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OIML R 142

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Automated refractometers: Methods and means of verification

Réfractomètres automatisés:
Méthodes et moyens de vérification

OIML R 142 Edition 2008 (E)

ORGANISATION INTERNATIONALE DE MÉTROLOGIE LÉGALE

INTERNATIONAL ORGANIZATION OF LEGAL METROLOGY

Contents

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Automated refractometers

Methods and means of verification

1 Scope

1.1 This Recommendation applies to working automated refractometers powered by mains or battery, which have either just been manufactured, are in service, or which have just been repaired.

1.2 This Recommendation lays down the methods and means of verification for working automated refractometers used in the determination of the relative refractive index of liquids, solids and their dispersion, as well as quantities that are functionally related to the refractive index, for example, the mass fraction of solutions.

1.3 The purpose and intended scope of the working automated refractometers are given in Table 1 (See Annex 1).

Automated refractometers functioning on the basis of interference and goniometric methods for the measurement of the composition of liquid media based on the difference in refractive indices between a controlled solution and a standard one, as well as specialized refractometers, are not covered by the scope of this Recommendation.

Note: The statements of the present Recommendation do not affect OIML R 124 "Refractometers for the measurement of the sugar content of grape must".

2 Terminology

2.1 Refractometer

Instrument for measuring the refractive index. If the refractometer is provided with another scale or an additional scale calibrated in the units of the fraction of soluble dry substances in aqueous solutions, which are recognized by the international organizations, e.g. the International Sucrose Mass Fraction Scale, $\%_{mass}$ (Brix), then the refractometer shall be accompanied by a conversion table for the refractive index values.

2.2 Working automated refractometer

Instrument in which the test sample is supplied manually or automatically to the device in the continuous mode, depending on the technological process.

2.3 **A** working automated refractometer may be equipped with a built-in microprocessor displaying the measurement data. It may also be connected to one or several secondary indicating devices, printing units and other auxiliary devices, including a universal computer.

2.4 Basic metrological terms used in this Recommendation are given in Annex 6.

3 Classification

3.1 The specifications and metrological characteristics of the most common refractometers are given in Table 1 (See Annex 1).

3.2 Refractometers measuring the refractive index are subdivided into the following types:

3.2.1 Pulfrich refractometers with a V-shaped prism based on measurement of the deviation angle β_λ of a refracted beam passing through a prism system from a test material (a) and a measuring prism (b) (Fig. 1).

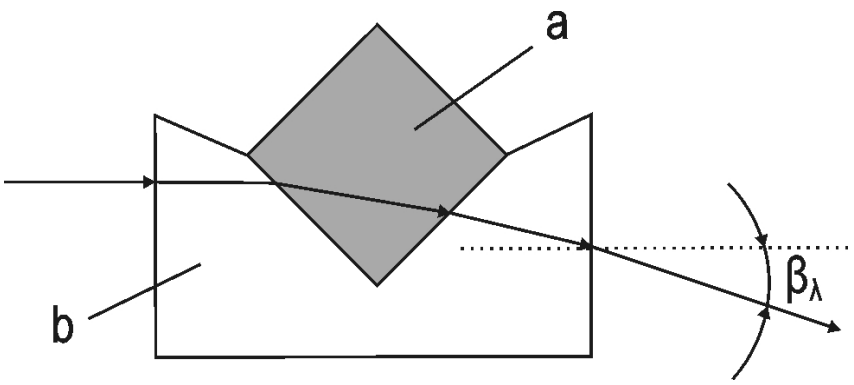

Figure 1

3.2.1.1 The refractive index $n(\lambda)$ of the test prism (a) for the wavelength λ is calculated by the formula:

$$n(\lambda) = \sqrt{N_\lambda^2 + \sin\beta_\lambda \sqrt{N_\lambda^2 - \sin^2\beta_\lambda}} \qquad (1)$$

where:

N_λ is the refractive index of a measuring prism for the wavelength λ;

β_λ is the angle between the emergent beam and the normal to the entry surface of a measuring prism.

3.2.1.2 A test sample shall be a rectangular parallelepiped with the section side minimum 17 mm.

The thickness of the sample shall be from 4 to 20 mm depending on the transparency of the material and the radiation intensity of the source.

The angle between the active faces of the sample shall be 90° ± 1'.

3.2.2 Refractometers that measure the refractive index by the total internal reflection method (TIR refractometers), which are based on the determination of the critical angle of the total internal

reflection, the light being reflected from the boundary of the test sample that is in contact with the measuring prism having a higher refractive index than the refractive index of the test sample.

3.2.3 TIR refractometers are subdivided into the following types depending on their design:

3.2.3.1 Pulfrich refractometers.

The critical angle i_λ of the beam emerging from the measuring prism (b) is measured to determine the refractive index $n(\lambda)$.

A Pulfrich refractometer uses line spectrum light. Replaceable measuring prisms with a prism angle of $\chi = 90°$ (Fig. 2) as well as a prism angle of $\chi = 60°$ are applied.

