

Australian Standard[®]

**Coal and coke—Analysis and
testing**

**Part 24: Guide to the evaluation of
measurements made by on-line coal
analysers**

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RECONFIRMATION
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Coal and coke—Analysis and testing
Part 24: Guide to the evaluation of measurements made by on-line coal analysers

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NOTES

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PREFACE

This Standard was prepared by the Standards Australia Subcommittee on Coal Evaluation, in collaboration with the Subcommittee on Coal Sampling and under the supervision of the Committee on Coal and Coke.

In preparing this document, reference was made to the ISO working draft *Solid mineral fuels—Evaluation of the measurement performance of on-line analysers*, August 1995. Additionally, reference was made to the draft ASTM *Standard guide to the evaluation of measurements made by on-line analysers*, Draft #9, May 23, 1994.

The objective of this Standard is to provide users of on-line coal analysers with guidance to the interpretation of the measurements made, so that the performance of analysers, which offer significant benefits to the coal industry, can be evaluated.

The term ‘informative’ has been used in this Standard to define the application of the Appendices to which it applies. An ‘informative’ appendix is for information and guidance only.

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FOREWORD

With the introduction of on-line analysers to the coal industry, coal analysis in real time has become technologically possible. Immediate on-line quality measurement offers substantial commercial benefit to coal producers and consumers.

In order for users to accept the analyses generated by on-line analysers, it is necessary to have an understanding of the limitations of both the on-line analysers and conventional sampling/laboratory practice.

This Guide outlines the procedures necessary for assessing the performance of on-line analysers, by comparison with the results of conventional sampling and laboratory techniques.

Detailed descriptions of statistical methods have been avoided in this Guide, and for more information reference should be made to the many papers on the subject.

STANDARDS AUSTRALIA

Australian Standard

Coal and coke—Analysis and testing

Part 24: Guide to the evaluation of measurements made by on-line coal analysers

1 SCOPE This Guide sets out recommended practices for the evaluation of on-line coal analysers.

It defines various categories of analyser and describes their operation. It also describes appropriate techniques of statistical assessment and makes specific recommendations concerning the procedures for evaluation and calibration maintenance of on-line analysers.

This Guide will—

- (a) clarify terminology;
- (b) present an overview of different kinds of analysers commercially available;
- (c) provide general observations regarding interpretation of measurements by on-line analysers; and
- (d) present to prospective users options available to evaluate performance of on-line analysers.

NOTE: A bibliography of reference texts appropriate to the various sections of this Guide is given in Appendix A.

2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS

2243 Safety in laboratories

2418 Coal and coke—Glossary of terms

4264 Coal and coke—Sampling

4264.1 Part 1: Higher rank coal—Sampling procedures

4264.3 Part 3: Lower rank coal—Sampling procedures

4264.4 Part 4: Determination of precision and bias

Code of practice for the safe use of radiation gauges—National Health and Medical Research Council

3 DEFINITIONS For the purpose of this Standard, the definitions given in AS 2418 and those below apply.

3.1 Absolute dynamic precision—a measure of the extent of agreement between test results, obtained from measurements made over a defined period of time on a set of samples of coal presented to the analyser in its normal operational mode by a comparative test method that eliminates bias and random errors attributable to the reference method.

3.2 Accuracy—the closeness of agreement between an observation and the true value. The accuracy of a result is not to be confused with its precision. A determination can be made with great precision so that the standard deviation of a number of determinations on the same consignment of coal may therefore be low, but the results will be accurate only if they are free from bias. Thus, accuracy includes the effect of precision and bias.

3.3 Bias—the tendency to obtain a value that is either persistently higher or persistently lower than the reference value; in practice, the difference between the reference value and the average result obtained from a large number of determinations (see AS 4264.4).

3.4 By-line type installation—a configuration in which a representative sample of the process stream is diverted by means of a suitable sampling system for presentation to an on-line analyser.

3.5 Comparative test method—a method of test in which the values obtained from the analyser are compared with corresponding values obtained from one or more reference methods.

3.6 Comparison period—that period of time for which a gross sample is collected and data from the analyser logged, to provide, in each case, single values for the measurand which are subsequently associated in a comparative test method.

NOTE: The period may be based on the typical time to produce a particular mass of coal, e.g. a train load, or in a period which coincides with plant operations, e.g. a shift, or some other period which is convenient to, or preferred for a specific evaluation procedure.

