SLOVENSKI STANDARD
SIST ISO 12787:2012

01-april-2012

Kozmetika - Analizne metode - Merila za validacijo analiznih rezultatov z uporabo kromatografskih tehnik

Cosmetics - Analytical methods - Validation criteria for analytical results using chromatographic techniques

Cosmétiques - Méthodes analytiques - Critères de validation pour les résultats analytiques utilisant des techniques chromatographiques

Ta slovenski standard je istoveten z: **ISO 12787:2011**

ICS:

71.100.70	Kozmetika. Toaletni pripomočki	Cosmetics. Toiletries

SIST ISO 12787:2012 **en,fr**

INTERNATIONAL STANDARD

ISO 12787

First edition
2011-12-15

Cosmetics — Analytical methods — Validation criteria for analytical results using chromatographic techniques

Cosmétiques — Méthodes analytiques — Critères de validation pour les résultats analytiques utilisant des techniques chromatographiques

Reference number
ISO 12787:2011(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

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ISO 12787 was prepared by Technical Committee ISO/TC 217, *Cosmetics*.

Cosmetics — Analytical methods — Validation criteria for analytical results using chromatographic techniques

1 Scope

This International Standard defines validation criteria with which analytical results obtained from the analysis of cosmetic products should comply in order to give confidence in performance, reliability and quality of the final result. It sets out an analytical approach that can be used by a single laboratory to carry out chromatographic analyses on a given sample, or samples.

2 Terms and definitions

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

2.1 General

2.1.1
analyte
substance being subjected to analysis

2.1.2
bias
difference between the expectation of the test results and an accepted reference value

2.1.3
recovery
ratio between the quantity of analyte found by a particular analytical method compared to the quantity of analyte expected

2.1.4
post-extraction spiked matrix standards
PoEMS
samples taken through the entire extraction procedure and spiked with the analyte of interest at the end of the extraction immediately before, or very close to, detection

NOTE PoEMS are also called “Matrix-Matched Standards” or “Fortified Analytical Solutions (FAS)” and are used for determination of the bias.

2.1.5
pre-extraction spiked matrix standards
PrEMS
samples spiked with the analyte of interest at the beginning of the analytical procedure

NOTE PrEMS are also called “Spikes” or “Fortified Analytical Portions (FAP)” and are used for calibration and quantification of the target analytes in samples (extraction recovery).

2.1.6
matrix effect
combined effect of the presence of one or more components of a sample other than the analyte on the measured quantity of the analyte

NOTE The matrix effect could increase or decrease the chromatographic peak area for a same analyte concentration.

2.1.7
extraction yield
ratio between the quantity of analyte extracted during the extraction process from the sample matrix compared to the quantity of analyte present in the sample

2.1.8
solvent standard calibration curve
analyte calibration curve obtained from the analyses of at least five different standard calibration levels prepared in the solvent

2.1.9
control standard
independent standard solution used to verify the solvent standard calibration curve

2.2 Terms relating to validation criteria for analytical results

2.2.1
accuracy
closeness of agreement between a test result (the average value obtained from a large series of test results) and an accepted reference value

NOTE The accuracy is often expressed in terms of bias.

2.2.2
LoD
limit of detection
lowest amount of an analyte that can be reliably distinguished from zero with reasonable statistical certainty

2.2.3
LoQ
limit of quantification
lowest amount of an analyte that can be determined with an acceptable level of uncertainty under the stated conditions of test

2.2.4
linearity
ability of the method to obtain test results proportional to the concentration of the analyte

2.2.5
measurement uncertainty
MU
parameter, associated with the result of a measurement, that characterizes the dispersion of values that could be reasonably attributed to the measurand

2.2.6
precision
closeness of agreement between independent test results obtained under stipulated conditions

NOTE Precision depends only on the distribution of random errors and does not relate to the true value or the specified value.

2.2.7
working range
interval between the upper and lower concentration (amounts) of analyte in the sample for which it has been demonstrated that the analytical procedure has a suitable level of certainty

2.2.8
repeatability
precision under repeatability conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time

2.2.9
intermediate precision
precision under conditions where independent test results are obtained with the same method on identical test items in the same laboratory by different operators using different equipment on different days

2.2.10
reproducibility
precision under reproducibility conditions, i.e. conditions where independent test results are obtained with the same method on identical test items from different laboratories at different times

2.2.11
selectivity
ability of a method to determine accurately and specifically the analyte of interest in the presence of other components in a sample matrix under the stated conditions of the test

2.2.12
sensitivity
change in the response of a measuring instrument divided by the corresponding change in the stimulus

2.2.13
specificity
ability of a method to measure only what is intended to be measured

2.2.14
target concentration
analyte concentration used as a reference for the determination of the analyte concentration in the sample

2.2.15
validation
confirmation of examination and provision of objective evidence that the particular requirements for a specified intended use are met

2.2.16
asymmetry
factor describing the shape of a chromatographic peak

NOTE The theory assumes a Gaussian shape and that peaks are symmetrical.