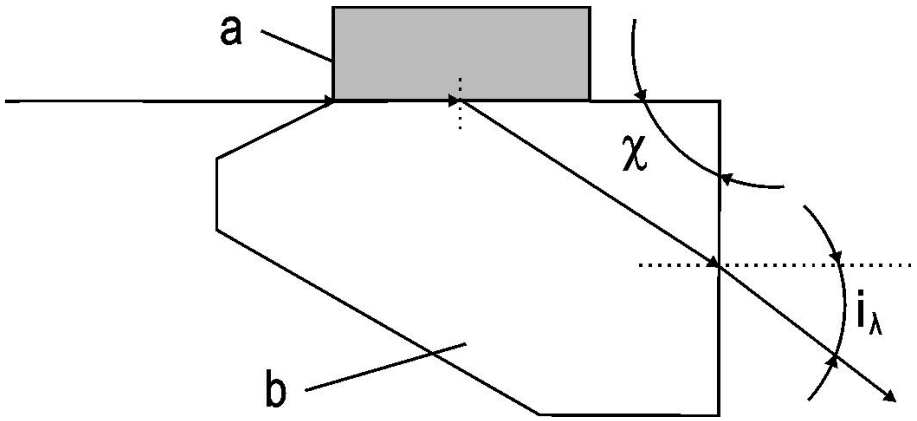

Figure 2

3.2.3.1.1 The refractive index $n(\lambda)$ of the test sample (a) with a measuring prism (b) refracting angle of 90° is calculated by the formula:

$$n(\lambda) = \sqrt{N_\lambda^2 - \sin^2 i_\lambda} \qquad (2)$$

where:

N_λ is the refractive index of the measuring prism (b) for the wavelength λ;

i_λ is the angle between the emergent beam and the normal to the entry surface of the measuring prism.

3.2.3.1.2 The test sample (a) shall be in the form of a rectangular plate having minimum dimensions 15 mm × 15 mm × 4 mm.

The angle between the active faces shall be 90° ± 10'. Facets and pop-offs on the right angle edge are not allowed.

The flatness tolerance of the working surfaces of the test sample (a) shall not exceed two fringes by 1 cm with a maximum local deviation of 0.5 fringe.

The surfaces of the active faces shall be polished. The roughness parameter is $R_z \leq 0.050$ μm.

The useful volume of the sample shall have no bubble clusters or inclusions.

The immersion liquid used for lapping the sample shall have a refractive index larger than that of the test sample (a), but not exceeding the refractive index of the measuring prism (b).

3.2.3.2 Abbe refractometers

An Abbe refractometer has a measuring prism (b) with a prism angle, φ, of about 60° (Fig. 3).

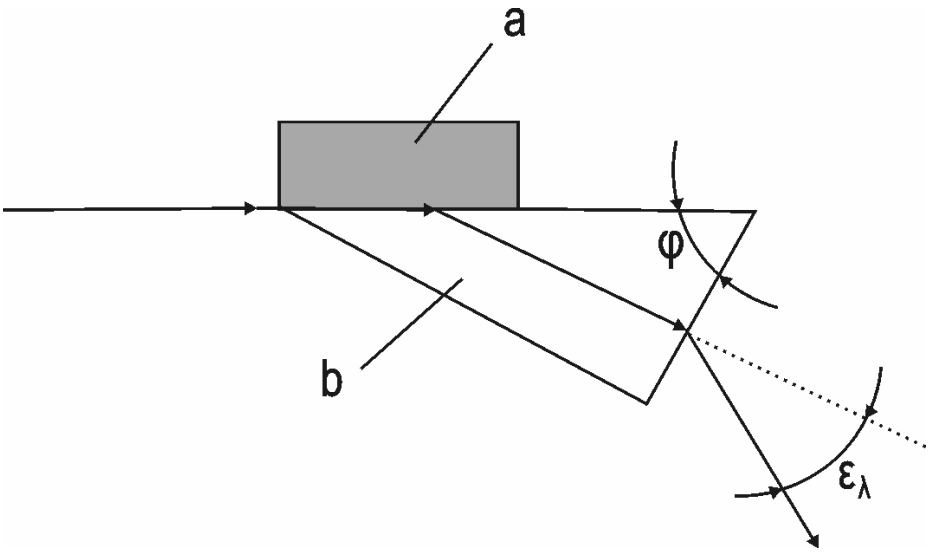

Figure 3

The Abbe refractometer is also equipped with an illuminating prism and an additional system of compensating prisms that allow for measurements to be made in white light (daylight or electric light).

3.2.3.2.1 The refractive index $n(\lambda)$ of the test sample (a) for the wavelength λ is calculated by the formula:

$$n(\lambda) = \sin\varphi\sqrt{N_\lambda^2 - \sin^2\varepsilon_\lambda} + \cos\varphi\sin\varepsilon_\lambda \qquad (3)$$

where:

φ is the measuring prism refracting angle;

N_λ is the measuring prism refractive index;

ε_λ is the critical angle of the beam exit.

3.2.3.3 Dipping refractometers

The measuring elements of immersion refractometers have the shape of a bevel-cut cylinder (Abbe prism) or a cylindrical rod (made of sapphire) with one flat end face and the other end face spherical.

During immersion into the liquid to be analyzed, beams are submitted to a total internal reflection on the surface of the dividing ridge between the liquid and the measuring element at an angle which is greater than the critical one. The dependence of photodiode signal on the critical angle value due to that effect allows the calculation of the refractive index of the liquid to be analyzed.

3.2.3.4 Refractometer with broken total internal reflection (BTIR)

Refractometer that measures the refractive index by the BTIR method which is based on the determination of the energy and polarization patterns of light reflected from the boundary of the test medium and the measuring component.

4 Units

4.1 The refractive index (N) of a medium is the ratio of the velocity of light in a vacuum "c" to the velocity of light in the medium "v". N is called the absolute refractive index. It is a dimensionless quantity:

$$N = \frac{c}{v}$$

The velocity of light in vacuum is c = 299792458 m/s.

The vacuum refractive index is $N_0 \equiv 1$.

