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Fluorspar — Method of determining the precision of sampling and sample preparation

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Spaths fluor — Méthode de détermination de la fidélité de l'échantillonnage et de la préparation des échantillons

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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.

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International Standard ISO 9499 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

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Fluorspar — Method of determining the precision of sampling and sample preparation

1 Scope

This International Standard specifies a method for the determination of the precision of fluorspar sampling and sample preparation carried out by the methods specified in ISO 8868.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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ISO 8868:1989, *Fluorspar — Sampling and sample preparation.*

3 General conditions

3.1 Number of lots

In order to ensure a reliable result, it is recommended that the precision determination be carried out on more than 20 lots of fluorspar of the same type from the same source; however, if this is impracticable, at least 10 lots shall be sampled. If the number of lots available for the precision determination is not sufficient, each lot may be divided into several part-lots to produce 10 or more part-lots, and the determination carried out on each part-lot, considering each part-lot as a separate lot, in accordance with ISO 8868.

3.2 Number of increments and number of gross samples

The minimum number of increments required for the precision determination shall be twice the number specified in ISO 8868. Thus if the number of increments required for the routine sampling is n, which are combined to form one gross sample, the minimum number of increments required for the precision determination shall be $2n$ and these are combined separately into two gross samples of n increments each.

NOTE 1 If this is impracticable, n increments may be taken and the increments divided into two sets, each comprising $n/2$ increments. (See clause 6.)

3.3 Sample preparation

Preparation of the sample shall be carried out in accordance with ISO 8868.

3.4 Frequency of determination

It is recommended that, even when a series of precision determinations has been conducted prior to routine sampling operations, a precision determination should be carried out occasionally in order to check for a possible change in precision in the methods for sampling, sample preparation and testing.

3.5 Design of method

Because of the large amount of work involved in this determination, it is recommended that it be designed as a part of the routine work of sampling and testing.

4 Method

4.1 Sampling procedure

The sampling procedure shall be selected from the three categories of sampling defined in ISO 8868, i.e. systematic sampling, stratified sampling and two-stage sampling.

4.1.1 Systematic sampling

4.1.1.1 The number of increments n shall be selected from table 3 of ISO 8868:1989, depending on the mass of the lot and the grade of fluorspar;

4.1.1.2 The sampling interval shall be calculated by dividing the tonnage of the lot by $2n$, thus giving an interval equal to one-half of the interval used for routine sampling. The sampling interval, in tonnes, thus calculated shall be rounded down to the nearest whole number.

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4.1.1.3 The increments shall be taken at the regular sampling interval calculated in 4.1.1.2, starting at a random point in the lot.

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4.1.1.4 The increments shall be placed alternately in two containers, A and B. Thus, two gross samples, A and B, will be formed, each comprising n increments.

EXAMPLE 1

a) Suppose that a lot of 2 000 t of acid-grade fluorspar is being discharged on belt conveyors. The minimum number of increments n required is 15, as shown in table 3 of ISO 8868.

b) The sampling interval for taking increments is given by the equation

$$\frac{2\,000}{15 \times 2} = 66{,}7 \rightarrow 66 \text{ t}$$

c) Thus increments are taken at 66 t intervals. The point at which the first increment is taken within the first 66 t sampling interval is determined by random selection. If this point is taken as 20 t from the start of discharge operations, subsequent increments will be taken at 86 t (= 20 + 66), 152 t (= 20 + 66 × 2), etc. Since the whole lot amounts to 2 000 t, 30 increments will be collected.

d) The increments are placed alternately in containers A and B, and combined separately into two gross samples, A and B, each comprising 15 increments (see figure 1).