3.7 Interrogation process—that procedure that elicits from the coal process stream a measurable response related, specifically or by inference, to the quantity of the measurand.

3.8 Interrogation volume—that volume of the coal process stream in which the detected response to the interrogation process originates.

3.9 Interrogation zone—that part of the analyser installation through which the coal process stream passes and in which it is subjected to the interrogation process.

3.10 Measurand—the quality parameter being measured.

3.11 On-belt type installation—a configuration in which the whole of the process stream is presented to an on-line analyser.

3.12 On-line analyser—an instrument for the measurement, continuously or intermittently, of one or more quality parameters of coal while it is undergoing processing or handling, to give data rapidly and automatically.

NOTE: This Guide is not applicable to measurements by analysers used for slurries.

3.13 Precision—a measure of the extent to which observations within a set agree with each other; expressed as twice the standard deviation (95% confidence limit).

3.14 Reference test method—a method of sampling, sample preparation and analysis in accordance with the relevant parts of AS 1038 and AS 4264 which is expected to give, for the coal process stream under test, a result that is a best estimate of the true value.

3.15 Reference value—a value that serves as an agreed reference for comparison, and which is obtained from a test carried out by a reference method.

3.16 Relative dynamic precision—a measure of the extent of agreement among test results, obtained from the measurements made over a defined period of time, on a set of samples of coal presented to the analyser in its normal operational mode, with results obtained by a comparative test method which eliminates bias but includes random errors attributable to the reference method.

3.17 Static precision—a measure of the extent to which test results, obtained from the measurements made over a defined period of time, on a reference standard placed in the interrogation zone of the analyser, agree with each other.

4 SAFETY For information on laboratory safety, reference should be made to the relevant parts of AS 2243.

Reference is also made to the ‘Code of practice for the safe use of radiation gauges (National Health and Medical Research Council, 1982).’

5 ANALYSER INSTALLATIONS

5.1 General The wide range of analyser types and possible installation configurations demand that specific details of each must be taken into consideration when making an evaluation in accordance with this Standard.

The majority of on-line coal analysers depend upon the existence of a quantitative relationship between the measurand and the degree of absorption or scattering of a beam of radiation in the X-ray, gamma-ray, microwave or optical energy regions. The source, sample and detector may be arranged in transmission or backscatter geometry.

A second group depends on a quantitative relationship between the measurand and the emission of gamma radiation either from natural sources within the fuel or as a result of activation by an outside source, such as a neutron source.

The coal to be analysed may be transported through or past the analyser on a conveyor belt or within the confines of a chute or pipe. In most designs, analysers are physically non-invasive and, except for the means by which the process stream is transported and profiled, non-contacting with the solid fuel.

NOTE: Appendix B gives examples of types of on-line analyser systems.

5.2 Methods of analysis

5.2.1 X-ray/gamma backscatter The X-ray backscatter technique was one of the first developed to measure mineral matter and relate it to ash. It has the disadvantage of requiring careful sample preconditioning of the coal stream and has been largely superseded by gamma-ray backscatter devices and dual-energy gamma-ray transmission meters.

5.2.2 Dual-energy gamma-ray transmission Dual-energy gamma-ray transmission is one of the most widely applied systems for direct measurement of mineral matter and the derived ash of bulk consignments. It relies on the transmission of two different energies of gamma rays through the coal. It can be applied directly to conveyor belts at almost any accessible location. Variation in mineralogy can limit the accuracy for some coal types.

5.2.3 Natural gamma Natural gamma relies on the fact that some of the elements found in coal mineral matter are naturally radioactive. Measurement of the natural gamma rays emitted by the coal can give an indication of the level of ash. Provided that the ash mineralogy is favourable, it can be used direct on-belt for a similar suite of applications as the dual-energy technique.

5.2.4 Prompt gamma-neutron activation analysis (PGNAA) PGNAA measures the individual elements in the coal by recording the gamma rays emitted when the coal is irradiated with neutrons. It can measure most elements in coal and offers the best accuracy of all the current measurement techniques.

5.2.5 Electrical capacitance Electrical capacitance is a system that responds to the moisture content of the sample. The electrical capacitance of the coal is measured at radiofrequency, which correlates with the moisture content. In recent applications, it has given way to the more accurate microwave technique.

5.2.6 Microwave In microwave analysis, a beam of microwave radiation is transmitted through the coal stream to be analysed. Measurement of the phase shift and attenuation of the signal allows moisture content to be determined.

