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Monolithic (unshaped) refractory products —

Part 2: **Sampling for testing**

Produits réfractaires monolithiques (non façonnés) —

Partie 2: Échantillonnage

Reference number
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Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1927-2 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 1927 consists of the following parts, under the general title *Monolithic (unshaped) refractory products*:

— *Part 1: Introduction and classification*

— *Part 2: Sampling for testing*

— *Part 3: Characterization as received*

— *Part 4: Determination of consistency of castables*

— *Part 5: Preparation and treatment of test pieces*

— *Part 6: Measurement of physical properties*

— *Part 7: Tests on pre-formed shapes*

— *Part 8: Determination of complementary properties*

Monolithic (unshaped) refractory materials —

Part 2:
Sampling for testing

1 Scope

This part of ISO 1927 gives guidance on the sampling of monolithic (unshaped) refractory materials for the purpose of inspection and testing for quality and general information on the reduction and treatment of samples prior to testing. It covers all materials formulated as monolithic refractory materials.

NOTE The term "monolithic" is the preferred term, whereas "unshaped" is commonly used in Europe. For the purposes of this part of ISO 1927, the terms "monolithic" and "unshaped" can be used interchangeably.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5022, *Shaped refractory products – Sampling and acceptance testing*

ISO 8656-1, *Refractory products — Sampling of raw materials and unshaped products — Part 1: Sampling scheme*

ISO 10725, *Acceptance sampling plans and procedures for the inspection of bulk materials*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
batch
quantity of material from which a sample is to be achieved for testing to determine the quality of the material

NOTE A batch consists of material characterized as being of the same type, composition, grading and which, as far as practical, has been manufactured under the same conditions.

3.2
consignment
quantity of material supplied at one time

NOTE A consignment may consist of one or more batches or parts of batches.

3.3
unit package
packaged part of a batch which can be a bag or a big bag (castables, gunning material, ramming mixes), a carton (plastics), wrapped block (tap-hole mixes), a drum or a can (injection material, refractory grout)

NOTE A pallet is not a unit package.

3.4
increment
quantity of material taken at one time from a larger quantity

3.4.1
elementary increment
quantity of material taken at one time from a unit package

NOTE This operation repeated a number of times will constitute a package increment after mixing.

3.4.2
package increment
increment that is representative of the unit package

NOTE 1 It can be the unit package itself or the result of mixing a certain number of elementary increments.

NOTE 2 The mass and number of elementary increments which are necessary to form the package increment are defined in accordance with ISO 8656-1.

3.4.3
laboratory increment
package increment that has been reduced by an approved method

3.4.4
test-piece increment
test bars or cylinders obtained by shaping the laboratory increment necessary to carry out several physical tests

EXAMPLE Castable test bars for cold modulus of rupture testing.

3.5
sample
one or more increments taken from a batch which are to be used to provide information on the batch and to allow a decision concerning the quality of the batch

3.5.1
batch sample
set of package increments representative of the batch

NOTE The number of package increments which are to form the batch sample should be agreed by the principal parties involved. ISO 5022 or other sampling schemes may be used.

3.5.2
laboratory sample
set of laboratory increments

NOTE The number of laboratory increments is the same as the number of package increments.

3.5.3
test-piece sample
set of test-piece increments

NOTE The number of test increments can be higher than the number of laboratory increments and is governed by international test standards.

4 Sampling scheme

4.1 General principles

4.1.1 It is essential that the adoption of a particular sampling scheme be agreed by the principal parties and that a detailed sampling plan be documented and made available to those responsible for the taking and testing of the increments. The basic framework of a scheme requires decisions and documentation on the following:

a) the constitution and description of the total material to be sampled;

b) the identification of batches and quantities which make up the total material;

c) the type of packaging and mass content for each type of material;

d) the parties responsible for sampling and testing who may be third parties;

e) the location, timing and method of sampling;

f) the level of sampling, population of increments;

g) the properties to be measured;

h) the methods of test (reference to International Standard number);

i) the criteria for assessing values of measured properties for deciding batch quality.

4.1.2 In all cases during sampling, increment division, preparation and storage of the increments, care shall be taken to protect against any changes in the properties to be tested.

4.1.3 Sampling shall be performed under the supervision of a person having adequate experience on sampling. The sampler shall be approved either by the interested parties or by the appropriate body or bodies. The sampler shall be informed of the aim of the sampling.

