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Aluminium ores — Experimental methods for checking the bias of sampling

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10226 was prepared by Technical Committee ISO/TC 129, *Aluminium ores*, Sub-Committee SC 1, *Sampling*.

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Aluminium ores — Experimental methods for checking the bias of sampling

1 Scope

This International Standard specifies experimental methods for checking the bias of sampling of aluminium ores, when the sampling is carried out in accordance with the procedures specified in ISO 8685.

NOTE 1 These methods may also be applied for checking the bias of sample preparation, when the sample preparation is carried out in accordance with the specifications of ISO 6140.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 6140:—¹⁾, *Aluminium ores — Preparation of samples*.

ISO 8685:—¹⁾, *Aluminium ores — Sampling procedures*.

3 General

3.1 In the experimental methods given in this International Standard, the results obtained from the method to be checked (referred to as "Method B") are compared with the results of a reference method (referred to as "Method A") which is considered to

produce practically unbiased results, from technical and empirical viewpoints.

In the event that there is no significant difference in a statistical sense between the results obtained from Method B and those obtained by Method A, Method B may be adopted as a routine method.

NOTE 2 In this International Standard, bias is assessed by application of the *t*-test (one-sided) at the 5 % significance level, by determining whether the difference between the results of Method A and of Method B are due to random chance variations or to whether the results are statistically different.

The number of paired sets of measurements shall not be less than 20. The number of data sets required depends on the standard deviation of the differences based on 20 data sets and the value of the bias, δ , to be detected as specified in clause 5.

Any chemical or physical quality may be used. The most commonly used characteristics are alumina, silica and moisture content. Bias may not always be determined for just one parameter, therefore several parameters, preferably those which would subsequently be of interest, should be determined to ensure that there is no bias. Characteristics to be tested need to be determined before the experiment begins. When increments for Method A and Method B can be taken from closely adjacent portions of the ore, it is recommended that sample preparation and testing be carried out on each increment individually. A comparison should never be made using combined data for increments, subsamples or gross samples.

The method for analysis of experimental data described in clause 5 may also be applied for checking a possible significant difference in the result obtained from the samples of one lot collected at different places, for example, a loading point and a discharging point.

1) To be published.

3.2 It is recommended that, even after a series of experiments has been conducted, the experiments should be repeated at regular intervals and whenever there is a change in ore quality. The experiment should also be repeated when there are changes in equipment or ore supply.

4 Sampling and sample preparation methods

4.1 Sampling

The reference method (Method A) for checking the bias of sampling is the stopped-belt method. The method to be checked (Method B) shall be compared with Method A using the same material.

EXAMPLE: Mechanical sampling (see ISO 8685)

Method A: Stopped-belt sampling.

Method B: Take each increment from the moving conveyor with a mechanical sampler.

4.2 Sample preparation

Methods for making up a pair of samples, preparation of samples and testing shall be as given in 4.2.1 and 4.2.2.

4.2.1 Increments obtained from one lot, in accordance with Methods A and B, are made up into two samples A and B.

4.2.2 Samples A and B are subjected in the same manner to sample preparation as specified in ISO 6140 and to measurement as specified in the relevant International Standards separately, and a pair of measurements obtained.

The above procedure is performed on 20 or more pairs of sample (see 3.1).

5 Analysis of experimental data

5.1 Determination of the standard deviation of the differences

5.1.1 Denote individual measurements obtained in accordance with Methods A and B, x_{Ai} , x_{Bi} , respectively.

5.1.2 Calculate the difference, d_i , between x_{Ai} and x_{Bi} using the equation

$$d_i = x_{Bi} - x_{Ai} \text{ with } i = 1, 2, \dots, k \quad \dots (1)$$

where k is the number of paired data sets.

5.1.3 Calculate the mean, \bar{d} , of the differences to one decimal place further than that used in the measurements themselves:

$$\bar{d} = \frac{1}{k} \sum d_i \quad \dots (2)$$

5.1.4 Calculate the sum of the squares, SS_d , and the standard deviation, s_d , of the difference:

$$SS_d = \sum d_i^2 - \frac{1}{k} \left(\sum d_i \right)^2 \quad \dots (3)$$

$$s_d = \sqrt{SS_d / (k - 1)} \quad \dots (4)$$

5.2 Determination of the required number of data sets, n_r , for experiment

Calculate the values of the standardized difference, D , using the equation

$$D = \frac{\delta}{s_d} \quad \dots (5)$$

Then determine, from table 1, the value of n_r corresponding to the value of D .

When $n_r \leq k$, proceed as in 5.3. When $n_r > k$, carry out additional experiments on $(n_r - k)$ data sets.

This procedure shall be repeated until the number of paired sets of data becomes equal to or greater than the value of n_r specified in table 1.