5.3 Installation configurations

5.3.1 General Two categories of configurations for the installation of on-line analysers are possible. The choice between the two for any particular application depends on the type of analyser appropriate to the measurand and parameters of the product and the plant, such as particle size and flow rate. They are here referred to as on-belt and by-line installations.

An on-belt installation is a system in which the analyser interrogates a ‘band’ of the process stream for which the analytical information is required. None of the coal is removed from the stream, but the system may contain conditioning steps, such as mixing and profiling, prior to interrogation by the analyser.

A by-line installation is a system in which a portion of the process stream is diverted to the analyser by means of a suitable sampling process. The diverted portion of the stream may be subsequently subjected to sample preparation procedures, such as crushing and dividing, and conditioned before presentation to the analyser. After interrogation, the substream is normally returned to the main process stream.

5.3.2 On-belt analysers On-belt analysers (see Figure 1) are mounted directly on the conveyor belt carrying the full stream of coal. The coal is interrogated by sensors mounted above or below the conveyor. Some types of analyser require contact with the coal or the conveyor, other types do not.

This type of analyser is limited to a narrow analysis ‘band’ and assumes uniformity across the belt width. Because in many cases the analysis region may be only a small part of the total stream, accuracy may be limited by the variation in presentation of coal to the analyser. Variation in coal thickness, coal profile and segregation of coal in the analysis region all may contribute to a lack of precision and accuracy.

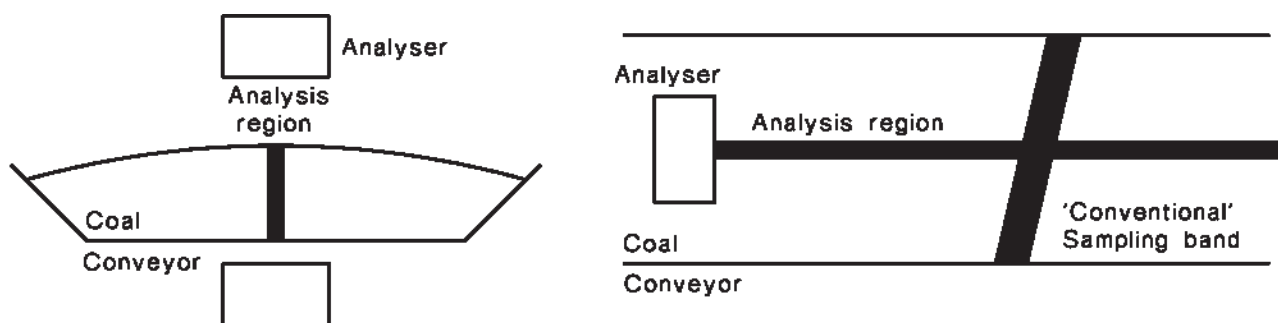


FIGURE 1 ON-BELT ANALYSER

5.3.3 By-line analysers By-line analysers (see Figure 2) require a mass flow rate of material substantially less than the full stream of coal. A sampling system is required to sample the full stream to provide the required mass flow rate of coal for the analyser (see AS 4264.1).

The analyser usually incorporates a chute or tube to carry the coal through the analysis region, although a small conveyor belt may sometimes be used. The chute or tube configuration generally provides better presentation of the coal to the analyser than can be achieved by an on-belt analyser.

A disadvantage of this type of analyser is that only incremental samples are analysed. The performance of the analyser is therefore dependent on—

- (a) sample increments free of relative bias from the sampling system; and
- (b) the precision achieved by sampling.

In many cases, a secondary sampler may be installed before or after the analyser to provide a subsample for laboratory analysis.

NOTE: See also Appendix B.

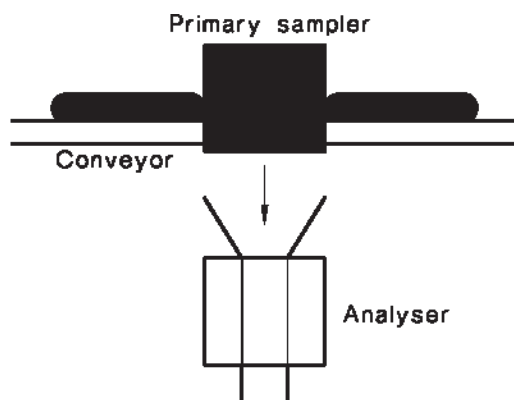


FIGURE 2 BY-LINE ANALYSER

Certain types of by-line analyser have sufficient mass flow rate capacity to handle the full stream (see Figure 3) in some smaller mass flow rate applications. The large flow rates encountered in the coal industry limit the number of applications of this type.