4.1.4 When individual batches are identified, agreement should be made between the parties on whether, or to what degree, a large batch should be subdivided into smaller batches. This may be undertaken to avoid the possibility of the whole of a large batch being rejected because of a problem with a proportion of it.

4.1.5 When a sample is required for third-party certification of factory production control as the product is being made, the sample shall be achieved by the same method that the producer uses to obtain a sample for production control purposes.

4.1.6 Where required, the consignment may be subdivided into individual test batches, for example, if it is clear that the consignment consists of various batches or should be treated in separate partial quantities.

4.1.7 The sampling framework is presented in Figures A.1 to A.3.

4.2 Procurement of the batch sample

4.2.1 Method

Identify the test batch, i.e. of the consignment or part of the consignment to be sampled (nature of the product, mass, transport conditions, etc.).

Identify the unit package. The average mass (m) of this unit package shall be known.

Obtain the number of package increments which form the batch sample as agreed between parties. The sampling scheme of ISO 5022 can be agreed by the parties if the unit packages, considered as equivalent to shaped pieces, weigh less than 35 kg.

Randomly, select this number of the unit packages from the batch.

Proceed to sample the selected unit packages, if their mass is more than 35 kg. This means obtain a package increment of each of these unit packages. In this case, ISO 8656-1 shall be applied to determine the mass and the number of elementary increments which are necessary to obtain the package increment:

a) Estimate the maximum grain size of the material. This estimation is important because the minimum mass of the elementary increment shall be determined, taking into account the maximum grain size of the material in order to avoid systematic errors during sampling (see Table 1).

Table 1 — Minimum mass of elementary increment depending on the maximum grain size

Maximum grain size mm	Minimum mass of elementary increment
10	500 g
3	200 g
1	50 g

NOTE 1 The masses of the elementary increments relate to a bulk density greater than 1 g/cm^3. For lower bulk densities, the mass of the elementary increment can be determined by multiplying the numerical value in Table 1 by the bulk density of the material.

NOTE 2 Special agreements should be made in the case of very lumpy products. In the case of pre-ground or pre-homogenized material, the minimum mass of the elementary increment can be determined, not from the grain size of the coarsest aggregate, but from the maximum size of the grains of the material before aggregating.

NOTE 3 The actual elementary increment masses should depend on the sampling equipment and the tests to be performed. This is the case for monolithic products if the physico-mechanical properties of test pieces taken from these products are to be determined.

b) Determine the mass of the elementary increment in accordance with ISO 8656-1, taking into account the minimum quantities required for the tests which are to be performed.

c) Classify the test batch in a quality variation class because the number of increments taken for testing from a batch shall be determined taking into account the deviations in the properties of the material.

The mean value and the standard deviation of a given property and type of monolithic product, designated respectively by μ and σ, define the coefficient of variation $C_V = 100\ \sigma/\mu$ of this property, expressed in practice as a percentage.

The values of the coefficient of variation are divided into three classes:

— $0 < C_V < 5$ %, small variation, class 1;

— 5 % $< C_V <$ 15 %, medium variation, class 2;

— 15 % $< C_V <$ 30 %, large variation, class 3.

The property which presents the highest value of the coefficient of variation is used to determine the number of elementary increments.

If the coefficient of variation is unknown or if it is greater than 30, use the class 3 values (large variations).

The number of elementary increments to be taken is determined in accordance with Table 2.

Table 2 — Minimum number n of elementary increments

Class of deviations of the property %			Mass m of unit packages
Class 1	Class 2	Class 3	
$C_V < 5$	$5 < C_V < 15$	$15 < C_V < 30$	10^3 kg
4	4	8	$m < 1$ or $= 1$
4	6	12	$1 < m < 5$

The value of $\boldsymbol{n}$ given in Table 2 is a minimum and generally the actual number should not be less than that specified in the table.

Package increment mass is equal to the minimum mass of elementary increment multiplied by the minimum number of increments.

If the fixed mass for the package increment is less than the mass required for the various tests, increase either the mass of the elementary increment or the number of the increments so as to obtain a sufficient amount.

NOTE For unit packages exceeding 5 000 kg, the sampling may be agreed between the principal parties or based on ISO 10725.

