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Shaped refractory products — Sampling and acceptance testing

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Foreword

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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.

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Shaped refractory products — Sampling and acceptance testing

1 Scope and field of application

This International Standard gives directives for sampling shaped refractory products and for obtaining, from a sample of the smallest possible size, the most precise assessment possible, of the quality of a consignment.

The methods described below make it possible to carry out an acceptance test based on an assessment of the extent to which the specifications have been observed, but do not make it possible to determine whether the accepted consignment is suitable for a given application or to compare different qualities of parts for this same purpose.

This International Standard applies to products manufactured from refractory materials.

It may be applied when the parties concerned have agreed to do so and have therefore, by common consent, made a choice between the various possibilities put forward in this International Standard, and have specified the various parameters (see 3.2) which must be defined in order to permit the application of the methods described.

It is also possible to apply the directives forming the subject of this International Standard while modifying, by prior agreement between the parties concerned, those values which, particularly in the tables, do not follow from statistical laws (see 3.3).

2 Statistical terminology and symbols

2.1 population : The totality of items under consideration. Each of the batches formed in accordance with 3.1 represents a population.

2.2 size of the population : Number of items in the population (symbol : N).

2.3 sample : One or more items taken from a population and intended to provide information on the population and possibly to serve as a basis for a decision on the population or the process which had produced it.

2.4 size of the sample : Number of items in the sample (symbol : n).

2.5 observed value : The value of a characteristic determined as a result of an observation or test (symbol for the observed value having the number i : x_i).

2.6 extreme values :

x_{max} : largest observed value in a sample;

x_{min} : smallest observed value in a sample.

2.7 (arithmetic) mean : The arithmetic mean of the observed values in a sample is their sum divided by the size of the sample.

$$\bar{x} = \frac{1}{n}(x_1 + x_2 + \ldots + x_n) = \frac{1}{n}\sum_{i=1}^{n} x_i$$

The mean value of the population is designated by the symbol μ.

2.8 standard deviation : The standard deviation is the quantity most commonly used in statistics to characterize dispersion. It is the square root of the variance.

The standard deviation of the sample is given by the formula :

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2}$$

The standard deviation of the population is designated by the symbol σ.

In practice it is generally not convenient to compute $\bar{x}$ and s using the above formulae. Computations are made easier and their results improved using equivalent but different formulae (see [2]).

2.9 confidence interval : When it is possible to define two functions T_1 and T_2 of the values observed such that, when θ is a population parameter to be estimated, the probability

$$P\,|T_1 \leqslant \theta \leqslant T_2| = 1 - \alpha$$

where $1 - \alpha$ is a fixed number which is positive and less than 1, the interval between T_1 and T_2 is a confidence inverval for θ.

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The limits T_1 and T_2 of the confidence interval are random variables which, as such, may have different values for each sample.

In a large series of samples, the frequency of the cases in which the interval will include θ will be approximately equal to $1 - \alpha$.

2.10 confidence level : The value $1 - \alpha$ of the probability associated with a confidence interval.

2.11 statistical tolerance interval : An interval for which it can be stated with a given level of confidence that it contains at least a specified proportion of the population.

When both limits are defined by statistics, the interval is two-sided. When one of the two limits is not finite or consists of the absolute boundary of the variable, the interval is one-sided.

2.12 inspection by attributes : A method which consists in taking note, for every item of a population or of a sample taken from this population, of the presence or absence of a certain qualitative characteristic (attribute) and in counting how many items have or do not have this characteristic.

The characteristics inspected by attribute are, for example, cracks or other defects which are visible on the outside, or else defects which are revealed on sawing or by a sonic test.

2.13 inspection by variables : A method which consists in measuring a quantitative characteristic for each item of a population or of a sample taken from this population.

The measurable characteristics are, for example, the results of dimensional measurements, of chemical analysis or of physical tests.

2.14 single sampling : A type of sampling which consists of taking only one sample per batch.

2.15 sequential sampling : A type of sampling which consists in taking successive items, or sometimes successive groups of items, but without fixing their number in advance, the decision to accept or reject the batch being taken, as soon as the results permit it according to rules laid down in advance.

2.16 acceptable quality level (AQL) : A quality level which, in a sampling plan, corresponds to a specified, but relatively high, probability of acceptance.

