BS EN 16123:2013

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Characterization of waste — Guidance on selection and application of screening methods

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National foreword

This British Standard is the UK implementation of EN 16123:2013.

The UK participation in its preparation was entrusted to Technical Committee B/508/3, Characterization of waste.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

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Characterization of waste - Guidance on selection and application of screening methods

Caractérisation des déchets - Lignes directrices relatives au choix et à l'application des méthodes de dépistage

Charakterisierung von Abfall - Anleitung für Auswahl und Anwendung von Screening-Verfahren

This European Standard was approved by CEN on 7 December 2012.

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Foreword

This document (EN 16123:2013) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2013, and conflicting national standards shall be withdrawn at the latest by August 2013.

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

The standardization of the selection and application of screening methods is obligatory to make such methods available to legislation. In addition to common laboratory methods, which are standardized individually, a framework approach is chosen for screening methods. The application of screening methods within this framework guarantees the reliability of results required by legal regulations.

In order to fulfil legal requirements, it is of high importance to document all decision steps of method selection, the applicability testing, the application of the method and the evaluation.

This European Standard refers to the use of screening methods on solid materials (waste) as sample matrix; the corresponding standard for water is ISO 17381.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This document provides guidance on the use of screening methods for waste characterisation. One field of application of screening methods is “on-site verification” as recommended in the Landfill Directive (1999/31/EC) and the Landfill Decision (2003/33/EC).

Screening methods are of increasing interest in processes such as entrance control because, in addition to standardized methods, they allow fast verification of the documented waste characteristics.

1 Scope

This European Standard gives guidance on the selection and application of screening methods for waste characterisation. The aim of this document is to set up criteria as to when the different kinds of screening methods may be applied for the analysis of a certain parameter in waste and which steps are required to prove their suitability.

This document does not recommend any particular screening method, but confirms the principles of its selection and application.

2 Normative references

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

EN 16192, *Characterization of waste — Analysis of eluates*

3 Terms and definitions

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

3.1
reference method
method which is performed in accordance with national or international Standards and is not necessarily comparable with screening methods

3.2
on-site verification
third level of inspection according to the Landfill Directive and the Landfill Decision to ensure that the waste accepted at a landfill is the same as described in the accompanying documents and that it is in accordance with the basic characterisation and/or compliance testing

3.3
screening
application of any analytical semi-quantitative method for exploratory analysis

3.4
screening method
method which is used (often on-site) to quickly explore a given area or test a set of samples and obtain data on sample characteristics

[SOURCE: ISO 12404, modified]

4 Principles

This standard defines the whole process from the selection of the screening method, applicability, fit-for-purpose testing, fulfilment of the acceptance criteria, quality control, and documentation of the measurement results.

5 Typical areas for application of screening methods

5.1 General

Screening methods constitute a useful addition to standard procedures in the following areas.

5.2 Support of sampling/sample preparation processes

Screening methods may be used for:

— selection of the most suitable analytical method (concentration range, interferences);

— pre-selection of samples for analysis in the laboratory;

— provision of information about accompanying compounds relevant for sample preparation.

5.3 On-site verification

Characteristics of sampled waste are verified, e.g. during transport or at the entrance of waste treatment plants and landfills.

5.4 Monitoring of processes

Screening methods can be used:

— to monitor and control processes (e.g. success of treatment processes);

— to perform quality control on a treatment plant.

5.5 Identification of homogeneity/heterogeneity of bulk material

Screening methods may be applied to measure “leading” parameters in huge charges of waste to identify whether the material is homogenous or not.

5.6 Survey of contaminated sites (hot spot identification)

Screening methods are useful to identify contaminated areas in contamination-suspected sites.

5.7 Identification of sources of contamination

Screening methods can be useful to identify the source of a contaminant or content in a material stream.

5.8 Safety issue

Screening methods can be used to detect potentially toxic compounds (e.g. gases, radioactivity, explosives) which may be hazardous to the personnel taking and processing samples.

6 Selecting a screening method

6.1 Selection criteria

The following selection criteria should be taken into consideration when selecting the appropriate method. The different criteria shall be weighted depending on the intended application. The decision-making process and the results have to be documented by the user (see flowchart in Annex A and documentation aid in Annex B).

Prerequisites are:

— one known parameter or a set of known parameters;

— aim of determination;

— matrix (solid/liquid waste).

6.1.1 Sampling/sample pretreatment/preparation

— direct measurement (e.g. handheld XRF-systems);

— pretreatment/preparation (e.g. extractions, separation) particularly for solid waste.

Most screening methods require the provision of the analyte in an extract/eluate, which therefore requires sample pretreatment. Pretreatment shall be carried out in accordance with EN 16192.

