INTERNATIONAL STANDARD

ISO 8868

First edition
1989-09-15

Fluorspar — Sampling and sample preparation

Spaths fluor — Échantillonnage et préparation des échantillons

Reference number
ISO 8868 : 1989 (E)

Contents

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

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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8868 was prepared by Technical Committee ISO/TC 175, *Fluorspar.*

Annex A forms an integral part of this International Standard.

Fluorspar — Sampling and sample preparation

1 Scope

This International Standard specifies sampling methods for a lot of fluorspar and methods for the preparation of samples taken from the lot for the purpose of determining the quality characteristics of the lot. It covers

a) the underlying theory;

b) the basic principles, and;

c) the basic requirements for the sampling device and its operation.

Details of different sizes of riffle divider are given in annex A.

The methods of sampling and sample preparation specified are applicable to all grades of fluorspar, i.e. acid grade, ceramic grade and the three metallurgical grades (concentrate, briquettes and gravel).

The methods are applicable to the sampling of fluorspar from conveyors, wagons and containers, ships and stockpiles, and from bagged material at the time of loading or discharging of a lot.

Samples are prepared for the determination of the following quality characteristics:

a) particle size distribution;

b) moisture content;

c) chemical composition.

2 Definitions

For the purposes of this International Standard, the following definitions apply.

2.1 lot: Definite quantity of fluorspar, the quality characteristics of which are to be determined.

2.2 increment: Quantity of fluorspar taken at one time from a larger body of fluorspar. There are two types of increment as follows:

a) a quantity of fluorspar taken at one time from a lot using a sampling device.

b) a quantity of fluorspar taken using the increment division method.

2.3 partial sample: A quantity of fluorspar consisting of a composite of two or more increments taken from a lot. This includes also a composite of two or more complete increments which have been individually crushed and/or optionally divided, as necessary, in accordance with this International Standard and prior to combining.

2.4 gross sample: A quantity of fluorspar consisting of a composite of all of the increments taken from a lot. This includes also the composite of all of the increments or all of the partial samples which have been individually crushed and/or optionally divided, as necessary, in accordance with this International Standard and prior to combining.

2.5 manual sampling: Sampling by human effort with sampling devices, including mechanically assisted devices.

2.6 mechanical sampling: Sampling by mechanical means.

2.7 test sample: Any sample, for the determination of the chemical composition, moisture content or particle size distribution, which is prepared in accordance with the specified method for that type of sample.

NOTE — If the entire quantity of a test sample is subjected to the test, the test sample may also be called the test portion.

2.8 test portion: Representative part of a test sample which is actually subjected to the tests for the determination of the chemical composition, moisture content or particle size distribution.

2.9 sample for chemical analysis: Sample taken for the determination of the chemical composition of the lot or part of the lot.

2.10 moisture sample: Sample taken for the determination of the moisture content of the lot or part of the lot.

2.11 particle size sample: Sample taken for the determination of the particle size distribution of the lot or part of the lot.

2.12 nominal top size, w_5: Particle size corresponding to the size of the opening of the sieve on which approximately 5 % (m/m) of the fluorspar charge is retained.

NOTE — The nominal top size of the lot may be ascertained either from past experience or by experiment. However, if no information is available, a visual estimation is acceptable.

2.13 stratified sampling: Sampling of a lot, which can be divided into several parts (called strata), by specific means in such a way that specified proportions of the sample are drawn from the different strata. The method is applied to sampling of lots from wagons or containers and from ships or stockpiles.

2.14 systematic sampling: Sampling in which increments are taken from a lot at regular intervals. There are two types of systematic sampling as follows.

2.14.1 systematic sampling on a mass basis: Systematic sampling in which the sampling interval used is a mass interval.

2.14.2 systematic sampling on a time basis: Systematic sampling in which the sampling interval used is a time interval.

