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Iron ores — Experimental methods for checking the bias of sampling

Minerais de fer — Méthodes expérimentales de contrôle de l'erreur systématique d'échantillonnage

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Foreword

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International Standard ISO 3086 was prepared by Technical Committee ISO/TC 102, *Iron ores*.

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This second edition cancels and replaces the first edition (ISO 3086-1974), of which it constitutes a technical revision.

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1 Scope and field of application

This International Standard specifies experimental methods for checking the bias of the sampling of iron ores, when the sampling is carried out in accordance with the methods specified in ISO 3081 and ISO 3082.

NOTE — These methods may also be applied for checking the bias of sample preparation when the sample preparation is carried out in accordance with the methods specified in ISO 3082 or ISO 3083.

2 References

ISO 3081, *Iron ores — Increment sampling — Manual method.*

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method.*[1)]

ISO 3083, *Iron ores — Preparation of samples — Manual method.*

3 General conditions

3.1 In the experimental methods given here, 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.

3.2 In the event that there is no significant difference in a statistical sense between the results obtained by method B and method A, then method B may be adopted as a routine method, provided that agreement on this subject is reached between the parties concerned.

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

3.3 The number of consignments or lots shall not be less than 20. The number of tests required depends on the standard deviation of the differences based on 20 tests and the value of the bias, δ, to be detected.

NOTE — The value of the bias, δ, to be detected shall be decided by agreement between the parties concerned, taking into account, from the technical and economic viewpoints, the various precisions of the methods to be adopted in the experiment, i.e. sampling, sample division and measurement precisions. As a guide, a bias value of half the overall precision, β_{SDM}, may be appropriate unless otherwise agreed.

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

1) At present at the stage of draft.

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

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.

Examples for the experiments are given in 4.1.1 to 4.1.4.

4.1.1 Example 1: Sampling from conveyors
(see ISO 3081)

Method A: Take each increment from the full width and thickness of the ore stream on the stopped conveyor at a specified place for a length of belt more than three times the maximum particle size or more than the width of the smallest increment shovel (60 mm), whichever is the greater.

NOTE — In order to apply this method, it is essential to have a length of accessible conveyor belt.

Method B: Take each increment from a point selected at random each time within the ore stream.

4.1.2 Example 2: Mechanical sampling
(see ISO 3082)

Method A: Take each increment according to method A of 4.1.1.

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

4.1.3 Example 3: Sampling from wagons
(see ISO 3081)

Method A: Take each increment according to method A of 4.1.1.

Method B: Either

a) take each increment with a sampling probe or boring sampler from the top surface of the ore loaded on a wagon; or

b) take each increment at random from the new surface of the ore exposed during loading on to or discharging from a wagon.

4.1.4 Example 4: Sampling from ships
(see ISO 3081)

Method A: Take each increment according to method A of 4.1.1.

Method B: Take each increment according to the procedure given in ISO 3081.

4.2 Sample preparation

Methods for making up a pair of gross samples, preparation of samples and testing shall be as given in 4.2.1 to 4.2.3.

4.2.1 Increments obtained from one consignment in accordance with method A and method B are made up into two gross samples A and B.

4.2.2 The gross samples A and B are subjected in the same manner to sample preparation as specified in ISO 3082 or ISO 3083 and testing as specified in the relevant International Standards separately, and a pair of measurements obtained.

4.2.3 The above procedure is performed on 20 or more consignments or lots (see 3.3).

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NOTE — 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. Thus the comparison can profitably be made on 20 or more individual pairs of measurements to check the bias of the sampling. The above comparison of measurements should be made on pairs of increments taken from several consignments, preferably of the same type of ore. However, it is not permitted to combine a number of paired results, originating from both increments and gross samples. It should be either a number of pairs from increments or ones from gross samples.

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 method A and method B, x_{Ai}, x_{Bi}, respectively.

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

$$d_i = x_{Bi} - x_{Ai}, \qquad i = 1, 2, \ldots, k \qquad \ldots (1)$$

where k is the number of paired sets of measurements.

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

$$\bar{d} = \frac{1}{k} \sum d_i \qquad \ldots (2)$$

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

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

$$s_d = \sqrt{SS_d/(k-1)} \qquad \ldots (4)$$

5.2 Determination of the required number of consignments or lots, n_r, for experiment

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

$$D = \frac{\delta}{s_d} \qquad \ldots (5)$$

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

When $n_r \leqslant 20$, proceed as in 5.3. When $n_r > 20$, carry out additional experiments on $(n_r - 20)$ consignments or lots.

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

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

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

NOTE — Table 1 is taken from DAVIES, O.L. (Ed.) *The Design and Analysis of Industrial Experiments*, 1956, pp. 606-607, and lists values of n_r for D at $\alpha = 0,05$ 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.