4.2 When measuring the refractive indices of liquids and solids, one usually determines their relative refractive indices n in relation to the air in the laboratory environment under standard conditions.

Standard conditions for the measurement of relative refractive indices are:

- temperature, T = (20 ± 2) °C;
- atmospheric pressure, P = (101 325 ± 1 000) Pa;
- relative humidity, f = (50 ± 30) %.

The air refractive index n_a under standard conditions has the following values:

- for Na green line Hg (λ = 546.1 nm) – 1.0002726;
- for Na doublet yellow line (λ = 589.3 nm) – 1.0002719;
- for He/Ne laser red line (λ = 632.99 nm) – 1.0002712.

The relationship between the absolute refractive index N and the relative refractive index n is the following:

$$N = n_a \cdot n$$

where:

n_a is the absolute refractive index of air in the laboratory environment in the process of measurement.

The dependence of the refractive index on the temperature, $n\ (t)$, for t = 20 °C, is designated as n^{20}.

The dependence of the refractive index n on the wavelength is designated, for example, as n_D, where:

$D = \frac{D_1 + D_2}{2}$ is the average wavelength of the Na-lamp doublet yellow line;

D_1 is 589.6 nm;

D_2 is 589.0 nm.

The wavelength, its designation and the corresponding chemical elements spectral lines are shown in Table 2 (Annex 2).

Note: Modern instruments usually utilize LEDs with a wavelength close to the sodium D-line (e.g. λ = 590 nm) as radiation sources.

5 Metrological requirements

5.1 For refractometers with several measuring ranges (multiple-range refractometers), the values of the main metrological characteristics shall be set for each range.

5.2 For a given range of influence quantities, the maximum permissible error (MPE) shall be specified in the refractometer operating manual.

5.3 Measurements shall be taken in one of the spectral regions (UV, visible or IR spectral region) at the fixed monochromatic wavelengths indicated in Table 2 (Annex 2).

In the visible spectral region the light monochromatization shall be realized basically for the spectral lines: C (λ_c = 656.3 nm), C′ ($\lambda_{c'}$ = 643.8 nm), D (λ_D = 589.3 nm), d (λ_d = 587.6 nm), e (λ_e = 546.1 nm), F(λ_F = 486.1 nm), F' ($\lambda_{F'}$ = 480.0 nm).

5.4 When a refractometer is intended to control optical glasses, measurements shall basically be realized for the following spectral lines: F' and C' (cadmium lamp), e (mercury lamp) and d (helium discharge tube).

5.5 When a refractometer is designed for the measurement of the refractive indices of liquids, measurements shall basically be realized for the following spectral lines: D (sodium lamp) and F, C (hydrogen discharge tube).

Note: For refractometers operated at wavelengths different from the sodium D spectral line (λ = 589.3 nm), the refractive index values can be evaluated in n_D (reduced to n_D), using the dispersion formula, if necessary. The mass fraction ($\%_{mass}$) should be corrected to take into account the dispersion and its dependence on the mass fraction of a test liquid sample.

5.6 When a refractometer is intended to simultaneously measure the refractive indices of solids and liquids, the measurement shall be taken for the spectral lines indicated in 5.4 and 5.5.

Notes: 1. When a refractometer operates outside the visible spectral region, the radiation lines indicated in Table 2 (Annex 2), as well as LEDs, are used. The wavelengths may be different from those indicated in 5.4.

2. It is permitted to utilize a continuous spectrum source with interference filters or a monochromator (particularly in the spectral region with a wavelength greater than 1.5 μm).

5.7 For all refractometers indicated in Table 1 (Annex 1), if the temperature of a test medium deviates from the standard value 20 °C, the main normalizing parameters shall be maintained on condition that the temperature correction is applied. The correction value shall be indicated in the specifications of the refractometer.

6 Technical requirements

6.1 Appearance of a refractometer

The appearance of a refractometer shall meet the following requirements:

6.1.1 Painted metal (or plastic) surfaces shall be clean and have no cracks, dents, chips or stains.

6.1.2 Non-painted surfaces shall have a rust-proof coating (chromium-plating, nickel-plating, etc.)

6.1.3 Faces of the instrument shall be rounded.

6.1.4 A manufacturer's name or trademark, a type and a serial number shall be indicated on the instrument.

6.2 Movable parts of the instrument shall move smoothly.

6.3 Optical parts of the instrument shall have no scratches, black spots or other defects.

6.4 The materials for a measuring prism or a cuvette, mandrels and other parts shall be selected on the basis of the field of application (purpose) of the refractometers. They must be chemically resistant to the influence of chemical substances to be analyzed, which, in turn, shall be protected from the direct influence of the environment.

6.5 The refractive index scale interval of automatic digital refractometers shall not exceed $1 \cdot 10^{-4}$, and shall not be greater than 0.1 $\%_{mas}$ (Brix) for the mass concentration.

6.6 The working temperature range shall be specified in the operating manual. The scale interval of utilized thermometric sensors is 0.5 °C, 0.2 °C, 0.1 °C, or less.

6.7 Thermostatic chambers shall be hermetically sealed and shall be equipped with a built-in thermometer or device for stabilizing and measuring the temperature of a measuring prism or the temperature in the vicinity of the liquid surface where it is in contact with the measuring prism.

6.8 Refractometers with external power sources (mains) shall have an emergency power supply.

6.9 To verify the refractometers, plane-parallel plates, prisms and refractometric liquids shall be utilized.

6.10 A refractometer shall be able to operate continuously during at least 8 hours. The time needed to set the operating mode shall not exceed 30 minutes even when switching the range of the refractometer.

