TECHNICAL REPORT

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Plastics — Guide to the writing of test methods

Plastiques — Guide pour la rédaction des méthodes d'essai

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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.

The main task of technical committees is to prepare International Standards. In exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

— type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;

— type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;

— type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 13883, which is a Technical Report of type 3, was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

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

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Introduction

The title of this Technical Report could lead one to assume that test methods issued by different organizations are not always ideal, and that they lead to results which are doubtful or not easily comparable between laboratories.

If this observation may be considered somewhat pessimistic, it appears that many test methods include ambiguities or technical inaccuracies or propose unjustified variants for either the sampling or for one or several of the steps in the procedure or in the expression of the test result.

When such situation is observed, the operator or the supervisor must make choices. Providing the choice is consistent, the results for the operator and between operators within the laboratory, will be comparable. However, when comparisons are made between laboratories, the choice that has been made can often cast doubts on the analysis and maybe even result in its rejection.

Thus, it is important when writing test methods to try to eliminate any ambiguity and also to provide the users of the methods with sufficient information needed to obtain valuable data.

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Plastics — Guide to the writing of test methods

1. SCOPE OF THIS TECHNICAL GUIDE

This is a guide devoted to test methods.

It defines the essential elements which must be present in a method and also those which shall not be included but which the operator must find in other documents, for example, the specification of the product being tested.

This guide also proposes some important definitions in the field of sampling and also in relation to the different results which are generated by the method.

2. THE RIGHT TERMS TO REMEMBER

- **Batch** : A definite quantity of product made up of elementary units of the same type and produced under conditions assumed to be constant. A batch may constitute all or part of any particular order. (Ref ISO 1886).

- **Sample** : A given number of elementary units which have been selected at random with a view to performing a test either directly on these units or on one or more specimens taken from the units. (Ref ISO 1886).

- **Elementary unit** : The smallest normally commercially available entity of a given product. (Ref ISO 1886).

- **Laboratory sample** : A certain part of one elementary unit from which one or several specimens will be selected for the analysis.

  Note 1 : Such intermediate sampling is generally performed when the elementary unit cannot be easily transported to the laboratory. The procedure for preparation of such a laboratory sample shall be provided either in the test method or in the test order instructions.

  Note 2 : For certain test types, it may become necessary to prepare, starting either from one elementary unit or from a sample for laboratory, one "secondary sample for laboratory" in which test specimens will be taken for the test itself. A typical case is represented by laminated plaques which are manufactured with a resin and a reinforcing material in view to measure mechanical properties.

- **Specimen (also called Test piece)** : The quantity of material submitted to the test itself.

- One **reading :** one piece of information, usually numerical, obtained using one measuring device or through a visual observation.

- One **measurement** : The result of a test on one specimen.

  Note : One unique measurement can comprise several readings. A typical case is the determination of loss on ignition.

- **One determination** : It is the outcome of the test method which will be expressed by one individual result.

3. THE NEEDS OF THE USER OF A TEST METHOD

3.1. Who are the main users ?

- Essentially the persons performing an analysis.

- But also :
  . The person who establishes a specification.
  . The person who purchases a product.

3.2. But, in practice, who really uses the ISO test methods ?

Industrial practice shows that ISO test methods are not necessarily found on the desk of the operator. They are often kept by the Laboratory Manager for writing up methods which are internal to the company.

These internal methods differ from ISO methods by the fact that :

- They are generally simpler, keeping only the paragraphs that are essential for the operator, in relation to the specific product tested in this laboratory.
- On the other hand, they contain information about the way to handle equipment that is used in the laboratory for that method.
- They are written in the local language if it differs from the 3 standard ISO languages (English, French, Russian).

In any case, for these internal methods, the applicable ISO standards constitute the reference and, consequently, must be such that they can be used without any divergent technical interpretation from user to user.

3.3. What does the main user need at the level of the test method ?

Clear information, complete, without any ambiguity or possible misinterpretation of what is required to run one analysis, either :

- on one elementary unit or a part of such a unit, or
- a certain number of elementary units, or
- on a batch which, as an example must be inspected for acceptance.

Attention : A difference must be made between :

- what shall be provided in the test method itself
- from what must be stated in other documents.

Hence, the analysis of a given quantity of a product comprises essentially 3 steps :

- the sampling of this "quantity" of product which can be made of units (or part of units) or different sizes, different shapes, or different presentations,
- the test itself,
- the writing of the test report with all details requested by the person ordering the analysis.

The person performing such an analysis must be in possession of accurate information regarding these 3 steps. The major question for this person is to know where to find the information.

3.4. Note

Outside of the problem related to what the test method should include, one must insist on :

- the need to write simple, clear and well built methods,
- the appropriate choice for the requirements related to the accuracy of the test apparatus (not excessive, not too limited).

In the specific field of accuracy of testing equipment, it is recommended to refer to the requirements specified in the ISO 9000 series of standards.

These standards insist on the necessity to proceed to a calibration of the testing equipment. This automatically implies that a given accuracy level be defined in the method and subsequently that this level can be achieved by the equipment used.

