

JIS

JAPANESE INDUSTRIAL STANDARD

**Particulate materials—
General rules
for methods of sampling**

JIS M 8100—1992

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**In the event of any doubt arising,
the original Standard in Japanese is to be final authority.**

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Particulate materials - General rules
for methods of sampling

M 8100-1992

1. Scope This Japanese Industrial Standard specifies the general matters of the following (1) to (4) methods for determining the average values (hereafter referred to as the "average quality") of the chemical constituents, moisture contents, particle size, physical characteristics and other characteristics of a lot of particulate materials.

- (1) Methods of sampling
- (2) Methods of preparing the sample to be tested
- (3) Methods of determining the moisture content
- (4) Methods of determining the particle size

Remarks 1. This Standard gives a guide line in preparing each standard of sampling methods of various kinds of particulate materials (see Annex 1).

2. This Standard is applicable to the case of carrying out the sampling of particulate materials and the like which have not been specified in JIS, as appropriate.

3. The following standards are cited in this Standard.

JIS Z 8401-Rules for rounding off of numerical values

JIS Z 8801-Test sieves

JIS Z 8815-Test sieving

4. Corresponding international standards to this Standard are given as follows:

ISO 3082: 1987 Iron ores - Increment sampling and sample preparation - Mechanical method

ISO 3084: 1986 Iron ores - Experimental methods for evaluation of quality variation

ISO 3085: 1986 Iron ores - Experimental methods for checking the precision of sampling

ISO 3086: 1986 Iron ores - Experimental methods for checking the bias of sampling

2. Definitions For the purposes of this Standard, the following definitions apply:

- (1) particulate materials The aggregates of powdery or lump materials and their mixed materials.
- (2) lot A certain quantity of the particulate materials for determination of the average quality.
- (3) increment A unit quantity of the particulate materials taken by one action, as a rule, from the lot by a sampling device.
- (4) sub-sample The sample produced by collecting several number of increments.

Remarks: After preparing increments individually, as required, the sample produced by collecting several number of those is sometimes called the sub-sample.

- (5) gross sample The sample of which all of the increments taken from lot, or all of the sub-samples have been collected.

Remarks: The sample of which the increments or sub-samples have been collected after each preparation is sometimes called the gross sample.

- (6) random sampling Sampling the increments so that the unit body or unit quantity composing the lot will be included in the sample at the same probability.

Remarks: Sampling the increments at random from the whole lot.

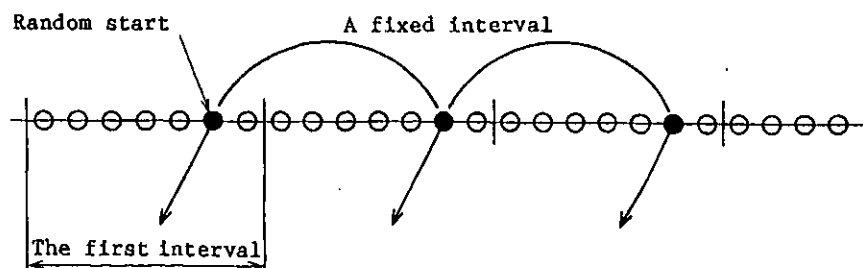
- (7) stratified sampling Sampling the increments at random from each stratum by dividing the lot into several strata.

Remarks: It is called the stratified proportional sampling to sample the number of increments in proportion to the size of stratum.

- (8) systematic sampling Sampling the increment at fixed interval in quantity or in time from the lot.

Remarks: For systematic sampling, it is called the random start to take the first increment at random from the first interval (see Fig. 1).

Fig. 1. Systematic sampling



- (9) two-stage sampling Divide the lot into several parts (the primary sampling units) and for the first stage, select several parts of them at random and next, take several number of increments (the secondary sampling units) respectively at random out of the parts selected for the second stage.
- Remarks: It is called the multi-stage sampling to take the increments repeatedly not less than 2 stages.
- (10) sampling on belts The method to take the increments from the belt conveyors or its chutes when the lot is carried by the belt conveyors or the like.
- (11) sampling from freight cars The method to take the increments from freight cars or lorries when the lot is delivered by freight cars or lorries.
- (12) sampling from containers The method to take the increments from the containers when the lot is delivered by containers (bags, drum cans or the like).
- (13) sampling from ship's hold The method to take the increments from ship's hold or cargo-handling implements during stevedoring when the lot is delivered by shipping.
- (14) preparation of sample Preparation of the samples for constituent test, moisture test, particle size test, physical characteristic test, etc. by size reduction, reducing and mixing of the sample, as required.
- (15) reduction To reduce the quantity of sample.
- (16) sample for constituent General term of the sample taken for the constituent analysis.
- Remarks: The sample for constituent prepared for the purpose of analysis is called the sample for constituent test.
- (17) sample for moisture content General term of the sample taken for the measurement of moisture content.
- Remarks: The sample for moisture content prepared for the measurement of moisture content is called the sample for moisture content test.
- (18) sample for particle size General term of the sample taken for the measurement of particle size.
- Remarks: The sample for particle size prepared as required for the measurement of particle size is called the sample for particle size test.

- (19) sample for physical characteristics General term of the sample taken for the measurement of physical characteristics.

Remarks: The sample for the physical characteristics prepared for the measurement of physical characteristics is called the sample for physical characteristic test. The sample for physical characteristic test may be called such as the sample for ○○ strength test in place of physical characteristics.

- (20) combined use of sample The combined use of sample is to use for the measurements of not less than 2 kinds of characteristics by dividing the sample.

- (21) duplicate use of sample The duplicate use of sample is to use all or its part of sample, which has been used for the measurement of a certain characteristics, for the measurement to obtain other characteristics.

- (22) whole through sieve size The size of the largest particle of sample.

Remarks: It is expressed by the size of the smallest sieve aperture through which the whole quantity of sample passes.

- (23) maximum particle size The size of aperture of sieve equivalent to 5 % of the residual rate of the sample on the sieve.

- (24) error The difference between the measured value and the true value.

Remarks: The error caused by sampling is called "sampling error", the error caused by sample preparation is called "preparation error", and the error caused by measurement is called "measurement error".

The errors are usually classified into precision and bias.

- (25) precision The degree of dispersion of measured values.

- (26) bias The difference between the center of distribution of measured values and the true value.

3. Symbols For the purposes of this Standard, the following symbols apply:

μ : population mean of characteristics of a lot

$\bar{x}$: arithmetic average value of measured values

R : range (difference between the maximum value and the minimum value among the measured values)

$\bar{R}$: average value of range

M : number of the primary sampling units constituting a lot

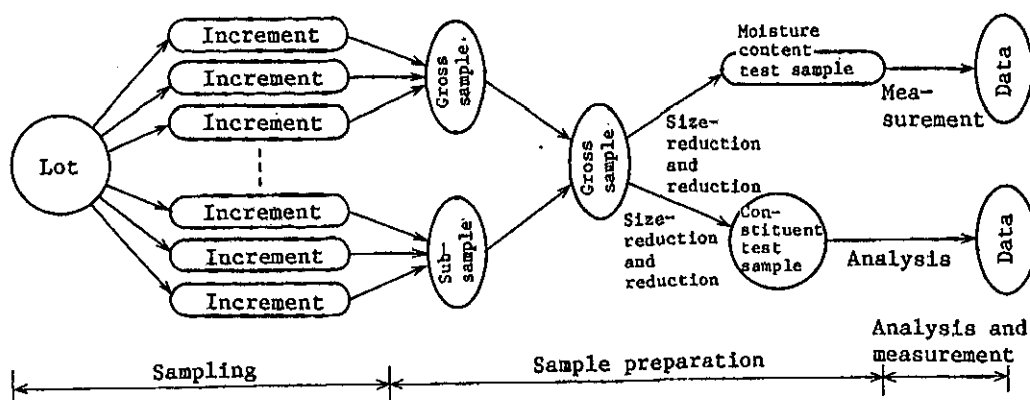
- m : number of the primary sampling units sampled for the first stage in the case of two-stage sampling
- n : number of increments taken from a lot
- n_w : number of increments taken for the second stage from the primary sampling unit in the case of two-stage sampling
- d_2 : coefficient to obtain the standard deviation from the average value $\bar{R}$ of range
- D_4 : coefficient to obtain the upper control limit of 3σ relative to the range from the average value of the range, $\bar{R}$
- σ : precision indicated by the standard deviation
- σ_b : dispersion among primary sampling units or among strata expressed by the standard deviation
- σ_w : dispersion among the increments in the primary sampling unit or in the stratum expressed by the standard deviation
- σ_i : dispersion among the increments in a lot which is expressed by the standard deviation
- σ_s : sampling precision expressed by the standard deviation
- σ_p : preparation precision expressed by the standard deviation
- σ_M : analysis or measuring precision expressed by the standard deviation
- CV : coefficient of variation
- β : precision of 2σ
- K_1 : cost to sample a primary sampling unit
- K_2 : cost to sample a secondary sampling unit (increment)