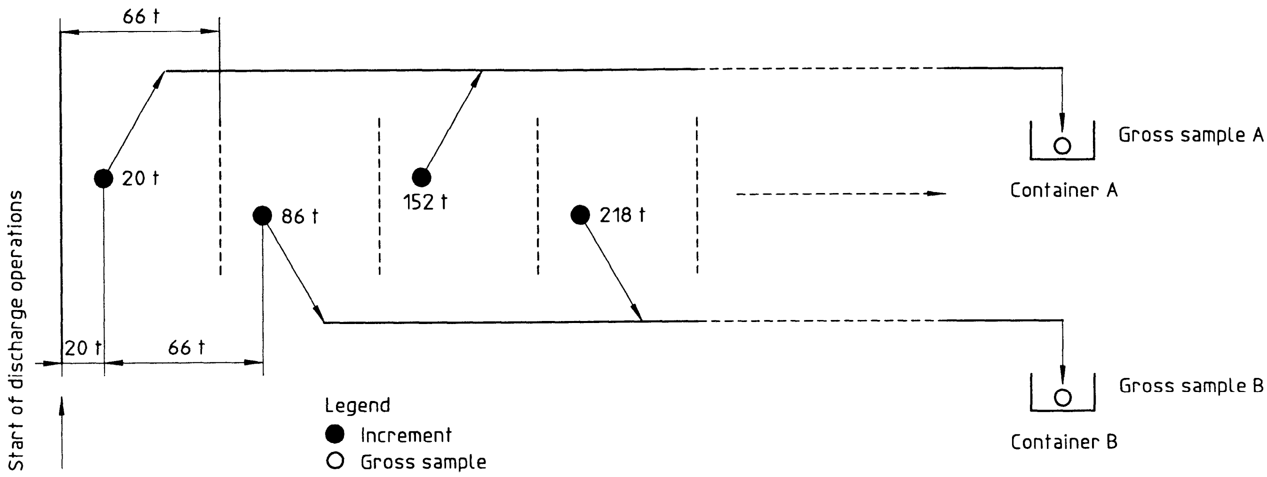

Figure 1 — Schematic diagram for example 1

4.1.2 Stratified sampling

4.1.2.1 In the case where the number of wagons or containers [hereinafter referred to as wagon(s)], i.e. the number of strata k forming one lot, is not more than the number of increments n given in table 3 of ISO 8868, the number of increments n_w to be taken from each wagon (stratum) shall be obtained by the formula $n_w = n/k$ and shall be rounded up to the next highest whole number.

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4.1.2.2 In all other cases, twice the n_w increments, i.e. $2n_w$, shall be taken from each wagon.

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4.1.2.3 The $2n_w$ increments taken from each wagon shall be separated at random into two partial samples, each of n_w increments (see figure 2).

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4.1.2.4 Each of the two partial samples from each wagon shall be combined separately to form two gross samples, A and B, respectively, each comprising n ($= kn_w$) increments.

EXAMPLE 2

a) Suppose that a lot of metallurgical-grade fluorspar in gravel form is delivered in eleven 60 t wagons. The minimum number of increments required for the 660 t lot is 30, as shown in table 3 of ISO 8868. The minimum required number of increments to be taken from each wagon in routine sampling is calculated and rounded up to the nearest whole number as follows:

$$\frac{30}{11} = 2,7 \rightarrow 3$$

b) Thus six (= 3 × 2) increments are taken from each wagon in the precision determination.

c) The six increments are separated at random into two partial samples, each consisting of three increments.

d) Each of the two partial samples from the 11 wagons is combined separately to form two gross samples, A and B, respectively, each comprising 33 (= 3 × 11) increments (see figure 2).