2.15 two-stage sampling: Sampling by which primary sampling units are first selected from a lot and then secondary sampling units are taken from those selected primary sampling units. The method is applied to sampling from wagons or containers and from bagged material.

2.16 coefficient of variation, CV: Ratio of the standard deviation to the absolute value of the arithmetic mean.

NOTE — The ratio may be expressed as a percentage.

2.17 sample preparation: Process of preparing the sample for the determination of its quality characteristics. Sample preparation includes sample division, crushing, mixing and sometimes predrying and may be performed at several stages.

2.18 sample division: The process in sample preparation whereby the mass of a sample is reduced by partition or extraction.

2.19 proportional-mass division: Type of sample division for obtaining divided samples the masses of which are proportional to the mass of the sample or increment to be divided (such as might be obtained by using a riffle or mechanical splitters).

2.20 constant-mass division: Type of sample division resulting in divided increments which have a coefficient of variation equal to or less than 20 %, where the source material had a coefficient of variation greater than 20 %.

2.21 split use of sample: Method whereby the sample is split into two or more parts, which are used individually for the determination of two or more quality characteristics.

2.22 multiple use of sample: Method whereby the sample in its entirety is used for the determination of one quality characteristic. The same sample in its entirety, or part of this sample, is then used for the determination of another different quality characteristic.

3 General procedure

3.1 General procedure for sampling

Sampling shall be carried out while a lot is being transferred.

The general procedure for sampling shall be as follows:

a) identify the lot to be sampled;

b) ascertain the nominal top size of the lot;

c) determine the mass of increment to be taken according to the nominal top size;

d) determine the minimum number of increments to be taken from the lot;

e) determine the type of interval to be used for taking increments by systematic sampling and stratified sampling and the interval to be used for selecting the wagons or containers or the bags in the case of two-stage sampling;

f) determine the place of sampling and the method of taking increments;

g) take increments during the whole period of handling of the lot.

3.2 General procedure for sample preparation

The test samples for the required determinations shall be prepared from the increments taken according to the following general procedure:

a) determine whether the test sample is to be prepared from each increment or each partial sample or the gross sample according to the requirements for the determination of the quality characteristics;

b) determine whether the sample is to be in split use or in multiple use;

c) select the method of sample division to be used at each stage of the sample preparation;

d) establish the processes to be used in the sample preparation, including the processes of division, crushing, mixing and predrying (if necessary);

e) prepare the test sample according to the procedure established in d).

4 Fundamentals of sampling and sample preparation

4.1 Overall precision

The methods presented in this International Standard are designed to achieve the overall precisions shown in table 1, at a

Table 1 — Overall precision

Quality characteristic	Overall precision for the following grades of fluorspar %		
	Acid and ceramic	Metallurgical	
		Concentrate	Briquettes and gravel
Calcium fluoride content	0,5	1	1
Silica content	0,05	0,8	0,8
Moisture content	0,5	0,2	0,2
− 5 mm particle size fraction, 10 % on average	—	—	5

probability level of 95 %, with respect to the mean values of the calcium fluoride content, the silica content, the moisture content and the − 5 mm particle size fraction of a lot. However, the overall precision required may be agreed upon between the parties concerned.

The overall precision obtained is a measure of the combined precision of sampling, sample preparation and measurement and also depends on the method of constitution of samples and the number of determinations (see 4.5).

4.2 Mass of increment

4.2.1 The minimum mass of each increment shall be as specified in table 2 according to the nominal top size of a lot.

Table 2 — Minimum mass of increment

Nominal top size, w_5 mm	Minimum mass of increment kg
$160 < w_5$	30
$100 < w_5 \leqslant 160$	20
$50 < w_5 \leqslant 100$	10
$20 < w_5 \leqslant 50$	5
$10 < w_5 \leqslant 20$	2
$w_5 \leqslant 10$	1

4.2.2 The increments shall be taken in such a manner as to ensure that they are of "almost uniform mass", i.e. that the variation in mass shall be less than 20 % in terms of the coefficient of variation. However, when systematic sampling on a time basis is applied, the mass of individual increments shall be proportional to the flow rate of fluorspar.