4. General matters

4.1 Summary of sampling and sample preparation The summary of sampling and sample preparation shall be as follows (see Fig. 2):

- (1) Decide the lot intended for sampling and sample preparation.
- (2) Take the required number of increments with the specified size out of the whole lot at random or systematically during movement of lot.
- (3) Prepare the test sample by size reduction and reducing every increment, every sub-sample or every gross sample as required.

Fig. 2. Summary of sampling and sample preparation (An example)



4.2 Handling of sample Cares shall be taken avoid losing or deteriorating a sample throughout the period of sampling, sample preparation and measurement.

Further, the devices to be used shall be sufficiently cleaned so that foreign substances can not be mixed in the sample.

(1) **Sample container** The sample container shall be as follows:

- (a) The container used for transporting or storing various kinds of samples shall be such that it can accommodate complete quantity of samples and be clean and rigid and further, it can be covered with a lid or be sealed securely.
- (b) Specially, the container of sample for moisture content shall be airtight and be made of materials free from hygroscopic property and more over, its inside shall be free from corrosions and the like.

Remarks: The straw bag, hemp bag, etc. are unsuitable for the container of sample for moisture content.

(2) **Marking of sample for constituent test** The sample for constituent test shall be marked on the package with the following items, as a rule:

- (a) Name of article and name of lot
- (b) Name of sample or its symbol
- (c) Dates of sampling and sample preparation
- (d) Name of factory of sampling and sample preparation
- (e) Name of responsible person for sampling and sample preparation

- (3) Storage of sample The storage of sample shall be as follows:
- (a) The constituent test sample shall be stored after preparing and sealing up, as a rule, for three months for domestic trade and for six months for foreign trade.
 - (b) Cares shall be taken for the storage place of the sample to be free from being affected by temperature, humidity, direct rays of the sun, etc. The sample shall be sealed up at the time of storage and cares shall be taken particularly on the materials of container for the sample easily oxidized.
- (4) Sending of sample The sample other than the constituent test sample shall not be sent, as a rule. However, when it is sent inevitably, the sending method shall be as agreed upon between the parties concerned with delivery.

4.3 Determination of average quality The average quality shall be determined in accordance with any one of the following methods:

- (1) When measurement is performed on gross sample, the measured value shall be taken as the determined value of the average quality of the lot.
- (2) When measurement is performed on each sub-sample, the weighted average of measured values shall be taken as the determined value of the average quality of the lot.
- (3) When measurement is performed on each increment, the average of measured values shall be taken as the determined value of the average quality of the lot.

4.4 Rounding-off of numerical value The rounding-off of numerical value shall be in accordance with JIS Z 8401.

4.5 Other matters

- (1) When a part of the method specified in this Standard cannot be applied, the alternative method can be specified in each standard if it can be confirmed by the check experiment that there is no bias and the specified precision can be satisfied.
- (2) The experiment to check the sampling method shall be carried out, as a rule, in accordance with Annex 3 to Annex 7 periodically or as required, and whether the sampling work is carried out according to the standard, or whether the specified precision is attained even when the process of work and the lot have been varied or not shall be inspected.
- (3) When the water spray treatment has been carried out for preventing dusts at the stevedoring of lot, the method to determine the moisture content shall be specified in each standard or be as agreed upon between the parties concerned with delivery.

5. Sampling method

5.1 Size of lot The particulate materials for one delivery and of the same brand shall be taken as one lot, as a rule.

As required, the maximum limit of size of one lot may be specified, as agreed upon between the parties concerned with delivery.

5.2 Characteristics and precision

5.2.1 Characteristics The characteristics to specify the precision shall be the representative characteristics to determine the value of the intended particulate materials or the overall characteristics⁽¹⁾.

Note (1) The overall characteristics are the characteristics in which the characteristics of not less than two kinds are considered at the same time and it is sometimes expressed in an amount of money, for example.

5.2.2 Overall precision The overall precision is expressed by two times the value of the standard deviation ($2\sigma_{SPM}$).

Remarks: Therefore, when taking the overall precision as β_{SPM} , the probability of the population mean value of lot included in the determined value $\pm \beta_{SPM}$ of lot is about 95 %.

5.2.3 Level of precision When designing the sampling method, sample preparation method, measuring or analyzing method, the level of precision used for each characteristic shall be any one of the levels in Table 1, as a rule. The level of precision is expressed by the coefficient of variation [$CV = (\sigma_{SPM}/\bar{x}) \times 100 \%$].

Table 1. Level of overall precision

Number of level	Level of precision (CV %)
1	20
2	10
3	5
4	2
5	1
6	0.5
7	0.2
8	0.1

5.3 Quality variation

5.3.1 Classification of quality variation The classification of quality variation shall be expressed by any one of the following standard deviation:

- (1) The standard deviation (σ_i) among increments in the lot, for simple random sampling.
- (2) The standard deviation (σ_w) in the stratum, for the stratified sampling and the systematic sampling.
- (3) The standard deviation (σ_w) among increments in the primary sampling unit and the standard deviation (σ_b) among the primary sampling units, for two-stage sampling.

5.3.2 Classification of magnitudes of quality variation The magnitudes of quality variation shall be classified into large, medium and small on the basis of the investigations of the standard deviation in the stratum, the standard deviation among strata and the standard deviation among increments in the lot which are required for each characteristic or representative characteristics (see Annex 3 and Annex 4).

Remarks 1. When the magnitude of quality variation of the intended particulate materials is unknown, those materials shall be dealt as that the quality variation is "large", as a rule, until that magnitude is known.

2. When the magnitude of quality variation is unknown, the division of magnitude of quality variation shall be determined by the investigation as soon as possible.

5.4 Carry-out time of sampling The sampling time of increment shall be as follows:

- (1) Carry it out during movement of lot at the time of delivery, as a rule.
- (2) Carry it out when the mass of lot is weighed or at the time as soon as possible before or after that time.

5.5 Classification of sampling The sampling shall be divided into the following 4 classes according to the means of transportation or the condition of lot.

Further, the methods of not less than 2 classes can be used jointly for one lot.

- (1) Belt sampling
- (2) Freight car sampling
- (3) Container sampling
- (4) Ship's hold sampling

For the screw conveyor, apron conveyor, pipe transportation, etc., the belt sampling; for the barge and others, the ship's hold sampling; for the motor lorry or truck, the freight car sampling, for the drum can or other containers, the container sampling; for the bucket conveyor, the container or freight car sampling shall be applied respectively, as appropriate.

5.6 Increments

5.6.1 Size of increment The size of increment shall be sufficient for being free from bias at the time of sampling.

It is necessary to decide the size of increment so that the maximum particle size of lot shall be included in the increment at random.