If sample preparation is necessary, follow the principles of EN 15002 taking into account CEN/TR 16130.

6.1.2 Parameter definition

— individual species (e.g. total Fe, Fe^{2+}, Fe^{3+});

— group parameters (e.g. total organic carbon (TOC), absorbable organically bound halogens (AOX));

— in the case of on-site verification, the parameters are typically defined by declaration or based on the experience of the staff.

6.1.3 Field of application

— specified decision value (e.g. limit value, target value);

— concentration range;

— matrix;

— method limitations/interferences.

6.1.4 Boundary conditions

— rapidity (in relation to aim of determination);

— mobility;

— costs;

— quality target of analysis;

— frequency of use (continuous, once only);

— qualification of staff;

— legal stipulations;

— availability and/or ease of acquisition;

— infrastructural conditions.

6.2 Fit-for-purpose test

In a second step, after passing these selection steps in 6.1, the selected method has to pass a fit-for-purpose test as described in Clause 8.

In case of frequently repeated tasks, the most suitable screening method should be identified and applied, the necessary equipment kept ready and the procedure documented in a standard operation procedure. Selection and fit-for-purpose testing has therefore to be performed only once.

6.3 Quality targets

The general quality target of analytical questions is its ability to establish the relationship between the analytical result and its confidence interval on the one hand, and the decision values on the other. This relationship with the decision values means that the analytical method to be used is subject to requirements regarding the quality of the analytical results. These requirements are task-related and shall be defined before the screening method is applied. The definition of these quality targets forms the basis for the selection of the appropriate method.

7 Applicability conditions for screening methods

7.1 General

Availability of complete information on a screening method of choice is a prerequisite of applicability. This clause deals with the most important points that should be apparent from the accompanying documentation of a method. All information, either supplied or separately obtainable (enclosed leaflet, application documents, etc.) shall be easily comprehensible and should be written in understandable language.

7.2 Field of application

— parameters (e.g. total Fe, Fe^{2+}, Fe^{3+});

— measurement range/graduation; “zero” may not be stated for the lower limit of the operating range;

— matrix;

— matrix interferences, measures to be taken for their prevention or elimination;

— temperature range, pH range, other physical conditions;

— storage and shelf life of the reagents.

7.2.1 Principle of the measurement

— chemical reaction or physical concept.

7.2.2 Instruction for method setup

— description of supplied reagents (e.g. composition, indication of hazards);

— description of supplied equipment, such as test vessel, metering device or colour scale;

— description of how and with which measuring instrument the evaluation may be performed;

— additional reagents required for the application (e.g. acid for pH adjustment);

— additional equipment required for the application (e.g. thermo reactor for chemical oxygen demand).

7.2.3 Sampling and samples

— description of sampling and of sample preparation;

— description of sample quantity and volume.

7.2.4 Measurement steps

— health and safety precautions;

— handling; step-by-step (pictogram), introduction, training;

— reaction time (interval);

— ascertainment of results;

— cleaning and maintenance instructions.

7.2.5 Statement of results

— number of digits after the decimal point;

— precision/accuracy;

— conversion table, conversion factors;

— recommended methods for assessment of results.

7.2.6 Disposal instructions

— waste, waste water, hazardous waste;

— return to the manufacturer.

7.2.7 Characteristic data of the method

— calibration;

— available certificate of analysis; reference to products for quality assurance by the user (control standard, inter-laboratory tests).

7.2.8 Literature references

— description of procedure;

— additional information, examples of possible applications.

8 Fit-for-purpose testing

8.1 General

In general, fit-for-purpose testing means proving whether a method of choice provides results that are related to the corresponding reference method. The intensity and type of fit-for-purpose testing depends on the quality targets defined according to 6.3 and the type of screening technique used.

Three modules of testing may be applicable:

— reproducibility testing;

— exclusion of false negative results;

— testing of individual comparability.

These three modules can be combined depending on the quality target. Reproducibility testing is always required the first time a special test application is introduced.

Reproducibility testing can easily be combined with the testing of individual comparability.

Where the producer (or user) of the screening test publishes data of successful fit-for-purpose tests under comparable conditions, these data may be referred to and may reduce the effort of the current test.

Where screening methods which deliver a concentration range or yes/no-results, instead of discrete values only, reproducibility testing and evaluation of false negatives applies.

8.2 Reproducibility testing

One (or more) typical homogenised waste sample(s), containing a known amount of analyte (verified by use of the reference method) is (are) analysed in six replicates, i.e. six complete approaches, by use of the screening method. The data set is evaluated according to the quality target. The result of this testing can be used to express the precision of the method.

