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**Plastics — Production quality control —  
Statistical method for using single  
measurements**

*Plastiques — Contrôle de qualité en production — Méthode statistique  
pour l'utilisation de mesurages uniques*

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Tel. + 41 22 749 01 11  
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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 25337 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

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## Introduction

Many ISO standard test methods specify that measurements be made in duplicate or even in more than duplicate. In some cases, repeated operations are also specified. However, for production quality control it is common practice to carry out single measurements. For many production environments, replicates and/or repeated operations, as specified in many ISO standards, are time-consuming and expensive and could result in an undesirable increase in production costs. Furthermore, the laboratory response time could also increase unacceptably.

This International Standard presents a statistical method for using only single measurements in a production environment, in accordance with ISO test method standards which specify at least duplicate measurements and/or repeated operations. This International Standard is likely to be of interest to companies using ISO test methods in production quality control and recorded in their quality management system. This International Standard is not intended to be used for publishing data, for marketing purposes or for the development of customer specifications/designations.

Provided the statistical computations support the reduction in the number of replicates for a particular test, reduction from duplicate replicates to a single measurement is possible, as is reduction in the number of replicates for a test result to half, or even less than half, the specified number of replicates. A reduction in the variability of a test method might be necessary before a reduction in the number of test replicates can be considered<sup>1)</sup>.

Technical Committee ISO/TC 61, *Plastics*, considers that such a model might not only be of importance in the field of plastics but also of interest in other fields in which ISO test methods are used in production quality control.

Single measurements are often used for quality control tests. The “single-measurement model” presented in this International Standard forms a basis for

- carrying out single measurements for production quality control;
- reducing the number of replicate tests carried out and/or modifying test methods using multiple test samples in order to reduce laboratory costs and decrease the laboratory response time;
- modifying test methods which specify repeated operations (as is usually the case with, e.g., drying to constant mass) to give a test method which involves only one operation, leading to shorter response times and lower laboratory costs;
- handling results that lie outside the production and/or acceptance limits;
- achieving cost savings by harmonizing material production limits with the test methods used, taking into account the precision of the test method and the production capacity.

Furthermore, when specified by the responsible authority, the single-measurement model described in this International Standard can be used as a reference in, for example, product specifications, in sales agreements and in communicating with customers.

---

1) An Excel-based tool for the calculation of repeatability and reproducibility parameters has been developed by ISO/TC 61/SC 5 and is expected to become available on the ISO web site for on-line calculations at some future point in time, initially at a password-protected location reserved for those participating in the development of ISO standards. The calculation model is based on that of ISO 5725 and is applicable to balanced and unbalanced data sets.

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# Plastics — Production quality control — Statistical method for using single measurements

## 1 Scope

This International Standard describes a statistical, so-called single-measurement, model (SD model) for using single measurements for production quality control purposes at a producer's manufacturing site, even if a standard test method specifies replicate measurements. The statistical model is also applicable to test methods which call for repeated operations. A general approach to the precision statement in test method standards which produce numerical results is also described. The statistical model is only applicable to test methods which give results that follow a normal (i.e. Gaussian) distribution.

The principle of the statistical model is based on the determination of upper and lower production limits, taking into account the accuracy and the reproducibility of the test method, the latter being added to the production limits in order to define an area outside the production limits in which a test result can fall owing to the nature of the production process and/or the test method.

This International Standard is designed for project managers, heads of laboratories and production managers. However, the support of a statistician is highly recommended, and sometimes indispensable, for providing the necessary technical backup and statistical analysis.

If this International Standard is used in combination with a test method standard, this needs to be clearly indicated in all relevant documents, e.g. product specifications, production process specifications and contracts.

## 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 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5725-1 and the following apply.

### 3.1

#### production process standard deviation

$s_p$

standard deviation for the production process

### 3.2

#### production and test method standard deviation

$s_{P\&T}$

combined standard deviation for the production process and test method

### 3.3

#### operator standard deviation

$s_O$   
square root of the operator variance,  $s_O^2$ , where the operator variance is the average of the weighted squares of the individual operator standard deviations

NOTE All operators are assumed to have essentially the same level of variability when following a specified test procedure. This assumption is more likely if the test results are obtained within a short period of time. Therefore, the operator variance can be calculated by averaging the squares of the operator standard deviations. The operator standard deviation can then be calculated as the square root of the operator variance.