The size of increment shall be expressed in mass.

- Remarks
1. At the time of sampling, the increment shall be taken so that its size shall become nearly uniform for each one. The "nearly uniform" means that the dispersion of size of increment is less than 20 % as coefficient of variation.
 2. When the increment cannot be taken so that its size shall become nearly uniform, nearly fixed quantity shall be collected respectively from each increment at an appropriate stage of sample preparation.
 3. When measuring each increment one by one, the size of increment may not necessarily be constant.
 4. When the mass of sample which has been prepared from the gross sample in accordance with the specified method becomes not more than the mass of the specified test sample, the size of increment shall be made larger than that of the specification or the number of increments shall be increased.

5.6.2 Devices and apparatus for taking increments The devices and apparatus for taking increments shall be as follows:

- (1) The shovel for taking increments shall be a shovel with larger capacity than the capacity specified in Table 2, as a rule, according to the maximum particle size of lot.

Further, the opening part shall be not less than three times the maximum particle size.

Table 2. Capacity of shovels for taking increments

Maximum particle size mm	Capacity ml	No. of shovel
150	About 35000	150
125	About 21000	125
100	About 11000	100
71	About 3700	70
50	About 1600	50
40	About 730	40
31.5	About 380	30
22.4	About 270	20
16	About 180	15
10	About 120	10
5	About 70	5
2.8	About 35	3
1	About 16	1
0.25	About 2	0.25D

Remarks 1. The shovel No. 0.25 D shall be used only for reduction of increments.

2. An example of dimensions of shovels for taking increments is shown in the Attached Fig. 1.

(2) Mechanical sampling apparatus (see Annex 2)

- (a) Installation of apparatus The sampling apparatus shall be installed at a place through where the whole quantity of lot passes when moving that lot.

Remarks: For example, install it on the main belt conveyor or at the chute and the like.

- (b) Sampler The size of opening part of sampler shall be such that the maximum particle size in the lot can be taken easily (size of not less than three times the maximum particle size, as a rule).

Further, the sampler shall be so constructed that the samples do not flow over from the sampler or that the samples are not blocked at the chute.

Functioning of the sampler in belt sampling, as a rule, on a plane surface vertical to a flow direction shall be thoroughly capable of dealing with the spread of the flow. The functioning speed in the flow shall be determined so that no bias shall be generated in characteristic value and it shall be almost constant.

Remarks: Fluctuation in functioning speed of the sampler should preferably be the average value $\pm 5\%$.

- (c) Sampling interval The sampling interval should preferably be changeable.

Remarks: When the increment is taken at the fixed time interval, it shall be taken as follows:

- (1) The size of increment to be taken shall be in proportion to the flow rate.
- (2) As to reduction, proportional reduction (reduction at the fixed rate) shall apply.

- (d) Safety Sufficient cares shall be taken on the safety of workers in designing and installing the mechanical sampling apparatus.

- (e) Maintenance and monitoring The mechanical sampling apparatus should preferably be so constructed that the maintenance and cleaning are easy throughout all the system such as sampler, conveyor, hopper, crusher, etc. and its materials are corrosion proof.

Further, each function should preferably be easily monitored during operation.

- (f) Check of bias After installing the mechanical sampling apparatus, it is necessary for the mechanical sampling to confirm as soon as possible that the samples taken by this apparatus are free from bias (see Annex 6 and Annex 7).

- (g) Check of overall precision The mechanical sampling apparatus should preferably be so constructed that the check experiment of overall precision can be carried out easily (see Annex 5).

(3) Other sampling devices

- (a) The increment shall be free from bias.
- (b) The capacity shall be not less than that of Table 2.

5.6.3 Determining method of number of increments The minimum required number of the increments taken from lot shall be specified for each division of the required overall precision ($\beta_{SPM}=2\sigma_{SPM}$) of each characteristics such as the representative characteristics and others or the overall characteristics and of the quality variation of lot (σ_i , σ_b and σ_w).

If required, the minimum required number of increments shall be specified on every size, every required precision and every class of characteristics of the lot. The number of increments (n) to be taken shall be obtained by the following procedure of calculation after selecting the level of overall precision (CV%).

(Procedure 1) Determine the level of precision (CV%) in accordance with Table 1.

(Procedure 2) Calculate the standard deviation (σ_{SPM}) from (CV%),

$$CV = \frac{\sigma_{SPM}}{\bar{x}} \times 100 \text{ therefore, } \sigma_{SPM} = \frac{CV \times \bar{x}}{100}$$

or obtain the standard deviation (σ_{SPM}) from the overall precision (β_{SPM})

$$\beta_{SPM} = 2\sigma_{SPM} \text{ therefore, } \sigma_{SPM} = \frac{\beta_{SPM}}{2}$$

(Procedure 3) Calculate σ_s using σ_P and σ_M obtained separately from σ_{SPM} which has been obtained by the procedure 2.

$$\begin{aligned} \sigma_s &= \sqrt{\sigma_{SPM}^2 - (\sigma_P^2 + \sigma_M^2)} \\ &= \sqrt{\left(\frac{CV \times \bar{x}}{100}\right)^2 - (\sigma_P^2 + \sigma_M^2)} \end{aligned}$$

or

$$\sigma_s = \sqrt{\left(\frac{\beta_{SPM}}{2}\right)^2 - (\sigma_P^2 + \sigma_M^2)}$$

(Procedure 4) According to the type of sampling, determine the number of increments n to be taken by rounding off the figures below decimal point.

(a) Random sampling

$$\sigma_s = \frac{\sigma_i}{\sqrt{n}} \quad n = \left(\frac{\sigma_i}{\sigma_s}\right)^2$$

(b) Stratified sampling

$$\sigma_s = \frac{\sigma_w}{\sqrt{n}} \quad n = \left(\frac{\sigma_w}{\sigma_s}\right)^2$$

(c) Systematic sampling

$$\sigma_s = \frac{\sigma_w}{\sqrt{n}} \quad n = \left(\frac{\sigma_w}{\sigma_s}\right)^2$$

(d) Two-stage sampling

$$\sigma_s = \sqrt{\frac{(M-m)}{M-1} \cdot \frac{\sigma_b^2}{m} + \frac{\sigma_w^2}{mn_w}}$$

$$n_w = \sqrt{\frac{K_1}{K_2} \cdot \frac{\sigma_w}{\sigma_b}} \quad m = \frac{M\sigma_b^2 + (M-1)\sigma_b\sigma_w\sqrt{\frac{K_2}{K_1}}}{(M-1)\sigma_s^2 + \sigma_b^2}$$

Remarks: The n_w and m are applicable to the case where the size of primary sampling unit and the number of increments taken from the primary sampling unit are fixed.

Informative reference: In the stratified sampling, the sample should be preferably so stratified that the inside of the stratum becomes as uniform as possible and a difference between the strata increases.

- Remarks 1. When the sampling is carried out at fixed interval in the systematic sampling, the sampling interval is determined by dividing the size of lot N [for example tonnage (t)] by the number of increments n obtained from the formula (c). There are cases where the specified number of increments cannot be taken completely by reasons of material handling or there are cases where the materials to be sampled cannot be found on the belt even when the time of sampling has come. In consideration of such cases, it should be preferable to increase the number of increments more than the calculated value and select a short sampling interval.
2. Taking increments at fixed interval in the systematic sampling method, for example at a 100 t interval, can be approximately considered as the stratified sampling method, provided that the respective increment is taken from each stratum divided by 100 t unit. Accordingly, the calculation can be made under the consideration of the approximate stratified sampling method.
 3. For two-stage sampling, $\frac{M-m}{M-1} \approx 1$ may be taken when $\frac{m}{M} \leq 0.1$.
 4. For two-stage sampling, $n_w = 1$ when $K_1 = 0$.
 5. The values used for σ_b , σ_b and σ_w shall be the intermediate representative values of each division of the magnitude of quality variation such as large, medium, small and the like.
 6. For n , m and n_w , it should be preferable to carry out the check experiment of precision in accordance with Annex 5.