4.2.2 Apparatus to take increments

General requirements and suggested designs for apparatus are given in ISO 8656-1. It is possible to use a sampling box, sampling tube, sampling spear or scoop.

— In all cases, the width of the opening shall be not less than 10 mm and not less than three times the upper aggregate size.

— The length of the sampling box opening shall be larger than the depth of the stream of material to be sampled. Its depth shall be such that no particles are lost by rebounding out of the box.

— The length of the sampling tube or spear shall be 1 000 mm to 2 000 mm.

4.2.3 Method to take increments

4.2.3.1 Sampling from a big bag

Sampling from a big bag is very difficult because:

a) the mass of this large amount of product prevents the use of the sampling tube or spear;

b) during transportation, segregation can occur.

Consequently, the optimum conditions of sampling accuracy are obtained only if the elementary increments are taken from the material in movement.

An elementary increment shall be taken by passing the sampling box through the discharge stream in a uniform movement, making sure that the complete cross-section of the stream of materials is intercepted. Shovels or scoops shall not be used for sampling of moving materials.

In the case of certain prepared monolithic products (for example, castables) where it is known that the material is delivered in unblended form or where it is evident that the material has segregated during transportation, it is necessary to take the elementary increment(s) after mixing the full big bag. This sampling method is expensive as it results in large quantities of materials being handled.

As soon as elementary increments are obtained, they shall be mixed to form a package increment.

4.2.3.2 Sampling from cans, wrapped blocks (mass limited to 35 kg)

These unit packages are considered as equivalent to shaped pieces. According to 4.2.1 c), remove for inspection a number of containers, taking them at random from the lot.

4.3 Size reduction of the increments

4.3.1 General

The procedures described in 4.3.2 and 4.3.4 shall preferably be used for the preparation of the laboratory sample from the batch sample. The method described in 4.3.3 may be used when other methods are not suitable or the devices are not available. All the methods may also be used in combination, for example, by using quartering for the first few reduction stages followed by riffling.

For mixing, and other operations that require a work surface, use a clean, flat, hard surface such as a sampling tray or a glass plate.

The minimum mass of each laboratory increment is that necessary to carry out all the tests plus an amount as a reserve, if required.

Wrapped plastic blocks containing a binding liquid shall be reduced by first removing the outer surface to a depth of 10 mm.

Items of apparatus for reduction are described in ISO 8656-1. Riffle boxes or mechanical dividers are both used.

If big bag sampling is requested by the customer, the sampling shall be performed by experienced personnel. The exact sampling procedure shall be agreed between the parties.

4.3.2 Reduction of a package increment using a riffle box

Put the package increment into one of the riffle box receptacles and place the other two in position. Pour the material from the long side of the riffle box down the centre-line of the riffle box. Discard the material that falls into one of the other two receptacles. Repeat as many times as needed to reach the required size of laboratory increment.

If the package increment is too large to go into the riffle box receptacle, divide the package increment up into sub-increments that are small enough, reduce each by the same number of riffling stages, then combine the reduced sub-increments to obtain a package increment.

The number of slots of the riffle box shall be even and not less than eight. The width of the slots shall be at least twice the upper aggregate size to avoid bridging.

4.3.3 Reduction of a package increment by quartering

Place each package increment on the work surface.

Thoroughly mix the package increment by heaping it up to form a cone, and turning it over with the shovel to form a new cone. Repeat this operation three times. When forming the cones, deposit each shovelful on the peak of the new cone in such a way that the material runs down all sides of the cone and is evenly distributed so that the different sizes become well-mixed.

Flatten the third cone, by inserting the shovel repeatedly into the peak of the cone, to form a flat heap which has a uniform thickness and diameter. Keep the shovel vertical, and lift it clear of the cone after each insertion.

Quarter the flat heap along two diagonals intersecting at right angles. Discard one pair of opposite quarters and shovel the remaining quantity into a stockpile.

Repeat the process of mixing and quartering until the required size of laboratory increment is obtained.

Repeat the total process for each package increment.

The set of laboratory increment corresponds to the laboratory sample.

4.3.4 Increment reduction using a mechanical divider

Increment reduction can be achieved in different ways, i.e. by division of an increment into equal parts or by extraction of a representative part of the increment. Before starting the procedure, make sure that the apparatus is clean. Set the feed rate so that at least 35 revolutions are completed by the time the hopper is emptied. If this is not achieved, recombine the sub-increment and repeat the increment reduction with a lower feed rate.