### 3.4

#### within-laboratory repeatability standard deviation

$s_{rLab}$   
standard deviation of test results obtained with the same method on identical test items in the same laboratory within a short interval of time by the same operator using the same equipment

### 3.5

#### within-laboratory reproducibility standard deviation

$s_{RLab}$   
standard deviation of test results obtained with the same method on identical test items in the same laboratory by different operators, preferably using different equipment, over a longer period of time

### 3.6

#### $k_w$ -factor

factor, taken from a standard normal distribution table, for determination of the warning limits

### 3.7

#### $k_a$ -factor

factor, taken from a standard normal distribution table, for determination of the acceptance limits

## 4 Precision of test methods

For ISO standard test methods which produce numerical results, the precision of the test method should preferably be determined and stated in the applicable International Standard. The complete description of the statistical method relevant to the determination of the precision of a test method, the relevant definitions, the responsibilities of the different participants and the statistical evaluation are discussed in the various parts of ISO 5725.

Knowledge of ISO 5725 is necessary to carry out a repeatability and reproducibility investigation (RRI), but this mainly concerns the statisticians who normally provide the technical backup for an inter-laboratory test programme and carry out the statistical analysis.

For standardized test methods which produce numerical results, the precision statement for the test method is very important for the user regarding, e.g.

- the precision to be expected from the test method;
- communication between the interested parties;
- comparison with (internal) laboratory performance;
- use as a basis for inter-laboratory comparisons;
- harmonization of test methods between interested parties;
- statistical process control.

A practical guide, specially designed for project leaders and heads of laboratories, to the determination of the precision of a test method is given in Reference [9] (see the Bibliography). However, it should be noted that the support of a statistician is recommended, and sometimes indispensable, for providing the technical backup necessary for the RRI programme and the statistical analysis of the data obtained.

## 5 Single measurements for production quality control purposes

### 5.1 General

Many ISO standards specify that measurements be made twice or even more. In many test method standards, repeated operations are also specified. The reported test result is the average, or another appropriate function such as the median, of the individual observations. The object of the procedures of this International Standard is to use single measurements for production quality control purposes within a producer's manufacturing site in a considered statistical manner, even if a standard test method specifies replicate measurements.

For production quality control, it is common practice to carry out single measurements since, in many production situations, replicate measurements are too time-consuming and expensive and the laboratory response time could also increase unacceptably.

For production quality control purposes, single measurements based on and with reference to this International Standard and statistically validated are permitted when specified by the responsible authority, even if the test method standard specifies replicate measurements.

For test methods where a set of specimens ( $> 2$ ) is considered as giving a single test result, the model can in some cases also be used to reduce the number of specimens in a set. However, it should be noted that, for test methods where a set of specimens is considered as giving a single test result, the number of test specimens is, in most cases, the minimum necessary to obtain a reliable test result, due to the spread in results caused by inhomogeneity in the sample, preparing the test specimens, etc.

Reduction in the variability of a test method might be necessary before a reduction in the number of test replicates can be considered. Therefore, any reduction in the number of test specimens in a set that is considered as giving a single test result shall only be carried out in close cooperation with a statistician.

For test methods which specify repeated procedures or operations carried out on a sample, such as drying to constant mass, the SD method can also be used to reduce the number of operations to possibly only one operation. A statistician should preferably be consulted for the necessary statistical guidance.

The effect on production limits of making duplicate and triplicate measurements as opposed to single measurements is shown by two examples given in Clauses B.2 and B.3 in Annex B.

### 5.2 Variability of the production process and test method

The variability of measurements made on items from a production process consists of the variability of the production process itself and the variability of the test method used to check the production process.

The lower and upper production limits,  $L_{PL}$  and  $U_{PL}$ , which include the variability of the test method, are calculated for a stable production period.

For a non-standard normal distribution production process, the lower and upper production limits shall be calculated by a statistician.

For a standard normal distribution production process, the standard deviation,  $s_{P\&T}$  (the production and test method standard deviation), can be calculated for a stable production period.  $s_{P\&T}$  can also be determined from production data over, for example, a period of one year. However, data from production runs in which irregularities or outliers are known to have occurred shall not be used. For a two-sided precision interval of 99,73 %, the production data will be within the range  $\pm 3s_{P\&T}$  ( $k$ -factor = 3) relative to the average value. For

other levels of precision, the correct  $k$ -factor can be found in tables of the standard normal distribution function. The data should preferably be evaluated by a statistician.

Other methods of determining or specifying the production limits, e.g. by mutual agreement between customer and supplier, may also be used, preferably in close consultation with a statistician.

### 5.3 Within-laboratory reproducibility

The test method needs to be such that it can be used to detect variations in the production process. Therefore, the precision of the test method should be, at most, 30 % of the combined variability of the production process and test method (see 5.2), and preferably less than 10 %. The test method precision necessary will depend on the importance of controlling the production process (see Annex B).

