



SLOVENSKI STANDARD
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Aluminijeve rude - Poskusno ugotavljanje heterogenosti razporeditve vzorcev

Aluminium ores -- Experimental determination of the heterogeneity of distribution of a lot

Minerais alumineux -- Détermination expérimentale de l'hétérogénéité de distribution d'un lot

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determination of the heterogeneity of
distribution of a lot**

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*Minerais alumineux — Détermination expérimentale de l'hétérogénéité
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 6139 was prepared by Technical Committee ISO/TC 129, *Aluminium ores*, Sub-Committee SC 1, *Sampling*.

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Aluminium ores — Experimental determination of the heterogeneity of distribution of a lot

1 Scope

This International Standard specifies experimental methods for evaluating the heterogeneity of distribution of aluminium ores, for the purpose of determining the minimum number of primary increments and consequently the sampling scheme.

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 8685:1992, *Aluminium ores — Sampling procedures*.

ISO 10277:—¹⁾, *Aluminium ores — Experimental methods for checking the precision of sampling*.

3 General

3.1 Origin of heterogeneity of distribution

The heterogeneity of distribution is a measure of the distribution variability of the aluminium ore and hence the manner in which the particles are distributed throughout the lot. It depends on the natural variability of the aluminium ore being mined, how well it is blended, and how it is subsequently handled. It can be reduced by mixing, but it can never be completely eliminated. Unlike the heterogeneity of constitution, the heterogeneity of distribution is not a function of sample mass. Its contribution to the sampling vari-

ance can be reduced only by increasing the number of increments.

3.2 Method of estimating the heterogeneity of distribution

The heterogeneity of distribution, characterized by the distribution variance, is determined by experimentally measuring the sampling variance. However, because both the composition variance (V_C) and distribution variance (V_D) contribute to the measured sampling variance, it is necessary to separate these two components. This is achieved by measuring the sampling variance at two different increment masses.

Two methods of data analysis are permitted. The first (see 5.1) is based on Visman's theory of sampling which uses classical statistics. This method gives conservative estimates of the sampling variance and hence the distribution variance. The second method (see 5.2) is based on the variogram which takes into account serial correlation between adjacent increments and the spacing between increments. It gives a better estimate of the sampling variance and should be used to optimize the sampling scheme.

In both cases, the variance of sample preparation and analysis shall be determined separately in accordance with ISO 10277, and subtracted from the measured variances.

3.3 Characteristics measured

The quality characteristics chosen for measuring the heterogeneity of distribution should be those that are most relevant to the sampling operation. For aluminium ores these could be

- aluminium content, expressed as a percentage by mass of Al_2O_3 ;
- silicon content, expressed as a percentage by mass of SiO_2 ;

1) To be published.

— moisture content.

4 Collection of data

Regardless of which method of data analysis is used, the same data are required. The procedure for collecting the data is as follows:

- Calculate the minimum mass of increment required to give an unbiased sample in accordance with ISO 8685, subclause 7.1.
- Collect a minimum of 30 increments from the lot, preferably close to the proposed sampling interval.
- Prepare and analyse each increment separately.
- Increase the increment mass substantially (e.g. by a factor of 10) and repeat steps b) and c).

5 Calculation of distribution variance

5.1 Increment variance method

The sampling variance σ_S^2 is given by

$$\sigma_S^2 = \sigma_{QE_1}^2 + \sigma_{QE_2}^2 \quad (1)$$

where

$\sigma_{QE_1}^2$ is the short range quality fluctuation variance;

$\sigma_{QE_2}^2$ is the long range quality fluctuation variance.

In terms of the composition and distribution variances, equation (1) becomes

$$\sigma_S^2 = \frac{V_c}{nm_1} + \frac{V_D}{n} \quad \dots (2)$$

where

V_c is the composition variance for a 1 kg sample;

V_D is the distribution variance;

n is the number of increments;

m_1 is the increment mass, in kilograms.

The first term (V_c/nm_1) contributes only to $\sigma_{QE_1}^2$, while the second term (V_D/n) contributes to both $\sigma_{QE_1}^2$ and $\sigma_{QE_2}^2$.

The variance of the individual analyses (V_e) is given by

$$V_e = \frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1} \quad \dots (3)$$

where

x_i is the analysis value for increment i ;

\bar{x} is the mean value for all increments;

n is the number of increments.

In order to obtain the increment variance V_1 due to sampling only, the sample preparation and analysis variance V_{PM} shall be subtracted as follows:

$$V_1 = V_e - V_{PM} \quad \dots (4)$$

The estimated sampling variance is then given by

$$\sigma_S^2 = \frac{V_1}{n} \quad \dots (5)$$

Alternatively, equation (5) can be transposed to give the following equation for calculating the number of increments required to achieve a given sampling variance σ_S^2 :

$$n = \frac{V_1}{\sigma_S^2} \quad \dots (6)$$

If the increment mass is changed, the increment variance shall either be redetermined experimentally or recalculated from data collected at two different increment masses.

