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Iron ores - Experimental methods for checking the bias of sampling

铁矿石 校核取样偏差的实验方法

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1 Scope

When sampling is carried out by the method laid down in ISO 3082:2002, the experimental method of this part checks the sampling bias of iron ores taking the stopped-belt sampling of ISO 3082:2002 as the reference method.

A mechanical check of the sampling system before the bias test is recommended.

For a sampling system that does not fully conform to ISO 3082:2002 bias is normally not wanted; a bias check test may therefore be carried out when the main effects of the non-conformity with the conditions of ISO 3082:2002 are disputed. If one party holds that under certain particular conditions the bias could be large, a bias test should normally also be carried out when those conditions are used.

This part applies to the experimental method for checking the sampling bias of iron ores.

A note states that the method of analysis of the test data described here is applied in this part to: a) checking the bias of iron ore sample preparation, taking the sample preparation

of ISO 3082:2002 as the reference method; b) checking the bias of the size distribution of iron ore by sieving, taking the manual sieving of ISO 4701 as the reference method; c) checking the samples collected from one and the same lot at different places, such as the loading port and the unloading port, where the results obtained may differ appreciably.

2 Normative references

The following documents are indispensable for the application of the document. For dated references only the dated edition applies; for undated references the latest edition, all its amendments included, applies.

GB/T 20565-2006 Iron ores and direct reduced iron - Terminology (ISO 11323:2002, IDT); ISO 3082:2002 Iron ores - Sampling and sample preparation procedures; ISO 3085:2002 Iron ores - Experimental methods for checking the precision of sampling, sample preparation and measurement; ISO 4701 Iron ores and direct reduced iron - Determination of size distribution by sieving.

3 Terms and definitions

The terms and definitions given in GB/T 20565-2006 apply to the document.

4 Principle

The results obtained by the method under examination, called method B, are compared with those obtained by the reference method, called method A, which by technique and by experience is held not to produce bias. If there is no statistically significant difference between the results of method B and those of method A, method B may be adopted as the routine method. The difference is assessed, within a confidence interval of 90 %, by comparing the measured mean bias with the corresponding bias delta given in 5.2.

5 General conditions

5.1 The number of delivery lots tested shall not be lower than 10. Whether the number of test lots has to be increased depends on the results of the statistical analysis of the actual mean bias confidence interval and on the results of the outlier test carried out on at least 10 lots. A note adds that method A and method B are used to sample the same raw material and that the same method is then used to prepare and to measure the samples, so that a pair of test data is obtained.

5.2 The relevant bias delta shall be fixed in advance. When the relevant bias delta is already large enough, the measures taken to lower the mean bias are reasonable. As a principle the relevant bias delta may be smaller than the standard deviation of sampling, sample preparation and measurement determined according to ISO 3085:2002. A note adds that, if the purpose of the experiment is only to check sample preparation, the value of delta may

be smaller than the corresponding standard deviation determined according to ISO 3085:2002.

5.3 Quality characteristics such as total iron content, moisture content, size distribution and physical properties may be used.

6 Sampling and sample preparation methods

6.1 Sampling. The reference method for checking the sampling bias, method A, is the stopped-belt sampling of ISO 3082:2002. In method A each increment is cut, at the position laid down, from the ore stream on the stopped conveyor belt over the full width of the stream; the length of the cut shall be greater than three times the nominal top size or at least 30 mm, whichever is the larger. Method B, the method under examination, shall be carried out as far as possible according to ISO 3082:2002 and shall use the same raw material as method A; examples of method B are sampling with a mechanical sampler during movement and sampling during transfer, or sampling from the hold of a ship or from a wagon. Taking into account the fluctuation of the ore flow, the samples of method A and of method B shall be taken from parts as close to each other as possible.

6.2.1 From one and the same lot, the increments obtained by method A and by method B make up two gross samples A and B.

6.2.2 The two gross samples A and B shall be prepared by the same method according to ISO 3082:2002 and tested separately according to the relevant standards, so that a pair of measured values is obtained.