5.6.4 Method of taking increments The method of taking increments shall be as follows:

- (1) Use the sampling devices specified in 5.6.2, take the increment at random by one working action so that the size of increment shall become nearly uniform. However, when it is difficult to take by one working action, the increment may be taken by several actions from one place selected at random and these taken materials may be combined to constitute one increment.
- (2) Take the increment from the lot during material handling, as a rule.

Remarks: For taking increment inevitably from the stock-pile, it shall be confirmed by carrying out check experiment whether or not the bias exists and the precision is sufficient.

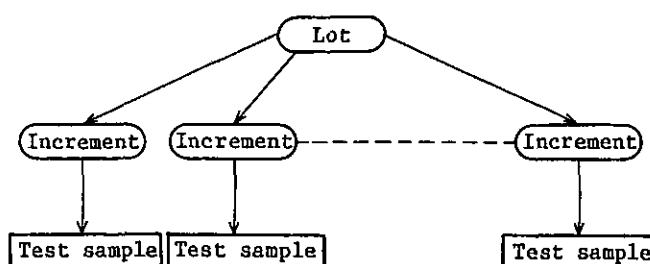
- (3) Do not change the sampling interval during the sampling work of one lot.
- (4) Even when the taking of the specified number of increments has been finished, the increment shall be continuously taken at the specified interval without discontinuing the taking of increments as long as the material handling of lot is carried out continuously.

5.7 Method for constitution of increments and its overall precision

5.7.1 When the increments are taken from whole lot

- (1) Prepare the test sample from each increment (see Fig. 3).

Fig. 3

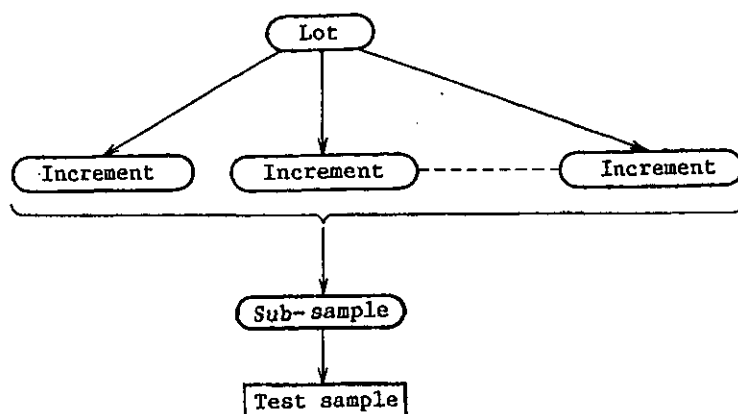


When the test sample has been analyzed one time, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{\sigma_w^2 + \sigma_p^2 + \sigma_M^2}{n}$

When the test sample has been analyzed l times, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{\sigma_w^2 + \sigma_p^2 + \sigma_M^2/l}{n}$

- (2) Prepare the test sample from each sub-sample (see Fig. 4).

Fig. 4



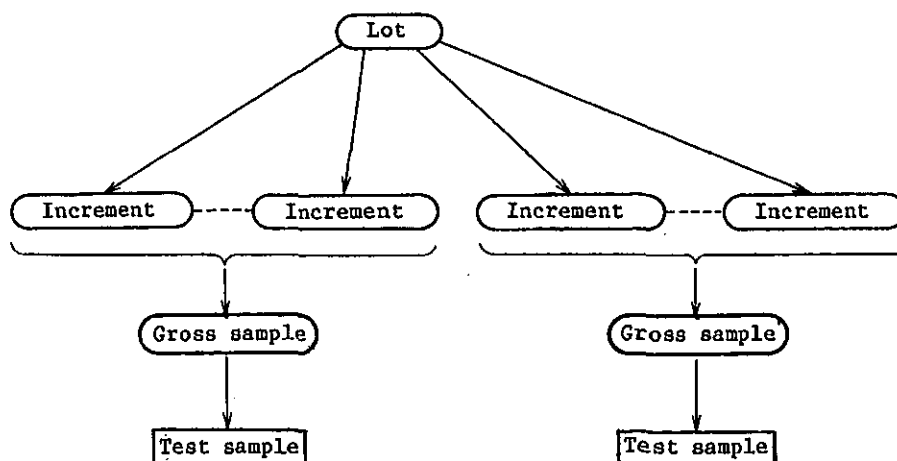
When the test sample has been analyzed one time, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{\sigma_w^2}{n} + \frac{\sigma_p^2 + \sigma_M^2}{k}$

When the test sample has been analyzed l times, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{\sigma_w^2}{n} + \frac{\sigma_p^2}{k} + \frac{\sigma_M^2}{kl}$

The symbol " k " represents the number of test samples.

- (3) Prepare the test sample from the gross sample (see Fig. 5).

Fig. 5.



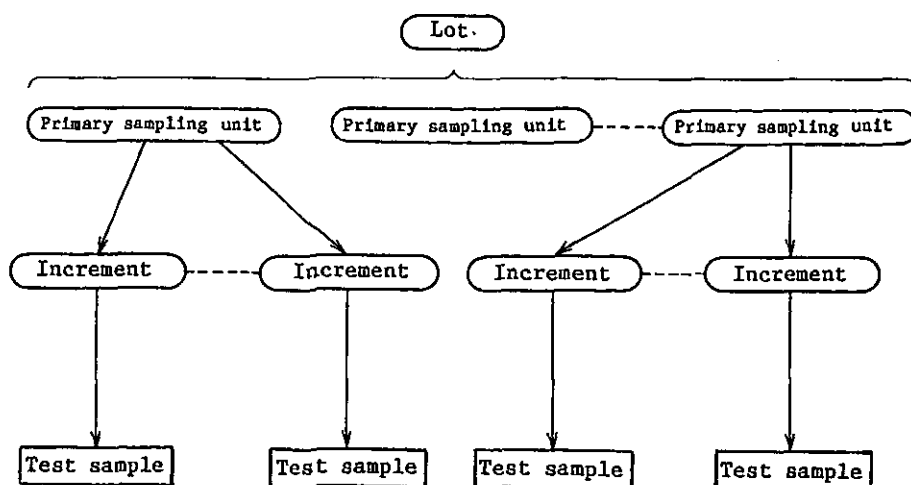
When the test sample has been analyzed one time, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{\sigma_w^2}{n} + \sigma_p^2 + \sigma_M^2$

When the test sample has been analyzed l times, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{\sigma_w^2}{n} + \sigma_p^2 + \frac{\sigma_M^2}{l}$

5.7.2 When some primary sampling units are taken from lot and next, some increments (secondary sampling units) are taken from this primary sampling unit

- (1) Prepare the test sample from each increment (see Fig. 6).

Fig. 6

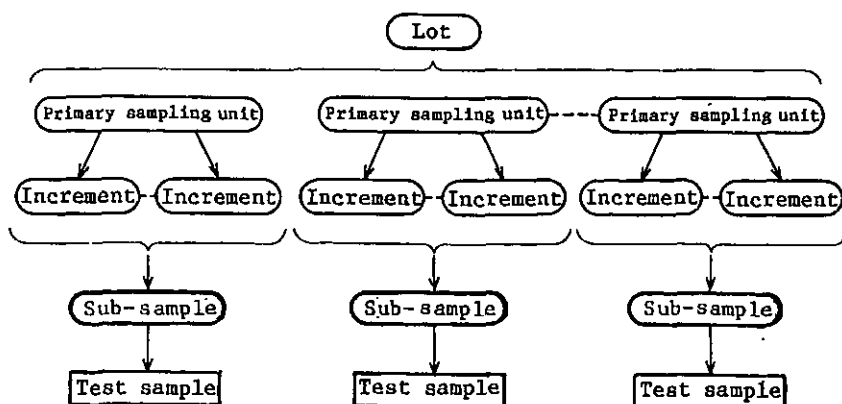


When the test sample has been analyzed one time, the overall precision
$$\left(\frac{\beta}{2}\right)^2 = \frac{M-m}{M-1} \cdot \frac{\sigma_b^2}{m} + \frac{\sigma_w^2 + \sigma_p^2 + \sigma_M^2}{mn_w}$$

When the test sample has been analyzed l times, the overall precision
$$\left(\frac{\beta}{2}\right)^2 = \frac{M-m}{M-1} \cdot \frac{\sigma_b^2}{m} + \frac{\sigma_w^2 + \sigma_p^2 + \sigma_M^2/l}{mn_w}$$

- (2) Collect the increments for each primary sampling unit to produce the sub sample, and prepare the test sample therefrom (see Fig. 7).