For a standard normal distribution production process, the production and test method standard deviation,  $s_{P\&T}$ , includes the standard deviation for the production process itself and the standard deviation for the test method:

$$s_{P\&T} = s_{\text{production}} + s_{\text{test method}}$$

The precision of the test method is defined here as the within-laboratory reproducibility of the test method. The within-laboratory reproducibility,  $s_{RLab}$ , takes into consideration the variability of the measurements made under the following conditions:

- on the same material;
- by several operators;
- if applicable, using more than one test apparatus;
- at different moments in time.

The within-laboratory reproducibility,  $s_{RLab}$ , of the test method can be determined from a within-laboratory reproducibility investigation as described in 5.4.

Comparison of the repeatability,  $s_r$ , of the test method with the within-laboratory reproducibility,  $s_{RLab}$ , gives insight into the performance of the test laboratory.  $s_r$  can be determined under the repeatability conditions described in Reference [9]. For other calculations, e.g. the calculation of  $s_{RLab}$  from series of measurements in several and/or different production runs, a statistician should be consulted.

### 5.4 Calculation of $s_{RLab}$ from a within-laboratory reproducibility investigation

$s_{RLab}$  can be calculated from a within-laboratory reproducibility investigation as described below.

$$s_{RLab} = \sqrt{s_O^2 + s_{rLab}^2}$$

where

$s_O$  is the operator standard deviation;

$s_{rLab}$  is the within-laboratory repeatability;

and where

$$s_O^2 = \left[ \frac{(T_2 \times T_3) - T_1^2}{T_3 \times (p-1)} - s_{rLab}^2 \right] \times \left[ \frac{T_3 \times (p-1)}{T_3^2 - T_4} \right]$$

and

$$s_{r\text{Lab}}^2 = \frac{T_5}{(T_3 - p)}$$

where

$$T_1 = \sum_1^p (n_i \times \bar{X}_i)$$

$$T_2 = \sum_1^p (n_i \times \bar{X}_i^2)$$

$$T_3 = \sum_1^p n_i$$

$$T_4 = \sum_1^p n_i^2$$

$$T_5 = \sum_1^p (n_i - 1) \times s_{O(i)}^2$$

and

$s_{O(i)}$  is the individual standard deviation for the  $i$ th operator;

$p$  is the number of operators;

$n_i$  is the number of measurements for the  $i$ th operator;

$\bar{X}_i$  is the mean value for the  $i$ th operator.

An example of such a calculation is given in Annex A.

A statistician should be consulted for evaluation of the data if  $s_{r\text{Lab}}^2$  is found to be negative.

**NOTE** An Excel-based tool for the calculation of repeatability and reproducibility parameters has been developed by ISO/TC 61/SC 5 and is expected to become available on the ISO web site for on-line calculations at some future point in time, initially at a password-protected location reserved for those participating in the development of ISO standards. The calculation model is based on that of ISO 5725 and is applicable to balanced and unbalanced data sets. The within-laboratory reproducibility,  $s_{r\text{Lab}}$ , can also be calculated using the same tool, but instead of the “laboratory” data the data from the operators is used.

## 5.5 Interpretation of single-measurement data for production quality control purposes

If the test method is capable of being used to detect variations in the production process, production quality control can be carried out using single measurements based on the following conditions:

- For a non-standard normal distribution production process, the lower and upper production limits,  $L_{\text{PL}}$  and  $U_{\text{PL}}$ , and the lower and upper warning limits,  $L_{\text{WL}}$  and  $U_{\text{WL}}$ , shall be determined by a statistician (see 5.2 and Figure 1).

The method described for determining the production limits by using  $\bar{X} \pm 3s_{\text{P\&T}}$  is commonly used, but other methods of determining the production limits may also be used.

- For a standard normal distribution production process, the production limits,  $L_{\text{P}}$ , for a two-sided precision interval of 99,73 % are given by

$$L_{\text{P}} = \bar{X} \pm 3s_{\text{P\&T}}$$

where

$\bar{X}$  is the mean value of the parameter measured for the stable production process;

$s_{P\&T}$  is the standard deviation for the production process and test method.

The measurement system or the production process can be such that individual measurement values lie outside the production limits. The warning limits,  $L_W$ , for quality control based on single measurements can be determined by using the combined production process and test method standard deviation,  $s_{P\&T}$ , and the within-laboratory reproducibility of the test method,  $s_{RLab}$ , as follows:

$$L_W = \bar{X} \pm (3s_{P\&T} + k_w \times s_{RLab})$$

where  $k_w$  is a constant for determining the warning limits, to be defined by the organization responsible for the production process.

NOTE A  $k_w$ -value of 1,28 represents a single-sided confidence interval of 90 % [see Figures 1 b) and 1 c)]. For other confidence intervals, the constant is taken from a standard normal distribution table.

