Academic Studies and the Coal Industry
The Sampling of Coal as a Bulk Commodity

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ABSTRACT  Nearly all of the standards which govern the taking of samples of coal for commercial evaluation may be regarded as "old generation" standards. In general, they have been developed from procedures which were in common use more than 50 years ago when the quantities of coal traded and used were considerably smaller than today, when the needs for accurate evaluation of qualities were much less and when coal handling systems were quite primitive by today's standards. Many of the national and international organisations responsible for the publication of coal sampling standards have been actively pursuing the review of existing documents and procedures with the aim of generating new standards more appropriate to modern practice and conditions. The Standards Association of Australia has taken a leading role in this work and a new Australian Standard in 8 parts was published in 1984/85. This new standard, whilst generally acknowledged as a considerable advance on the previous Australian Standard, published in 1975, must however be regarded as an interim measure and further work is proceeding with a view to incorporating, in the next edition, as many as possible of the advances in sampling theory which have occurred and been refined over the past 5 to 10 years into what must always be an essentially practical document.

This contribution examines some of the aspects of changes in the latest coal sampling standards, with particular reference to the new Australian Standard and attempts to identify some of the further changes that seem likely to be introduced in these same features in the next generation of standards. Future standards will be aimed at further improving the accuracy of sampling coal as a bulk commodity and allowing the development of sampling schemes which are appropriate to the massive infrastructure of the industry.

INTRODUCTION

Academic as well as practical studies of coal are of importance to the Industry. They range from those which lead to more efficient extraction and preparation to those which enhance the value of the product in the market place and the acceptance of the product for a broadening range of applications in the face of strong competition.

In the current climate of oversupply by buyers can afford to be more selective than they have been in the past. Small quality advantages may mean the difference between winning and losing contracts. Thus accurate sampling and analysis become more important than ever before. Significant advances have been made over the last decade, especially through introduction of instrumental techniques, in characterising both coking and steamings coals in terms of conventional parameters, while new techniques such as coal petrography are steadily gaining wider acceptance as a result of more standardised procedures.

Improvements in the testing arena will however be of limited value unless the procedures for taking accurate and representative samples can be brought to a similar standard. Accurate sampling is essential at all stages of the coal evaluation chain, especially in the ultimate stages of marketing and utilisation.

The large numbers of tests carried out to assess the various qualities of coals during their production, preparation, marketing and utilisation rely on samples taken according to principles laid down in a variety of national and international standards. The most prominent standards in use the international trade are British (B.S.), International (I.S.O.), and American (ASTM). All of the sampling schemes followed in Australian coal exports are designed to comply with all of them. Australian Standards (A.S.), which until recently have been little more than an endorsement of the relevant B.S. document, with generally minor amendment, have tended to be used mainly for internal purposes in the same way that Japanese (JIS), German (DIN) Soviet (GHOST) and others have been used in their respective countries and regions.

All of these standards recognise that coal comprises particles of infinitely varied shapes and sizes which have different physical and chemical properties, so that for a sample to be representative it must be collected by taking a
number of portions or increments distributed throughout the whole of the mass.

All of them emphasise that the whole of the bulk of the coal to be sampled should be exposed so that all parts are equally accessible and have an equal chance of being included in the sample: the most favourable sampling situation is where the coal is being conveyed on a belt or similar device so that it passes the sampling point as a stream.

Precautions for avoiding bias where there is a likelihood of periodic quality variations coinciding with the frequency of taking increments and where size segregation and other factors may result in some particles being excluded, are outlined.

They contain stringent requirements on the minimum masses of increments which should be taken in order to be reasonably representative of the surrounding material and the minimum number of increments to be taken in particular sampling situations to achieve a required standard of precision.

There is generally, inadequate coverage of procedures required in mechanical sampling systems and on-line sample preparation trains which are an essential part of the modern coal-handling plant, and of procedures which are necessary to maintain sample integrity through to the testing laboratory.

In short, most of the national and international standards in use today in the coal industry and in trade worldwide may be considered to be out of date to a greater or lesser extent and in need of review. This is a slow and somewhat difficult process in view of the sectional interests which are at play and the entrenched practices even at the national level. The task of developing a satisfactory standard at the international level to incorporate all the advances in sampling theory and practise of approximately the last 50 years is a daunting one. It is perhaps not surprising that the ISO technical committee which commenced this work in 1979, has progressed through eight drafts of the new standard before releasing a Draft Proposal Standard, which is at present in the course of preparation.