In case of screening methods that only provide a concentration range or yes/no-results, the test should deliver information whether the six replicates of the screening method always results in the same range or the same yes/no-information.

8.3 Exclusion of false negative results

In many cases of application, the screening method is used to pre-select samples. In these cases, it is important that the screening method does not give false negative results. False positives are not critical as in these cases a control by reference method delivers clarification.

In order to test the probability of false negative results, a test scenario according to the quality targets has to be designed.

Firstly, the precision has to be defined or derived from the actual precision (e.g. based on 8.2).

A set of typical samples covering the expected range of application is prepared (homogenised) and characterised by the reference method. All samples are also analysed by use of the screening method.

The data are evaluated under consideration of the precision. The probability of false negatives is calculated.

The number of samples analysed depends on the required accuracy of the test. Ten typical samples are considered the minimum.

In case of screening methods that only give concentration range or yes/no-results, the test should deliver information regarding the number of false negatives of repeated measurements of the screening method.

8.4 Testing of individual comparability

One (or more) typical homogenised samples are prepared and analysed both by the screening method and the reference method (six replicates).

The results are statistically evaluated according to the statistical procedure described in Annex C.

9 Analytical acceptance criteria

9.1 General

After a screening method has been proven to meet the given acceptance criteria, it may be used for the defined purpose. Some of these criteria are to be checked before using the test (starting criteria); others have to be continuously checked during the use of the method.

9.2 Starting criteria

— Selection process according to Clause 6 successfully completed and documented.

— Application criteria according to Clause 7 successfully evaluated and documented.

— Quality targets defined according to analytical task and documented.

— Individual fit-for-purpose scenario designed, successfully passed and documented.

— Quality assurance measures and corresponding quality acceptance criteria defined (Clause 10) and documented.

9.3 Continuous criteria

— Continuous monitoring of quality acceptance criteria completed and documented.

In case of deviations from the quality criteria, measures have to be taken and documented. Use of method may continue after re-covering quality criteria.

10 Quality assurance

Similar to routine procedures used in the laboratory, quality assurance of screening methods is subject to various requirements, depending on the handling and nature of the method in question. In case of a method used regularly (e.g. more than once per week), the quality assurance measures of the reference method may be used.

These may include:

— multiple testing;

— measurements of standards and possible reference materials;

— plausibility tests by standard addition;

— comparative tests with reference methods;

— interlaboratory tests.

Finally, the selection of suitable quality assurance measures depends upon the specific aim of the analysis. The decision concerning the extent of the measures to be implemented, the results of these measures and their assessment shall be documented.

11 Documentation

The application of this European Standard enables a qualified decision to be made regarding the most appropriate method of analysis for the task at hand. At the same time, however, the decision-making process shall be transparent and verifiable. For this reason, thorough documentation is especially important, starting at the beginning of the test and lasting until assessment of the analytical results. Systematic documentation provides objective proof of the quality of analysis.

The minimum requirements for documentation include:

— presentation of decision criteria in accordance with Clause 6;

— documentation of the qualifications of decision makers and personnel performing analysis;

— documentation of individual quality assurance measures;

— documentation of continuous quality assurance measures;

— sampling record;

— written report of the analysis, including:

  — indication of measured values with clear identification of samples;

  — indication of the equipment used;

  — deviations from the operating procedure, if applicable;

  — assessment of results;

  — pretreatment.

Annex A
(informative)