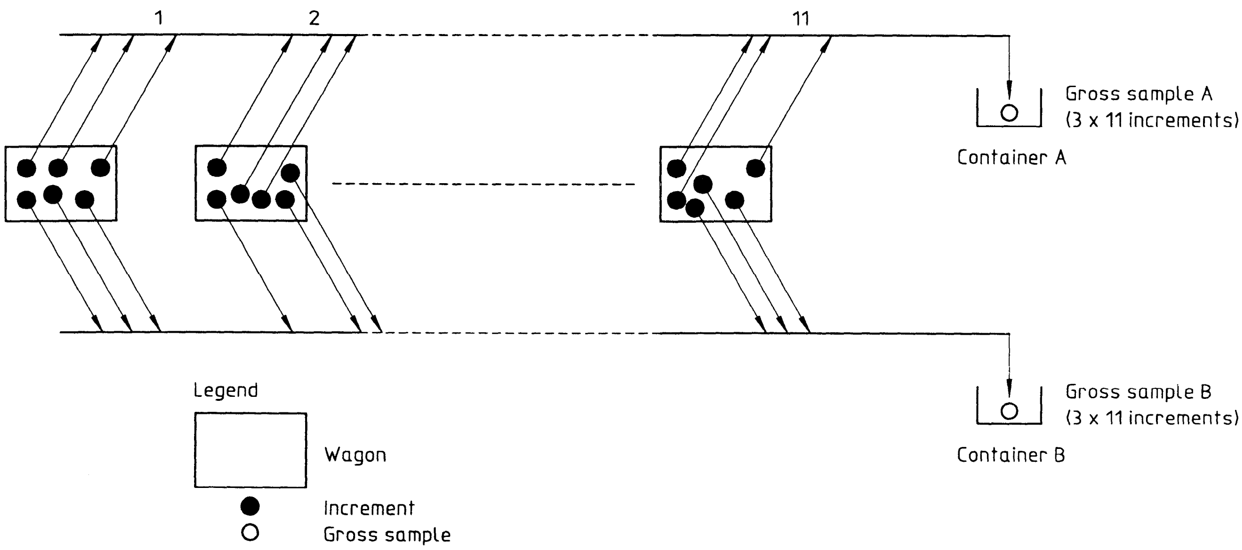

Figure 2 — Schematic diagram for example 2

4.1.3 Two-stage sampling

4.1.3.1 If the number of wagons forming one lot is more than the number of increments n required by table 3 of ISO 8868, n wagons shall be selected at random from the lot.

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4.1.3.2 An additional n wagons shall be selected at random from the same lot independently. It is possible for the same wagons to be included in each independent selection.

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4.1.3.3 One increment shall be taken from each of the wagons selected.

4.1.3.4 All of the increments taken from the wagons selected in accordance with 4.1.3.1 shall be combined to form gross sample A.

All of the increments taken from the wagons selected in accordance with 4.1.3.2 shall be combined to form another gross sample B.

4.2 Sample preparation and testing

The two gross samples A and B taken in accordance with 4.1 shall be prepared separately and tested by the type 1, type 2 or type 3 procedure described in 4.2.1, 4.2.2 and 4.2.3, respectively.

4.2.1 Type 1 preparation/testing procedure (see figure 3)

4.2.1.1 The two gross samples A and B shall be prepared separately to provide two test samples from each, A_1, A_2 and B_1, B_2.

4.2.1.2 Each of the four test samples shall be tested in duplicate, the total of eight tests being run in random order.

NOTE 2 The type 1 procedure gives the precisions for sampling, preparation and measurement separately.