Progress, at the national level, is slightly easier to achieve. In order to most avoid later conflict most of the groups responsible for the development of standards in the main coal producing and consuming countries prefer to defer final decisions on contentious issues until some clear indication of the directions which the new international standard will follow emerge.

In Australia however the situation was perhaps a little more urgent than elsewhere on account of the rapid advances in the coal industry during the 1960s and 1970s to the point where today's production of black coal has reached about 130 million tonnes of raw coal and over 100 million tonnes of saleable product, about three quarters of which is exported to an ever increasing range of markets and one quarter of which is consumed locally. Almost all of this massive tonnage requires sampling, much of it two to three times and to varying levels of accuracy with the highest levels of accuracy being required at the end of the processing and marketing train where it passes from producer to receiver. The fact that such a high proportion of Australia's total output of black coal is exported to the markets of the world has meant that the sampling infrastructure has had to be built up to keep pace with the development of facilities to handle such quantities. At the Kooragang Island Coal Loader at Newcastle for example, which is one of the largest of such installations in the world, the designed maximum loading rate is 10500 tonnes per hour. There are many others in Australia which approach this high level of throughput. These installations require massive and sophisticated sampling plants which, apart from the purely mechanical problems which they introduce, create some very special problems in sampling.

It is not surprising that there are considerable difficulties in translating the principles which are enunciated in standards developed over fifty years ago in relation to individual increments of a few kilograms and handling rates of a few hundred tonnes per hour at most to situations where the individual increment will be many hundreds of kilograms and rates many thousands of tonnes per hour.

In these circumstances the Standards Association of Australia's Committee on Coal and Coke, working under the direction of the Mineral Standards Board in 1979 saw the need for an urgent revision of the existing standard which had been published in 1975. The resulting work lead to the publication of a new Australian Standard, covering both coal and coke in several parts during 1984 and 1985:

Part 1 : Guide to the Use of Parts 2 to 8
Part 2 : Hard Coal - Sampling from Moving Streams
Part 3 : Coke - Sampling from Moving Streams
Part 4 : Hard Coal - Sampling from Stationary Situations
Part 5 : Coke - Sampling from Stationary Situations
Part 6 : Hard Coal - Preparation of Samples
Part 7 : Coke - Preparation of Samples
Part 8 : Determination of Precision and Bias

INCREMENT MASSES

Most of the existing coal sampling standards specify a minimum increment mass calculated from an empirical equation relating mass to maximum particle size as a linear relationship, e.g.:

\[ P = 0.06D \]

where \( P \) = minimum mass of the increment in kilograms
\( D = \) nominal topsize of the coal in millimetres

This approach results in very variable numbers of particles depending on topsize, i.e. in most cases a 100 fold increase in particle numbers for the 10-fold reduction in topsize from 50mm to 5mm which is the normal range over which bulk coal
is sampled. The equation is not applicable to material larger than about 150mm where less than one particle would be accommodated in the minimum increment. This means that some modification to the basic formula must be introduced. But even so the number of particles is still in most cases very considerably less than when sampling small coal and would result in much lower precision if the same rules regarding the number of increments taken from a testing sample were followed.

The new Australian Standard - and others under review - use an equation for determining increment mass based on the cube of particle topsize and include a density factor e.g:

$$P = 3 \times 10^{-5} \rho D^3$$

where $P$ and $D$ are defined as above and $\rho$ = average density of the coal in kilograms per cubic metre

$$3 \times 10^{-3} = \text{a factor obtained from the relationship}$$

$$10\pi t^2 \times 10^{-3} \text{ where } t = \text{Student's } t$$

This formula, while theoretically more appropriate, still results in major practical problems which in some circumstances, tend to outweigh the advantages. The advantages are that with small particle sizes (say 5 - 10mm) which are sampled at the secondary and tertiary stages of a mechanical sampling plant after primary increments have been crushed, the minimum mass required will be smaller than required by the old standards but the disadvantages are that with large coal (say 150mm or greater) which may require sampling at for example the mine, the minimum mass will be extremely large.

One solution to this problem may be to discard the principle of minimum increment masses altogether and replace it with the concept of minimum sample mass based on the measured co-efficient of variation between individual particles for the quality characteristic under consideration e.g.