Fig. 7

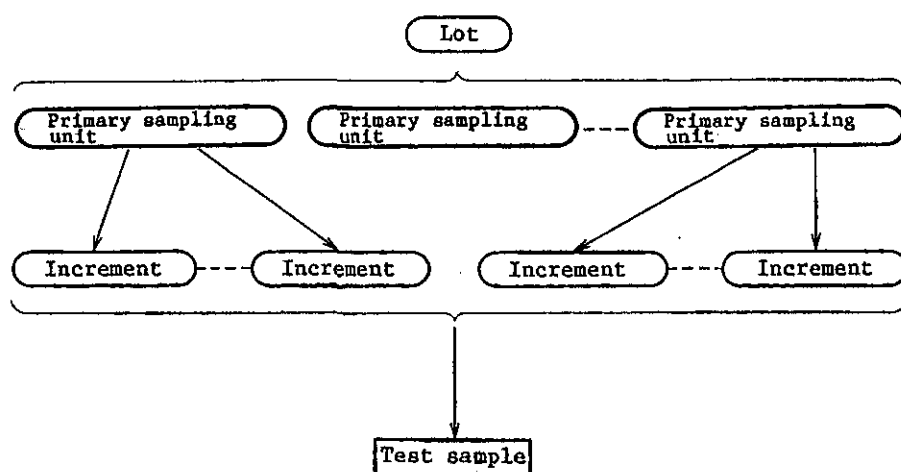


When the test sample has been analyzed one time, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{M-m}{M-1} \cdot \frac{\sigma_b^2}{m} + \frac{\sigma_w^2}{mn_w} + \frac{\sigma_P^2 + \sigma_M^2}{k}$

When the test sample has been analyzed l times, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{M-m}{M-1} \cdot \frac{\sigma_b^2}{m} + \frac{\sigma_w^2}{mn_w} + \frac{\sigma_P^2 + \sigma_M^2/l}{k}$

(3) Prepare the test sample from the gross sample (see Fig. 8).

Fig. 8



When the test sample has been analyzed one time, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{M-m}{M-1} \cdot \frac{\sigma_b^2}{m} + \frac{\sigma_w^2}{mn_w} + \sigma_P^2 + \sigma_M^2$

When the test sample has been analyzed l times, the overall precision $\left(\frac{\beta}{2}\right)^2 = \frac{M-m}{M-1} \cdot \frac{\sigma_b^2}{m} + \frac{\sigma_w^2}{mn_w} + \sigma_P^2 + \frac{\sigma_M^2}{l}$

5.8 Combined use and multiple use of sample The sample can be used in combination for particle size, for water content and for constituent, as required. When the sample is used in combination for these purposes, the sample meeting the characteristics of a largest number of increments shall be used. However, when the mass of sample is insufficient for the sample preparation and measurement of each characteristic to be in combination, the increment shall be increased up to not less than the required quantity by making the size of increment larger or by increasing the number of increments to be taken.

Further, the sample for particle size test can be used in duplication for the constituent.

Remarks: The mutual multiple uses of the sample for particle size and sample for moisture content, the sample for moisture content and sample for constituent, these samples and sample for other characteristics, and other than those, the samples for each kind of characteristics shall be specified in each standard.

5.9 Belt sampling

5.9.1 Taking place of increment When the lot is moved by a belt conveyor, the increment shall be taken from the specified place on the belt or its chute.

5.9.2 Number of increments The minimum required number of increments taken from one lot shall be determined in accordance with 5.6.3.

5.9.3 Taking method of increments The taking method of increments shall be as follows:

- (1) Carry out the systematic sampling by the random start.

The sampling interval of increments shall be determined in accordance with Remarks 2. of 5.6.3.

- (2) When taking the increments after stopping the belt conveyor, take the increment of quantity of not less than the specified size at the specified place of conveyor. In this case, take the whole quantity along the moving direction of belt ranging over the full flow width of belt, which is not less than three times the maximum particle size and not less than 30 mm in width at least, as one increment.
- (3) Take the increments ranging over the full flow width of belt conveyor by the sampler on the belt or at its chute when they are gathered from the belt conveyor under operation.

Remarks: When taking in the full flow width is impossible, after confirming that it is free from bias, select a place at random in the full flow width at every time when the increments are taken on the belt conveyor or its chute and take the increments by the specified sampler.

5.10 Freight car sampling

5.10.1 Taking place of increment Take the increment from the new surface of cargo, from the freight car under material handling, which as appeared during the material handling.

Remarks 1. The increment may be taken from the device for material handling and the like during handling. For hopper car, it should preferably be avoided taking the increments during the unloading because of the sampling accompanied by the possibilities of inclusion of bias and the danger due to the work.

2. It shall be avoided, as a rule, taking the increments only from the surface of cargo which is loaded as it is in the freight car.

5.10.2 Number of increments The minimum required number of increments to be taken from one lot, number of freight cars to be selected out and number of increments to be taken from one freight car shall be determined in accordance with 5.6.3.

5.10.3 Method of taking increments The method for taking increments shall be as follows:

- (1) The freight car to take the increments shall be selected as the first stage of the two-stage sampling and its selecting method shall be in accordance with the random sampling or the systematic sampling. When the number of freight cars is small, the increments shall be taken from all of freight cars in accordance with the stratified proportional sampling.
- (2) In order to take the increments from the freight car, determine the taking place at random and take the increments from the new surface of cargo which has appeared during material handling.
- (3) In order to take several increments from the freight car under material handling by the systematic sampling method, take the increments from the places determined at random on the new surface which appears at every fixed quantitative interval of material handling.

5.11 Container sampling

5.11.1 Taking place of increments When the lot is in the bags, drum cans or other containers, take the increments from those containers.

5.11.2 Number of increments The minimum required number of increments to be taken from one lot, number of containers to be selected and number of increments to be taken from one container shall be determined in accordance with 5.6.3.

5.11.3 Methods of taking increments

- (1) When the number of containers is small, the increments shall be taken from each container by the stratified sampling. When the number of containers is especially small, all the quantity of contents in all containers may be taken as the sample.
- (2) When the number of containers is large, a container shall be selected as the first stage of two-stage sampling and the increment shall be taken from it. The selecting method of container for the first stage shall be at random or in accordance with the systematic sampling. When the capacity of container is small, each container selected at the first stage may be taken as the increment.
- (3) When the increment is taken from the container, it shall be in accordance with either one of the following:
 - (a) Take the increment from a random place after emptying the container of contents at a place free from foreign substances.
 - (b) Take the increment from a place selected at random from the container.

5.12 Ship hold sampling

5.12.1 Place of taking increments During the stevedoring of lot by means of a material handling device such as grab bucket, straw basket, etc., take the increments out of the handling device, or from the place directly after Stevedoring in the ship hold.

5.12.2 Number of increments The minimum required number of increments to be taken from one lot shall be determined in accordance with 5.6.3.