For measurement values outside the production limits but within the range

$$\bar{X} \pm (3s_{P\&T} + k_w \times s_{RLab})$$

the organization responsible for the production process shall have written procedures on how to handle the product. These procedures could include

- repeating the measurement;
- in the case of batchwise production, carrying out the measurement on a homogenized batch;
- acceptance of the product based on wider production limits.

Measurement values outside the confidence interval  $\bar{X} \pm (3s_{P\&T} + k_w \times s_{RLab})$  are considered to be caused by the production process.

The lower and upper production limits,  $L_{PL}$  and  $U_{PL}$  (corresponding to  $\bar{X} \pm 3s_{P\&T}$ ), based on a two-sided precision interval, include 99,73 % of all production data (see Figure 1).

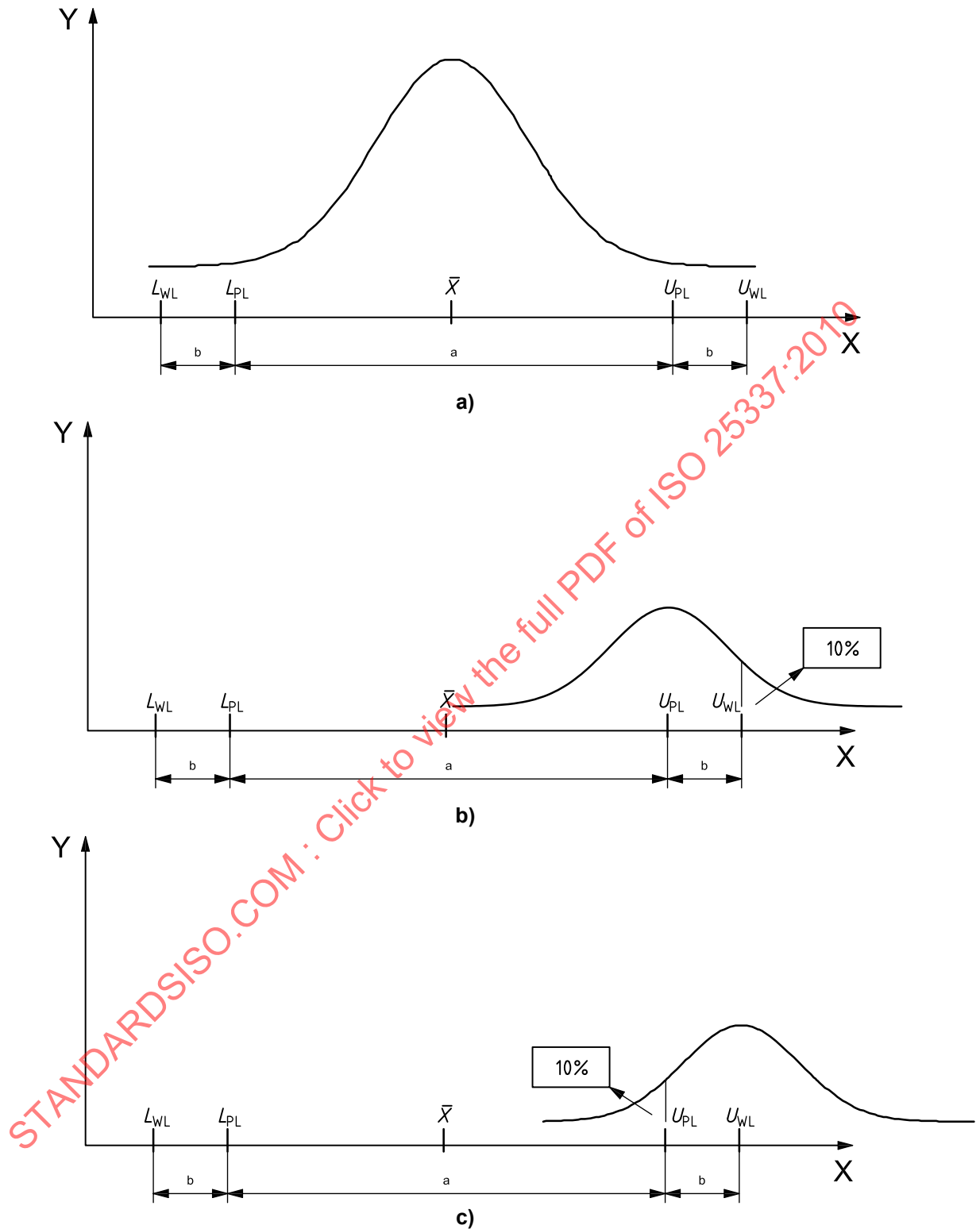
For an absolute real value equal to  $L_{PL}$  or  $U_{PL}$ , there is a probability of, at the most, 10 % that a single measurement value outside the warning limits will result from the measurement system [see Figure 1 b)]. The combined probability for the production process and test method is, at the most, 0,013 5 %. In such a case, the measurement is incorrectly indicated as being outside the warning limits.