It is the maximum proportion of defective units in the batch, such that batches in which the percent defective does not exceed this values, are regarded as "good" and will very probably be accepted if a sampling plan is applied.

2.17 limiting quality (LQ) : A quality level which, in a sampling plan, corresponds to a specified and relatively low probability of acceptance (usually 10 %).

It is the proportion of defective units in the batch, such that batches in which the percent defective exceeds this value are regarded as "bad" and will very probably be rejected if a sampling plan is applied.

2.18 producer's risk : For a given sampling plan, the probability of rejecting a batch in which the proportion of defective items has a value fixed by the plan.

It is the probability α of rejecting a batch when the proportion of defective units in this batch equals the acceptable quality level AQL (or when its mean value is equal to the guaranteed value μ_G for the mean).

2.19 consumer's risk : For a given sampling plan, the probability of accepting a batch in which the proportion of defective items has a value fixed by the plan.

It is the probability β of accepting a batch when the proportion of defective units is equal to the limiting quality LQ (or when its mean value equals $\mu_G + \Delta\mu$ or $\mu_G - \Delta\mu$).

2.20 operating characteristic curve (OC) : A curve showing, for a given sampling plan, the probability of acceptance of a batch as a function of its actual quality.

3 General considerations and preliminary conditions for sampling

3.1 Subdivision of consignments into batches

Consignments which correspond to a large tonnage shall be subdivided into batches of 100 to 500 t made up in accordance with the objectives which are being aimed at. These batches shall be sampled and subjected to tests separately and they may be accepted separately.

It will also be necessary to subdivide into batches a consignment which comprises products belonging to different classes or in which the items have been obtained by different methods of manufacture.

Moreover, a consignment shall also be subdivided into batches according to sizes, masses and, if necessary, the shapes of the items, if the producer and consumer are agreed in thinking that these factors influence the characteristics investigated.

For the purpose of making up batches in terms of the masses of the items, it is often desirable to divide the items into the following three categories :

- category 1 : items up to 15 kg;
- category 2 : items ranging from 15 to 35 kg;
- category 3 : items in excess of 35 kg.

The making up of batches from a consignment may be facilitated if the items are marked in such a way as to indicate the period during which they have been manufactured.

If a batch is declared to be non-complying it is possible to subdivide it into smaller batches by applying the criteria indicated above which might not have been taken into account when making it up, in order to ensure greater uniformity of each of the new batches made up, and these may be subjected to acceptance separately. This procedure may only be applied after a

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new agreement has come into operation between the producer and the consumer, and it is expedient to make sure that the new sampling plans which will be operated provide, for both parties, similar guarantees to those which would result from the first plan used.

3.2 Properties inspected

3.2.1 Specifications relating to the properties inspected

For each of the batches made up as indicated above, each of the properties inspected by attributes is characterised by a proportion of defective units in the batch, and each of the measurable properties is characterised by a mean value and by a standard deviation.

Statistical control of a production in respect of its quality shows that, over a period of time, the mean value (μ) of a property undergoes fluctuations which are due to inevitable variations in the raw materials, their preparation and the methods of casting and firing. The standard deviation (σ), on the other hand, generally varies less.

When the specifications are drawn up, a mean value μ_G is guaranteed by the producer : the producer guarantees, depending upon the nature of the property, that the mean value of each batch is either less than or equal to μ_G or else greater than or equal to μ_G.

The delivery contract must therefore specify, for each class of product :

— the properties on the basis of which acceptance or rejection of batches will be decided;

— for each of these properties, the specification which will be employed.

This specification may assume various forms. It may consist of fixing down :

— in the case of sampling by attributes, a maximum percentage of defective parts (which takes the form of the fixing of an acceptable quality level : AQL). The corresponding sampling plans are dealt with in clause 4.

— in the case of sampling by variables :

— a guaranteed value (μ_G) for the mean. The corresponding sampling plans are dealt with in 5.3 and 5.5, or,

— a limit value for the individual values (an upper limit T_s or a lower limit T_i, according to the properties).

In this event, the delivery contract must also lay down an acceptable quality level (AQL). The corresponding sampling plans are dealt with in articles 5.4 and 5.6, or,

— a downwardly limited and an upwardly limited value for the mean value or the individual values. The sampling plans corresponding to bilateral protection of this kind are not given in the present document.

A batch will comply with requirements if it really belongs to the class laid down in the order or specifications and if the values found for each of the properties investigated, following application of the sampling plans described below, result in a conformity decision.

