INTERNATIONAL
STANDARD

**ISO
6139**

First edition
1993-08-01

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

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

Reference number
ISO 6139:1993(E)

# Foreword

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

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland
Printed in Switzerland

# 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 heterogenity of distribution is not a function of sample mass. Its contribution to the sampling variance 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 $SiO_2$;

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[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:

a) Calculate the minimum mass of increment required to give an unbiased sample in accordance with ISO 8685, subclause 7.1.

b) Collect a minimum of 30 increments from the lot, preferably close to the proposed sampling interval.

c) Prepare and analyse each increment separately.

d) 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 $\sigma_S^2$ is given by

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

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

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

where

$V_c$ is the composition variance for a 1 kg sample;

$V_D$ is the distribution variance;

$n$ is the number of increments;

$m_I$ is the increment mass, in kilograms.

The first term $(V_c/nm_I)$ 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

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

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 $V_I$ due to sampling only, the sample preparation and analysis variance $V_{PM}$ shall be subtracted as follows:

$$V_I = V_e - V_{PM} \qquad \dots (4)$$

The estimated sampling variance is then given by

$$\sigma_S^2 = \frac{V_I}{n} \qquad \dots (5)$$

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

$$n = \frac{V_I}{\sigma_S^2} \qquad \dots (6)$$

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

$$V_I = \frac{V_c}{m_I} + V_D \qquad \dots (7)$$

Rewriting this for the two different increment masses $m_{I_1}$ and $m_{I_2}$ used for data collection gives

$$V_{I_1} = \frac{V_c}{m_{I_1}} + V_D \qquad \dots (8)$$

$$V_{I_2} = \frac{V_c}{m_{I_2}} + V_D \qquad \dots (9)$$

The solution to these two equations is

$$V_c = \frac{m_{I_1} m_{I_2} (V_{I_1} - V_{I_2})}{m_{I_2} - m_{I_1}} \qquad \dots (10)$$

$$V_D = \frac{m_{I_2} V_{I_2} - m_{I_1} V_{I_1}}{m_{I_2} - m_{I_1}} \qquad \dots (11)$$

Once $V_c$ and $V_D$ are known $V_I$ 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

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

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:

$$V(t) = V_e(t) - V_{PM} \qquad \ldots (13)$$

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.

$$V(t) = A + Bt \qquad \ldots (14)$$

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

$$A = V(1) \qquad \ldots (15)$$

$$B = 0 \qquad \ldots (16)$$

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

$$A = 2V(1) - V(2) \qquad \ldots (17)$$

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

where $\Delta t$ is the interval between increments.

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

$$A = 0 \qquad \ldots (19)$$

$$B = \frac{V(1)}{\Delta t} \qquad \ldots (20)$$

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

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

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 $\sigma_S^2$ can be calculated by transposing equation (21) and solving for $n$, which gives:

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

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:

$$A = \frac{V_c}{m_l} + V_r \qquad \ldots (23)$$

where

$V_c$ is the composition variance for a 1 kg sample;

$V_r$ is the short-range distribution variance.

Thus equation (21) becomes

$$\sigma_S^2 = \frac{V_c}{nm_l} + \frac{V_r}{n} + \frac{BQ}{6n^2} \qquad \ldots (24)$$

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

$$V_D = V_r + \frac{BQ}{6n} \qquad \ldots (25)$$

Rewriting formula (23) for the two different increment masses $m_{l_1}$ and $m_{l_2}$ used for data collection gives

$$A_1 = \frac{V_c}{m_{l_1}} + V_r \qquad \ldots (26)$$

$$A_2 = \frac{V_c}{m_{l_2}} + V_r \qquad \ldots (27)$$

The solution to these two equations is

$$V_c = \frac{m_{I_1} m_{I_2}(A_1 - A_2)}{m_{I_2} - m_{I_1}} \qquad \ldots (28)$$

$$V_r = \frac{m_{I_2} A_2 - m_{I_1} A_1}{m_{I_2} - m_{I_1}} \qquad \ldots (29)$$

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

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

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

$$\begin{aligned} \sigma_S^2 &= \frac{V_I}{n} \\ &= \frac{0{,}348}{60} \\ &= 0{,}005\,8 \end{aligned}$$

Thus

$$\sigma_S = 0{,}076 \text{ \% } Al_2O_3$$

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

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

### 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 $\sigma_S^2$ for the average of the 60 analyses in table 1 is then given by equation (21) for systematic sampling, i.e.:

$$\begin{aligned} \sigma_S^2 &= \frac{A}{n} + \frac{BQ}{6n^2} \\ &= \frac{0{,}222}{60} + \frac{2 \times 10^{-5} \times 6\,000}{6 \times 60^2} \\ &= 0{,}003\,7 \end{aligned}$$