For an absolute true value equal to the lower warning limit,  $L_W$ , or the upper warning limit,  $U_W$ , there is a probability of, at the most, 10 % that a single measurement value between  $L_{PL}$  and  $U_{PL}$  will result from the measurement system [see Figure 1 c)]. The combined probability for the production process and test method is, at the most, 0,013 5 %. In such a case, the measurement is incorrectly indicated as being within the production limits.

For an absolute real value between  $L_{PL}$  and  $L_W$  or between  $U_{PL}$  and  $U_W$ , there is a probability decreasing from 50 % to 10 %, starting from the relevant production limit, that a single measurement value within the production limits  $L_{PL}$  and  $U_{PL}$  will result from the measurement system [see Figures 1 b) and 1 c)].

For an absolute true value between the relevant production limit and warning limit, the combined probability for the production process and test method is between 0,065 % and 0,013 %, respectively. In such a case, the measurement is incorrectly indicated as being within the production limits [see Figures 1 b) and 1 c)].

A method of reducing the risk of accepting a nonconforming product is given in Annex C.

**Key**

|          |                                                |           |                                                       |
|----------|------------------------------------------------|-----------|-------------------------------------------------------|
| X        | measurement result                             | $U_{PL}$  | upper production limit                                |
| Y        | frequency of occurrence of a particular result | $U_{WL}$  | upper warning limit                                   |
| $L_{WL}$ | lower warning limit                            | $\bar{X}$ | mean value corresponding to stable production process |
| $L_{PL}$ | lower production limit                         |           |                                                       |
| a        | Production range ( $\bar{X} \pm 3s_{P\&T}$ ).  |           |                                                       |
| b        | Warning area.                                  |           |                                                       |

**Figure 1 — Interpretation of single measurements for product quality control**

## 6 Procedure

When using single measurements, the following procedure shall be followed. For a non-standard normal distribution production process, the procedure shall start at step 5), where the production limits and the warning limits shall be determined by a statistician (5.2, 5.5, Figure 1).

- 1) Determine the production process and test method standard deviation,  $s_{P\&T}$ , from production data over, e.g., a period of one year or a stable production period, using basic statistics. Data from production runs which are known to be affected by irregularities and outliers shall not be used.
- 2) Determine the within-laboratory reproducibility standard deviation, given by

$$s_{RLab} = \sqrt{s_O^2 + s_{RLab}^2},$$

from a within-laboratory reproducibility investigation (see 5.4) or, in consultation with a statistician, from other available production data.

Report the value of  $s_{RLab}$  as a percentage of  $s_{P\&T}$ .

- 3) Determine the production limits:

$$\text{lower production limit } L_{PL} = \bar{X} - 3s_{P\&T}$$

$$\text{upper production limit } U_{PL} = \bar{X} + 3s_{P\&T}$$

Other methods of determining or specifying the production limits, e.g. by mutual agreement between customer and supplier, may also be used, preferably in close consultation with a statistician.

- 4) Determine the warning limits for a one-sided confidence interval of 90 %:

$$\text{lower warning limit } L_{WL} = \bar{X} - 3s_{P\&T} - k_w \times s_{RLab}$$

$$\text{upper warning limit } U_{WL} = \bar{X} + 3s_{P\&T} + k_w \times s_{RLab}$$

Report the confidence interval, if different from 90 %.

A  $k_w$ -value of 1,28 represents a single-sided confidence interval of 90 %. For other levels of precision, the constant is taken from a standard normal distribution table and shall be reported.

- 5) For test methods specifying a number of test specimens greater than two, considered as being a single set, report the reduction in the number of specimens in the set.
- 6) For a measurement result lying between the production limits,  $L_{PL}$  and  $U_{PL}$ , the product shall be considered to be within the defined production limits. The measurement results shall be recorded in the production quality system.
- 7) For a measurement result between the production limits and the warning limits, i.e. between  $L_{PL}$  and  $L_{WL}$  or between  $U_{PL}$  and  $U_{WL}$ , the product shall be considered to be outside the defined production limits (i.e. nonconforming), caused by the measurement system and/or the production process. The organization responsible for the production process shall have written procedures on how to handle such cases and how to determine the reason for the nonconformity. The measurement results, the conclusions and the actions taken shall be recorded in the production quality system.
- 8) For a measurement result outside the warning limits,  $L_{WL}$  and  $U_{WL}$ , the deviation is considered to be caused by the production process. The product shall be rejected and the measurement results shall be recorded in the production quality system.
- 9) The "responsible authority" shall specify the use of the method described in this International Standard in their production quality system. This International Standard shall be referenced in product or process specifications, contracts, inspection instructions and other documents and the provisions set forth herein shall apply. The "responsible authority" shall be designated in one of the above documents.