6.11 The error-free running time of released automated refractometers shall be established on the basis of reliability calculations and shall be specified in the operating manual.

7 Verification

7.1 Preparation and conditions of verification

7.1.1 During verification the safety requirements related to the operation of the electrical installations shall be met.

7.1.2 Verification of refractometers using volatile or toxic liquids (refractometric or immersion) shall be carried out in a room equipped with active exhaust ventilation.

7.1.3 A refractometer shall be installed in such a manner that there is a sufficient space for heat irradiation and air circulation. It shall not be installed closer than 1.5 m from a central heating radiator or air conditioning unit.

7.1.4 A refractometer with line supply (mains) shall be verified 30 minutes after being switched on.

7.1.5 A refractometer shall not be exposed to direct sunlight.

7.1.6 During verification, a refractometer shall not be subject to vibration, shaking or shocks, nor to external electric or magnetic fields, which may influence its operation.

7.1.7 Permissible concentrations of interfering and aggressive components in ambient air, at the place at which verification is carried out, shall not exceed the values specified in the national regulation.

7.1.8 Before verification, a refractometer that is not equipped with a temperature adjustment function shall be kept in a laboratory at a temperature between +18 °C and +22 °C for at least 12 hours, for stabilization.

7.1.9 Maintenance is performed in accordance with the instruction manual, before subsequent verification.

7.1.10 The verification means shall be prepared in accordance with the instruction manual.

7.2 Performance of verification

7.2.1 External examination

During external examination, the following shall be determined:

- completeness of a refractometer in accordance with the requirements of its instruction manual;
- absence of defects preventing readability of inscriptions, markings and digital scale reading of a refractometer;
- absence of mechanical failures of an enclosure, digital display and connecting cables;
- absence of chips, scratches and contaminations on visible optical parts of a refractometer;
- presence of the manufacturer's name or trade mark, the type and the serial number.

7.2.2 A refractometer that does not comply with the requirements above is not subject to further verification.

7.3 Testing

7.3.1 During testing, the refractometer's operation shall be checked according to the operating manual, using standard means of verification that have verification (calibration) certificates.

7.3.2 The following points shall be verified during testing:

- correspondence of the measuring range of the refractometer to that specified in the operating manual (checked by a single measurement of the refractive index for the extreme values of the working range, one of which is n_D^{20}, using the verification means indicated in 7.4).

  Note:

  1. The refractometer working range claimed for verification may depend on measurement problems and may be lower than the underlying construction capabilities.
  2. Distilled water and/or sucrose solutions should be used to establish the correspondence of the working range to that specified in the operating manual for refractometers with a mass concentration measuring scale, $\%_{mass}$ (Brix) (see Annexes 4, 5 and 7).

- correlation of the refractive index of distilled water (n_D^{20} = 1.33299) with the initial value of the sucrose mass fraction scale (0.00 $\%_{mass}$). For refractometers with both scales, the refractive index scale having the lower part of its measurement range around 1.3;
- correct functioning of the refractometer software and correctness of the information displayed;
- operation of the device for the measurement and stabilization of the measuring prism temperature. The readings shall correspond to (20 ± 0.1)°C.

7.4 Means and methods of verification

The means and methods of verification of refractometers are the following:

- solid samples: parallel-sided plates in the measuring range 1.4 – 1.9 and trihedral acute-angled and rectangular glass prisms in the measuring range 1.2 – 1.4.

Note: The requirements related to standard parallel-sided plates and trihedral acute angled and rectangular prisms are presented in the manufacturer's specifications of the above means of verification.

- liquid samples: refractometric liquids and sucrose solutions.

Note: 1. As an example of liquid samples, Table 3 (Annex 3) gives the list of refractometric samples for the verification of refractometers within the measurement range from 1.3 to 1.7 with the nominal refractive index values.

2. The verification means should be approved by the national and international metrology organizations as certified reference materials (CRMs) and be accompanied by a certificate containing the names, values of the refractive indices $n(\lambda)$ relative to air, temperature correction factors for the refractive index under the working temperature other than 20 °C (for liquid samples), the production date, the batch number and the expiration date.

7.4.1 Verification by means of solid samples

An optimum amount of immersion liquid with a diameter of about 1 mm is put on the polished work surface of a well-cleaned standard plate or prism (according to the provisions of the operating manual and on the basis of practical experience of operating the refractometer). As an example, the following liquids could be used as immersion liquids:

- pure α-bromine naphthalene ($n_D^{20} \approx 1.66$) for standard plates and prisms with $n_D^{20} < 1.66$;
- pure methylene iodide ($n_D^{20} = 1.74$) for standard plates with $1.66 < n_D^{20} < 1.7$;
- pure sulfur-saturated methylene iodide ($n_D^{20} = 1.78$) for standard plates with $n_D^{20} > 1.74$.

The standard plate or prism is laid on the measuring prism of the refractometer such that the immersion liquid may be equally distributed and is ground to obtain an optical contact. The refractive index is measured. Measurements are repeated five times. The standard plate or prism is removed each time and is placed on the refractometer measuring prism. The average refractive index is calculated from the five values obtained. These measurements are repeated for each standard plate or prism.

7.4.2 Verification on the basis of liquid samples using standard refractometric liquids

Double distilled water with the conductivity $(1.0\ldots..1.1)\cdot 10^{-6}\ \Omega^{-1}\text{cm}^{-1}$ is used to verify the lower limit considering that the lowest point of the refractometer measuring range corresponds to refractive index 1.3.