5.12.3 Method of taking increments Determine the taking interval in accordance with 5.6.3 Remarks 2, and take the increments by the systematic sampling.

5.13 Sampling for particle size

5.13.1 Place of taking increments The place of taking increments shall be in accordance with 5.9 to 5.12. However, if there is the crushing equipment in the way of lot movement, the sampling shall be carried out before crushing at the time of receiving and after crushing at the time of shipping, as a rule.

5.13.2 Number of increments The minimum required number of increments to be taken from one lot shall be determined in accordance with 5.6.3.

5.13.3 Method of taking increments The method of taking increments shall be in accordance with 5.9 to 5.12.

5.14 Sampling for moisture content

5.14.1 Place of taking increment The place of taking increments shall be in accordance with 5.9 to 5.12. However, the sample for moisture content shall be taken at a place as near as possible, before or after the place where the mass of lot is weighed because the moisture content is easy to change.

5.14.2 Number of increments The minimum required number of increments to be taken from one lot shall be determined in accordance with 5.6.3.

5.14.3 Method of taking increments The method of taking increments shall be in accordance with 5.9 to 5.12. When there are differences in moisture contents in part of lot, for example, among the upper part, middle part and lower part of the freight car, the stratified sampling shall be carried out.

Further, when the part of ship bottom and the like are particularly watery, it should be preferable to carry out the stratified sampling for the preparation of increments.

Remarks: Cares shall be taken on the container of sample, the place for storage and the treatment method of sample so that the moisture content will not vary by evaporation, wetting with rain, etc. in the course from the sampling to the measurement.

6. Method of sample preparation

6.1 Preparation of sample The outline of the method of sample preparation shall be as follows (see Fig. 9 and Fig. 10):

- (1) Crush and reduce the sample as required to prepare the test sample.
- (2) When the sample is extremely wet, dry it preparatively.
- (3) The reduction shall be carried out by an appropriate reduction method in accordance with a specified reduction standard.

The sample for constituent is reduced by using the sample of not more than 31.5 mm particle size of which all quantity have passed through the test sieve, and its standard shall be in accordance with 6.5.2 and 6.5.6.

The reduction standard of the sample for moisture content shall be specified in each standard.

The reduction standard of the sample for particle size shall be in accordance with 6.8.

The reduction standard of the sample for physical characteristics and the sample for other characteristics shall be specified in each standard.

- (4) The number of times to transfer the samples for particle size and for physical characteristics and the fall to transfer shall be minimized to avoid crushing of sample overall stages of work of sampling, sample preparation and test sample.
- (5) It is desired that the sample preparation apparatus be integral with the usual sampling apparatus, but when an appropriate place cannot be found near the sampling apparatus or when the increments taken from several places are treated by one sample preparation apparatus, it may be separated from the sampling apparatus.

The sample preparation apparatus, which has been arranged in one united body with the sampling apparatus and carried out the treatment for every increment, shall be capable of treating the increments in shorter time than the time interval to take the increments.

Remarks 1. When the sample after the particle size test is used in duplication for the constituent test, the duplicate uses shall be carried out as follows, for example: Carry out the particle size test by using the whole quantity of sample at first, and collect all of the samples divided by particle size next. If possible, after mixing them sufficiently, crush and reduce them to prepare the sample for constituent test.

2. The preparation of sample including a great amount of particles impossible to crush such as metals shall be specified in each standard.

- (6) Cares shall be taken so that during preparation of sample, a part of sample can not be dispersed, and the dusts from surroundings and other foreign substances can not be mixed.

Fig. 9. When test sample for constituent test is prepared after particle size test

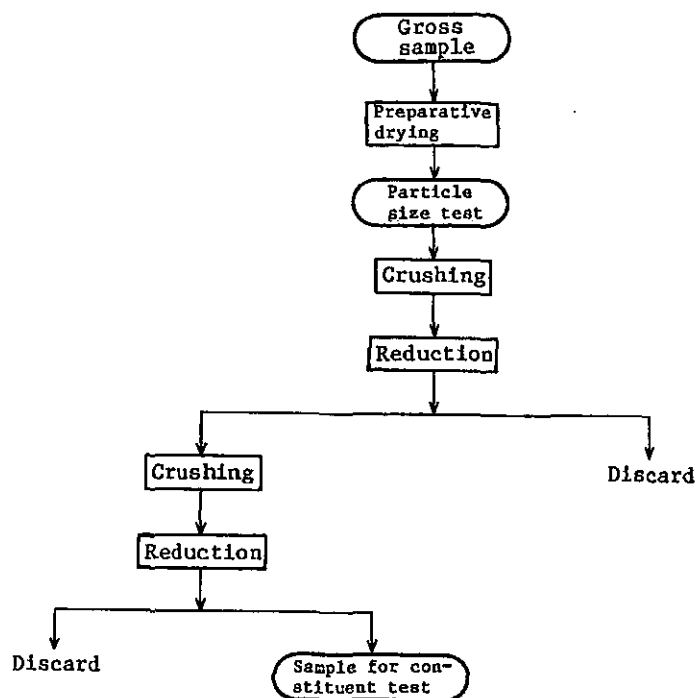
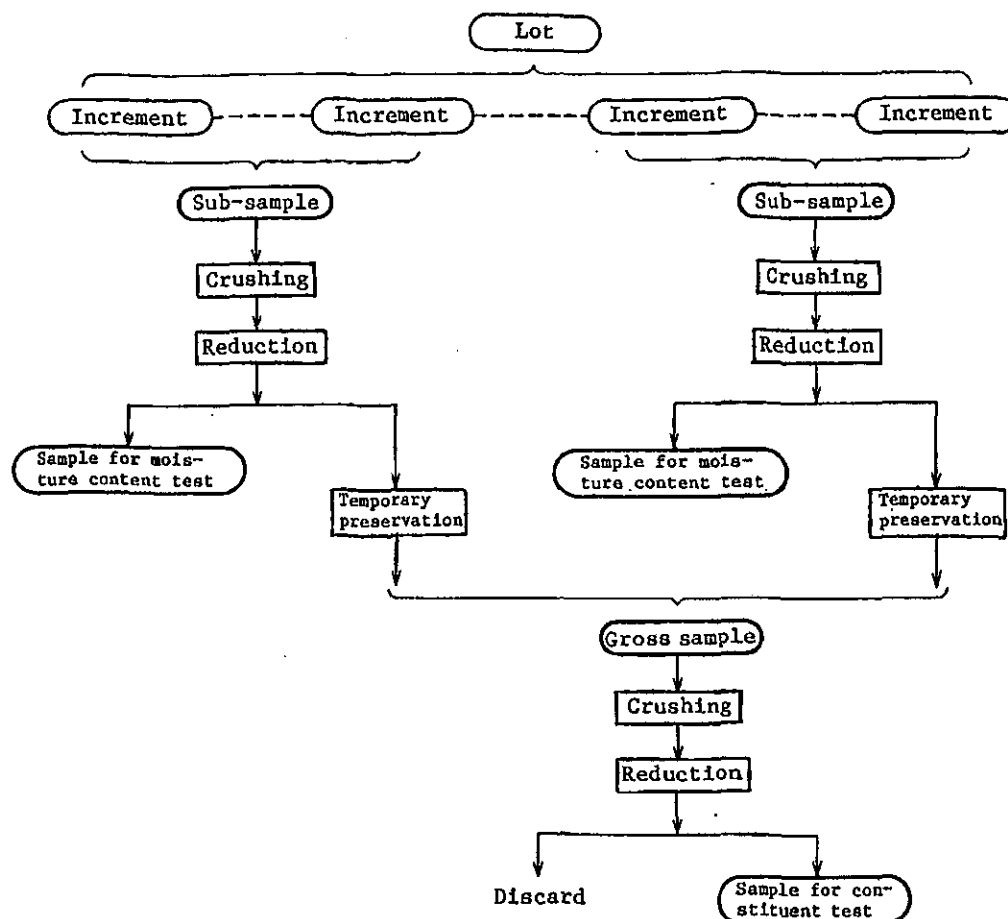


Fig. 10. When sample for moisture content test and sample for constituent test are prepared



6.2 Precision of preparation The precision of preparation of main characteristics of sample shall be CV 0.05 % to 5 %.

6.3 Predrying of samples

6.3.1 Summary When the screening, crushing and reduction of samples are impracticable because of the excessive moisture of the samples, dry them to such a degree that the sample can be processed using sieve, crusher and reduction apparatus under the specified conditions (temperature, period of time, etc.) in the individual standard by the room drying or the drying apparatus, etc. In the case of adding to the moisture content (%) obtained in 7., calculate the predrying moisture content (%).