Decision making process

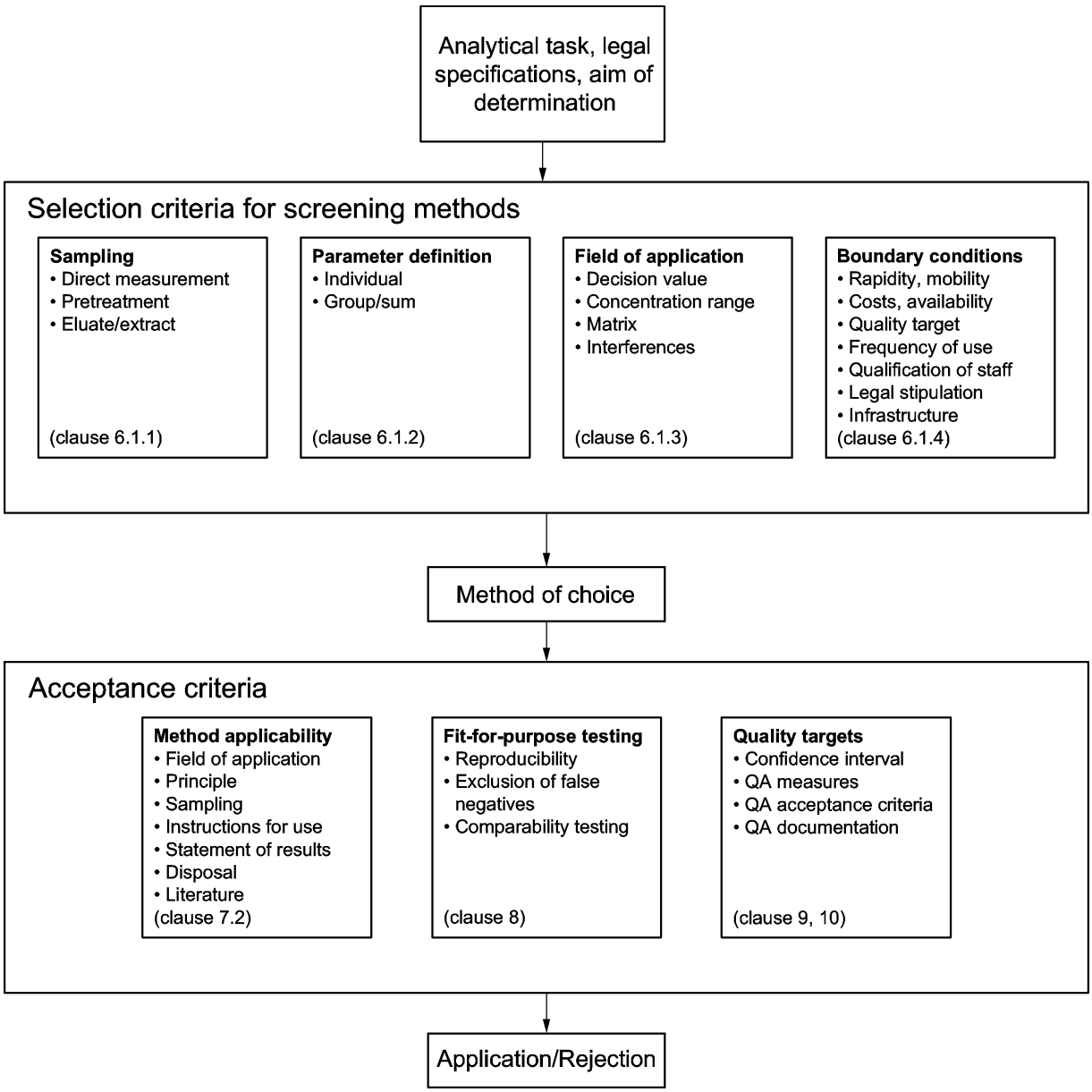

Figure A.1 — Flow chart

Annex B
(informative)

Example of documentation aid/check list

Short description of analytical task: **Definition of the quality target:**

Selection criteria

Parameter definition

Relevance please tick	Aspect	Applicability to method of choice
☐	Specified analyte:	☐
☐	Group or sum parameter:	☐

Field of Application

☐	Decision value:	☐
☐	Concentration range:	☐
☐	Matrix:	☐
☐	Expected interferences:	☐
☐	Method limitations:	☐

Sampling, sample pretreatment

☐	Direct measurement:	☐
☐	Sampling required:	☐
☐	Sample pretreatment required:	☐
☐	Eluate/Extract required:	☐

Boundary conditions

☐	Rapidity (in relation to aim of determination)	☐
☐	Mobility	☐
☐	Costs	☐
☐	Quality target of analysis	☐
☐	Frequency of use (continuous, once only)	☐
☐	Qualification of staff	☐
☐	Legal stipulations	☐
☐	Availability and/or ease of acquisition	☐
☐	Infrastructural conditions	☐
☐	Others:	☐

Applicability conditions

relevant	condition	applied
☐	Temperature range, pH range, other physical conditions	☐
☐	Storage and shelf life of the reagents	☐
☐	Chemical reaction or physical concept	☐
☐	Description of supplied reagents (e.g. composition, indications of danger)	☐
☐	Description of supplied equipment, such as test vessel, metering device or colour scale	☐
☐	Description of how and with which measuring instrument the evaluation may be performed	☐
☐	Additional reagents required for the application (e.g. acid for pH adjustment)	☐
☐	Additional equipment required for the application	☐
☐	Description of sampling and of sample preparation	☐
☐	Description of sample quantity	☐
☐	Health and safety precautions	☐
☐	Step-by-step (pictogram), introduction, training	☐
☐	Reaction time (interval)	☐
☐	Ascertainment of results	☐
☐	Maintenance instructions	☐
☐	Number of figures after decimal point	☐
☐	Precision/accuracy	☐
☐	Conversion table, conversion factors	☐
☐	Recommended methods for assessment of results	☐
☐	Waste, waste water, hazardous waste	☐
☐	Return to the manufacturer	☐
☐	Calibration	☐
☐	Available certificate of analysis	☐
☐	Description of procedure	☐
☐	Additional information, examples of possible applications	☐