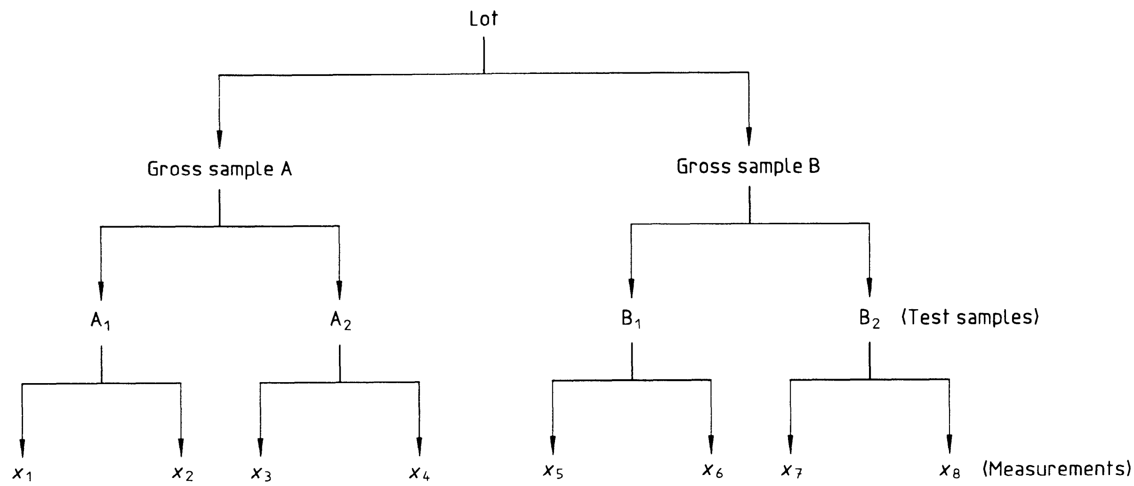

Figure 3 — Flowsheet for type 1 testing

4.2.2 Type 2 preparation/testing procedure (see figure 4)

4.2.2.1 Gross sample A shall be prepared to provide two test samples, A_1 and A_2, and from gross sample B one test sample shall be prepared.

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4.2.2.2 Test sample A_1 shall be tested in duplicate and the other test samples, A_2 and B, shall be tested individually.

NOTE 3 The type 2 procedure also gives the precisions for sampling, preparation and measurement separately. However, the precisions for preparation and measurement are inferior to those obtained using the type 1 procedure.

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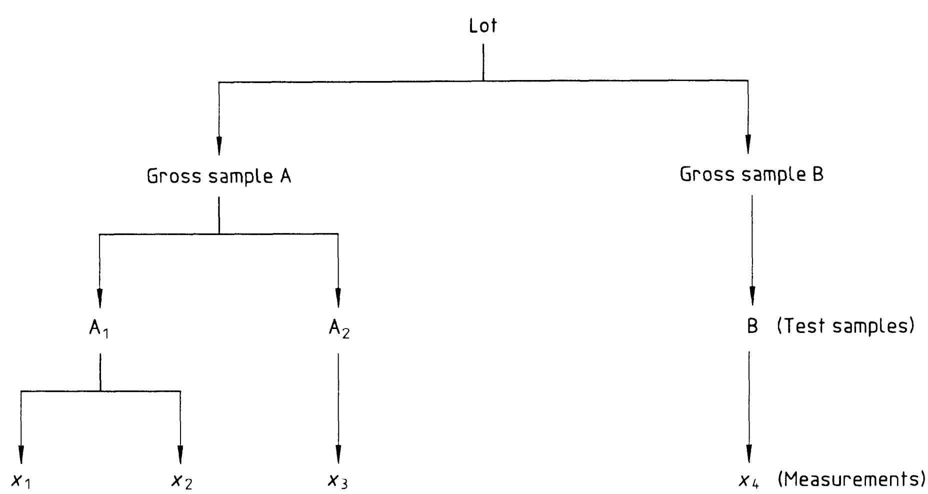

Figure 4 — Flowsheet for type 2 testing

4.2.3 Type 3 preparation/testing procedure (see figure 5)

4.2.3.1 One test sample shall be prepared from each of the gross samples A and B.

4.2.3.2 The two test samples A and B shall be tested individually.

NOTE 4 The type 3 procedure gives only the overall precision for sampling, preparation and measurement.

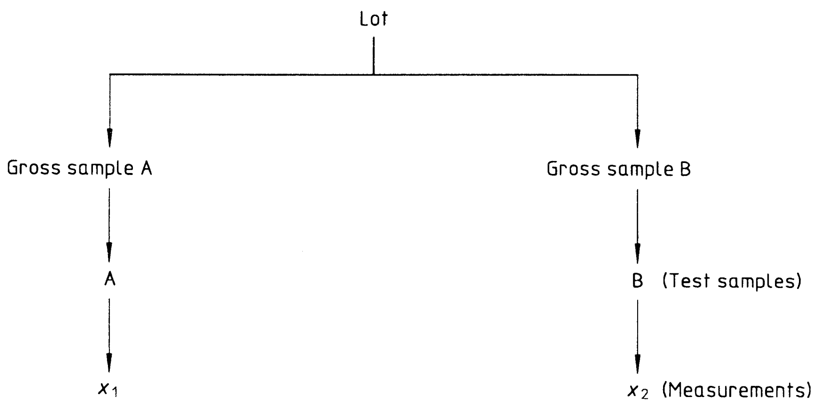

Figure 5 — Flowsheet for type 3 testing

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5 Analysis of experimental data

The test data obtained shall be analysed as specified below, depending on the type of preparation/testing procedure selected and regardless of the method of sampling used (systematic sampling, stratified sampling or two-stage sampling).