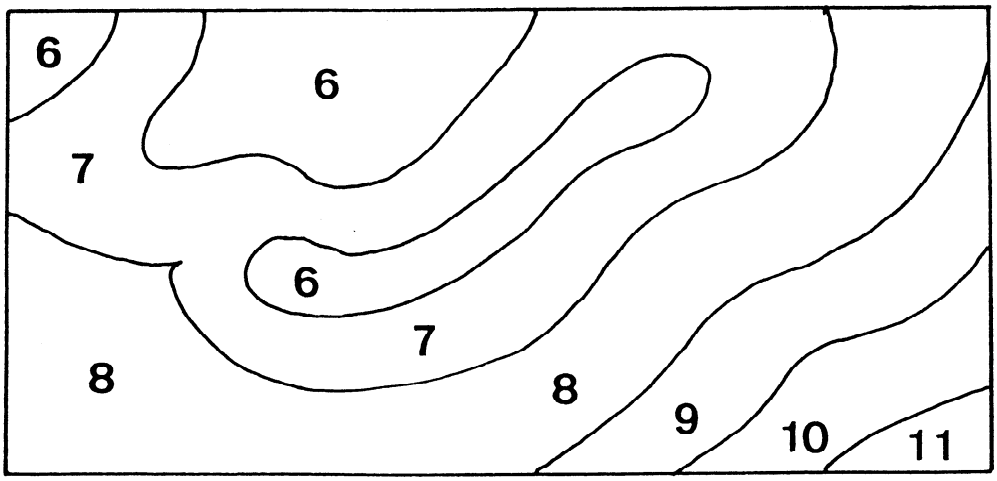
4. THE PUZZLE OF THE IDEAL SAMPLING

4.1. Shape, dimensions, orientation, etc, of the specimen.

Let us assume that a test must be performed on an article which has the form of the surface shown in figure 1.

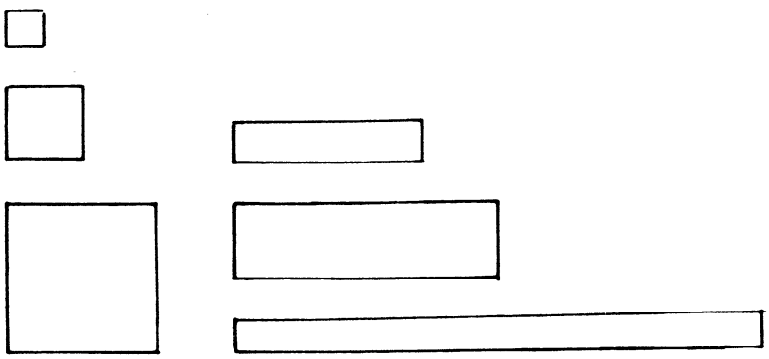
Let us consider that such surface can be characterized by a measurable property, the level of which is represented by the zones on the figure 1.

Figure 1



The first question to be solved is to define the type of specimen which must be chosen (its shape, dimensions, weight, orientation) in view to obtain the adequate information.
We may, as an example, consider several specimen types such as those shown in following figure 2.

Figure 2



It is obvious that, depending upon the local variability of the property, the information resulting from the test will be directly linked to the choice made for the specimen.

4.2. How many measurements for one determination ?

The 2nd question deals with the number of measurements which must be done for one determination.
This number depends essentially upon the variability of the measurement itself and basically is linked to :

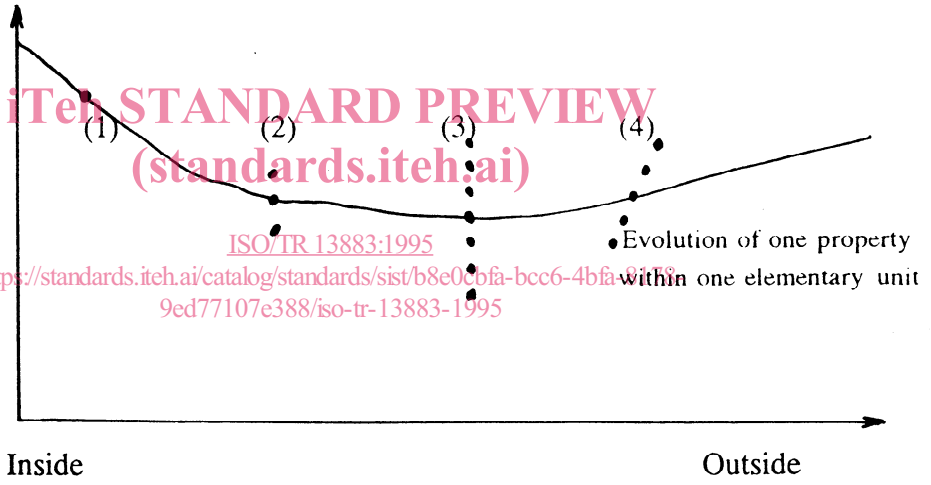
- the precision of the test equipment(s) utilized,
- the operator him(her)self and his qualification, his training, his visual acuteness, etc.

The objective is to reach a value which is the closest to the true value at the place on the elementary unit where the test is performed.

If the method has a high variability, the number of measurements shall be increased either on the same specimen (in the case of non-destructive testing) or on separate specimens which need to be as identical as possible (destructive testing). The average of the measurements done will constitute the individual result of the determination.
On the contrary, an accurate method (or a method for which the accuracy is judged sufficient) may be satisfied with one measurement on one specimen.

An illustration of the notion related to the number of measurements for one determination is provided on figure 3.

Figure 3



Different types of determination shemes

Legend : 1. : 1 measurement on 1 specimen : accurate method
2. : 3 measurements on 1 specimen : medium accuracy, non-destructive test
3. : N measurements on 1 specimen : low accuracy, non-destructive test
4. : N measurements on N specimens : low accuracy, destructive test

4.3. How many determinations within one elementary unit ?

Referring back to the figure 1, it is obvious that a 3rd question needs to be tackled. To judge the quality of the elementary unit, it is now necessary to define how many times, and where within the unit, the determination as described by the test method must be repeated.

For this purpose, one must take into account the variability within the elementary unit. This variability shall be taken in its broad sense and shall therefore consider :