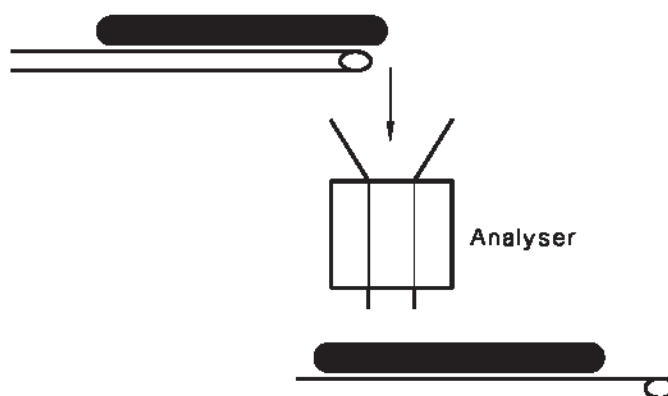


FIGURE 3 BY-LINE ANALYSER (FULL STREAM APPLICATION)

6 EVALUATION PROCEDURES

6.1 Introduction On-line analysers do not determine coal quality indicators directly; they make measurements by a comparative method in which results are obtained by comparison with a reference test method. However, due to the range of configurations for on-line analysers and their relationship to sampling/analysis systems (i.e. some share a primary sampler whereas for others both systems are completely separate), it is not possible to provide particular test methods to cover all situations; rather, the principles involved are outlined in this Guide with reference to certain situations. Some of the errors that are attributable to the analyser occur only as a result of operations on, or interactions, with the moving process stream. Consequently, it is essential that comparative test methods are undertaken while the whole system is operational, i.e. under dynamic conditions.

An understanding of the sources of variation that contribute to the errors of measurement of the analyser is essential for the evaluation of analyser performance. The validity of conclusions drawn about analyser performance depend upon the allocation of errors to their proper sources.

NOTE: Appendix C describes the principal sources of variations that are likely to occur in on-line analyser installations and associated sampling and analysis schemes.

To make a full evaluation of on-line analyser performance, it is necessary to address the following three interdependent aspects of analyser operation:

- (a) Instrument stability (see Clause 7).
- (b) Evaluation of calibration (see Clause 8).
- (c) Operational on-line measurement performance as follows:
 - (i) Relative dynamic precision (see Clause 9.2).
 - (ii) Absolute dynamic precision (see Clause 9.3).

The tests become more comprehensive from (a) to (c) above. If a comprehensive test such as that for relative dynamic precision meets the performance requirements, it is inferred that the less comprehensive parameters (i.e. stability and calibration) are within specification, although they have not been tested.

The procedures involved vary, but all rely on a common core of test methods and data analysis techniques.

Suitable test methods and their uses are summarized in Table 1.

TABLE 1
TESTS AND THEIR USES

Test method	Purpose of test			
	Stability	Calibration evaluation	Operational performance	
			Relative dynamic precision	Absolute dynamic precision
Reference material (see Note 1)	✓	✓		
2-way test (see Note 2)		✓	✓	✓ (where precision of reference method is known)
3-way test (see Note 3)				✓

NOTES:

- 1 A reference material is placed in the analyser measuring zone and periodic measurements are taken of the control parameter. The mean and standard deviation of the results are used to indicate changes to the analyser static performance.
- 2 The on-line analyser results are compared to laboratory analysis results on samples collected on line.
- 3 The on-line analyser results are evaluated with two independent laboratory analysis results collected on line.

6.2 Steps in evaluation

6.2.1 General In carrying out tests on an on-line analyser, the principal steps involved in an evaluation procedure are as follows:

- (a) Decide the aspect of analyser operation to be evaluated.
- (b) Establish the conditions for a suitable test method.
- (c) Carry out the test.
- (d) Apply appropriate statistical treatment.

6.2.2 *Conditions for tests*

6.2.2.1 *General* The following actions need to be undertaken initially to establish the conditions for conducting comparative tests:

- (a) Define the comparison period.
- (b) Determine the number of comparison periods.
- (c) Estimate the precision to be expected for the reference test method.

