NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB/T 19494.3-2023

[Including 2025XG1] Mechanical sampling of coal - Part 3: Determination of precision and bias test

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1 Scope

This document specifies the methods for mechanized sampling, sample preparation, and precision determination of coal analysis, as well as bias testing methods.

This document applies to the precision determination and bias test of mechanized coal sampling.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 19494.1, Mechanical sampling of coal - Part 1.Method for sampling

GB/T 19494.2, Mechanical sampling of coal - Part 2.Method for sample preparation

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 19494.1 and GB/T 19494.2 apply.

4 Sampling, sample preparation, and precision determination

of laboratory tests

4.1 Overview

When designing a sampling scheme with acceptable precision, there needs to be a correlation between precision and coal characteristics and sampling characteristics. The main correlation factors are coal variability (i.e., subsample variance), sample preparation and testing errors, number of subsamples and total number of samples, and specimen quality.

There are several methods for estimating precision, depending on the purpose of sampling and the sampling scheme and equipment used. There are two main cases.

a) For existing sampling systems, the purpose of testing is to check whether the sampling scheme can actually achieve the desired precision. If it cannot, it needs to be improved until the precision reaches the desired value. The most rigorous method for verifying the precision of routine sampling schemes is double sampling with multiple sampling units (see 4.4.1).

b) For a specific batch of coal, the purpose of the test is to estimate the achievable precision from the actual test results. In this case, the best method is the multiple sampling method (see 4.4.2).

The precision calculation formula presented in this document is based on the following assumptions: the quality variation of the sampled coal is random; and the quality observations are normally distributed. Although these two assumptions are not very accurate for some coal quality parameters, the degree of deviation between the actual state and the assumed state has no practical impact on the validity of the precision verification formula, because the statistical methods used in this document are not very sensitive to non-normal distributions.

Coal quality often exhibits a certain serial correlation, meaning that adjacent coals tend to have similar compositions, while coals that are far apart tend to have dissimilar compositions. The variance of the primary subsample obtained from tests may differ from the actual variance; therefore, the precision estimates obtained from the primary subsample variance and the variances of sample preparation and analysis will also differ from the actual results.

4.2 Relationship between sampling, sample preparation, overall precision of

analysis, and its influencing factors

4.2.1 Overview

Precision is the degree of agreement between results obtained by repeatedly performing a measurement procedure under specified conditions. It is a characteristic of the measurement method used. The smaller the random error of a method, the higher its precision. The precision value is usually taken as twice the population standard deviation.

If j total samples are taken from a sampling unit and prepared and tested separately, the estimation precision P of a single observation is calculated according to formula (1).

Where,

4.5.1 Overview

The sample preparation and testing precision verification methods provided in this clause are used to estimate the random errors (expressed as variance) at each stage of sample preparation, including the total variance of sample preparation and testing and the variance of each stage.

Each sample preparation stage generally includes steps such as crushing, mixing, and reduction. Sample preparation errors mainly arise from the reduction process and the final sampling of a certain number of grams from the test specimen. The most important factors affecting reduction error are the particle size distribution of the specimen before reduction and the amount of specimen retained after reduction.

Since ash content is the most sensitive parameter to coal particle size distribution, as long as the ash content variance meets the requirements, other parameters in industrial analysis and elemental analysis will generally also meet the requirements. However, total moisture and calorific value may need to be verified. Therefore, this clause uses ash content as an example for description. In actual verification, all coal quality parameters can be verified if necessary.

NOTE. These methods were originally designed for manual and non-integrated mechanical preparation. If sample preparation is performed in an integrated primary sampling/preparation system, the error of individual components may be difficult to determine. While it can be measured manually, such as by refeeding discarded material into the system, these methods are not representative of normal operation. Therefore, the variance of the entire sample preparation stage may have to be measured together with the variance of the primary subsample.

4.5.2.1 Basic requirements

The total variance VPT estimated according to procedure 4.5.3 can be judged by a target value. is generally proposed by the sample preparation technician, but shall meet the requirements of GB/T 19494.2.Individual reduction errors shall be estimated directly, judged by a target value or as part of the total variance.

Roughly estimated, the variance of a reduction stage is generally twice the variance of the test. Therefore, the total variance of a 3-stage sample preparation-testing procedure can be allocated in a 2.2.1 ratio to the variances of the two reduction stages and the variance of the test.

The final target variance value for the final testing stage can be obtained from the relevant analytical test method standards according to formula (18).

Where, r - the repeatability limit for analyzing the test method.

4.5.2.2 Offline sample preparation

The coal sample preparation method specified in GB/T 19494.2 requires that, under the condition that the sample quantity meets the requirements, the variance of ash content preparation and analysis shall be below 0.2.For most coals, especially when using mechanical reducers with a cutting number much greater than the minimum cutting number specified in the standard, a much lower variance can be obtained. Therefore, empirically speaking, if similar equipment is used to prepare samples of similar coals, it is possible to specify a relatively strict target value for the total variance.

The worst-case variance of a single reduction stage (0.08 for ash) is taken as the maximum reduction variance. This variance value will decrease if mechanical reduction is used.

4.5.2.3 Online sample preparation

When certain procedures of specimen preparation are performed in a system combined with a primary subsampler, sample preparation error may be associated with the primary subsample variance (VI). In this case, the residual VPT may be smaller than the variance if all sample preparation is performed offline.

It is recommended to determine the actual total variance target value for online sample preparation based on experience. However, the maximum total variance target value shall be the sum of the worst single reduction stage variance and the test variance for each reduction stage (see 4.5.2.1).

4.5.3 Sample preparation and overall (total variance) verification procedures

The first step in the overall verification is to verify whether the total variance of sample preparation and testing exceeds the target value, that is, whether the difference between the two is statistically significant.

The verification method is as follows.

In the first stage of specimen reduction, a pair of double specimens are taken. Then, two test specimens are prepared separately (see Figure 3). Ten pairs of double specimens are taken in this way.

Let y be the average of the absolute differences of the results of 10 pairs of double Figure 3 -- Specimen preparation overall test