6.2.3 The steps above shall be repeated for 10 lots or more, as required by 5.1. When method A and method B can take their increments from neighbouring parts of the ore, it is recommended that the increments or the sub-samples be prepared and tested separately, so that 10 or more pairs of measured values can be compared, which is quicker than comparing the measured values of all the lots. The comparison shall use ore of the same kind, taking paired increments from a number of lots; increments and gross samples may not be paired together, and the comparison shall be made between a number of pairs of increments or between a number of pairs of gross samples. A note recalls that stopped-belt sampling is costly and inconvenient, so that the usual economical practice is to carry out paired sample preparation and testing with the greatest care, in order to reduce the number of stopped-belt samplings.

7 Analysis of the test data

7.1.1 The measured values obtained by method A and by method B are written X with the subscript Ai and X with the subscript Bi. When the same method is used for sample preparation and measurement, they may also stand for the mean of the data measured by one and the same method.

7.1.2 Formula (1) gives the difference d with the subscript i between X with the subscript Ai and X with the subscript Bi, as the value of method B minus the value of method A, for i running from 1 to k, where k is the number of pairs of measurements.

7.2.1 Formula (2) gives the mean of the differences, written d with a bar, carrying one decimal place more than the original values.

7.2.2 Formula (3) gives the sum of squares SS with the subscript d and formula (4) the standard deviation S with the subscript d, which carries one decimal place more than the process data.

7.3 Outlier test - Grubbs test. 7.3.1 The differences d with the subscript i are put in order from the smallest to the largest. 7.3.2 Formula (5) gives the Grubbs statistic G with the subscript k and formula (6) the statistic G with the subscript 1, where d with the subscript k is the largest of the differences and d with the subscript 1 the smallest. 7.3.3 The larger of G with the subscript k and G with the subscript 1 is selected. 7.3.4 That larger value is compared with the critical value of the Grubbs test at the 5 % confidence level given in Table 1.

Table 1, headed critical values of the Grubbs test, is printed as three side-by-side blocks of two columns, k and critical value at 5 %, and covers k from 6 to 23: 6 and 1.887; 7 and 2.020; 8 and 2.126; 9 and 2.215; 10 and 2.290; 11 and 2.355; 12 and 2.412; 13 and 2.462; 14 and 2.507; 15 and 2.549; 16 and 2.585; 17 and 2.620; 18 and 2.651; 19 and 2.681; 20 and 2.709; 21 and 2.733; 22 and 2.758; 23 and 2.781. A note refers, for the critical values of the Grubbs test at a wider range of numbers of observations and at other significance levels, to the paper of Grubbs, F. E. and Beck, G. and others of 1972 on the percentage points of the significance test of sample size and outlying observations, Technometrics 14, pages 847 to 854.

7.3.4.1 If the larger of G with the subscript k and G with the subscript 1 is smaller than or equal to that critical value, the data carry no outlier and the work goes on according to 7.5. 7.3.4.2 If it is larger than the critical value, then a) if G with the subscript k is the larger value, the largest difference d with the subscript k is the outlier, and b) if G with the subscript 1 is the larger value, the smallest difference d with the subscript 1 is the outlier.

7.3.5 The outlier is removed and the process described in 7.2 to 7.3.3 is repeated. 7.3.6 The larger of G with the subscript k and G with the subscript 1 is again compared with the critical value of Table 1 at the 5 % confidence level; 7.3.6.1 if it is smaller than or equal to the critical value the data carry no outlier and the work goes on according to 7.4; 7.3.6.2 if it is larger, a) the largest difference is the outlier when G with the subscript k is the larger value and b) the smallest difference is the outlier when G with the subscript 1 is the larger value. 7.3.7 If at least 60 % of the initial data are still kept, the work goes on according to 7.3.5. 7.3.8 If not, the outlier test is stopped, all the outlying values are restored and the work goes on according to 7.5.