2.2.17
resolution
ability of a column to separate chromatographic peaks, usually expressed in terms of the separation of two peaks

3 Principle

The ingredients of cosmetic products are variable and complex, mainly due to the type of formulation. General analytical methods exist, or are to be developed, to assess the quality of cosmetics. These generalized methods, some of which might not be strictly certifiable, are intended to be widely usable, comprehensible and transferable.

The application of analytical methods to cosmetic products requires a specific validation approach in order to ensure the reliability of the results. For cosmetic products, the choice and use of a general method for analytical testing has to be supported by validation criteria specific to the sample matrix in order to ensure the reliability of the results. In this context, this International Standard aims to propose specific validation criteria to be evaluated for the use of a general method for testing cosmetic products. Validation criteria for analytical results to be evaluated include specificity, selectivity, recovery, confidence interval, limit of detection, limit of quantification, precision, accuracy and linearity.

Validation criteria shall be determined for each sample matrix. If a similar matrix is used, validation criteria need only be determined on the samples first analysed and extended to other samples in the same concentration range. Accordingly, this approach would not necessarily be applied in routine testing of cosmetic products if validation criteria were previously obtained. Careful consideration should be given to the sample matrix when determining if additional validation is required.

4 General information

4.1 Matrix effect

If the sample were submitted to an extraction process before injection (e.g. liquid-liquid extraction or solid-phase extraction), the recovery obtained on the PrEMS, using the solvent calibration curve, would include both the sample matrix effect and the extraction yield of the process.

From an analytical point of view, it would be interesting to distinguish the matrix effect from the extraction yield resulting from the sample preparation (extraction of the analyte from the cosmetic matrix). Use of a PoEMS would allow one to distinguish between the matrix effect and the extraction yield.

Figure 1 indicates the importance of preparing a PoEMS, in addition to a PrEMS and a standard calibration curve, in order to obtain different validation criteria on the analytical results, such as the extraction yield and/or the matrix effect.

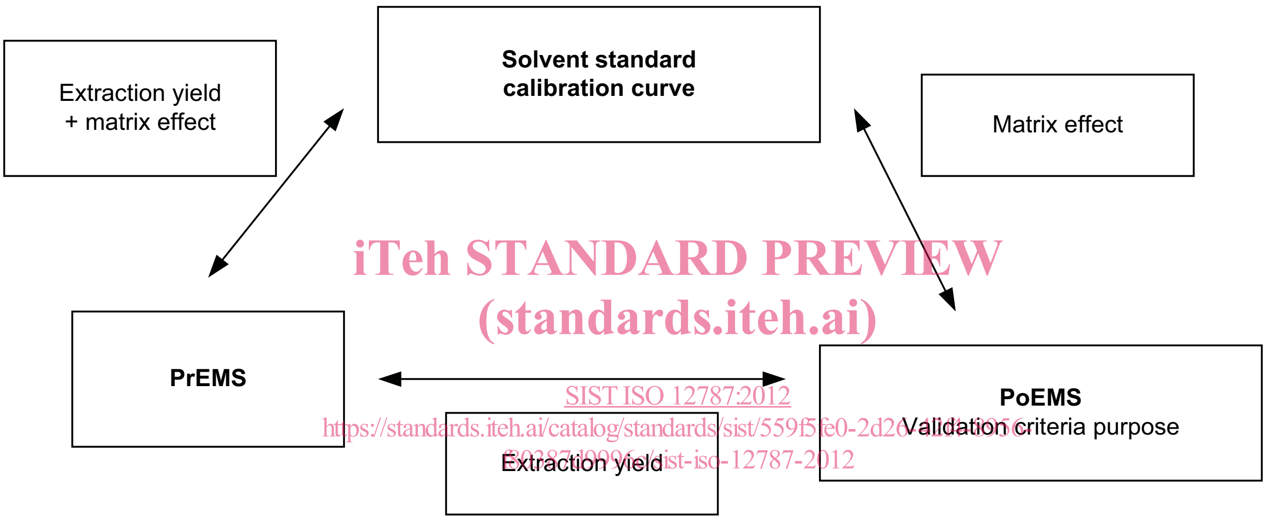

Figure 1 — Validation criteria for analytical results obtained using PrEMS, PoEMS and a solvent calibration curve

If an extraction process is performed, the matrix effect is given by the PoEMS recovery (using the solvent standard calibration curve). The difference between PoEMS and PrEMS recoveries gives the extraction yield of the sample process.

If no extraction process is performed, the extraction yield is equal to 100 %, and the matrix effect is given by the PrEMS (or PoEMS) recovery. If the recovery obtained on PrEMS, using the solvent standard calibration curve, is significantly different from the expected value, a matrix effect should be suspected. Under these circumstances, it is recommended that the method of standard addition be used.

