SLOVENSKI STANDARD
SIST ISO/TR 7821:1995

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Tobak - Priprava identiļ nih vzorcev iz iste serije (dobra praksa za primerljive ġtudije izraļ unskih metod preskusov)

Tobacco - Preparation and constitution of identical samples from the same lot (Code of practice, for collaborative studies for evaluating methods of test)

Ta slovenski standard je istoveten z:

ICS:

65.160	Tobak, tobaļ ni izdelki in oprema	Tobacco, tobacco products and related equipment

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Tobacco — Preparation and constitution of identical samples from the same lot (Code of practice, for collaborative studies for evaluating methods of test)

0 Introduction

The repeatability and the reproducibility of a particular laboratory method are defined by ISO 3534. These characteristics may be determined by means of inter-laboratory tests as described in ISO 5725.

In addition to the description of the equipment and the methods of test to be used, often by reference to existing standards, the organization of such tests also includes the preparation of test materials, i.e. samples which will serve as media for the studies to be carried out.

It should be noted that, in this type of study, there is rarely any interest in the characteristics of the test material itself, provided that such characteristics are within the typical range for the materials commonly tested, and values are determined in this way only in order to obtain the quantitative data necessary for compiling a statistical report concerning the method of test.

Thus, in most cases, every effort is made to obtain lots or samples which are as similar to each other as possible, in such a way as to reduce the residual variance of the tests and, in the same way, to increase the accuracy of the conclusions which may be drawn from the global analysis of the results.

Since the individual test samples do not necessarily need to represent the population from which they are taken, but only have to be identical to each other, the method for their preparation can be quite different from those needed when the aim of the study is to obtain a knowledge of the original population.

In the case of certain industries, the chemical industry for example, it is relatively easy to make up samples which are practically identical to each other, as the structure of the material allows it to be divided up so as to conserve the microscopic or macroscopic homogeneity of the product, for example solutions, powders, etc.

In the case of tobacco, however, it is completely different, because the raw materials, and even the finished products, are affected by an intrinsic heterogeneity likely to cause considerable differences between samples if special precautions are not taken.

Tobacco, as a test medium, may occur in the following forms :

— powder;

— cut tobacco (scaferlati);

— cigarettes;

— leaves or strips.

The method for the preparation of samples of test media depends, in most cases, on the type of test to be carried out. Nevertheless, it seems possible to lay down a few general guidelines applicable to a large number of collaborative studies, depending on the form in which the material occurs.

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1 Scope and field of application

This Technical Report provides a practical framework for the operations of dividing a lot (not necessarily homogeneous) of tobacco or tobacco products into several similar samples intended for collaborative studies either within or between laboratories.

2 References

ISO 3534, *Statistics — Vocabulary and symbols.*

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

3 Preparation of samples

3.1 General

The division of a lot into samples which are similar to each other is very easy if the lot is (or can be made, according to its physical state) homogeneous, but may be more difficult if it is not. In this case, an appropriate sampling procedure has to be used in order that the influence of heterogeneity is as small as possible.

It is obviously not possible to describe in detail every case which may arise.

The aim of the following examples, therefore, is to illustrate the principles of, and to give guidance on, the operations to be carried out, which, moreover, appear simple to conceive bearing in mind

— on the one hand, the given aim, which is to make up samples which are as similar as possible;

— on the other hand, to take into account the fact that two units will probably have characteristics which are most similar if they are sampled from points which are physically close together.

3.2 Powder

The initial lot comprises N g.

It has to be divided up into n samples of $\frac{N}{n}$ g.

3.2.1 Principle

Each sample consists of a number of increments.

Each increment is taken separately from the lot, and should, in theory, be allocated at random to each of the n samples. In practice, it is sufficient to allocate the increments to each of the n samples in rotation, provided that the number of cycles of rotation (i.e. number of increments in each sample) is large.