4.3 Number of increments and precision of sampling

4.3.1 The minimum number of increments to be taken from a lot to attain the required precision of sampling shall be as specified in table 3 according to the mass of the lot.

Table 3 — Minimum number, N_{min}, of increments and precision of sampling (standard deviation), σ_S, for calcium fluoride content at the 95 % confidence level for all grades of fluorspar

Mass of lot, m tonnes	Gravel and briquettes		Concentrate, ceramic grade and acid grade	
	N_{min}	σ_S	N_{min}	σ_S
$13\,000 < m$	160	0,4	40	0,3
$6\,400 < m \leqslant 13\,000$	120	0,5	30	0,4
$3\,200 < m \leqslant 6\,400$	80	0,6	20	0,4
$1\,600 < m \leqslant 3\,200$	60	0,8	15	0,5
$750 < m \leqslant 1\,600$	40	1	10	0,6
$350 < m \leqslant 750$	30	1,1	7	0,8
$150 < m \leqslant 350$	20	1,3	5	0,9
$m \leqslant 150$	10	1,6	4	1

4.3.2 The values of the precision of sampling, σ_s, for calcium fluoride content shown in table 3 have been determined according to the corresponding minimum number of increments stipulated in table 3.

4.4 Method of taking increments

4.4.1 Each increment shall be taken at one time using a single motion of a sampling device. However, if this is difficult to achieve, the increment may be taken by using several motions of the sampling device from a point selected at random (with equal probability). The latter method shall be proven to have no bias with each type of sampling device in each grade of fluorspar before it is applied.

4.4.2 The increments shall be taken in such a manner as to ensure that they are of almost uniform mass as described in 4.2.2. When increments cannot be taken as almost uniform mass increments, each increment shall be prepared individually and the quality characteristics of each increment shall be determined. Alternatively, at an appropriate stage of the sample preparation, the divided increments of almost uniform mass may be combined into a partial sample or the gross sample. However, when systematic sampling on a time basis is applied, the mass of individual increments shall be proportional to the flow rate of fluorspar.

4.4.3 When the calculated mass of a sample is less than that required for preparing test samples (for the determination of particle size distribution etc.), the mass of each increment and/or the number of increments to be taken shall be increased.

4.5 Precision of sample preparation and overall precision

4.5.1 Sample preparation shall be carried out in accordance with clauses 7, 8 and 9.

4.5.2 The overall precision in terms of the standard deviation, σ_{SPM}, for the cases where division and measurement are carried out on the gross sample or each partial sample or on each increment may be calculated as follows.

4.5.2.1 When the gross sample is constituted for a lot and l determinations (e.g. chemical analyses) are carried out on the gross sample, the overall precision will be

$$\sigma_{SPM}^2 = \sigma_S^2 + \sigma_P^2 + \frac{\sigma_M^2}{l}$$

where

σ_S is the precision of sampling in terms of the standard deviation;

σ_P is the precision of sample preparation, in terms of the standard deviation, comprising the steps from the preparation of the gross sample into the test sample(s);

σ_M is the precision of measurement in terms of the standard deviation.

4.5.2.2 When k partial samples, each of which consists of an equal number of increments, are constituted and l determinations are carried out on each partial sample, the overall precision will be

$$\sigma_{SPM}^2 = \sigma_S^2 + \frac{\sigma_P^2 + \frac{\sigma_M^2}{l}}{k}$$

where σ_P is the precision of sample preparation, in terms of the standard deviation, comprising the steps from the preparation of the partial sample to the constitution of the test sample(s).