Fit-for-purpose testing

Relevance please tick	Test type	Result summary	Test passed
☐	Reproducibility		☐
☐	False negatives		☐
☐	Comparability		☐

Quality Assurance measures

Apply	Measure	Define measure and quality target:
☐	Measurements of standards and possible reference materials	
☐	Plausibility tests by standard addition	
☐	Comparative tests with reference methods	
☐	Inter-laboratory tests	
☐		
☐		

Acceptance criteria

☐ Selection process according Clause 6 successfully completed

☐ Application criteria according to Clause 7 successfully evaluated

☐ Quality targets defined according to analytical task

☐ Individual fit-for-purpose scenario designed, successfully passed

☐ Quality assurance measures and corresponding quality acceptance criteria defined (Clause 12)

Results:

Name of responsible person: _____

Date: _____ **Signature:** _____

Annex C
(informative)

Statistical tool for individual comparability – Equality of results from reference method and screening method: Mean value t-test for real samples

C.1 General

At least six sub samples of a representative homogenised material are analysed with both methods to be compared under the same conditions.

C.2 Test on outliers

The results of both series are checked according to Grubbs test on outliers. One outlier per series is accepted. The test value (*TV*) according to Grubbs is calculated as follows (Formula C.1):

$$TV = \frac{\left| X^{*} - \overline{X} \right|}{S} \qquad \text{(C.1)}$$

where:

TV is the test value;

*X** is the single value (possible outlier);

$\overline{X}$ is the mean value of a series;

s is the standard deviation of a series.

The test value is compared with the tabled value rM (*f* = *N*; *P* = 95 %). Where the test value is higher than the tabled value, the individual result under observation has been identified as outlier – and has to be eliminated accordingly. Mean value and standard deviation are calculated by the remaining data again and the Grubbs test is repeated. In case of detection of another outlier, the data series is not valid and a new set of data has to be established (minimum number of valid data: 5).

C.3 Homogeneity of variances

The variances of both data series are checked according to the variances-F-test.

The test value is calculated according to Formula C.2:

$$TV = \frac{\left(s_{\mathrm{S}}\right)^2}{\left(s_{\mathrm{R}}\right)^2} \qquad \text{(C.2)}$$

where:

TV is the test value;

s_S is the standard deviation of the screening series;

s_R is the standard deviation of the reference series.

The result is compared with the corresponding tabled value for F-distribution ($f_v = N_v - 1$, $f_R = N_R - 1$; $P = 99$ %).

Where the test value is higher than the tabled value a significant difference is proven. According to the current variations, no comparability is found. Where the test value is lower or equal to the tabled value no significant difference is found; i.e. the variation of the results is equal.

NOTE Where the variance of the screening method is significantly lower than the variance of the reference method the F-test is successfully passed.

C.4 Mean value t-test

After elimination of outliers the mean values of both data series are compared according to the mean value t-test. The test value is checked by use of Formulae C.3 and C.4:

$$TV = \frac{\left|\overline{X}_R - \overline{X}_S\right|}{s_d} \cdot \sqrt{\frac{N_R \cdot N_S}{N_R + N_S}} \quad \text{(C.3)}$$

and

$$s_d = \sqrt{\frac{(N_R - 1)\cdot s_R^2 + (N_S - 1)\cdot s_S^2}{N_R + N_S - 2}} \quad \text{(C.4)}$$

where

TV is the test value;

$\overline{X}_R$ is the mean value of reference series;

$\overline{X}_S$ is the mean value of screening series;

N_R is the number of data in reference series;

N_S is the number of data in screening series;

s_d is the mean standard deviation from both series;

s_R is the standard deviation of the reference series;

s_S is the standard deviation of the screening series.

The mean standard deviation from both series is compared to the tabled value of the t-distribution ($f = N_R + N_S - 2$; $P = 99$ %).

Where the test value is higher than the tabled value, no equality of the compared data sets is given. Where the test value is lower or equal to the tabled value, the result of the test is equality. Where the variation of the screening method is significantly lower than the variation of the reference method it is allowed to use s_R of the reference method instead of s_d in Formula C.3. Otherwise, a false non-equality may result. Depending on the

quality target of the analytical task, the user may be willing to change probabilities of the tests mentioned above. In case of deviations, the argumentation has to be plausible and well documented.

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