Note: For refractometers with a different lowest point of the refractometer measuring range, means of verification according to 7.4 should be utilized.

The refractive indices of double distilled water at the temperature of 20 °C are (λ = 589.3 nm) – n_D^{20} = 1.33299 and (λ = 546.1 nm) – n_e^{20} = 1.33447 for the sodium doublet yellow line and mercury green line, respectively.

The other points of the refractometer scale are verified with liquid standard refractometric samples covering the verified refractometer scale uniformly over the refractive index range.

Note: Liquid refractometric samples are chosen on the basis of the requirement for constant values of their refractive indices, low volatility, non-toxicity, non-hygroscopicity and the possibility

to receive them in pure form. The liquid refractometric samples are recommended to be kept in hermetic or sealed one-use glass ampoules. Each sample should have a label with the name, the refractive index value relative to air, temperature correction factor for the refractive index at the working temperatures other than 20 °C, the production date, the batch number and the expiry date. The ampoule capacity is determined by the manufacturer on the basis of adequacy of their application for verification of refractometers with the minimum consumption of the liquid sample.

7.4.3 Verification with liquid samples using refractometric liquids

Verification is carried out in the following way:

1) Put the optimum amount (recommended in the operating manual) of double distilled water on the surface of the measuring prism or into the measuring cuvette of the refractometer. Wait for stabilized temperature within the required values and measure the refractive index considering that the lowest point of the refractometer measuring range corresponds to refractive index 1.3.

2) Remove the double distilled water sample from the surface of the measuring prism or cuvette. Again, put double distilled water on the surface of the measuring prism or into the measuring cuvette. After the required temperature value has been established, make the measurement. Measurements are taken five times, the measuring sample being put on and removed from the surface of the measuring prism or cuvette each time. The average value of the refractive index is calculated from the five values obtained.

3) Measure one by one the refractive indices chosen from Table 3 (Annex 3) or other standard refractometric liquids (CRM), repeating the operations in 1) and 2).

Before each measurement the prism planes or the cell shall be cleaned with distilled water or ethyl alcohol, wiped with gauze or moisture absorbing paper and dried. Any hair residue or stains on the surfaces is not permitted. The time interval between measurements shall be at least 60 seconds including the time needed for cleaning and drying the surfaces of the prisms or the cell after each measurement.

7.4.4 Refractometers with scales numbered in the mass concentration values, $\%_{mass}$ (Brix), can also be verified using a fresh sucrose-water solution in accordance with Table 4 for sucrose-water solutions of the ICUMSA Specification and Standard SPS – 3 2000 (Annex 4). The corrections should be applied if the temperature deviates from 20 °C, using Table 5 (Annex 5). The procedure for the preparation of the sucrose-water solutions is given in Annex 7. The measurement procedure corresponds to 7.4.3 1) and 2) and the further processing of measurement results corresponds to 7.5.

7.5 Processing of measurement results (uncertainty budget)

7.5.1 The measurement results are processed in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM).

7.5.2 The deviation of the refractometer measurement result from the measurand value is determined from the result of multiple measurements ($I = 5$).

7.5.3 The error of the refractometer indication at each control point is calculated by the formula:

$$E = n_{D_{av}} - n_{D_0} \tag{4}$$

where:

n_{D_0} is the refractive index value of the standard verification means (e.g. CRM);

$n_{D_{av}}$ is the average value of the refractive indices of the standard means of verification obtained by measurements using the refractometer ($I = 5$).

7.5.4 The uncertainty components of the refractometer measurement results

The uncertainty components are the following:

- refractive index uncertainty of the standard verification means. This uncertainty is determined during the certification process;
- random dispersion of the measurement results;
- uncertainty of temperature maintenance, measurement temperature of the measuring prism and temperature dependence of the refractive index of the standard verification means (CRM) (if a liquid CRM is utilized).

7.5.5 The standard uncertainty of the refractive index values of the standard means of verification is estimated by Type A and is calculated by the formula:

$$u_A = \sqrt{\frac{1}{m(m-1)} \cdot \sum_{i=1}^{m} \left({}^{n}D_i - {}^{n}D_{av} \right)^2} \tag{5}$$

where:

$m = 5$ (number of measurements);

$n_{D\,av}$ is the average value of the refractive indices of the standard verification means obtained during the measurement using the refractometer.

7.5.6 Type B standard uncertainty:

$$u_B = \sqrt{u_{CRM}^2 + u_{reading}^2 + u_t^2} \tag{6}$$

where:

u_{CRM} is the standard uncertainty $u(k = 1)$ of the utilized CRM;

u_{reading} is the standard uncertainty of the refractometer reading;

u_{t} is the uncertainty due to temperature dependence.

7.5.7 The total standard uncertainty is calculated with the formula:

$$u_c = \sqrt{u_A^2 + u_B^2} \tag{7}$$

7.5.8 The expanded uncertainty *U(k = 2)* is calculated with the formula:

$$U(k = 2) = 2.u_c \tag{8}$$

Notes:

1) The coverage factor k is determined from the effective degrees of freedom v_{eff}. The estimation of the effective degrees of freedom v_{eff} of $u_c(y)$ is performed by the Welch-Satterthwaite formula:

$$v_{eff} = \frac{u_c^4(y)}{\sum_{i=1}^{N} \frac{c_i^4 u^4(x_i)}{v_i}}$$

2) The coverage factors k for different values of the effective degrees of freedom v_{eff} are presented in the table below.

v_{eff}	1	2	3	4	5	6	7	8	10	20	50	∞
k	13.97	4.53	3.31	2.87	2.65	2.52	2.43	2.37	2.28	2.13	2.05	2.00

7.5.9 The refractometer error (E) calculated with the formula in 7.5.3 shall be smaller than or equal to the MPE for each control point. The MPE is given in the operating manual of the refractometer.

$$\left| n_{D_o} - n_{D_{av}} \right| \leq MPE \tag{9}$$

7.5.10 The expanded uncertainty *U(k = 2)* shall be smaller than one third of the MPE:

$$U(k = 2) \leq \frac{MPE}{3} \tag{10}$$

8 Drawing up the verification results

8.1 The verification results are entered into the protocol.

8.2 The verification is considered successful if the requirements of clause 7 are met and the refractometer measurement results do no exceed the MPE specified in the operating manual.