Combining equations (2) and (5) gives

$$V_1 = \frac{V_c}{m_1} + V_D \quad \dots (7)$$

Rewriting this for the two different increment masses m_{1_1} and m_{1_2} used for data collection gives

$$V_{1_1} = \frac{V_c}{m_{1_1}} + V_D \quad \dots (8)$$

$$V_{1_2} = \frac{V_c}{m_{1_2}} + V_D \quad \dots (9)$$

The solution to these two equations is

$$V_c = \frac{m_{1_1} m_{1_2} (V_{1_1} - V_{1_2})}{m_{1_2} - m_{1_1}} \quad \dots (10)$$

$$V_D = \frac{m_{1_2} V_{1_2} - m_{1_1} V_{1_1}}{m_{1_2} - m_{1_1}} \quad \dots (11)$$

Once V_c and V_D are known V_1 can be recalculated for any increment mass using equation (7).

5.2 Alternative variogram method

Calculate the values of the experimental variogram $V_e(t)$ at lag t (in mass or time units) using the equation

$$V_e(t) = \frac{\sum_{i=1}^n (x_{i+t} - x_i)^2}{2N_t} \quad \dots (12)$$

where

- x_i is the analysis value for increment i ;
- x_{i+t} is the analysis value for increment $i+t$;
- N_t is the number of pairs of increments at lag t apart.

It is recommended that $v_e(t)$ be calculated for values of t between 1 and 20.

In order to obtain the corrected variogram $V(t)$ corresponding to the sampling process only, the sample preparation and analysis variance V_{PM} must be subtracted as follows:

$$V(t) = V_e(t) - V_{PM} \quad \dots (13)$$

A typical experimental variogram is shown in figure 1.

Practical experience shows that variograms which occur in practice can be adequately approximated by a straight line over the range from very small values of t to at least twice the spacing between increments, i.e.

$$V(t) = A + Bt \quad \dots (14)$$

where

- A is the random component of variance (the intercept) of the corrected variogram;
- B is the gradient (or slope) of the variogram.

The values of A and B are estimated by fitting a straight line to the first two points of the corrected variogram as follows:

- a) If $V(1) > V(2)$, then take

$$A = V(1) \quad \dots (15)$$

$$B = 0 \quad \dots (16)$$

where

$V(1)$ is the value of the variogram at lag 1;

$V(2)$ is the value of the variogram at lag 2.

- b) If $V(1) < V(2)$, then A and B are given by

$$A = 2V(1) - V(2) \quad \dots (17)$$

$$B = \frac{V(2) - V(1)}{\Delta t} \quad \dots (18)$$

where Δt is the interval between increments.

- c) If the estimate of A from equation (17) is negative, then take

$$A = 0 \quad \dots (19)$$

$$B = \frac{V(1)}{\Delta t} \quad \dots (20)$$

After A and B have been determined, the sampling variance can be calculated from the following equation for systematic sampling:

$$\sigma_S^2 = \frac{A}{n} + \frac{BQ}{6n^2} \quad \dots (21)$$

where

- Q is the mass of the lot;
- A is the intercept of the corrected variogram;
- n is the number of increments.

The first term (A/n) corresponds to $\sigma_{QE_1}^2$ in equation (1) while the second term ($BQ/6n^2$) corresponds to $\sigma_{QE_2}^2$.

The number of increments required to obtain a sampling variance of σ_S^2 can be calculated by transposing equation (21) and solving for n , which gives:

$$n = \frac{A + \sqrt{A^2 + \frac{2}{3} BQ\sigma_S^2}}{2\sigma_S^2} \quad \dots (22)$$

If the increment mass is changed, the intercept A on the corrected variogram must either be redetermined experimentally or recalculated from data collected at two different increment masses.

The intercept A can be expressed in terms of its constitution and distribution variance components as follows:

$$A = \frac{V_c}{m_1} + V_r \quad \dots (23)$$

where

V_c is the composition variance for a 1 kg sample;

V_r is the short-range distribution variance.

Thus equation (21) becomes

$$\sigma_S^2 = \frac{V_c}{nm_1} + \frac{V_r}{n} + \frac{BQ}{6n^2} \quad \dots (24)$$

The last two terms in equation (24) result from the distribution variance, i.e.:

$$V_D = V_r + \frac{BQ}{6n} \quad \dots (25)$$

Rewriting formula (23) for the two different increment masses m_1 and m_2 used for data collection gives

$$A_1 = \frac{V_c}{m_1} + V_r \quad \dots (26)$$

$$A_2 = \frac{V_c}{m_2} + V_r \quad \dots (27)$$

The solution to these two equations is

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$$V_c = \frac{m_1 m_2 (A_1 - A_2)}{m_2 - m_1} \quad \dots (28)$$

$$V_r = \frac{m_2 A_2 - m_1 A_1}{m_2 - m_1} \quad \dots (29)$$

Once V_c and V_r are known, A can be recalculated for any increment mass using equation (23).