3.2.2 Precautions to be taken

During the preparation of the samples, the following precautions should be taken :

a) Work in an atmosphere which has a relative humidity as close as possible to that corresponding to the equilibrium humidity of the material being handled.

b) Work as quickly as possible in order to limit the exchange of moisture between the atmosphere and the material if it is not possible to satisfy completely condition a).

3.2.3 Example

A gross sample of 1 kg of powder is to be divided into 10 samples of 100 g. The condition to be fulfilled for the collaborative studies is not that 10 samples of exactly 100 g each should be obtained, but that each sample of approximately 100 g should be similar, from the physical and chemical point of view, to each of the others.

3.2.3.1 Manual method

After mixing the gross sample as thoroughly as possible, place the pile on a tray or in a receptacle from which the material can be easily scooped out. Use a spoon, ladle or measure, of capacity between 5 and 10 g for example. Provide 10 boxes capable of holding at least 100 g each.

Taking a succession of increments, using the spoon, ladle or measure, feed, in turn, box 1, box 2, ..., box 10.

Each sample is thus made up of at least 10 (10 to 20) increments.

When the initial pile is almost exhausted, i.e. when there is only enough left for one round of 10 increments, it is recommended that distribution be finished with a measure of smaller capacity.

This method guarantees that the 10 samples thus constituted are as similar as possible. Any mixing of the gross sample will cause variation of particle size and density through the pile from top to bottom, and this should be taken into account when taking increments.

3.2.3.2 Automatic method

Use a mechanical device such that, at each revolution, the quantity attributed to each sample is less than or equal to one-tenth of the total mass of the final sample.

3.3 Cut tobacco (scaferlati)

3.3.1 General

Exactly the same principle as for powder is applicable. In practice, handling is more difficult as the tobacco may form tangles, especially if it is in long strands.

The temptation to make up the increments from wads or lumps should be resisted, and, instead, they should be broken down so that they can be divided between all the samples.

As a general rule, each sample should be made up of at least 10 increments (and more if possible). In addition, increments should always be distributed to each sample in a standard rotation.

Depending on the size of the lot, the increment may be of the order of a large handful, a handful or a pinch.

3.3.2 Special precautions

Cut tobacco (scaferlati) always tends to settle, i.e. handling causes a certain amount of separation of the short strands which collect at the bottom of the receptacle.

In order to avoid causing an artificial heterogeneity between samples when the long strands have been exhausted, it is necessary to distribute the short strands equally between samples using an instrument such as a scoop, spoon, etc., which prevents the loss of these strands at the time of distribution.

3.4 Cigarettes

3.4.1 Cigarettes to be sampled from the production machine.

It is known that there are serial correlations between the characteristics of cigarettes, i.e., on average, the probability of cigarettes being identical is greater the shorter the interval between their fabrication by the cigarette making machine.

For example, if 10 samples, each of 100 cigarettes, are to be prepared, proceed as follows :

a) take a series of 10 consecutive cigarettes and distribute them to the 10 samples to be made up;

b) repeat the operation 100 times.

NOTES

1 There is no disadvantage if there is a gap between taking increments (of 10 cigarettes), i.e. if the tenth cigarette of one increment and the first cigarette of the next increment are not successive cigarettes.

2 To save time, it is permissible to take increments successively. For example 20, 50, or even 100 successive cigarettes can be taken. In this case, it is undesirable to keep the cigarettes in bulk, and they should be kept in individual increments, labelled in the order of manufacture, and should be distributed in that order, rotating round the ten samples to be constituted in the usual manner. For this purpose, automatic sampling devices can be used.

Experience shows that making up samples in this way gives results as good as, or even better than, selection by mass or draw resistance as far as comparability of samples is concerned.

3.4.2 Cigarettes sampled from a bulk cigarette storage before packing

For the same reasons as described in 3.4.1, it is necessary to avoid constituting the samples by taking each of them in a group from the case.