8.3 If, according to the verification results, the refractometer is declared ready for service, it is stamped with a verification mark and/or a certificate of verification is issued.

8.4 Seals or stamps bearing the verification mark are put in places such that they prevent access to the adjustment components of the refractometer.

8.5 If, according to the verification results, the refractometer is rejected as defective, then its certificate of verification is abrogated and its verification marks are removed.

8.6 If the verification of a refractometer is not successful after its production, then the instrument is returned to the manufacturer for repair. In such a condition, the measuring instrument may be represented for verification.

Annex 1
(Informative)

Technical and metrological characteristics of the most common refractometer types

Refractometer technology	Refractive index measurement range	Combined standard measurement uncertainty		Purpose and prevailing scope
		refractive index $u_{c\,n}$	dispersion $u_{c\,D_n}$	
Refractometers with a V-shaped prism	1.20 – 2.50	$\pm 3 \cdot 10^{-5}$	$\pm 1 \cdot 10^{-5}$	Measurement of refractive index and dispersion of solids (mainly glasses) in the optomechanical, chemical, electronic and other industries
Pulfrich refractometers	1.20 – 2.10	$\pm 5 \cdot 10^{-5}$	$\pm 2 \cdot 10^{-5}$	Measurement of refractive index and dispersion of liquids and solids in the chemical, pharmaceutical, food, optomechanical and other industries
Abbe refractometers	1.20 – 2.10	$\pm 2 \cdot 10^{-4}$	$\pm 2 \cdot 10^{-4}$	Measurement of refractive index and average dispersion mainly of liquids in the chemical, pharmaceutical, food and other industries
Dipping refractometers	1.33 – 1.65	$\pm(2 \cdot 10^{-5} - 3 \cdot 10^{-4})$	–	Fast measurement of refractive index and mass fraction of liquids in the chemical, food and other industries
BTIR refractometers	1.20 – 2.10	$\pm 3 \cdot 10^{-4}$ ($10^{-3} < K < 10^{-2}$) $\pm 1 \cdot 10^{-4}$ ($\pm 1 \cdot 10^{-3}$) ($K < 10^{-3}$) K: absorption coefficient of test medium	–	Measurement of refractive index mainly of strongly absorbing media and mass fraction in the chemical, pharmaceutical, food and other industries

Table 1

Annex 2
(Informative)

Wavelengths and the corresponding spectral lines of chemical elements

Wavelength λ (in nm)	Wavelength designation	Chemical element
365.0_1	*i*	Hg
404.6_6	*h*	Hg
435.8_3	*g*	Hg
479.9_9	*F′*	Cd
486.1_3	*F*	H
546.0_7	*e*	Hg
587.5_6	*d*	He
589.2_9	*D*	Na
632.9_9	–	He/Ne (laser)
643.8_5	*C′*	Cd
656.2_8	*C*	H
694.3	–	$Cr+Al_2O_3$ (laser)
706.5_2	*r*	He
852.1_1	*s*	Cs
$1\,013.9_8$	*t*	Hg
1 060.0	–	Nd (laser)
$1\,128.6_6$	–	Hg
1 153.0	–	He/Ne (laser)
3 392.2	–	He/Ne (laser)
1 395.1	–	Hg
10 600.0	–	CO_2 (laser)

Table 2

Annex 3
(Informative)

Refractive index values of refractometric liquids (CRMs) used for verification of refractometers

Chemical substance	**Refractive index nominal value,** n_D
2.2.4 – trimethyl pentane	1.3914
methylcyclohexane	1.4238
cyclohexane	1.4262
toluene	1.4967
chlorbenzene	1.5245
o – nitrotoluene	1.5462
α – bromonaphthalene	1.6580
carbon tetrachloride	1.4602
n – heptane	1.3877
ethylene chloride	1.4448
benzene	1.5011
double distilled water	1.3330

Table 3

Note:

More accurate refractive index values of refractometric liquids relative to air at the temperature of 20 °C and their temperature coefficients should be indicated in the certificate as well as on labels according to certification results of these CRMs. The refractive index uncertainty of a CRM, determined in the process of certification should not exceed ± 2 10^{-5} for distilled water and ± 3 10^{-5} for other refractometric liquids,

Annex 4

(Informative)

International refractive index scale of ICUSMA (1974) for pure sucrose solutions at 20 °C and 589 nm