PrEMS and PoEMS preparations should be carried out under the following conditions:

— use a solvent compatible with the sample preparation;

— use the minimum possible amount of solvent to introduce the analyte in the test solution;

— depending on the sample type, spiked samples (PrEMS) should be prepared by mixing the analyte solution with the sample, allowing dispersion into liquid samples and penetration/adsorption onto non-liquid or solid samples (this step should be adapted if the analyte is highly volatile);

— perform the PrEMS and the PoEMS at the estimated analyte concentration within the calibration range.

This analytical approach should only be used if the compound added to the cosmetic matrix behaves similarly to the compound present in the matrix. If not, certified or well-characterized standard samples could be proposed as an alternative. Careful consideration should be given to the use of spiked samples with solid cosmetic products.

4.2 Decision tree

The decision tree, represented in Figure 2, indicates the proposed approach and the different steps to be performed.

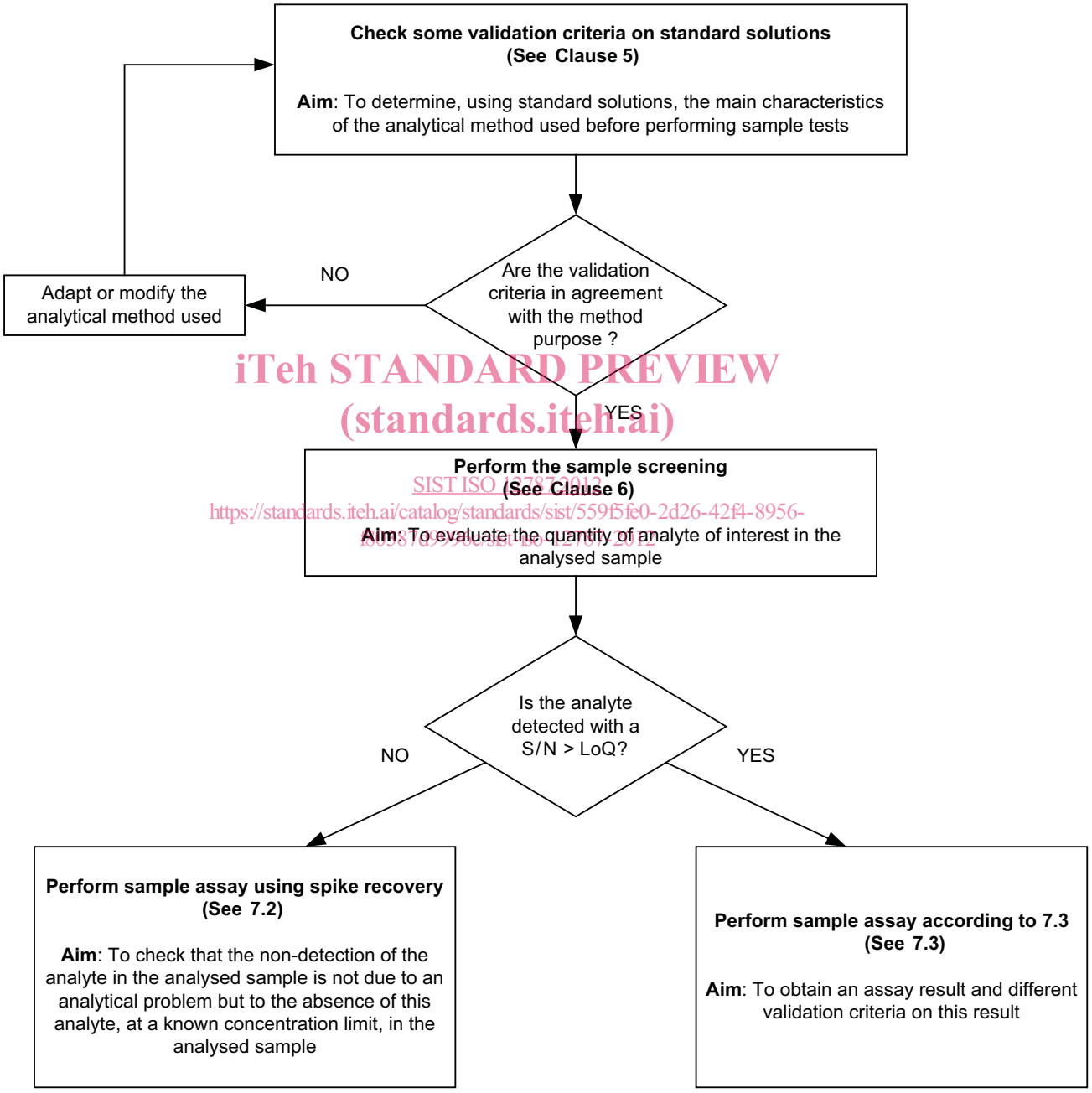

Figure 2 — Purpose of the approach and steps to be performed