not grind it.
2. Compare the colour of the mixed sieved sample with that of the standard colour sample. The comparison must be made under conditions of good and even illumination.
3. If there is an obvious colour difference between the sample dust and the standard colour sample, record whether it is lighter or not.
4. If there is no obvious colour difference, use a spatula to take a small portion of both the sample dust and standard colour sample and put them side by side on clean white paper. Press the dust portions flat with the spatula to form a smooth surface. If there is still no

distinguishable difference, treat the result as being no lighter than the standard colour sample.

5. Examine dust portions and record whether or not the sample is lighter in colour than the standard colour sample.

#### 4.2.4 Preparation of standard colour sample

1. Grind some dry coal dust or small dry coal from the seam for which the standard colour sample is being prepared so that it passes through the 250 micrometre sieve.
2. Determine the ash content of the sieved coal dust according to AS 1038.3:2000. The ash content should not exceed 20% by weight on a dry basis. This is to avoid the use of shale in the preparation of a standard colour sample.
3. Pass some of the dry stone dust of the type most widely used in the mine through the 250 micrometre sieve.
4. The standard colour sample must have a solid incombustible content equal to or greater than the total incombustible content required in a particular sample.
5. Weigh the sieved coal dust and sieved stone dust in proportions that will give the desired solid incombustible content.
6. Mix the dusts thoroughly by stirring, shaking or rolling the mixture, but do not grind the mixture.
7. Using the chemical method for roadway dusts, determine the solid incombustible content of the mixture and verify that it is not less than the required value.
8. At intervals of not more than 12 months, re-test the standard in the manner set out in (7) above and keep a record of the results of these tests; if the standard has a solid incombustible content that is less than that required in (4) above, replace the standard with a new one.
9. Prepare new standards every two years.
10. The mine operator should determine the need for new standards if conditions, mining practices or seam conditions change. The mine operator should determine if there is a need for different samples in different areas of the mine. There should be separate standards for each seam worked at the mine.

### 4.3 Analysis method - density

#### 4.3.1 Overview

The pycnometer determines the true density of powder samples by measuring the pressure difference when a known quantity of gas under pressure is allowed to flow from a precisely known reference volume into a sample cell containing the powdered material.

#### 4.3.2 Equipment required:

- gas pycnometer
- 250 micrometre aperture size sieve (the sieve must comply with AS 1152:1993, BS 410-1:2000, or ISO 3310-1:2000)
- nitrogen (compressed)
- stopwatch

#### 4.3.3 Method

1. Turn the pycnometer on and allow for the pressure transducer to warm up and stabilise in accordance with manufacturer's instructions.

2. Dry the dust sample (carefully in a microwave oven, for example) until it flows and can be sieved easily. Sieve the dust through a 250µm sieve.
3. Fill the sample cup with the available sieved dust, up to 50 g, and record the sample weight,  $m$ .
4. Insert the sample cup into the sample cell and seal unit.
5. Bring the sample to atmospheric pressure and zero the pressure gauge.
6. Pressurise the reference cell with nitrogen to approximately 17psi and allow to stabilise. Record the initial pressure,  $P_1$ .
7. Introduce the gas to the sample cell. After 30 seconds, record the final pressure,  $P_2$ .  
Note: accurate timing is important to achieving reproducible results.
8. Vent the pressure to bring the sample back to atmospheric pressure.
9. Repeat steps 6 to 9 twice more.
10. Calculate the volume of sample using the following equation:

$$V_S = V_C - V_R \left( \frac{P_1}{P_2} - 1 \right)$$

Where:

$V_S$  = Volume of the sample

$V_C$  = Volume of the sample cell. (note: sample cell volume provided by manufacturer.)

$V_R$  = Volume of the reference cell (note: reference cell volume provided by manufacturer.)

$P_1$  = Initial pressure

$P_2$  = Final pressure

11. Calculate sample densities from

$$d = \frac{m}{V_S}$$

Where:

$d$  = density (g/cc)

$m$  = sample mass (g)

$V_S$  = sample volume (cc)

12. Discard the first density result, and average the second and third density results.
13. From the average density calculate the solid incombustible content using the formula

$$I = 237.7 - \frac{375.9}{d}$$

Where:

$I$  = incombustible content (%)

$d$  = density (g/cc).

**Note:** the above method is indicative of the steps involved in the use of a pycnometer. Laboratories should develop and validate detailed methods specific to their model of pycnometer.

## REFERENCES

Documents that form part of this code:

Document		Application to this code	
Title	Content	Version	Part of code
AS 1038.3-2000 <i>Coal and coke – Analysis and testing – Proximate analysis of higher rank coal</i> Standards Australia	All	As at time of incorporation	4.2 Analysis method – Colorimetric
AS 1152:1993 <i>Specification for test sieves</i> Standards Australia	All	As at time of incorporation	3.1 Method for size analysis of stone dust 4.1 Analysis method – Chemical 4.2 Analysis method – Colorimetric 4.3 Analysis method – Density
BS 410-1:2000 <i>Test sieves. Technical requirements and testing. Test sieves of metal wire cloth</i> BSI	All	As at time of incorporation	3.1 Method for size analysis of stone dust 4.1 Analysis method – Chemical 4.2 Analysis method – Colorimetric 4.3 Analysis method – Density
ISO 3310-1:2000 <i>Test sieves - Technical requirements and testing - Part 1: Test sieves of metal wire cloth</i> ISO	All	As at time of incorporation	3.1 Method for size analysis of stone dust 4.1 Analysis method – Chemical 4.2 Analysis method – Colorimetric 4.3 Analysis method – Density