Table 4 gives the refractive index values against air with sucrose mass fraction

Table 4

Sucrose g/100 g	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0	1.332986	1.333129	1.333272	1.333415	1.333558	1.333702	1.333845	1.333989	1.334132	1.334276
1	1.334420	1.334564	1.334708	1.334852	1.334996	1.335141	1.335285	1.335430	1.335574	1.335719
2	1.335864	1.336009	1.336154	1.336300	1.336445	1.336590	1.336736	1.336882	1.337028	1.337174
3	1.337320	1.337466	1.337612	1.337758	1.337905	1.338051	1.338198	1.338345	1.338492	1.338639
4	1.338786	1.338933	1.339081	1.339228	1.339376	1.339524	1.339671	1.339819	1.339967	1.340116
5	1.340264	1.340412	1.340561	1.340709	1.340858	1.341007	1.341156	1.341305	1.341454	1.341604
6	1.341753	1.341903	1.342052	1.342202	1.342352	1.342502	1.342652	1.342802	1.342952	1.343103
7	1.343253	1.343404	1.343555	1.343706	1.343857	1.344008	1.344159	1.344311	1.344462	1.344614
8	1344765	1.344917	1.345069	1.345221	1.345373	1.345526	1.345678	1.345831	1.345983	1.346136
9	1.346289	1.346442	1.346595	1.346748	1.346902	1.347055	1.347209	1.347362	1.347516	1.347670
10	1.347824	1.347978	1.348133	1.348287	1.348442	1.348596	1.348751	1.348906	1.349061	1.349216
11	1.349371	1.349527	1.349682	1.349838	1.349993	1.350149	1.350305	1.350461	1.350617	1.350774
12	1.350930	1.351087	1.351243	1.351400	1.351557	1.351714	1.351871	1.352029	1.352186	1.352343
13	1.352501	1.352659	1.352817	1.352975	1.353133	1.353291	1.353449	1.353608	1.353767	1.353925
14	1.354084	1.354243	1.354402	1.354561	1.354721	1.354880	1.355040	1.355199	1.355359	1.355519
15	1.355679	1.355840	1.356000	1.356160	1.356321	1.356482	1.356642	1.356803	1.356964	1.357126
16	1.357287	1.357448	1.357610	1.357772	1.357933	1.358095	1.358257	1.358420	1.358582	1.358744
17	1.358907	1.359070	1.359232	1.359395	1.359558	1.359722	1.359885	1.360048	1.360212	1.360376
18	1.360539	1.360703	1.360867	1.361032	1.361196	1.361360	1.361525	1.361690	1.361854	1.362019
19	1.362185	1.362350	1.362515	1.362681	1.362846	1.363012	1.363178	1.363344	1.363510	1.363676
20	1.363842	1.364009	1.364176	1.364342	1.364509	1.364676	1.364843	1.365011	1.365178	1.365346
21	1.365513	1.365681	1.365849	1.366017	1.366185	1.366354	1.366522	1.366691	1.366859	1.367028
22	1.367197	1.367366	1.367535	1.367705	1.367874	1.368044	1.368214	1.368384	1.368554	1.368724
23	1.368894	1.369064	1.369235	1.369406	1.369576	1.369747	1.369918	1.370090	1.370261	1.370433
24	1.370604	1.370776	1.370948	1.371120	1.371292	1.371464	1.371637	1.371809	1.371982	1.372155
25	1.372328	1.372501	1.372674	1.372847	1.373021	1.373194	1.373368	1.373542	1.373716	1.373890
26	1.374065	1.374239	1.374414	1.374588	1.374763	1.374938	1.375113	1.375288	1.375464	1.375639