Further, when the above k partial samples are combined into the gross sample at an appropriate stage (e.g. at a particle size of − 10 mm) after individual sample preparation, and l determinations are carried out on the gross sample, the overall precision will be

$$\sigma_{SPM}^2 = \sigma_S^2 + \frac{\sigma_{P1}^2}{k} + \sigma_{P2}^2 + \frac{\sigma_M^2}{l}$$

where

σ_{P1} is the precision of sample preparation, in terms of the standard deviation, comprising the steps from the division of the partial samples to the constitution of the gross sample;

σ_{P2} is the precision of sample preparation, in terms of the standard deviation, comprising the steps from the preparation of the test sample(s) from the gross sample.

4.5.2.3 When l determinations are carried out on each increment, the overall precision will be

$$\sigma_{SPM}^2 = \sigma_S^2 + \frac{\sigma_P^2 + \frac{\sigma_M^2}{l}}{N}$$

where

σ_P is the precision of sample preparation, in terms of the standard deviation, comprising the processes of preparing the test sample(s) from the increments;

N is the number of increments.

Further, when all the increments are combined into the gross sample at an appropriate stage (e.g. at a particle size of − 10 mm) after individual sample preparation, and l determinations are carried out on the gross sample, the overall precision will be

$$\sigma_{SPM}^2 = \sigma_S^2 + \frac{\sigma_{P1}^2}{N} + \sigma_{P2}^2 + \frac{\sigma_M^2}{l}$$

where

σ_{P1} is the precision of sample preparation, in terms of the standard deviation, comprising the steps in the preparation of the gross sample from the increments;

σ_{P2} is the precision of sample preparation, in terms of the standard deviation, comprising the steps in the preparation of the test sample from the gross sample.

4.6 Constitution of samples

When samples are to be constituted from the increments, the following shall be taken into consideration:

a) the quality characteristics to be determined;

b) the overall precision to be achieved;

c) the coefficient of variation, CV, in the mass of increments taken by mass-basis sampling.

4.7 Division rules

In order to obtain an acceptable precision of sample preparation, the following aspects of sample division shall be considered:

a) the nominal top size of the sample to be divided;

b) the minimum mass of the sample after division, specified for each quality characteristic to be determined;

c) the method of division to be adopted.

4.8 Method of division

One or more of the following methods of sample division shall be conducted individually or jointly:

a) manual increment division method (see 8.1) (chocolate box method);

b) manual riffle division method (see 8.2);

c) coning and quartering method (see 8.3);

d) mechanical division method (see 8.4).

4.9 Split use and multiple use of sample

When a sample taken from the lot meets the respective requirements for the determination of the quality characteristics, the sample may be either in split use or in multiple use for obtaining the test samples for chemical analysis, moisture determination and determination of particle size distribution.

4.10 Comminution

Crushing and grinding shall be conducted with crushers and grinders suitable for the size and hardness of the particles of fluorspar. Crushers and grinders should be purged with fluorspar from the same source.

Care shall be taken not to induce any changes in the chemical composition, hydration etc. of the fluorspar.

4.11 Mixing

By mixing the sample thoroughly, it will be made homogeneous and consequently the errors in sample division can be lessened. The mixing may be carried out either by using a mechanical mixer or by hand. The mixer shall be selected to suit the sample and its particle size.

4.12 Predrying

When the sample is very wet or sticky and the sample preparation cannot be carried out, the sample may be predried by air drying or oven drying, at a temperature of 105 °C ± 5 °C, so that the sample preparation can be carried out without any difficulty.

4.13 Requirements for sampling and sample preparation

4.13.1 Sampling and sample preparation shall be carried out in such a manner that there shall be no contamination of the sample or introduction of foreign materials into the sample and no change in its quality characteristics.

4.13.2 Control experiments for checking the precision and bias shall be carried out from time to time on the procedures of sampling and sample preparation so that significant errors in the results due to the procedures may be detected.

5 Apparatus

5.1 Apparatus for sampling

Sampling devices capable of taking the specified mass of increment without introducing bias shall be provided. If an increment shovel is used as the sampling device for taking increments from a lot, it should be of the type and dimensions given in figure 1 and table 4.