A batch will not comply with requirements if it does not belong to the class laid down or if the values found for one or more than one of the properties investigated, following application of the sampling plans described below, result in a non-conformity decision.

3.2.2 Nature and number of the properties subjected to inspection — Efficiency of plans

The nature and number of the characteristic properties inspected depend upon the nature of the consignment, its intended use, all the risks which the producer and consumer agree to incur, and the expense which they agree to devote to sampling and testing.

In fact, the application of any sampling plan provides no certainty that the batch either is, or is not in conformity with the requirements : the probability of acceptance of a batch and its quality level are related through a function which is defined by the selected sampling plan.

This function is represented by the operating characteristic curve of the plan which, for convenience of use, is characterised by two points : one corresponds to the producer's risk α and the other to the consumer's risk β.

If the inspection deals with a single property, the sampling plans described below are such that :

- the producer's risk (α), which is associated with the acceptable quality level (AQL) fixed by the requirement (or which is associated with the guaranteed value μ_G for the mean) is always fixed, in the case of inspection by variables, at a value which is equal to or very close to 5 %. In the case of inspection by attributes, this risk is variable (see table 3).

- the consumer's risk (β) is associated with a batch quality level which depends directly upon the sampling plan selected for inspection. The values for this quality level (limiting quality) are given for a constant risk β = 10 % in tables 3, 9 and 10 and can be found, for different values of β, from the graphs giving the operating characteristic curves of the corresponding sampling plans (see figures 4, 5, 6 and 7).

It may be noted that the percentages of defective units associated with the consumer's risk in the various sampling plans are generally high as compared with the producer's risk. This is the result of economic considerations which induce both parties to cut the size of the inspected samples.

However when inspection is carried out on a number of quality characteristics the resulting risk increases for the producer and decreases for the consumer if it is assumed that a product subjected to inspection must meet all the requirements put on the individual properties tested to receive final acceptance. In this case, and assuming in addition that the quality characteristics

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involved in the various acceptance procedures are independent, table 1 gives the resulting values of α and β as a function of the number j of quality characteristics subjected to inspection.

In actual fact, the overall consumer's risk indicated in this table do not permit a full assessment of the severity of inspection. This severity is much better represented by the quality level (or percentage defective) LQ associated with a constant value of β.

This is shown in the example given in figure 1[1] (the points B_1, B_2, B_3 all correspond to $\beta = 10$ %).

In each case, only a knowledge of the complete operating characteristic curve of the plan makes it possible to know the value of one quality level corresponding to a given risk and to the number of properties inspected.

Furthermore, the properties of refractory products are not all independent of one another, and the values indicated in table 1 are therefore maximum values (for the producer) or minimum values (for the consumer).

They nevertheless lead to limitation of the number of properties inspected : the number of properties inspected by destructive tests (excluding chemical analysis) should not be greater than three.[2]

Furthermore, with the exception of methods dealt with in 5.3, the methods of sampling by variable are based on the theoretical hypothesis that the property measured is distributed in the batch in accordance with the normal law. In practice, the properties investigated will very rarely be distributed precisely in accordance with this law, but the efficiency of the methods of investigation is changed very little in cases where the distributions deviate only slightly from normal.

In cases of doubt, however, it is expedient to verify, with the aid of a statistical test (for example [2]), that the distribution of the property considered in the investigation may be regarded as normal.

Table 1 — Change in risks when the number of independant properties inspected increases

Number of properties j	Total producer's risk when the producer's risk α rises to 5 % for each property %	Total consumer's risk when the consumer's risk β rises to 10 % for each property
1	$\alpha_1 = 5{,}00$	$\beta_1 = 10$ %
2	$\alpha_2 = 9{,}75$	$\beta_2 = 1$ % $= 10^{-2}$
3	$\alpha_3 = 14{,}26$	$\beta_3 = 10^{-3}$
4	$\alpha_4 = 18{,}55$	$\beta_4 = 10^{-4}$
5	$\alpha_5 = 22{,}62$	$\beta_5 = 10^{-5}$
6	$\alpha_6 = 26{,}49$	$\beta_6 = 10^{-6}$
7	$\alpha_7 = 30{,}17$	$\beta_7 = 10^{-7}$

1) The operating characteristic curves shown in figure 1 apply in case where, for each property, the same sampling plan used, as defined by :

- a known standard deviation (σ);
- a one sided tolerance limit for individual values;
- an AQL of 4 %;
- a sample size of 10.