6.3.2 Sample The sample to be used shall be the whole quantity of the sample immediately after taking.

6.3.3 Apparatus

- (1) Drying apparatus Use the apparatus having a capacity in which the required quantity is accommodated and capable of adjusting to not higher temperature than that at which the sample does not deteriorate.

- (2) Balance The reciprocal sensibility of balance shall meet the required precision.

6.3.4 Operation The operation shall be carried out as follows:

- (1) Weigh the mass of the whole quantity of sample.
- (2) Level the sample so that the thickness of sample shall become uniform and dry it under the specified conditions (temperature, period of time, etc.).
- (3) Weigh the mass immediately after completion of drying.

6.3.5 Calculation Calculate the predrying moisture content A (%) to two places of decimals, as a rule, according to the following formula:

$$\text{Predrying moisture content } A (\%) = \frac{W - W_a}{W} \times 100$$

where, W : total mass of sample before drying

W_a : total mass of sample after drying

Remarks 1. Take care to avoid the loss of micro-powder by drying.

2. Take care on preservation after predrying for the sample having a large hygroscopicity.

6.4 Crushing of sample Crush the whole quantity of sample by an appropriate crusher up to the particle size that allows the whole quantity of sample to pass through the specified sieve.

Remarks: When the materials impossible to crush are mixed, for example, obtain the mass ratio independent of that part previously, produce the test sample separately to obtain each measured value and further, obtain the weighted mean taking the mass ratio which has been obtained previously as weight.

- (1) Selection of crusher Select the crusher having the type and capacity suited for the particle size of sample to be ground, particle size of sample after crushing and physical properties⁽²⁾ of sample.

Note ⁽²⁾ The physical properties are defined as hardness, rigidity, specific gravity, water content, stickiness, and others.

- (2) Cleaning The inside of crusher shall be cleaned before the sample is supplied.
- (3) Preliminary cleaning When crushing the different sample from the sample which has been crushed at the last time, it should be preferable to pass an appropriate quantity of particulate materials taken from the lot previously through.

- (4) Taking out of sample Take care to take out the whole sample remaining in the crusher.
- (5) Prevention of deterioration Take care to avoid the deterioration of sample due to the type of crusher or the heat generated by the long hour's continuous operation and the like.

6.5 Reduction of sample

6.5.1 Classification of reduction method The reduction method shall be classified into the following 5 methods:

- (1) Increment reduction method
- (2) Method by riffle sampler
- (3) Conical quartering method
- (4) Alternate shovelling method
- (5) Method by reductor

6.5.2 Increment reduction method

- (1) The size of shovel for the increment reduction and the thickness of the spread sample shall be as given in Table 3 according to the particle size of the sample.

Table 3. Particle size of sample size of shovel for increment reduction and thickness of spread sample

Particle sizes of whole-through sieve	Number of shovel	Thickness of spread sample mm
31.5 mm max.	30 D	40 to 50
22.4 mm max.	20 D	35 to 45
16.0 mm max.	15 D	30 to 40
10.0 mm max.	10 D	25 to 35
5.00 mm max.	5 D	20 to 30
2.80 mm max.	3 D	15 to 25
1.00 mm max.	1 D	10 to 20
250 μ m max.	0.25 D	5 to 10

Remarks 1. The size of shovel shall be as given in Attached Fig. 1.

2. In the case of increment reduction, the particulate materials of unit quantity which has been taken from the sample is called an increment.

(2) The number of increments shall be in accordance with the following standards:

(a) In the case of gross sample 20 increments or over

(b) In the case of sub-sample 10 increments or over

(c) In the case of each increment 4 increments or over

(3) When the increment reduction of gross sample is carried by hand, the procedures of reduction shall be carried out as follows (see Attached Fig. 2):

(Procedure 1) Spread the sample in square with thickness of Table 3 and uniformly on the flat board.

(Procedure 2) Divide this into 5 equal parts lengthwise and into 4 equal parts breadthwise.

(Procedure 3) Collect one increment from each division to be 20 increments in total by the use of the shovel for increment reduction (by selecting the place at random in each division) and take these as the sample after reduction. At this time, the increment shall be taken by thrusting the shovel to the bottom of sample stratum.

Remarks 1. When the quantity of sample after reduction larger than the quantity of sample taken by the above-stated method is required, enlarge the size of increment or increase the number of increments. When the number of increments are increased, the number of divisions is increased or the number of increments to be taken from one division is increased, and the number of increments to be taken from each division, however, shall be fixed.

2. The number of divisions for the reduction of each sub-sample shall be not less than 10 divisions and one increment shall be taken from each division.

3. The number of divisions for the reduction of each increment shall be not less than 4 divisions and one increment shall be taken from each division.

4. In the case of increment reduction, depending on its object, it should be preferable to take the increments with applying the padding board.

6.5.3 Method by use of riffle sampler

- (1) Depending on the particle size of sample, the riffle sampler with number shown in Table 4 shall be selected, as a rule.

Table 4. Particle size and classification of riffle sampler

Particle size of whole- through sieve mm		Classifi- cation of riffle sampler	Width of riffle mm
Over	Max.		
22.4	31.5	No. 60	60 ± 1
16.0	22.4	No. 50	50 ± 1
10.0	16.0	No. 30	30 ± 1
5.00	10.0	No. 20	20 ± 1
2.80	5.00	No. 10	10 ± 0.5
	2.80	No. 6	6 ± 0.5

- (2) The riffle sampler shall be as follows:
- (a) The Attached Fig. 3 gives an example of the size and structure of each part of riffle sampler.
 - (b) The angles θ of upper part and lower part between both inclined planes shall not be larger than 60 degrees.
 - (c) The number of riffles shall be an even number and be not less than 16, as a rule. However, the number of riffles of sampler with number of 60, 50 and 30 shall be not less than 12.
 - (d) The sample receiver shall be so constructed that the micro powders do not disperse from the outlet of riffle sampler.
 - (e) The inside of riffle samplers shall be smooth and free from rust.
- (3) The sample shall be put in the mineral-feed container and fallen uniformly on the body of riffle sampler to be divided into 2. Either one of the divided samples shall be selected at random and be taken as the sample after reduction.

Remarks: In using the riffle sampler, cares shall be taken so that the riffles shall not be packed.

- (4) The standard of sample reduction shall be in accordance with 6.5.7.

6.5.4 Conical quartering method

- (1) The procedures when reducing the sample in accordance with the conical quartering by hand shall be as follows (see Attached Fig. 4):
 - (Procedure 1) Heap up the crushed sample into a cone-form on the plane.
 - (Procedure 2) Flatten the circular cone of (Procedure 1) and repeat the operation of (Procedure 1) at other places once or twice.
 - (Procedure 3) Flatten the cone with pressing down vertically from the top and quarter the sample in a fan-form.
 - (Procedure 4) Take a pair of diagonal parts and join them to be taken as the sample after reduction.
- (2) The standard of sample reduction shall be in accordance with 6.5.7.

Remarks: When the particle size of sample to be reduced is large, pay attention to the segregation easily to be produced in the conical quartering method.