Table 1 — Required number of data sets, n_r , determined by the value of the standardized difference, D

Range of standardized difference D	Required number of data sets n_r	Range of standardized difference D	Required number of data sets n_r
$0,30 \leq D < 0,35$	122	$1,1 < D < 1,2$	11
$0,35 \leq D < 0,40$	90	$1,2 \leq D < 1,3$	10
$0,40 \leq D < 0,45$	70	$1,3 \leq D < 1,4$	8
$0,45 \leq D < 0,50$	55	$1,4 \leq D < 1,5$	8
$0,50 \leq D < 0,55$	45	$1,5 \leq D < 1,6$	7
$0,55 \leq D < 0,60$	38	$1,6 \leq D < 1,7$	6
$0,60 \leq D < 0,65$	32	$1,7 \leq D < 1,8$	6
$0,65 \leq D < 0,70$	28	$1,8 \leq D < 1,9$	6
$0,70 \leq D < 0,75$	24	$1,9 \leq D < 2,0$	5
$0,75 \leq D < 0,80$	21	$2,0 < D$	5
$0,80 \leq D < 0,85$	19		
$0,85 \leq D < 0,90$	17		
$0,90 \leq D < 0,95$	15		
$0,95 \leq D < 1,00$	14		
$1,00 \leq D < 1,10$	13		

NOTE — This table is taken from pages 606 and 607 of *The Design and Analysis of Industrial Experiments* published by Owen L. Davies in 1956. It lists values of n_r for D at the confidence level $\alpha = 0,05$ and $\beta = 0,05$, where α is the chance of assuming a statistical difference when none exists (i.e. the confidence level of the one sided t test) and β is the chance of assuming no statistical difference when a bias δ is present.

Table 2 — Value of t at 5 % significance level (one-sided t -test)

Number of paired data sets k	t	Number of paired data sets k	t
20	1,729	40	1,685
21	1,725	41	1,684
22	1,721	42	1,683
23	1,717	43	1,682
24	1,714	44	1,681
25	1,711	45	1,680
26	1,708	46	1,679
27	1,706	47	1,679
28	1,703	48	1,678
29	1,701	49	1,677
30	1,699	50	1,677
31	1,697	51	1,676
32	1,696	61	1,671
33	1,694	81	1,664
34	1,692	121	1,658
35	1,691	241	1,651
36	1,690	∞	1,645
37	1,688		
38	1,687		
39	1,686		

NOTE This table is taken from *Statistical Tables and Formulas with Computer Applications* (Japanese Standards Association, Tokyo, 1972).

5.3 Statistical test

Calculate the value of t_0 to the third decimal place, by rounding off the fourth decimal place:

$$t_0 = \frac{\bar{d}}{s_d/\sqrt{k}} \dots (6)$$

When the absolute value of t_0 is smaller than the value of t corresponding to k as indicated in table 2, conclude that the difference is not significant and that Method B can be adopted as a routine method.

6 Numerical examples of experiment

6.1 Numerical example 1 (δ : 0,2 % of the alumina content)

The numerical example shown in table 3 is the result of an experiment with a mechanical sampler carried out according to 4.1.

The magnitude of bias to be detected in the experiment is 0.2 % of the alumina content.

Table 3 — Numerical example 1

Data set number	Alumina content (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	59,20	59,00	0,20	0,040 0
2	59,75	59,67	0,08	0,006 4
3	62,00	61,74	0,26	0,067 6
4	62,62	63,16	-0,54	0,291 6
5	62,96	63,26	-0,30	0,090 0
6	60,02	59,92	0,10	0,010 0
7	63,17	63,11	0,06	0,003 6
8	63,91	63,87	0,04	0,001 6
9	59,98	60,42	-0,44	0,193 6
10	61,21	61,13	0,08	0,006 4
11	61,26	61,30	-0,04	0,001 6
12	58,98	59,22	-0,24	0,057 6
13	58,95	59,09	-0,14	0,019 6
14	61,97	61,89	0,08	0,006 4
15	59,36	58,88	0,48	0,230 4
16	63,74	64,24	-0,50	0,250 0
17	62,74	63,14	-0,40	0,160 0
18	60,47	60,33	0,14	0,019 6
19	62,55	63,03	-0,48	0,230 4
20	63,80	63,94	-0,14	0,019 6
Sum			-1,70	1,706 0

$$\bar{d} = \frac{1}{k} \sum d_i = \frac{-1,70}{20} = -0,085$$

$$S_d = \sum d_i^2 - \frac{1}{k} \left(\sum d_i \right)^2$$

$$= 1,706 0 - \frac{(-1,70)^2}{20} = 1,561 5$$

$$s_d = \sqrt{S_d / (k - 1)} = \sqrt{1,561 5 / 19} = 0,287$$

Thus

$$D = \frac{\delta}{s_d} = \frac{0,2}{0,287} = 0,696$$

Table 1 gives $n_r = 28$, thus the number of data sets in the experiment is insufficient. Therefore, an additional eight data sets should be collected and then the significance test should be carried out on a total of 28 data sets.

6.2 Numerical example 2 (δ : 0,15 % of the alumina content)

The numerical example shown in table 4 is the result of an experiment with a mechanical sampler carried out according to 4.1.