5.1 Type 1 preparation/testing procedure (see figure 3)

The 95 % probability values of the precision (hereinafter simply referred to as "the precision") for sampling, preparation and measurement shall be calculated in accordance with the procedure given in 5.1.1 to 5.1.7.

5.1.1 Let the eight measurements (such as % CaF_2) for the test samples prepared from gross samples A and B be designated by x_1, x_2, x_3, x_4, x_5, x_6, x_7 and x_8.

5.1.2 Calculate the mean and the range for each pair of duplicate measurements using the sets of equations (1) and (2), respectively:

$$\left.\begin{aligned}\bar{x}_1 &= \frac{1}{2}(x_1 + x_2)\\ \bar{x}_3 &= \frac{1}{2}(x_3 + x_4)\\ \bar{x}_5 &= \frac{1}{2}(x_5 + x_6)\\ \bar{x}_7 &= \frac{1}{2}(x_7 + x_8)\end{aligned}\right\} \quad \ldots (1)$$

$$\left.\begin{aligned} R_{11} &= |x_1 - x_2| \\ R_{13} &= |x_3 - x_4| \\ R_{15} &= |x_5 - x_6| \\ R_{17} &= |x_7 - x_8| \end{aligned}\right\} \quad \ldots (2)$$

5.1.3 Calculate the mean and the range for each pair of duplicate test samples using the sets of equations (3) and (4), respectively:

$$\left.\begin{aligned} \bar{\bar{x}}_1 &= \frac{1}{2}(\bar{x}_1 + \bar{x}_3) \\ \bar{\bar{x}}_5 &= \frac{1}{2}(\bar{x}_5 + \bar{x}_7) \end{aligned}\right\} \quad \ldots (3)$$

$$\left.\begin{aligned} R_{21} &= |\bar{x}_1 - \bar{x}_3| \\ R_{25} &= |\bar{x}_5 - \bar{x}_7| \end{aligned}\right\} \quad \ldots (4)$$

5.1.4 Calculate the mean and the range for the pair of gross samples A and B using equations (5) and (6), respectively:

$$\bar{\bar{\bar{x}}} = \frac{1}{2}(\bar{\bar{x}}_1 + \bar{\bar{x}}_5) \quad \ldots (5)$$

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$$R_3 = |\bar{\bar{x}}_1 - \bar{\bar{x}}_5| \quad \ldots (6)$$

5.1.5 Calculate the overall mean and the means $\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$ of the ranges for all the lots for which the precision determination is being carried out using equations (7) to (10), respectively:

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$$\bar{\bar{\bar{\bar{x}}}} = \frac{1}{k}\sum \bar{\bar{\bar{x}}} \quad \ldots (7)$$

$$\bar{R}_1 = \frac{1}{4k}\sum (R_{11} + R_{13} + R_{15} + R_{17}) \quad \ldots (8)$$

$$\bar{R}_2 = \frac{1}{2k}\sum (R_{21} + R_{25}) \quad \ldots (9)$$

$$\bar{R}_3 = \frac{1}{k}\sum R_3 \quad \ldots (10)$$

where k is the number of lots and Σ covers all the relevant lots.

For the preparation of control charts for means and ranges, calculate the control limits using the sets of formulae (11) and (12).

Control limits for $\bar{x}$-chart:

$$\left.\begin{aligned} &\bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_1 \\ &\bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_2 \\ &\bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_3 \end{aligned}\right\} \quad \ldots (11)$$