6.5.5 Alternate shovelling method The procedure of the alternate shovelling method shall be as given in Attached Fig. 5.

Further, the standard for reduction shall be in accordance with 6.5.7.

6.5.6 Method by reductors It shall be confirmed preliminarily that the reductor is sufficient in precision and is free from bias.

Further, the standard of reduction shall be in accordance with 6.5.7.

Remarks: The apparatus used for the sample reduction shall be cleaned sufficiently before use. When the reduction of sample different from the sample which has been reduced at the previous time is carried out, it should be preferable to clean by passing through an appropriate quantity of particulate materials which has been separately taken from its lot preliminarily.

6.5.7 Standard of sample reduction The relation between the particle size that allows the whole quantity of sample to pass through and the mass of sample after reduction shall be in accordance with Table 5, as a rule. However, the relation in the case of increment reduction shall be in accordance with 6.5.2.

Table 5. Particle size and standard of reduction

Particle size of whole-through sieve	Mass of sample after reduction kg		
	In the case of gross sample	In the case of sub-sample	In the case of each increment
31.5 mm max.	280 min.	140 min.	80 min.
22.4 mm max.	140 min.	70 min.	40 min.
16.0 mm max.	70 min.	35 min.	20 min.
10.0 mm max.	35 min.	17 min.	10 min.
5.00 mm max.	8 min.	4 min.	2.5 min.
2.80 mm max.	2 min.	1 min.	0.6 min.
1.00 mm max.	0.5 min.	0.5 min.	0.3 min.
420 μ m max.	0.1 min.	0.1 min.	0.1 min.
250 μ m max.	0.05 min.	0.05 min.	0.05 min.

Remarks 1. The mass of sample in this table is the mass in the case where the bulk density of sample is 1.

2. The sample shall not be reduced to not more than the mass of sample shown in this Table.

6.6 Preparation of constituent test sample The preparation of constituent test sample shall be as follows:

- (1) Crush and reduce the increment, sub-sample or gross sample. When the specified particle size has been obtained, prepare the specified number of samples with specified mass.
- (2) Put this sample in a container to seal it. Take this sealed sample as the sample for constituent test.
- (3) The number of the samples for constituent test shall be not less than 4 samples for seller, buyer, umpire and preservation, etc.

6.7 Preparation of moisture content test sample The preparation of moisture content test sample shall be as follows:

- (1) Crush and reduce the increment, sub-sample or gross sample and produce the sample with the specified particle size and specified mass for the moisture content test sample.

- (2) The number of moisture content test samples shall be not less than one in the cases of increment and sub-sample and be not less than 2 in the case of gross sample, as a rule.
- (3) In the following cases, the moisture content test sample should preferably be prepared for each increment or each sub-sample.
 - (a) When the sample is watery (%)
 - (b) When the quality variation of moisture content (%) is remarkably large by rainfall, snowfall and the like.
 - (c) When a long time is required for the sampling of one lot.
- (4) Because the sample for moisture content is easy to yield bias by the change of moisture content at the time of crushing, reduction, and mixing, etc., the predrying of sample should preferably be carried out immediately after taking as far as possible.

Further, it is necessary to treat the sample for moisture content, with paying attention to the handling place, the equipment, the methods of crushing and reduction, and to lessen the number of times of crushing and reduction as far as possible.

6.8 Preparation of particle size test sample The preparation of particle size test sample shall be as follows:

- (1) Prepare the particle size test sample from the sample for particle size without crushing.
- (2) By the use of whole quantity of the sample or by the reduction method of 6.5.1, reduce the sample in accordance with the standard of reduction and produce the particle size test sample.
- (3) The standard of reduction of (2) shall be according to the following formulae:

- (a) In the case of gross sample

$$\begin{cases} M_s = \frac{Q}{\beta_{PM}^2} \times \frac{S}{5000} \\ Q = 2.5 \times 10^{-6} G (100 - G) L^3 \sqrt{(Y/L)} \end{cases}$$

where, M_s : mass of the particle size test sample (kg)

β_{PM} : required precision of reduction test

S : apparent density (kg/m³)

G : percentage (%) containing the specified particle size division

L : particle size that allows the whole quantity of sample to pass through (mm)

Y : specified particle size (mm)

(b) In the case of sub-sample

$$\frac{M_s}{k} \text{ (kg)}$$

where, k : number of sub-samples constituting the gross sample

M_s : mass of test sample in the case of gross sample (kg)

(c) In the case of increment

$$\frac{M_s}{n} \text{ (kg)}$$

where, n : number of increments constituting the gross sample

M_s : mass of test sample in the case of gross sample (kg)

7. Moisture content determining method

7.1 Summary Dry the moisture test sample to become a constant weight at the specified temperature in the thermostatic oven. Calculate the moisture content (mass %) of each sample from the loss on drying and determine the moisture content (mass %) of lot in accordance with 7.7.

7.2 Sample Use the moisture content test sample prepared in accordance with 6.7.

7.3 Apparatus The following items shall be previously determined in each standard:

(1) Drying basin [class, form, material and base area⁽³⁾]

Note ⁽³⁾ It should be preferable to determine the base area so that the thickness of sample shall become not more than 30 mm.

(2) Thermostatic oven (range of temperature capable of using, temperature reversion time, tolerance of holding temperature, etc.)

(3) Balance (reciprocal sensibility, required precision)

7.4 Operation The operation shall be as follows:

(1) Transfer the moisture content test sample prepared in accordance with 6.7 into the drying basin of known mass (W_1) and level the sample so that its thickness shall become uniform, and weigh the whole mass (W_2).

(2) Put the moisture content test sample in the thermostatic oven adjusted to the specified temperature preliminarily and dry it. At this time, specify the drying conditions⁽⁴⁾ suitable for the particulate materials susceptible to influence of oxidation or the like.

Note ⁽⁴⁾ The drying condition is defined as heat source of drying, ventilating condition, atmosphere, etc.

- (3) Continue to dry the sample until it becomes a constant weight. The constant weight means that the loss on drying becomes not more than a fixed percentage (or a fixed mass) per a fixed time.

Remarks 1. When the required time to attain a constant weight could be obtained by carrying out the experiment for each brand, it may be considered that the sample attained a constant weight in that required time.

2. When the rate of loss on drying does not become not more than a fixed value, the drying condition shall be decided as agreed upon between the parties concerned with delivery.

- (4) Weigh the whole mass (W_3) of sample while it is heated immediately after completion of drying.

7.5 Calculation Calculate the moisture content B (%) according to the following formula and obtain it to two places of decimals, as a rule.

$$B = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

7.6 Tolerance It should be preferable to previously fix the tolerance for the difference of moisture contents B (%) when the test of 2 samples has been carried out.

Remarks: In the case of one test sample, the test method shall be controlled sufficiently and be checked at times because the confirmation of tolerance is impossible.

7.7 Determined values The determination value shall be as follows:

(1) When predrying is not carried out

- (a) In the case of gross sample, find the arithmetical average of 2 values B (%) of rate of loss on heat drying and take that value which has been rounded off to the first place of decimal in accordance with JIS Z 8401 as the moisture content (%) of lot.
- (b) When the loss on heat drying has been obtained for each sub-sample, obtain the weighted average taking the stratum of which each test value is representative, the mass of primary sampling unit or the number of increments composing sample as weight and take this value as moisture content (%) of lot.
- (c) When the rate of loss on heat drying has been obtained for each increment, find the arithmetical average of each test value and take it as moisture content (%) of lot.

- (2) When predrying has been carried out Add the predrying moisture content A (%) obtained in 6.3 to the moisture content C (%) to be obtained from (1)(a) to (c) by the following formula, round off the number to the first place of decimal and take it the moisture content D (%) of lot. However, in this case, C (%) shall be the value calculated to two places of decimals.

$$D = A + \frac{100 - A}{100} \times C$$