The magnitude of bias to be detected in the experiment is 0,15 % of the alumina content.

Table 4 — Numerical example 2

Data set number	Alumina content (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	49,50	49,00	0,50	0,250 0
2	50,05	49,67	0,38	0,144 4
3	52,10	51,74	0,36	0,129 6
4	53,32	53,16	0,16	0,025 6
5	53,26	53,06	0,20	0,040 0
6	50,32	49,92	0,40	0,160 0
7	53,47	53,11	0,36	0,129 6
8	53,91	53,57	0,34	0,115 6
9	50,28	50,02	0,26	0,067 6
10	51,51	51,13	0,38	0,144 4
11	51,56	51,30	0,26	0,067 6
12	49,28	49,02	0,26	0,067 6
13	48,95	48,75	0,20	0,040 0
14	51,97	51,59	0,38	0,144 4
15	49,36	48,88	0,48	0,230 4
16	54,04	53,75	0,29	0,084 1
17	53,04	52,80	0,24	0,057 6
18	50,77	50,42	0,35	0,122 5
19	52,85	52,62	0,23	0,052 9
20	53,80	53,53	0,27	0,072 9
Sum			6,30	2,146 8

$$\bar{d} = \frac{1}{k} \sum d_i = \frac{+6,30}{20} = +0,315$$

$$SS_d = \sum d_i^2 - \frac{1}{k} \left(\sum d_i \right)^2$$

$$= 2,146 8 - \frac{(6,30)^2}{20} = 1,162 3$$

$$s_d = \sqrt{SS_d / (k - 1)} = \sqrt{1,162 3 / 19} = 0,092$$

Thus

$$D = \frac{\delta}{s_d} = \frac{0,15}{0,092} = 1,63$$

Table 1 gives $n_r = 6$, thus the number of data sets in the experiment is sufficient.

$$t_o = \frac{\bar{d}}{s_d/\sqrt{k}} = \frac{+0,315}{0,092/\sqrt{20}} = +15,312$$

$$|t_o| > t$$

Therefore, it is concluded that there is a significant bias in Method B and action to eliminate this bias should be taken.

6.3 Numerical example 3 (δ : 0,3 % of the moisture content)

The final numerical example shown in table 5 is the result of an experiment to test the effects of particle size and mass of test samples upon moisture content. In this experiment, samples of mass less than 1 kg and of particle size less than 10 mm (Method B) were compared with samples of mass less than 5 kg and of particle size less than 22,4 m (Method A).

The magnitude of bias to be detected in the experiment is 0,3 % of the moisture content.

$$\bar{d} = \frac{1}{k} \sum d_i = \frac{-0,57}{20} = -0,028 \quad (\text{standards.iteh.ai})$$

$$\begin{aligned} SS_d &= \sum d_i^2 - \frac{1}{k} \left(\sum d_i \right)^2 \\ &= 1,609\ 5 - \frac{(-0,57)^2}{20} = 1,593\ 3 \end{aligned}$$

$$s_d = \sqrt{SS_d/(k-1)} = \sqrt{1,593\ 3/19} = 0,290$$

Thus

$$D = \frac{\delta}{s_d} = \frac{0,3}{0,290} = 1,03$$

Table 1 gives $n_r = 13$, thus the number of data sets in the experiment is sufficient.

$$t_o = \frac{\bar{d}}{s_d/\sqrt{k}} = \frac{-0,028}{0,290/\sqrt{20}} = -0,432$$

$$t = 1,729 \text{ for } k = 20 \text{ from table 2}$$

$$|t_o| < t$$

Therefore, the difference, $d = -0,028$ is not significant at the 5 % level and is negligibly small. Method B could then be adopted as a routine method.

Table 5 — Numerical example 3

Data set number	Moisture content (%)		$d_i = x_{B_i} - x_{A_i}$	d_i^2
	x_{B_i}	x_{A_i}		
1	12,64	12,99	-0,35	0,122 5
2	11,47	11,60	-0,13	0,016 9
3	12,35	12,27	0,08	0,006 4
4	12,70	12,75	-0,05	0,002 5
5	10,64	10,59	0,05	0,002 5
6	11,78	11,63	0,15	0,022 5
7	10,55	10,91	-0,36	0,129 6
8	12,92	13,29	-0,37	0,136 9
9	12,75	12,85	-0,10	0,010 0
10	12,09	12,36	-0,27	0,072 9
11	13,73	13,38	0,35	0,122 5
12	12,93	12,83	0,10	0,010 0
13	12,37	12,68	-0,31	0,096 1
14	12,09	12,27	-0,18	0,032 4
15	11,94	12,02	-0,08	0,006 4
16	12,24	11,54	0,70	0,490 0
17	12,11	11,62	0,49	0,240 1
18	10,36	10,46	-0,10	0,010 0
19	11,80	12,07	-0,27	0,072 9
20	12,14	12,06	0,08	0,006 4
Sum			-0,57	1,609 5

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