(1) using a formula based on the measured coefficient of variation between individual particles of the quality characteristic under consideration

$$m_G = 0.03 \left( \frac{C_v}{S} \right)^2 \rho D^3$$

where

$m_G$ = minimum gross sample mass in kilograms

0.03 = rounded value of the coefficient $10\pi t^2 \times 10^{-3}$ for $t = 2.262$

$C_v$ = coefficient of variation between particles of the quality characteristic under investigation

$\beta_S$ = required relative sampling precision at the 95 percent confidence level expressed in percent

$\rho$ = density of the ore particles (not bulk density) in tonnes per cubic metre

$D$ = nominal top size of the ore in the lot in millimetres, or

(2) considering coal as a binary type ore consisting of mineral and gangue particles only and applying the following formula derived by Gy

$$m_G = (100 - c) \left[ (100 - c) \rho_c + \rho_g \right] lfgb^3$$

where

$m_G$ = minimum gross sample mass in kilograms

c = concentration of the mineral species containing the quality characteristic of interest in percent

$\rho_c$ = density of mineral particles containing the quality characteristic of interest in tonnes/m$^3$

$\rho_g$ = density of the gangue particles in tonnes/m$^3$

$f$ = particle shape factor (normally 0.5)

g = size range factor (between 0.25 and 1.00)

$D$ = nominal top size of the ore in millimetres

l = liberation factor

$= \sqrt{D_j/D}$, where $D_j$ is the nominal top size in millimetres at which complete liberation occurs

S = required relative sampling precision at the 95 percent confidence level in percent

This approach would introduce the necessity to know much more about the actual material under consideration, which is also of importance in other factors of the sampling process. It would obviate the need to assume that the material will behave in much the same manner as any other coal or that it can be allocated to one of the limited number of classes all the numbers of which are assumed to have similar sampling characteristics.

Even if it were feasible to carry out the necessary test programs to determine all of the appropriate co-efficients of variation of all of the coals one may wish to sample there may still be difficulties. The sampling devices and systems to perform the tasks on a routine basis are usually not capable of infinite variation and some compromise has to be found.

INCREMENT NUMBERS

The number of increments to be taken from a quantity of coal and the manner in which they are grouped to form samples for analysis is a function of the variability of the lot and the precision required in the final test result. This variability depends on the amount of segregation present in the bulk for which the test result is required, the particle size range of the coal and the size of the parcel. Furthermore it is influenced by a wide range of geological, mining, preparation and handling factors. The desired precision will be determined by commercial factors.

Most sampling standards have been developed around a "reference" standard of precision

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generally set at ± 10% relative precision and the sampling of quantities up to 1000 tonnes with certain variations. The variations are designed to take account of the difficulties of sampling in some situations such as stockpiles or wagons compared with the preferred situation of a falling stream where parts of the mass should have an equal chance of being taken and different "conditions" of coal, e.g. cleaned, uncleaned, sized, graded and blended.

Whilst it would have been difficult to conceive of the very large quantities of coal in individual consignments today, the old standards do provide a mechanism for adjusting the number of increments when it is necessary to sample consignments larger than 1000 tonnes e.g. by multiplying the number of increments specified for a mass of 1000 tonnes by the formula

\[ \frac{\text{consignment mass (in tonnes)}}{1000} \]

But generally preference is given to simply divide the total quantity into a series of 1000 tonne portions and treating each of them individually. Standards also provide mechanisms for adjusting the precision to a level different from the ± 10% "reference" usually through formulae which are of limited value unless the chosen precision is fairly close to the "reference" standard e.g.

\[ n_d = n_f \left( \frac{4A_1^2}{5A_2^2 - A_1^2} \right) \]

where \( n_d \) = number of increments required for the desired precision
\( n_f \) = number of increments for 1000 tonnes, specified according to the condition of coal and the sampling situation
\( A_1 \) = "reference" standard of precision
\( A_2 \) = desired precision

These approaches have been considered unsatisfactory and inappropriate to modern practice and the more stringent uses to which analyses performed on the samples are put, for the following reasons.

1. The "reference" standard at ± 10% relative precision is too low (numerically high) for most commercial purposes and tends to encourage very imprecise sampling.

2. The "initial" numbers of increments are set in relation to impractically low masses of consignments (1000 tonnes) and the procedures for adjusting increment numbers for more practical masses will lead to loss of sample integrity, in particular loss of moisture which is in most cases, particularly in a commercial transaction, one of the most important test parameters.