NOTE — Other sampling devices, including mechanically assisted devices and mechanical samplers, may be used to take increments. These devices should have a minimum opening equivalent to the dimension *a* given in table 4 or at least three times the nominal top size.

5.2 Apparatus for sample preparation

The following apparatus shall be provided for sample preparation:

a) crushers and grinders;

b) drying ovens, if necessary, in which the temperature at any point in the oven can be regulated to ± 5 °C of the desired temperature;

c) mixers, if necessary;

d) riffle dividers, details of which are given in the annex;

e) shovels for increment division, details of which are given in figure 1 and table 4;

f) equipment for mechanical sample preparation, if necessary.

6 Method of sampling

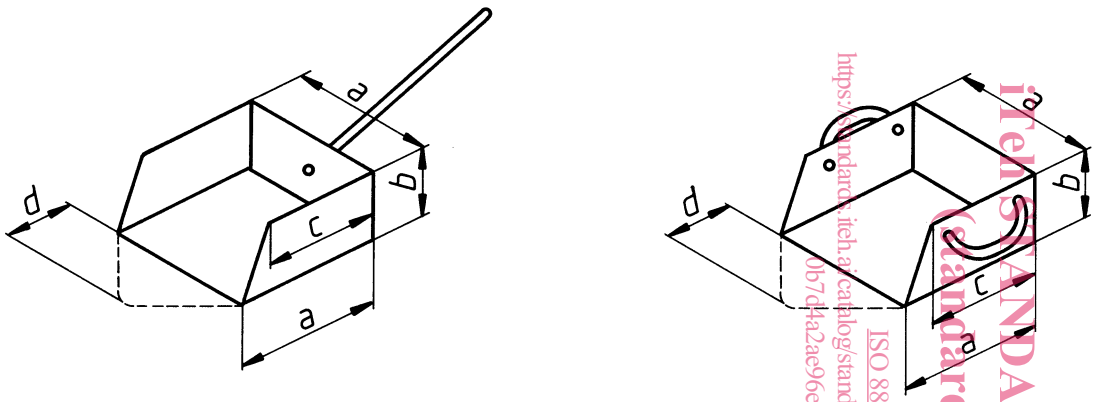
The method of increment sampling used shall be as specified in either 6.1, 6.2, 6.3 or 6.4 as appropriate.

6.1 Sampling from conveyors

6.1.1 When the increment is taken from a conveyor which is stationary, a section of "adequate length" in the direction of the flow and of the full width and thickness of the fluorspar stream shall be taken from the specified position. The adequate length shall be sufficient to ensure that a minimum mass of increment as specified in table 2 can be taken, and shall be more than three times the nominal top size, with a minimum value of not less than the width of the smallest sampling increment shovel (i.e. 60 mm).

6.1.2 When the increment is taken from a moving conveyor, the full width and thickness of the flowing fluorspar stream shall be taken using a mechanically assisted device or mechanical sampler.

6.1.3 When the effect of segregation in particle size is known to be not significant at the point of sampling and there is no pulsation in the flow, the individual increments may be taken



NOTE — The shovel may have a triangular tip, which is convenient for insertion of the shovel into the fluorspar. However, shovels for increment division should have the triangular tip cut off; such shovels are identified by the letter D in the shovel number (see tables 4 and 7).

Figure 1 — Examples of increment shovels

Table 4 — Dimensions of increment shovels

Shovel number	Nominal top size mm	Dimensions of increment shovel mm			
		a	*b*	*c*	*d*
150D	150	450	190	380	170
125D	125	380	160	320	150
100D	100	300	130	260	120
70D	71	200	100	170	80
50D	50	150	75	130	65
40D	40	120	65	100	50
30D	31,5	90	50	80	40
20D	22,4	80	45	70	35
15D	16	70	40	60	30
10D	10	60	35	50	25
5D	5	50	30	40	0
3D	2,8	40	25	35	0
1D	1	30	15	25	0
0,5D	0,5	20	10	20	0
0,25D	0,25	15	10	12	0

from the points selected at random either on the stationary conveyor or within the flowing stream.