4.4 Test-piece increment achievement

A laboratory increment shall undergo modification (e.g. mixing with water for a castable) and/or shaping to carry out subsequent testing (physical and mechanical properties). As soon as the laboratory increment has been modified and/or shaped, it is called the test-piece increment. In general, each test standard defines the number of test bars which are necessary to validate the tests. The total number of test bars necessary to perform the test represents the test-piece sample. This number can be bigger or equal to the number of laboratory increments.

When the laboratory increment mass is still too big, reduce it to sufficient quantities for each test in the testing programme with the same method as in 4.3.1 and mark for identification.

Ensure that there is sufficient material for all the tests required and for reserve material and that the quantity for each test is consistent with the minimum requirement for the maximum grain size of the material.

The use of an increment to determine several properties is allowed, provided that the result(s) of the test is (are) not changed by the test or the preceding tests.

5 Marking, package, storage of increments

5.1 Marking

The increments shall be clearly and durably marked. Marking shall include

a) a unique code, or

b) identification of the increments, place of sampling, date of sampling, and designation of the material.

5.2 Package

The package and laboratory increment shall be packed in such a way that their condition at the time of sampling is preserved. If necessary, in order to preserve the moisture or volatiles content, airtight containers shall be used.

Laboratory increments for dispatch or transport to third parties, and increments to be held in reserve, shall be securely sealed using a method which will ensure that the condition of the material is maintained, and that properties are not affected.

5.3 Storage

Monolithic refractory materials can be modified between the date of manufacturing and the date of sampling. No increments shall be stored for longer than the shelf life of the product.

Modification can occur for many reasons.

a) Dry powdered materials, such as castables or dry gunning materials, contain binders which are always very hygroscopic. Hard balls can appear in the bags. These balls which represent a higher maximum grain size than the main aggregate have to be removed with a screen one size coarser than the maximum grain size before sampling. But, in themselves, these balls are an indicator of the material age and their presence shall be reported. For this type of material, it will be necessary to protect increments from water/moisture.

b) Ready-to-use ramming mixes contain a liquid added during manufacturing (water or resins). Ageing often results in a loss of moisture. For this reason, increments shall be preserved in an airtight plastic bag. During sampling, the sometimes lumpy appearance of these materials shall be taken into account. In this case, it is necessary to increase the mass of increment to avoid segregations.

c) Ageing of plastic mass and tap-hole mixes material frequently occurs because these materials contain organic binders (tar, pitch, resin). During ageing, a hard crust progressively develops and has to be removed from the block before forming test pieces. It is necessary to take into account the estimated crust mass to determine the increment mass.

d) For injection material and refractory grout, similar precautions to those described above for other materials shall be taken. However, as they usually contain a higher proportion of binder, extra care may be required.

This type of material runs a risk of its components segregating (between liquids and solids). Consequently, it is necessary to mix the material before sampling.

After sampling, the increments shall be preserved in a cool room, reduced and tested, as soon as possible. In the case of tap-hole mix or resin-bonded material, increments can be stored in a refrigerator if the time between sampling and testing exceeds one day.

NOTE 1 The conditions of storage for phosphate-bonded materials should be decided by agreement between the parties.

NOTE 2 Refractory mixes (e.g. harth ramming mixes, hot repair mixes, gunning mixes) with a high lime content tends to ‘dust’.

6 Sampling report

The sampler shall prepare a sampling report for each laboratory increment and also for each group of laboratory increments from a single source. The sampling report shall refer to this part of ISO 1927 and state:

a) the sampling report identification (serial number);

b) the laboratory increments identification mark(s);

c) the date and time of sampling;

d) the source of sampling point or identification of the batch sampled;

e) the sampling procedure;

f) the name of the sampler.

Depending on the circumstances, other information can be relevant and shall be included.

Annex A
(informative)

Example of sampling

A.1 General

Open porosity is determined on a 100 tonnes batch of castable (max. grain size: 10 mm).

A.2 Batch supplied in 25 kg sacks

Batch supplied in 25 kg bags

According to the mass of the unit package (less than 35 kg), the parties decide to follow ISO 5022.