These curves are graphic representations of the following function :

$$P = \Phi_j\,[(U_{1-p} - K)\sqrt{n}]$$

where

P is the probability of accepting the batch on the basis of all quality characteristics inspected;

Φ is the distribution function of the standardized normal distribution;

U_1 is the standardized deviation corresponding to a probability p;

p is the proportion of defective items in the batch subjected to inspection;

K is the constant defined by the sampling plan used;

j is the number of properties subjected to inspection.

2) It will be possible to use the items which have supplied the highest or lowest values when subjected to these three investigations, for determining certain other characteristics for information purposes.

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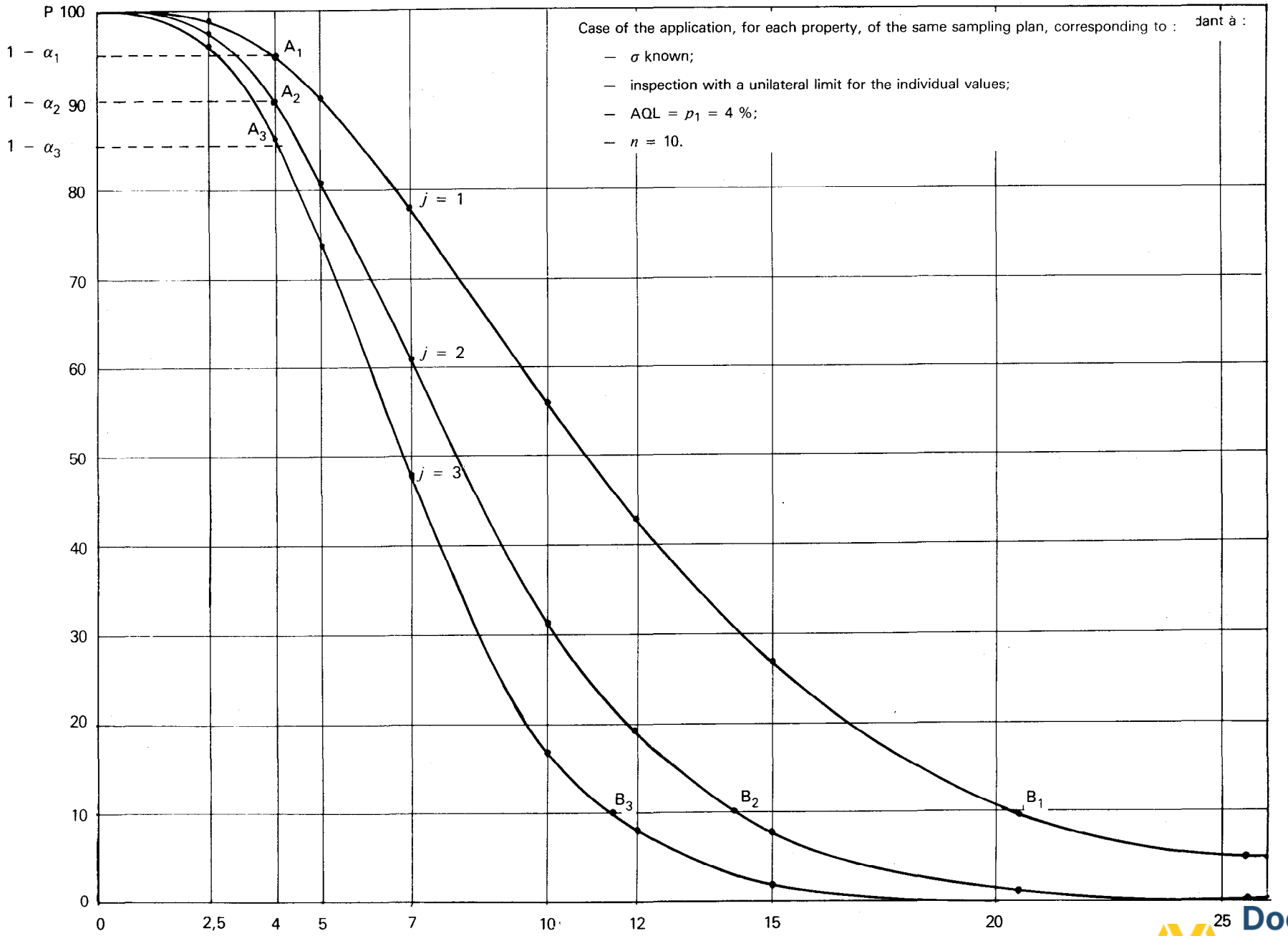