## Annex A (informative)

### Example of the calculation of $s_{R\text{Lab}}$ from a within-laboratory reproducibility investigation

| Measurement | Operator           |      |      |      |      |      |      |      |   |    |
|-------------|--------------------|------|------|------|------|------|------|------|---|----|
|             | 1                  | 2    | 3    | 4    | 5    | 6    | 7    | 8    | 9 | 10 |
|             | Measurement values |      |      |      |      |      |      |      |   |    |
| 1           | 0,71               | 0,69 | 0,66 | 0,67 | 0,70 | 0,73 | 0,71 | 0,70 |   |    |
| 2           | 0,71               | 0,67 | 0,65 | 0,65 | 0,69 | 0,74 | 0,71 | 0,65 |   |    |
| 3           | 0,70               | 0,68 | 0,65 | 0,66 | 0,66 | 0,73 | 0,69 | 0,68 |   |    |
| 4           | 0,71               |      | 0,69 |      | 0,71 |      | 0,67 | 0,65 |   |    |
| 5           | 0,71               |      | 0,66 |      | 0,69 |      | 0,68 | 0,69 |   |    |
| 6           | 0,70               |      | 0,65 |      |      |      | 0,65 | 0,66 |   |    |
| 7           | 0,71               |      | 0,69 |      |      |      | 0,68 | 0,65 |   |    |
| 8           |                    |      | 0,66 |      |      |      |      |      |   |    |
| 9           |                    |      | 0,65 |      |      |      |      |      |   |    |
| 10          |                    |      | 0,69 |      |      |      |      |      |   |    |
| 11          |                    |      | 0,68 |      |      |      |      |      |   |    |
| 12          |                    |      | 0,65 |      |      |      |      |      |   |    |
| 13          |                    |      | 0,69 |      |      |      |      |      |   |    |
| 14          |                    |      | 0,66 |      |      |      |      |      |   |    |
| 15          |                    |      |      |      |      |      |      |      |   |    |

| Operator details                          |         |         |         |         |         |         |         |         |  |  |
|-------------------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|--|--|
| No. of measurements, $n_i$                | 7       | 3       | 14      | 3       | 5       | 3       | 7       | 7       |  |  |
| Mean value, $\bar{X}_i$                   | 0,71    | 0,68    | 0,67    | 0,66    | 0,69    | 0,73    | 0,68    | 0,67    |  |  |
| $n_i \times \bar{X}_i$                    | 4,95    | 2,04    | 9,33    | 1,98    | 3,45    | 2,20    | 4,79    | 4,68    |  |  |
| $n_i \times \bar{X}_i^2$                  | 3,50    | 1,39    | 6,22    | 1,31    | 2,38    | 1,61    | 3,28    | 3,13    |  |  |
| $s_{\text{Operator}(i)}, s_{\text{O}(i)}$ | 0,005   | 0,010   | 0,017   | 0,010   | 0,019   | 0,006   | 0,021   | 0,021   |  |  |
| $(n_i - 1) \times s_{\text{O}(i)}^2$      | 0,000 1 | 0,000 2 | 0,003 9 | 0,000 2 | 0,001 4 | 0,000 1 | 0,002 8 | 0,002 7 |  |  |
| No. of operators, $p_i$                   | 1       | 1       | 1       | 1       | 1       | 1       | 1       | 1       |  |  |

| Parameter calculations                             |             |
|----------------------------------------------------|-------------|
| $T_1 = \Sigma(n_i \times \bar{X}_i)$               | 33,42       |
| $T_2 = \Sigma(n_i \times \bar{X}_i^2)$             | 22,812 612  |
| $T_3 = \Sigma n_i$                                 | 49          |
| $T_4 = \Sigma n_i^2$                               | 395         |
| $T_5 = \Sigma[(n_i - 1) \times s_{\text{O}(i)}^2]$ | 0,011 388 1 |
| $\bar{X} = T_1/T_3$                                | 0,682 040 8 |
| $p = \Sigma p_i$                                   | 8           |

| Repeatability and reproducibility report                    |                                     | ISO 5725 method |
|-------------------------------------------------------------|-------------------------------------|-----------------|
| Within-laboratory repeatability standard deviation          | $s_{r\text{Lab}}$                   | 0,017           |
| Operator standard deviation                                 | $s_{\text{O}}$                      | 0,020           |
| <b>Within-laboratory reproducibility standard deviation</b> | <b><math>s_{R\text{Lab}}</math></b> | <b>0,026</b>    |
| Overall mean value                                          | $\bar{X}$                           | 0,68            |