AQL = 4 %.

Acceptance factor $K = 1{,}23$.

Because individual values T_S and T_i are given with an unknown standard deviation, the batch sample size is fixed at 18.

Eighteen bags are taken randomly among about 4 000 bags:

— the 18 bags form the batch sample;

— one bag is a package increment.

To obtain a laboratory increment from each bag, it is necessary to reduce the package increment with an appropriate method.

With a 5 kg mix, it is possible to manufacture 2 test bars (size B) that is, 2 test-piece increments for each package increment.

A.3 Batch supplied in 1 tonne big bags

The parties agree to consider that the batch sample size is fixed to 8 (the number of increments for 100 tonnes is fixed between 4 and 16 in accordance with ISO 8656-1).

It is necessary to obtain a package increment from each selected big bag to form the batch sample. For that purpose, the number of elementary increments is determined in accordance with ISO 8656-1.

According to the fact that open porosity often presents a coefficient of variation between 5 % and 15 %, four elementary increments are taken (see Table 2). The mass of each elementary increment is fixed at 500 g in accordance with Table 1.

The elementary increments will have to be taken during big bag charging.

Mixed together, these 4 elementary increments will form the package increment representative of the big bag.

Because the full mass of each package increment is not big enough to manufacture 2 test bars, it is necessary to increase the number of elementary increments or the mass of each elementary increment.

In both cases, even if the final total mass of package increment is different: 25 kg (1) or 2 kg (2), the test bar set (test-piece sample) is representative of the batch.