Figure 1 — Example of operating characteristic curves when several properties are investigated simultaneously

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3.3 Sampling process and use of the sampling plans described in clauses 4 and 5

Every sampling plan shall be prepared, and its execution supervised, by experts who are as well acquainted with the problems of the production and use of the products as with the problems of sampling.

The taking of samples shall be carried out in such a way that all the items in the batch have the same probability of being selected and tested.

The efficiency of a sampling plan depends solely on the number of sampled units, n, whatever the size, N, of the batch, provided that n/N is less than 10 %. Tables 3, 4, 6, 9 and 10 (or the operating characteristic curves also given in this International Standard) shall be used to determine, on the basis of the required efficiency, what the sample size should be.

If experience shows that the quality of the manufacturer's production corresponds to the agreements, it is possible, when batches of the same quality are frequently subjected to acceptance procedures, either to choose a plan with lower efficiency which implies the use of a smaller sample size or to reduce the number of batches inspected while retaining a plan having the same efficiency. The same applies when statistical quality control charts are available (see 3.5).

Similarly, if it is desired to reduce the proportion of defective units associated with a given consumer's risk value, it is necessary to choose a sampling plan with higher efficiency which implies the use of a larger sample size.

The sample size, n, indicated in the tables corresponds to that number of results relating to one of the properties inspected, which must be available in order to decide whether the batch is in conformity as regards the said property.

Each method of test must define what constitutes a result. Thus according to the nature of the test, a result may be constituted either by the value obtained by applying the method of test on a single occasion, or by a value which is deduced from the values obtained by repeating the test one or more times under the conditions prescribed by the method. Each of the n "results" must be obtained from a different item.

It is therefore necessary to calculate, from the sample size corresponding to each of the properties which will be inspected, the number of items which will have to be selected while taking into account :

— the number of properties which will be inspected;

— the specifications of each of the methods of test which will be used;

— the fact that each of the items selected may, or may not, be used for investigating a number of properties;

— the possibility of problems during the handling of the items selected or during the tests;

— the way in which it is intended to settle any disagreement between the producer and the consumer : in this connection, it is recommended that the number of items selected should permit the making-up of a reserve sample for use in the event of arbitration.

3.4 Treatment of the items selected

The distribution of the items selected, their possible apportionment between the various parties concerned (producer, consumer, arbitrator) and the constitution of a reserve sample shall be indicated in the terms of the transaction as well as, if necessary, the method of sub-dividing the test pieces.

3.5 Use of statistical control charts

The tests carried out by the consumer may be considerably reduced if the manufacturer regularly plots statistical control charts of the quality of his production, and places these charts at the disposal of his customers.

It is therefore expedient that the pattern of the control chart used should be selected in such a way that it can be used with equal success for production and for the inspection of a consignment : a selection of works on this subject is given in annex D.

Control charts can be used for controlling the mean value, the standard deviation, the tolerance or the percentage of defective units.

A further advantage of regular use of control chart is that, in certain cases it supplies a good estimate of the standard deviation of the quality characteristic.

4 Sampling for non-destructive tests

4.1 Inspection of the external appearance

The specification precisely defines what should be regarded as a defective item after examination of its external appearance.

It therefore specifically states the defects, such as cracks, blemishes, deformations, firing defects, etc., which will be taken into consideration.

The acceptable proportion of defective items (AQL) is also fixed by agreement between the two parties. This proportion may often be fixed at 4 % in the case of ordinary bricks and mass-produced items, and at 1,5 % for items having complicated shapes.

The external appearance is inspected by attributes.

The sampling plans to be taken into consideration, which are defined by the sample size, n, and the acceptance number, c, may be taken from ISO 2859 [40]. Table 3 gives a selection of sampling plans for AQL's of the order of 6,5, 4,0 or 1,5. This table also gives, in column 4, the probability of acceptance P for different proportions p of defective units in the batch.

The number y of defective pieces in the sample having a size n is determined.

If $y \leqslant c$, the batch is in conformity;

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