5.3 Statistical test

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

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

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

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

Number of paired sets of measurements, k	t	Number of paired sets of measurements, 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 — Table 2 is taken from JSA *Statistical Tables and Formulas with Computer Applications*, Tokyo, Japanese Standards Association, 1972.

6 Numerical examples of experiment

6.1 Numerical example 1 (δ : 0,2 % total iron content)

The numerical example shown in table 3 is the result of an experiment with a mechanical sampler carried out in accordance with 4.1.2.

The magnitude of bias to be detected in the experiment is 0,2 % in total iron content by agreement between the parties concerned.

Table 3 — Numerical example 1 (for ore A)

Consign-ment No.	Total iron content, %		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	63,54	63,34	0,20	0,040 0
2	63,94	63,86	0,08	0,006 4
3	64,02	63,76	0,26	0,067 6
4	63,90	64,44	−0,54	0,291 6
5	63,73	64,03	−0,30	0,090 0
6	63,72	63,62	0,10	0,010 0
7	63,51	63,45	0,06	0,003 6
8	63,91	63,87	0,04	0,001 6
9	63,96	64,40	−0,44	0,193 6
10	63,84	63,76	0,08	0,006 4
11	63,72	63,76	−0,04	0,001 6
12	63,64	63,88	−0,24	0,057 6
13	63,97	64,11	−0,14	0,019 6
14	63,98	63,90	0,08	0,006 4
15	63,58	63,10	0,48	0,230 4
16	63,74	64,24	−0,50	0,250 0
17	63,86	64,26	−0,40	0,160 0
18	63,95	63,81	0,14	0,019 6
19	63,69	64,17	−0,48	0,230 4
20	63,80	63,94	−0,14	0,019 6
		Sum	−1,70	1,706 0

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

$$SS_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 1,706\,0 - \frac{(-1,70)^2}{20} = 1,561\,5$$

$$s_d = \sqrt{SS_d/(k-1)} = \sqrt{1,561\,6/19} = 0,286\,7 \approx 0,287$$

Thus

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

From table 1, $n_r = 28$ is obtained.

Therefore, an additional set of eight experiments should be undertaken and then the significance test carried out on a total of 28 data sets.

6.2 Numerical example 2 (δ : 0,1 % total iron content)

The numerical example shown in table 4 is the result of an experiment on a mechanical sampler carried out according to 4.1.2.

The magnitude of bias to be detected in the experiment is 0,1 % in total iron content by agreement between the parties concerned.

Table 4 — Numerical example 2

Consign-ment No.	Name of iron ore	Total iron content, %		$d_i = x_{Bi} - x_{Ai}$	d_i^2
		x_{Bi}	x_{Ai}		
1	F	59,20	59,00	0,20	0,040 0
2	E	59,75	59,67	0,08	0,006 4
3	C	61,80	61,74	0,06	0,003 6
4	B	63,02	63,16	−0,14	0,019 6
5	B	62,96	63,06	−0,10	0,010 0
6	E	60,02	59,92	0,10	0,010 0
7	B	63,17	63,11	0,06	0,003 6
8	A	63,91	63,87	0,04	0,001 6
9	E	59,98	60,02	−0,04	0,001 6
10	D	61,21	61,13	0,08	0,006 4
11	D	61,26	61,30	−0,04	0,001 6
12	E	58,98	59,02	−0,04	0,001 6
13	F	58,95	59,05	−0,10	0,010 0
14	C	61,97	61,89	0,08	0,006 4
15	F	59,06	58,88	0,18	0,032 4
16	A	63,74	63,75	−0,01	0,000 1
17	B	62,74	62,80	−0,06	0,003 6
18	E	60,47	60,42	0,05	0,002 5
19	B	62,55	62,62	−0,07	0,004 9
20	A	63,80	63,83	−0,03	0,000 9
			Sum	+0,30	0,166 8

$$\bar{d} = \frac{1}{k}\sum d_i = +0,30/20 = +0,015$$

$$SS_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 0,166\,8 - \frac{(0,30)^2}{20} = 0,162\,3$$

$$s_d = \sqrt{SS_d/(k-1)} = \sqrt{0,162\,3/19} = \sqrt{0,008\,5}$$

$$= 0,092\,4 \approx 0,092$$

Thus

$$D = \frac{\delta}{s_d} = \frac{0,1}{0,092} = 1,08$$

From table 1, $n_r = 13$ is obtained, and thus $k = 20$ is sufficient for the experiment.

$$t_o = \frac{\bar{d}}{s_d/\sqrt{k}} = \frac{0,015}{0,092/\sqrt{20}} = 0,721$$

$t = 1,729$ for $k = 20$ from table 2

$|t_o| < t$

Therefore, the bias is judged to be less than 0,1 % in total iron content and method B could be adopted as a routine method.