**Formulae:**

$$s_O^2 = \left\{ \left[ (T_2 \times T_3) - T_1^2 \right] / \left[ T_3 \times (p-1) \right] - s_{r\text{Lab}}^2 \right\} \times \left\{ \left[ T_3 \times (p-1) \right] / (T_3^2 - T_4) \right\}$$

$$s_{r\text{Lab}}^2 = T_5 / (T_3 - p)$$

**Remark 1:**

A problem might be encountered in calculating  $s_R$  and/or  $s_r$  if it is necessary to take the square root of a negative number. Where variance component estimates are involved, negative variances can occur. They are most probably caused by one of the following reasons:

- the variability in the data might be large enough to produce a negative estimate, even though the true value of the variance component is positive;
- the data contain outliers;
- a different model for interpreting the data might be more appropriate (with some statistical models for variance component analysis, negative estimates are an indication that some of the data is negatively correlated).

In such cases, a statistician should be consulted to assist in evaluation of the data.

**Remark 2:**

If there is only one measurement for an operator, this data point should be discarded since it would give a value for  $(n_i - 1)$ , and hence also for  $T_5$ , of zero.

## Annex B (informative)

### Examples of the effect on production limits of making duplicate and triplicate measurements as opposed to single measurements

#### B.1 General

Examples of the effect of making duplicate and triplicate measurements compared to single measurements are given below and illustrated in Figures B.1 and B.2.

As shown in the examples, the effect on the production limits of duplicate and triplicate measurements is significant for a test method with a standard deviation which is large in comparison with the standard deviation for the production process. However, for a test method with a standard deviation of  $\leq 30$  % of the standard deviation for the production process, the effect on the production limits is only small.

#### B.2 Determination of glass content when standard deviation for test method is greater than that for production process

|                                                             |        |
|-------------------------------------------------------------|--------|
| — Mean value of glass content                               | 33 %   |
| — Production standard deviation, $s_P$                      | 0,68 % |
| — Test method standard deviation, $s_{RLab}$                | 0,86 % |
| — Production and test method standard deviation, $s_{P\&T}$ | 1,10 % |

$$s_{P\&T} = \sqrt{s_P^2 + s_{RLab}^2}$$

For a confidence interval of 99,73 %, based on the combined standard deviation for the production process and the test method, the production and test method limits are calculated using the formula

$$3 \times \sqrt{s_P^2 + (s_{RLab}/\sqrt{n})^2}$$

Single measurements:  $3 \times \sqrt{s_P^2 + (s_{RLab}/\sqrt{1})^2} = \pm 3 \times 1,10 \% = \pm 3,30$

Duplicate measurements:  $3 \times \sqrt{s_P^2 + (s_{RLab}/\sqrt{2})^2} = \pm 3 \times 0,91 \% = \pm 2,74$

Triplicate measurements:  $3 \times \sqrt{s_P^2 + (s_{RLab}/\sqrt{3})^2} = \pm 3 \times 0,84 \% = \pm 2,53$

#### B.3 Determination of glass content when standard deviation for test method is less than $0,3 \times$ that for production process

|                                                             |        |
|-------------------------------------------------------------|--------|
| — Mean value of glass content                               | 33 %   |
| — Production standard deviation, $s_P$                      | 1,05 % |
| — Test method standard deviation, $s_{RLab}$                | 0,33 % |
| — Production and test method standard deviation, $s_{P\&T}$ | 1,10 % |

For a confidence interval of 99,73 %, based on the combined standard deviation for the production process and the test method, the production and test method limits are calculated, using the formula given in Clause B.2, as follows:

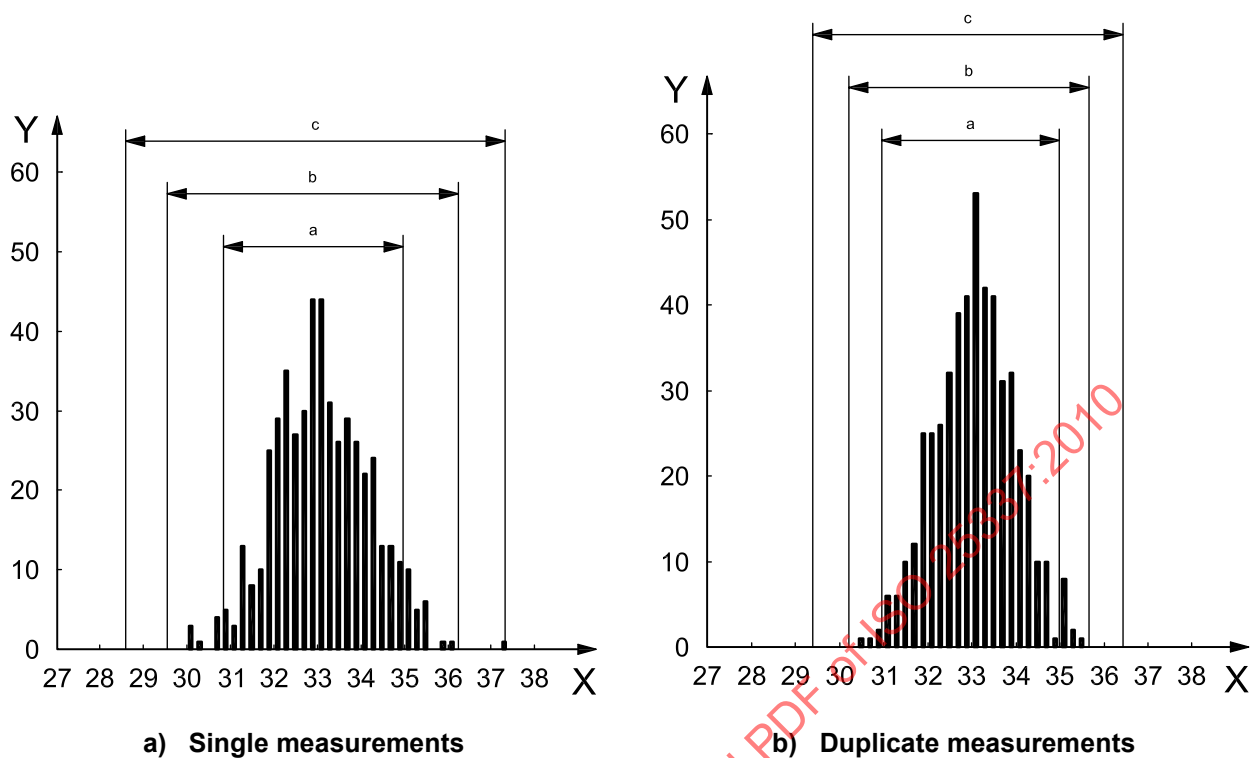
Single measurements:  $3 \times \sqrt{[s_P^2 + (s_{RLab}/\sqrt{1})^2]} = \pm 3 \times 1,10 \% = \pm 3,30$

Duplicate measurements:  $3 \times \sqrt{[s_P^2 + (s_{RLab}/\sqrt{2})^2]} = \pm 3 \times 1,08 \% = \pm 3,24$

Triplicate measurements:  $3 \times \sqrt{[s_P^2 + (s_{RLab}/\sqrt{3})^2]} = \pm 3 \times 1,07 \% = \pm 3,21$

**Table B.1 — Comparison of the effect on the production limits of making single, duplicate and triplicate measurements**

| Measurements | Production and test method limits<br>$\pm 3 \times \sqrt{[s_P^2 + (s_{RLab}/\sqrt{n})^2]}$ | Warning limits<br>$\pm(3s_{P\&T} + 1,3s_{RLab})$ | Production and test method limits<br>$\pm 3 \times \sqrt{[s_P^2 + (s_{RLab}/\sqrt{n})^2]}$ | Warning limits<br>$\pm(3s_{P\&T} + 1,3s_{RLab})$ |
|--------------|--------------------------------------------------------------------------------------------|--------------------------------------------------|--------------------------------------------------------------------------------------------|--------------------------------------------------|
|              | $s_{P\&T} = 1,10 \%$<br>$s_P = 0,68$<br>$s_{RLab} = 0,86 \%$                               |                                                  | $s_{P\&T} = 1,10 \%$<br>$s_P = 1,05$<br>$s_{RLab} = 0,33 \%$                               |                                                  |
| Single       | $\pm 3,30$                                                                                 | 4,41                                             | $\pm 3,30$                                                                                 | 3,73                                             |
| Duplicate    | $\pm 2,74$                                                                                 | 3,85                                             | $\pm 3,24$                                                                                 | 3,66                                             |
| Triplicate   | $\pm 2,53$                                                                                 | 3,64                                             | $\pm 3,21$                                                                                 | 3,63                                             |

**Key**

X glass content (%)

Y number of determinations giving a particular glass content

a Production limits.

b Production and test method limits.

c Warning limits.

**Single measurements**

Mean glass content: 33,00 %

Production and test method standard deviation,  $s_{P\&T}$ : 1,10

Test method standard deviation: 0,86

Number of determinations: 500

**Figure B.1 — Determination of glass content — Comparison of effect on production limits of making single rather than duplicate measurements ( $s_{R\text{Lab}}/s_{P\&T} = 0,78$ )**