Thus

$$\sigma S = 0{,}061 \text{ \% } Al_2O_3$$

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

$$n = \frac{A + \sqrt{A^2 + \frac{2}{3} BQ\sigma_S^2}}{2\sigma_S^2}$$

$$= \frac{0{,}222}{2 \times (0{,}1)^2} +$$

$$\frac{\sqrt{(0{,}222)^2 + \frac{2}{3} \times 2 \times 10^{-5} \times 10\,000 \times (0{,}1)^2}}{2 \times (0{,}1)^2}$$

$$= \frac{0{,}222 + \sqrt{0{,}049\,3 + 0{,}001\,3}}{0{,}02}$$

$$= 22$$

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

**Table 1 — $Al_2O_3$ content of individual increments taken from an aluminium ore at 100 tonne intervals**

| Increment number | $Al_2O_3$ content % |
|---|---|
| 1 | 56,4 |
| 2 | 56,1 |
| 3 | 55,8 |
| 4 | 56,7 |
| 5 | 56,4 |
| 6 | 56,5 |
| 7 | 56,4 |
| 8 | 57,3 |
| 9 | 57,4 |
| 10 | 56,4 |
| 11 | 56,8 |
| 12 | 56,3 |
| 13 | 56,8 |
| 14 | 57,9 |
| 15 | 56,5 |
| 16 | 57,1 |
| 17 | 55,5 |
| 18 | 56,0 |
| 19 | 56,9 |
| 20 | 56,2 |
| 21 | 55,8 |
| 22 | 56,1 |
| 23 | 55,9 |
| 24 | 56,5 |
| 25 | 56,8 |
| 26 | 56,4 |
| 27 | 56,3 |
| 28 | 56,2 |
| 29 | 56,5 |
| 30 | 56,5 |
| 31 | 56,3 |
| 32 | 56,0 |
| 33 | 56,3 |
| 34 | 56,4 |
| 35 | 56,3 |
| 36 | 56,1 |
| 37 | 57,5 |
| 38 | 56,6 |
| 39 | 56,5 |
| 40 | 56,4 |
| 41 | 56,6 |
| 42 | 56,5 |
| 43 | 56,2 |
| 44 | 57,2 |
| 45 | 56,7 |
| 46 | 56,6 |
| 47 | 56,4 |
| 48 | 56,6 |
| 49 | 55,7 |
| 50 | 55,4 |
| 51 | 57,0 |
| 52 | 56,0 |
| 53 | 57,1 |
| 54 | 55,5 |
| 55 | 55,6 |
| 56 | 55,4 |
| 57 | 55,8 |
| 58 | 53,9 |
| 59 | 55,4 |
| 60 | 55,7 |

**Table 2 — Experimental variogram points for data in table 1**

| Lag | Variance |
|---|---|
| 1 | 0,264 |
| 2 | 0,266 |
| 3 | 0,280 |
| 4 | 0,275 |
| 5 | 0,284 |
| 6 | 0,303 |
| 7 | 0,332 |
| 8 | 0,311 |
| 9 | 0,312 |
| 10 | 0,323 |
| 11 | 0,331 |
| 12 | 0,394 |
| 13 | 0,411 |
| 14 | 0,361 |
| 15 | 0,328 |
| 16 | 0,331 |
| 17 | 0,357 |
| 18 | 0,327 |
| 19 | 0,355 |
| 20 | 0,366 |