3. They assume that all coals in the same general category or condition will have the same or similar increment variance and therefore the same sampling characteristics, e.g. that all uncleaned coals will be more variable than all cleaned coals which is not necessarily the case. In fact with modern coal cleaning practices it is often the highest grade and most refined products which are the most difficult to sample.

Because of the problems outlined above, many of the newer standards including the new Australian Standard focus greater attention on the variability of the actual coal to be sampled. They set out fairly straightforward procedures for establishing a sampling scheme which will achieve the desired level of precision through determinations of primary increment variance (normally of ash but other critical parameters may be substituted) and the variance of sample preparation and testing. Using this approach the number of increments required to achieve the desired precision for the lot to be sampled may be calculated from the equation

\[ n = \frac{V_I}{\left( \frac{\alpha_{\text{SPPT}}}{2} \right)^2 - V_{\text{PT}}} \]

where \( n \) = number of increments
\( V_I \) = increment variance
\( V_{\text{PT}} \) = variance of preparation and testing
\( \alpha_{\text{SPPT}} \) = overall precision

Despite these changes of approach, which give a theoretically more correct determination of the numbers of increments to be taken, there are still problems in practice. For example if the quantity of coal to be sampled is to be treated as a single entity (one gross sample for testing) the number of primary increments to be taken may be quite large, even for relatively low levels of overall precision and it may not be possible to take increments at the required frequency. In addition, if the selected or desired precision is high (numerically low) in relation to variance of preparation and testing then the number of increments cannot be calculated.

In these circumstances it becomes necessary either to select a new overall precision (worse than that originally selected) or to divide the consignment into a number of sampling units and to perform preparation and testing on each of them individually and to average the results. In this case the number of increments required from each of the sampling units may be calculated from the equation

\[ n = \frac{V_I}{n_s \left( \frac{\alpha_{\text{SPPT}}}{2} \right)^2 - V_{\text{PT}}} \]

where \( n \), \( V_I \), \( V_{\text{PT}} \) and \( \alpha_{\text{SPPT}} \) are defined as above and \( n_s \) = number of sampling units.

It is usually possible, within the practical limitations of any particular sampling system to achieve a desired or commercially acceptable precision using different combinations of increment numbers and sampling units.
The method for determining quality variation based on measuring the variance $V_1$ of a particular quality characteristic for all increments from the lot assumes that the quality of the coal varies in a random manner throughout the mass and that the observations will follow a normal distribution. This is not strictly correct as coal flowing in a stream will frequently display serial correlation i.e. long-term trends superimposed on the random short-term variations. The value of $V_1$ and thus the number of increments required for the desired precision may therefore be overestimated.

New approaches, including the use of variograms and the calculation of fundamental sampling error as described by Gy (1982), Royle (1983), Holmes (1985), Lyman (1986) and others have been proposed to overcome these difficulties and it is expected that they will be incorporated in future editions of coal sampling standards.

DIVISION OF PRIMARY INCREMENTS

Earlier standards did not permit division of a primary increment prior to crushing but with the much larger masses handled in modern systems this would be impractical. Therefore new systems have been introduced for dividing these quantities into a mass suitable for crushing. This is usually done as an online function.

MECHANICAL ASPECTS

The older standards contained insufficient information on critical design factors for components of a mechanical sampling system, in particular cutter velocities and cutter apertures and the angles at which cutters intersect the stream of coal. The factors have a significant bearing on the bias which may be introduced. Limitations are now being put on these factors but to date have been determined on a mainly theoretical basis.

MASS BASIS/TIME BASIS

Most of the standards have shown procedures for taking increments on a random basis or on a systematic basis. The random basis, provided it is done within fixed strata, has some advantage in countering systematic errors due to periodic quality variation. It is usually not applicable in mechanical sampling situations because of the additional capacity required to handle the "bunching" of increments. Therefore most sampling involves the taking of increments at regular intervals, usually on a time basis. However this is not applicable in systems which are subject to tonnage surges or show a wide variation in feed rate. In these cases the increments should be taken at uniform mass intervals.

CO-ORDINATION OF STANDARDS

Standards for the sampling of mineral commodities other than coal have likewise developed in a variety of directions. Although there may be some special requirements for each commodity there is no fundamental reason why the basic principles and equipment should be different. The need to "standardise" standards has been clearly recognised by the groups responsible for the development of sampling standards for coal and coke, iron ores, aluminium ores, heavy mineral sands, copper, lead and zinc ores and concentrates, ferroalloys, oil shales and alumina in Australia.

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