Step 1: Procurement of the batch sample

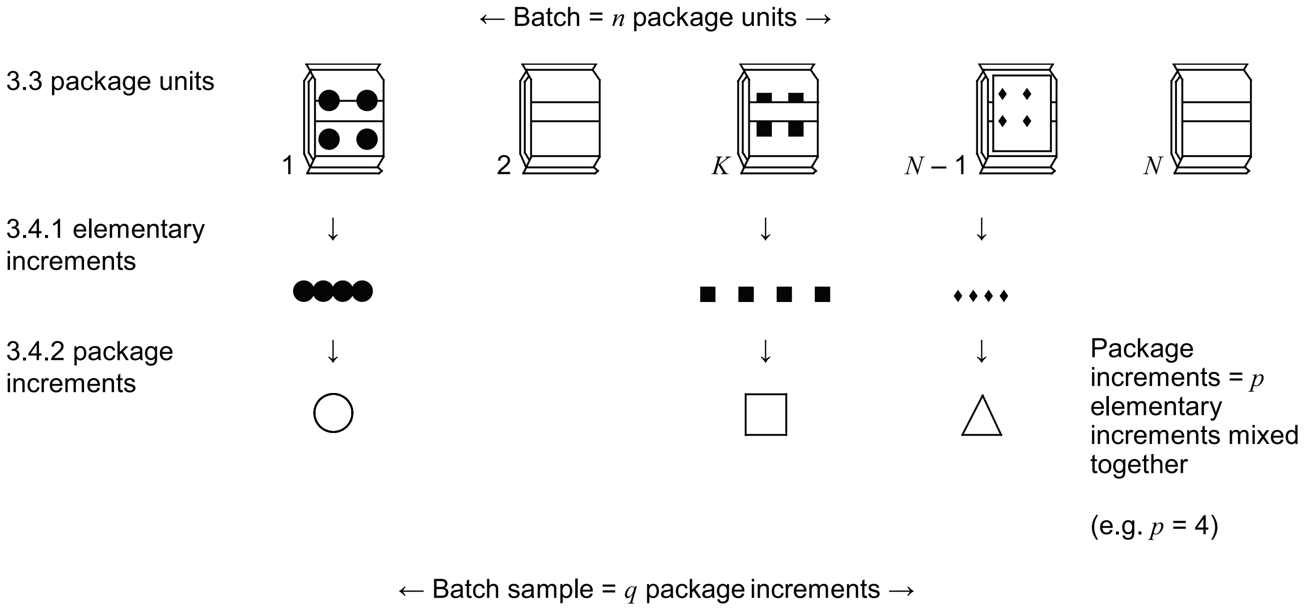


Figure A.1 — Sampling framework — Step 1

Step 2: Size reduction of increments

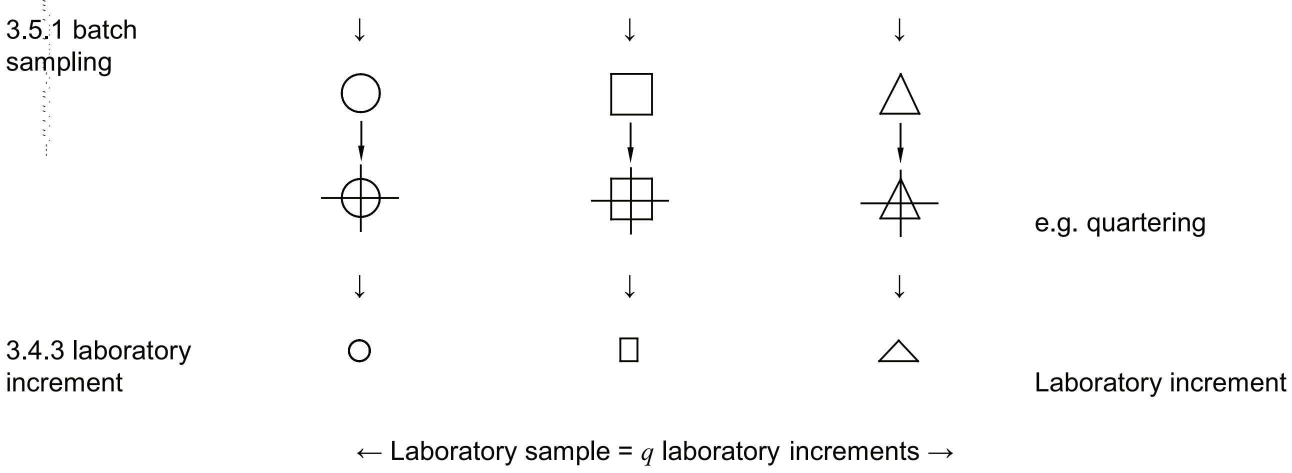
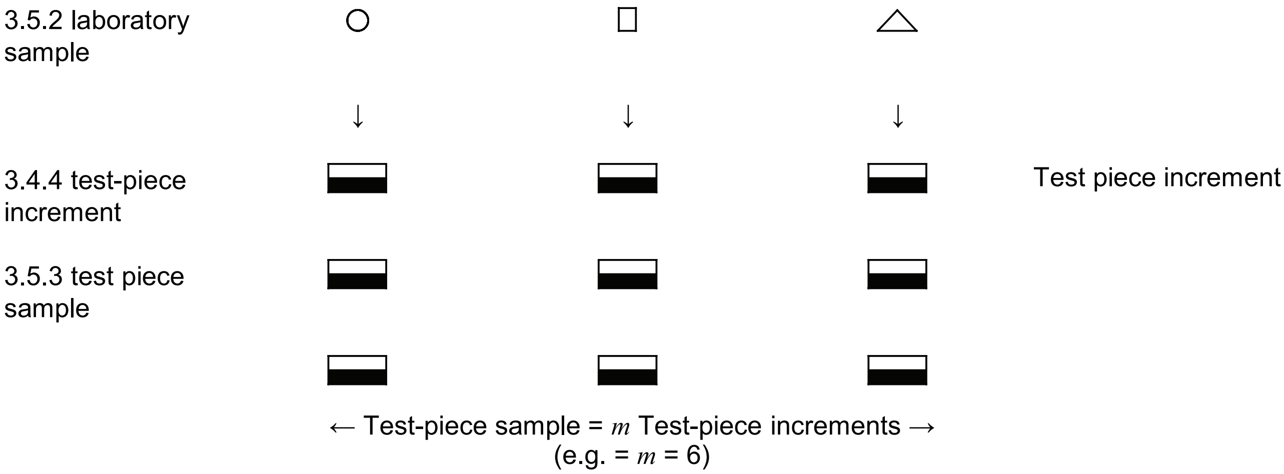


Figure A.2 — Sampling framework — Step 2

Step 3: Shaping



p is fixed in accordance with ISO 8656-1

q is fixed by the parties concerned or in accordance with ISO 5022

m is depends on the *q* value and ISO test standards

Figure A.3 — Sampling framework — Step 3

ICS 81.080

Price based on 11 pages