6.3 Numerical example 3 (δ : 0,15 % total iron content)

The numerical example shown in table 5 is the result of an experiment on a mechanical sampler carried out according to 4.1.2.

The magnitude of bias to be detected in the experiment is 0,15 % in total iron content by agreement between the parties concerned.

Table 5 — Numerical example 3 (for ore B)

Consign-ment No.	Total iron content, %		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	63,46	62,96	0,50	0,250 0
2	63,41	63,03	0,38	0,144 4
3	62,98	62,62	0,36	0,129 6
4	63,32	63,16	0,16	0,025 6
5	63,26	63,06	0,20	0,040 0
6	63,36	61,96	0,40	0,160 0
7	63,47	63,11	0,36	0,129 6
8	63,49	63,15	0,34	0,115 6
9	63,44	63,18	0,26	0,067 6
10	63,27	62,89	0,38	0,144 4
11	62,80	62,54	0,26	0,067 6
12	63,09	62,83	0,26	0,067 6
13	63,15	62,95	0,20	0,040 0
14	63,18	62,80	0,38	0,144 4
15	63,60	63,12	0,48	0,230 4
16	63,77	63,48	0,29	0,084 1
17	63,04	62,80	0,24	0,057 6
18	63,12	62,77	0,35	0,122 5
19	62,85	62,62	0,23	0,052 9
20	63,22	62,95	0,27	0,072 9
		Sum	6,30	2,146 8

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

$$SS_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 2{,}146\,8 - \frac{(6{,}30)^2}{20} = 0{,}162\,3$$

$$s_d = \sqrt{SS_d/(k-1)} = \sqrt{0{,}162\,3/19} = 0{,}092\,4 \approx 0{,}092$$

Thus

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

From table 1, $n_r = 6$ is obtained, and thus the number of data sets in the experiment is sufficient.

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

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

$$|t_o| > t$$

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

6.4 Numerical example 4 (δ : 0,3 % moisture content)

The final numerical example shown in table 6 is the result of an experiment for effects of particle size and mass of test samples upon moisture content. In this experiment, samples of mass 1 kg and particle size − 10 mm (method B) were compared with samples of mass 5 kg and particle size − 22,4 mm (method A).

The magnitude of bias to be detected in the experiment is 0,3 % in moisture content by agreement between the parties concerned.

Table 6 — Numerical example 4

Consign-ment No.	Name of iron ore	Moisture content, %		$d_i = x_{Bi} - x_{Ai}$	d_i^2
		x_{Bi}	x_{Ai}		
1	A	2,64	2,99	−0,35	0,122 5
2	B	1,47	1,60	−0,13	0,016 9
3	C	2,35	2,27	0,08	0,006 4
4	D	2,70	2,75	−0,05	0,002 5
5	E	0,64	0,59	0,05	0,002 5
6	F	1,78	1,63	0,15	0,022 5
7	C	0,55	0,91	−0,36	0,129 6
8	G	3,92	4,29	−0,37	0,136 9
9	H	4,75	4,85	−0,10	0,010 0
10	A	4,09	4,36	−0,27	0,072 9
11	A	3,73	3,38	0,35	0,122 5
12	I	4,93	4,83	0,10	0,010 0
13	I	5,37	5,68	−0,31	0,096 1
14	J	7,09	7,27	−0,18	0,032 4
15	K	6,94	7,02	−0,08	0,006 4
16	L	8,24	7,54	0,70	0,490 0
17	M	8,11	7,62	0,49	0,240 1
18	N	0,36	0,46	−0,10	0,010 0
19	O	1,80	2,07	−0,27	0,072 9
20	P	7,14	7,06	0,08	0,006 4
			Sum	−0,57	1,609 5

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$$\bar{d} = \frac{1}{k}\sum d_i = -0{,}57/20 = -0{,}028$$

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

$$s_d = \sqrt{SS_d/(k-1)} = \sqrt{1{,}593\,3/19} = 0{,}289\,5 \approx 0{,}290$$

Thus

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

From table 1, $n_r = 13$ is obtained, and thus $k = 20$ in the experiment is sufficient.

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

$t = 1,729$ for $k = 20$ from table 2

$|t_o| < t$

Therefore, the bias is judged to be less than 0,3 % in moisture content and method B could be adopted as a routine method.

7 Bibliography

The following International Stnadards are closely related to bias of sampling.

ISO 3084, *Iron ores — Experimental methods for evaluation of quality variation.*

ISO 3085, *Iron ores — Experimental methods for checking the precision of sampling.*

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