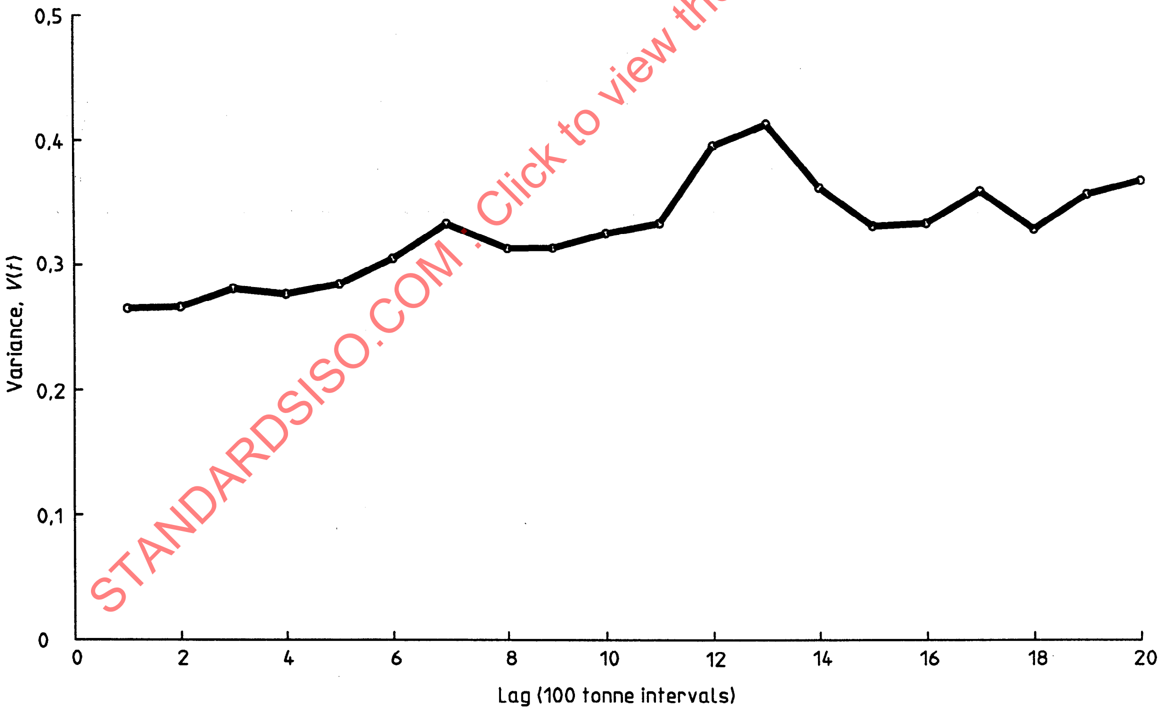

Figure 1 — Experimental variogram for $Al_2O_3$ content

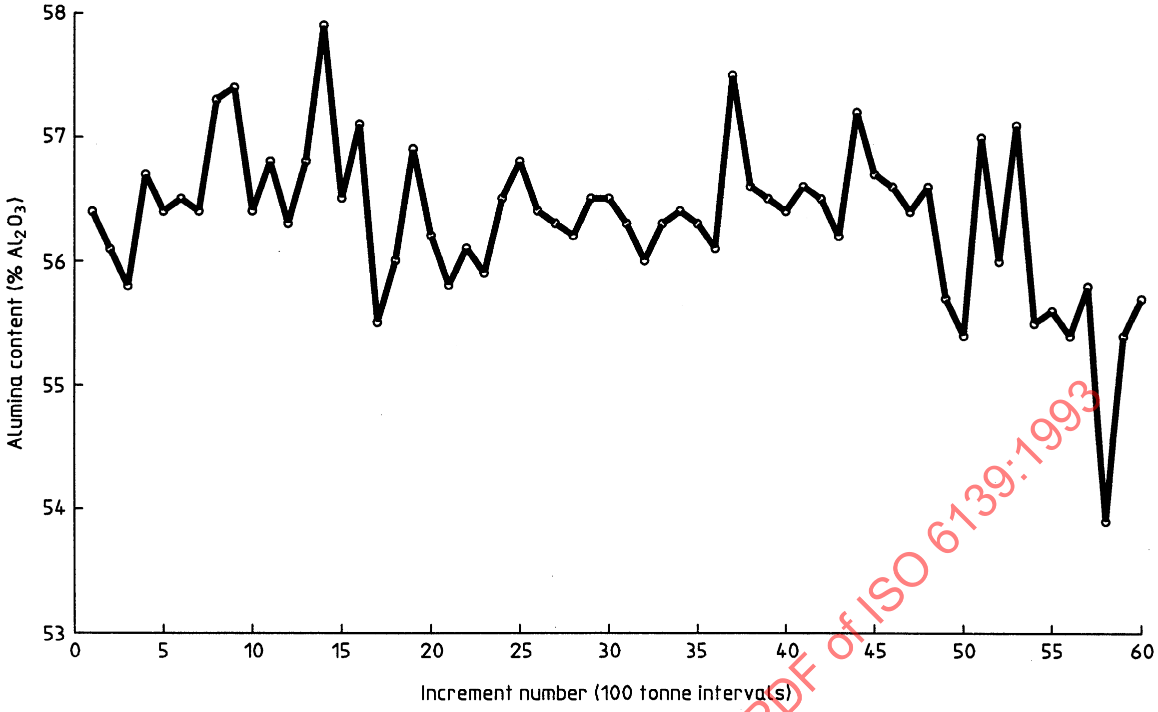

Figure 2 — $Al_2O_3$ content of individual increments taken from an aluminium ore at 100 tonne intervals