6.2.2.2 *Size of comparison period* As a basic guideline, the length of a comparison period should be sufficient to allow the collection of an adequate number of increments to give a reasonable precision to reference values and to minimize the error of the analyser due to instrumental and presentation variations.

This is particularly true for the evaluation of calibrations, where maximum precision of the reference values within the practical constraints of gross sample size and plant operations should be aimed for.

Absolute dynamic precision should be measured over periods that reflect the need, by the test method, to constrain the precision of the reference test method to be approximately the same as the expected analyser precision.

Relative dynamic precision should be measured over the period normally used for routine plant operations for which the data are required.

6.2.2.3 *Number of comparison periods* The number of comparison periods required is governed by the type of evaluation and the statistical treatment to be used. For determining RMSD (see paragraph E4 of Appendix E), there should be a minimum of 20, whereas for a three-way comparison (Grubbs estimator method) that minimum should be increased to 60.

Consideration also has to be paid to the expected precision of measurement; less precise values, such as may arise from short period comparison, require a larger number of sets of observations to infer statistical significance in the precision estimate.

It is imperative that the number of periods is large enough and the total duration of the test sufficient to cover the whole range of measurand values expected.

6.2.2.4 *Precision of reference samples* If it is not already known from previous experience with the particular coal and the sampling schemes that are to be used, the precision to be expected is estimated for a single sample for a period of the chosen size (see AS 4264.4 for methods for estimating precision of sampling).

For systems in which a mechanical sampling system is installed, the collection and preparation of the test samples representing the comparison period required shall be carried out in accordance with the relevant requirements of AS 4264.4. If the coal concerned is brown coal or lignite, due regard shall be made to any particular requirements of AS 4264.3. If the procedure is based on manual sampling, then the appropriate requirements of AS 4264.1 apply. The time at which each sample is collected shall be noted to ensure that proper synchronization of the corresponding data collected for the analyser is achieved.

Analysis of the test samples shall be carried out according to the relevant Australian Standard appropriate to the measurand concerned.

If a reasonable precision is not indicated, the conditions of the reference sampling procedure are adjusted (see AS 4264.4 for checking and adjusting sampling precision). Table 2 gives some typically expected values of precision for various measurands and types of on-line analysers. These values may be used if no firm values are suggested by the analyser manufacturer.

TABLE 2
TYPICAL RMSD VALUES OF PRECISION

Parameter	Dual energy	PGNAA
*Total moisture ar	0.5	0.5
Low ash < 12% d	0.3 - 0.5	0.2 - 0.5
High ash > 12% d	0.6 - 1.5	0.5 - 1.0
Sulfur < 0.8% d	nd	0.04
Sulfur > 0.8% d	nd	0.06
Specific energy MJ/kg d	±0.35	0.23

* By microwave technique.

† Derived from relationship to moisture/ash.

7 EVALUATION OF INSTRUMENT STABILITY

7.1 Introduction A prerequisite of accurate measurement by an on-line analyser is that the instrumentation is stable and contributes as little as possible to the total error of measurement.

A useful and rapid check of the analyser instrumentation may be obtained by making periodic measurements of the precision and the level of response of the analyser with respect to some reference standard. In some analysers, this check is carried out automatically.

Where possible, it is recommended that this test be carried out as part of the commissioning of the analyser to determine the initial stability (sometimes referred to as baseline stability). Once the initial stability is established, the subsequent stability is compared statistically to the initial stability and if any deviation is observed, investigation into instrument stability is required.

7.2 Initial stability measurement

7.2.1 General The precision of a number of readings taken with a static reference material in the analysing zone of the analyser is used as a measure of the instrument stability. This precision (static precision) is a function of the variations attributable to the instrumental characteristics of the analyser. It provides an indication of the limiting value of accuracy available from the analyser.

The reference material must be stable over a protracted period of time and provide a response that is typical of that expected from the solid fuel normally being processed. Suitable materials are air, perspex or aluminium blocks, synthetic coal samples.

7.2.2 Conditions of test The measurement period is first decided upon (using the manufacturer's recommendation).

Then, for the purpose of assessing the contribution of the instrumental characteristics to the total measurement precision of the analyser, the same total period is chosen as that which will be used in routine measurements for maintenance of performance (see Clause 9.2).

7.2.3 Procedure The procedure is as follows:

- (a) Place the reference standard into the interrogation zone of the analyser and make repeated measurements under the chosen conditions of test.