6 Numerical example

The data for the Al_2O_3 content of an aluminium ore shown in table 1 and figure 2 were obtained by taking increments at 100 tonne intervals and analysing each increment separately. The sample preparation and analysis error was estimated in a separate experiment and found to be 0,2 % Al_2O_3 .

6.1 Calculation of increment variance

Inserting the data from table 1 into equation (3) gives

$$V_e = 0,388$$

In addition

$$V_{PM} = (0,2)^2 = 0,04$$

Substituting the above values into equation (4) gives

$$\begin{aligned} V_1 &= V_e - V_{PM} \\ &= 0,388 - 0,04 \\ &= 0,348 \end{aligned}$$

The sampling variance (σ_S^2) obtained by averaging the 60 analyses in table 1 is then given by equation (5):

$$\begin{aligned} \sigma_S^2 &= \frac{V_1}{n} \\ &= \frac{0,348}{60} \\ &= 0,0058 \end{aligned}$$

Thus

$$\sigma_S = 0,076 \% \text{ Al}_2\text{O}_3$$

Alternatively, if a sampling error σ_S of 0,1 % Al_2O_3 is required, equation (6) gives the number of increments as follows:

$$\begin{aligned} n &= \frac{V_1}{\sigma_S^2} \\ &= \frac{0,348}{(0,1)^2} \\ &= 35 \end{aligned}$$

6.2 Calculation of the variogram

Inserting the data in table 1 into equation (12) gives the variogram plotted in figure 1. The numerical values are given in table 2.

Equations (13), (17) and (18) give the intercept A and slope B of the corrected variograms as follows:

$$\begin{aligned} A &= 2V(1) - V(2) - V_{PM} \\ &= 2 \times 0,264 - 0,266 - 0,04 \\ &= 0,222 \end{aligned}$$

$$\begin{aligned} B &= \frac{V(2) - V(1)}{\Delta t} \\ &= \frac{0,266 - 0,264}{100} \\ &= 2 \times 10^{-5} \end{aligned}$$

The estimated sampling variance σ_S^2 for the average of the 60 analyses in table 1 is then given by equation (21) for systematic sampling, i.e.:

$$\sigma_S^2 = \frac{A}{n} + \frac{BQ}{6n^2}$$

$$= \frac{0,222}{60} + \frac{2 \times 10^{-5} \times 6000}{6 \times 60^2}$$

$$= 0,0037$$

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$$\sigma_S = 0,061 \% \text{ Al}_2\text{O}_3$$

Alternatively, if a sampling error σ_S of 0,1 % Al_2O_3 is required for a 10 000 tonne lot, equation (22) gives the required number of increments as follows:

$$\begin{aligned} n &= \frac{A + \sqrt{A^2 + \frac{2}{3} BQ\sigma_S^2}}{2\sigma_S^2} \\ &= \frac{0,222}{2 \times (0,1)^2} + \\ &= \frac{\sqrt{(0,222)^2 + \frac{2}{3} \times 2 \times 10^{-5} \times 10000 \times (0,1)^2}}{2 \times (0,1)^2} \\ &= \frac{0,222 + \sqrt{0,0493 + 0,0013}}{0,02} \\ &= 22 \end{aligned}$$

This is significantly less than the number of increments calculated using the increment variance method.

Table 1 — Al₂O₃ content of individual increments taken from an aluminium ore at 100 tonne intervals

Increment number	Al ₂ O ₃ content %	Increment number	Al ₂ O ₃ content %
1	56,4	31	56,3
2	56,1	32	56,0
3	55,8	33	56,3
4	56,7	34	56,4
5	56,4	35	56,3
6	56,5	36	56,1
7	56,4	37	57,5
8	57,3	38	56,6
9	57,4	39	56,5
10	56,4	40	56,4
11	56,8	41	56,6
12	56,3	42	56,5
13	56,8	43	56,2
14	57,9	44	57,2
15	56,5	45	56,7
16	57,1	46	56,6
17	55,5	47	56,4
18	56,0	48	56,6
19	56,9	49	55,7
20	56,2	50	55,4
21	55,8	51	57,0
22	56,1	52	56,0
23	55,9	53	57,1
24	56,5	54	55,5
25	56,8	55	55,6
26	56,4	56	55,4
27	56,3	57	55,8
28	56,2	58	53,9
29	56,5	59	55,4
30	56,5	60	55,7

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