6.1.4 The interval used to take increments shall be uniform, on a mass basis, throughout the whole lot and shall not vary during the course of sampling.

6.1.4.1 The mass interval between taking increments shall be calculated using the following formula :

$$\Delta m = \frac{m}{N_{\text{min}}}$$

where

Δm is the mass interval, in tonnes, between taking increments;

m is the mass, in tonnes, of the lot;

N_{min} is the minimum number of increments determined in 4.3.1.

6.1.4.2 The actual mass interval used to take increments shall be smaller than the calculated mass interval, Δm, as calculated in 6.1.4.1, taking the convenience of operation into account.

6.1.4.3 If the flow of fluorspar is regular, the mass interval may be converted into an equivalent time interval.

6.1.5 The interval used to take increments in systematic sampling on a time basis should be smaller than the equivalent time interval used for mass-basis sampling.

6.1.6 The first increment shall be taken after a randomly selected tonnage of fluorspar has been handled within the first interval, after the start of the handling operation.

6.1.7 The subsequent increments shall be taken at the fixed time intervals until the handling operation of the lot has been completed.

6.2 Sampling from wagons or containers

6.2.1 Selection of wagons or containers and number of increments to be taken

6.2.1.1 When the number of wagons or containers constituting a lot is more than or equal to the minimum number of increments specified in 4.3.1, a number of wagons or containers equal to the number of increments shall be selected systematically or at random. One or more increments shall be taken from each wagon or container so selected.

6.2.1.2 When the number of wagons or containers constituting a lot is less than the minimum number of increments specified in 4.3.1, divide the minimum number of increments by the number of wagons or containers, round off the result to the next higher whole number and take that number of increments from each wagon or container.

6.2.2 Method of taking increments from wagons or containers

6.2.2.1 The increments shall be taken at random from the fresh surface of fluorspar exposed during the loading or the unloading of the wagons or containers.

6.2.2.2 When it is possible that there is some bias in the fluorspar distribution in the wagon or container between the top and the bottom, between the front and the rear, or between the left and the right, it is advisable to take the increments from each stratum divided or from different places in each of the wagons or containers selected.

6.2.2.3 There is a possibility of introducing some bias in sampling when the sampling is conducted with a sampling probe or boring sampler from the top surface of fluorspar loaded on wagons or containers and, accordingly this sampling method shall be applied only after it has been ascertained, by check experiments, that there is no bias present.

6.3 Sampling from ships or stockpiles

6.3.1 Sampling from ships

6.3.1.1 When and where to take increments

The increments shall be taken, during the handling operation, from the fresh surface of fluorspar exposed in the hold by the handling.

6.3.1.2 Method of taking increments

The minimum number of increments specified in 4.3.1 shall be taken from a ship. The number of increments to be taken from each hold of a ship shall be proportional to the mass of fluorspar in each hold. When the mass of fluorspar per hold is small, the increments may be taken at positions spaced regularly over the entire lot.

6.3.1.3 Taking increments at other places

Increments taken from the surface of fluorspar loaded in the hold are subject to significant bias and it is more desirable to take increments at other places, such as from conveyors or from handling equipment.

6.3.2 Sampling from stockpiles

6.3.2.1 The sampling of fluorspar from stockpiles shall be conducted in accordance with the method specified in 6.1, during the formation of the stockpiles or during the reclamation of the stockpiles for their transfer to other places.

6.3.2.2 Sampling shall not be conducted from stockpiles which are not being formed or reclaimed. If this were to be done, the precision of sampling would not be in accordance with the specifications of this International Standard and some significant bias would be introduced.