Instead, it is recommended that one of the two following procedures be used in order to constitute, for example, 10 samples of 100 cigarettes :

a) take successive cigarettes for each sample at random, for example using a table of random numbers to which, for example, the (approximate) coordinates are made to correspond to a point from where each cigarette is taken;

b) take each horizontal row of the storage bin or case as a primary sample and take from it one increment for each of the samples being made up. It is not necessary in this case for the whole row to be allocated; the main point is that each sample should contain cigarettes from every row selected as a primary sample.

Procedure a) is theoretically the most satisifactory, but it can be unwieldy to apply when there is a large number of samples (greater than 3 or 4), comprising a large number of cigarettes, to be constituted.

In current practice, procedure b), which is more rapid, is amply sufficient, because of the partial stratification which is involved, and thus compensates for the fact that the sampling is not totally at random.

3.4.3 Cigarettes in packets

Initial lots of cigarettes in packets may, as appropriate, be made up of :

- packets in bulk;
- cartons or units (for example units of 200);
- boxes.

The procedure to be used for sampling the lot for the purposes laid down in this Technical Report relies on observation of the following principles :

a) Take into account the fact that two cigarettes are, probably, most similar in their characteristics, if they originate from the same package unit.

b) Adapt the method of sampling to the volume of the samples to be made up.

Thus, in certain cases, the packet itself may be considered as an increment, whereas, in other cases, the packets themselves must be opened.

3.4.3.1 Example 1

The initial lot is made up of two boxes each containing 25 units of 10 packets of 20 cigarettes (boxes of $20 \times 10 \times 25 = 5\,000$ cigarette units, i.e. 250 packets, or a total of 10 000 cigarettes, 500 packets, 50 cartons).

If it is wished to make up, for example, 5 samples (as similar to each other as possible) of 20 packets from the initial lot of 500 packets, constitution of the samples by systematically sampling 2 units of 10 packets to make up each sample will obviously be avoided, since internally homogeneous samples would be obtained, but there would be greater differences between them than within them. The best procedure would be the following :

From each box, take at random 1 unit in 5. Thus 10 units (2×5) each of 10 packets will be obtained.

Each unit can be divided into 5 increments each of two packets, and each sample will then receive one increment from each unit.

In this case, the 5 samples of 2×10 packets will be replicates.

3.4.3.2 Example 2

From the initial lot described in 3.4.3.1, select 10 samples of 50 cigarettes each.

For obvious reasons, it is not recommended in this case that the packet should be kept as the unit of sampling.

Moreover, it is again recalled that the aim in view is not to make up samples representing the original lot but samples which are as similar as possible to each other.

Starting from these principles, and with a total number of cigarettes in all samples of 500, or 25 packets, the method to be used is as follows :

a) Select 3 units from one box. Keep two whole units, but remove half of the third unit to give a total of 25 packets.

b) Open each packet and distribute the cigarettes into 10 samples (2 cigarettes per sample).

c) In this manner, 10 samples each of 50 cigarettes are constituted and these will be as similar to each other as possible.

3.5 Leaves or strips

This case is called upon only as a last resort, because it poses difficult specific problems and because it is rare that leaves or strips themselves will constitute the material on which the collaborative, physical or chemical studies will be carried out.

Similar samples of leaves or strips are usually used only for tests concerning changes of industrial processes. These tests require the use of industrial production machinery (for example mixing boxes) and the sampling procedure is outside the scope of this Technical Report.

However, if the lot consists of leaves or strips, analysis of the actual requirements shows that it is possible, without disadvantage to the method of test, to modify the physical aspect of the material and to use one of the methods described in the above examples.

An example will illustrate this point, as it was an actual case which occurred some years ago.

There were various procedures to be tested for the determination of the moisture content of tobacco for different varieties of leaves[1] and each initial lot to be sampled was made up of leaves. Detailed analysis of the problem showed that sampling should not be carried out on the leaves, but after treating them and reducing them to cut tobacco.

Collaborative tests were carried out in good conditions with correctly sampled lots of cut tobacco.

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1) CCE, *Method for the determination of the moisture content of tobacco*, 91, 1972.