Sucrose g/100 g	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
27	1.375815	1.375991	1.376167	1.376343	1.376519	1.376695	1.376872	1.377049	1.377225	1.377402
28	1.377579	1.377756	1.377934	1.378111	1.378289	1.378467	1.378644	1.378822	1.379001	1.379179
29	1.379357	1.379536	1.379715	1.379893	1.380072	1.380251	1.380431	1.380610	1.380790	1.380969
30	1.381149	1.381329	1.381509	1.381690	1.381870	1.382050	1.382231	1.382412	1.382593	1.382774
31	1.382955	1.383137	1.383318	1.383500	1.383682	1.383863	1.384046	1.384228	1.384410	1.384593
32	1.384775	1.384958	1.385141	1.385324	1.385507	1.385691	1.385874	1.386058	1.386242	1.386426
33	1.386610	1.386794	1.386978	1.387163	1.387348	1.387532	1.387717	1.387902	1.388088	1.388273
34	1.388459	1.388644	1.388830	1.389016	1.389202	1.389388	1.389575	1.389761	1.389948	1.390135
35	1.390322	1.390509	1.390696	1.390884	1.391071	1.391259	1.391447	1.391635	1.391823	1.392011
36	1.392200	1.392388	1.392577	1.392766	1.392955	1.393144	1.393334	1.393523	1.393713	1.393903
37	1.394092	1.394283	1.394473	1.394663	1.394854	1.395044	1.395235	1.395426	1.395617	1.395809
38	1.396000	1.396192	1.396383	1.396575	1.396767	1.396959	1.397152	1.397344	1.397537	1.397730
39	1.397922	1.398116	1.398309	1.398502	1.398696	1.398889	1.399083	1.399277	1.399471	1.399666
40	1.399860	1.400055	1.400249	1.400444	1.400639	1.400834	1.401030	1.401225	1.401421	1.401617
41	1.401813	1.402009	1.402205	1.402401	1.402598	1.402795	1.402992	1.403189	1.403386	1.403583
42	1.403781	1.403978	1.404176	1.404374	1.404572	1.404770	1.404969	1.405167	1.405366	1.405565
43	1.405764	1.405963	1.406163	1.406362	1.406562	1.406762	1.406961	1.407162	1.407362	1.407562
44	1.407763	1.407964	1.408165	1.408366	1.408567	1.408768	1.408970	1.409171	1.409373	1.409575
45	1.409777	1.409980	1.410182	1.410385	1.410588	1.410790	1.410994	1.411197	1.411400	1.411604
46	1.411808	1.412011	1.412215	1.412420	1.412624	1.412828	1.413033	1.413238	1.413443	1.413648
47	1.413853	1.414059	1.414265	1.414470	1.414676	1.414882	1.415089	1.415295	1.415502	1.415708
48	1.415915	1.416122	1.416330	1.416537	1.416744	1.416952	1.417160	1.417368	1.417576	1.417785
49	1.417993	1.418202	1.418411	1.418620	1.418829	1.419038	1.419247	1.419457	1.419667	1.419877
50	1.420087	1.420297	1.420508	1.420718	1.420929	1.421140	1.421351	1.421562	1.421774	1.421985
51	1.422197	1.422409	1.422621	1.422833	1.423046	1.423258	1.423471	1.423684	1.423897	1.424110
52	1.424323	1.424537	1.424750	1.424964	1.425178	1.425393	1.425607	1.425821	1.426036	1.426251
53	1.426466	1.426681	1.426896	1.427112	1.427328	1.427543	1.427759	1.427975	1.428192	1.428408
54	1.428625	1.428842	1.429059	1.429276	1.429493	1.429711	1.429928	1.430146	1.430364	1.430582
55	1.430800	1.431019	1.431238	1.431456	1.431675	1.431894	1.432114	1.432333	1.432553	1.432773
56	1.432993	1.433213	1.433433	1.433653	1.433874	1.434095	1.434316	1.434537	1.434758	1.434980
57	1.435201	1.435423	1.435645	1.435867	1.436089	1.436312	1.436535	1.436757	1.436980	1.437203
58	1.437427	1.437650	1.437874	1.438098	1.438322	1.438546	1.438770	1.438994	1.439219	1.439444
59	1.439669	1.439894	1.440119	1.440345	1.440571	1.440796	1.441022	1.441248	1.441475	1.441701
60	1.441928	1.442155	1.442382	1.442609	1.442836	1.443064	1.443292	1.443519	1.443747	1.443976
61	1.444204	1.444432	1.444661	1.444890	1.445119	1.445348	1.445578	1.445807	1.446037	1.446267
62	1.446497	1.446727	1.446957	1.447188	1.447419	1.447650	1.447881	1.448112	1.448343	1.448575

Sucrose g/100 g	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
63	1.448807	1.449039	1.449271	1.449503	1.449736	1.449968	1.450201	1.450434	1.450667	1.450900
64	1.451134	1.451367	1.451601	1.451835	1.452069	1.452304	1.452538	1.452773	1.453008	1.453243
65	1.453478	1.453713	1,453949	1.454184	1.454420	1.454656	1454893	1.455129	1.455365	1.455602
66	1.455839	1.456076	1.456313	1.456551	1.456788	1.457026	1.457264	1.457502	1.457740	1.457979
67	1.458217	1.458456	1.458695	1.458934	1.459174	1.459413	1.459653	1.459893	1.460133	1.460373
68	1.460613	1.460854	1.461094	1.461335	1.461576	1.461817	1.462059	1.462300	1.462542	1.462784
69	1.463026	1.463268	1.463511	1.463753	1.463996	1.464239	1.464482	1.464725	1.464969	1.465212
70	1.465456	1.465700	1.465944	1.466188	1.466433	1.466678	1.466922	1.467167	1.467413	1.467658
71	1.467903	1.468149	1.468395	1.468641	1.468887	1.469134	1.469380	1.469627	1.469874	1.470121
72	1.470368	1.470616	1.470863	1.471111	1.471359	1.471607	1.471855	1.472104	1.472352	1.472601
73	1.472850	1.473099	1.473349	1.473598	1.473848	1.474098	1.474348	1.474598	1.474848	1.475099
74	1.475349	1.475600	1.475851	1.476103	1.476354	1.476606	1.476857	1.477109	1.477361	1.477614
75	1.477866	1.478119	1.478371	1.478624	1.478877	1.479131	1.479384	1.479638	1.479892	1.480146
76	1.480400	1.480654	1.480909	1.481163	1.481418	1.481673	1.481929	1.482184	1.482439	1.482695
77	1.482951	1.483207	1.483463	1.483720	1.483976	1.484233	1.484490	1.484747	1.485005	1.485262
78	1.485520	1.485777	1.486035	1.486293	1.486552	1.486810	1.487069	1.487328	1.487587	1.487846
79	1.488105	1.488365	1.488625	1.488884	1.489144	1.489405	1.489665	1.489926	1.490186	1.490447
80	1.490708	1.490970	1.491231	1.491493	1.491754	1.492016	1.492278	1.492541	1.492803	1.493066
81	1.493328	1.493591	1.493855	1.494118	1.494381	1.494645	1.494909	1.495173	1.495437	1.495701
82	1.495966	1.496230	1.496495	1.496760	1.497025	1.497291	1.497556	1.497822	1.498088	1.498354
83	1.498620	1.498887	1.499153	1.499420	1.499687	1.499954	1.500221	1.500488	1.500756	1.501024
84	1.501292	1.501560	1.501828	1.502096	1.502365	1.502634	1.502903	1.503172	1.503441	1.503711
85	1.503980									

Annex 5

(Informative)

Table 5 gives the mass fraction corrections to refractometric tables for sucrose solutions at 589 nm for temperatures different from 20 °C

Table 5

Temperature (°C)	Measured sucrose (mass fraction)																	
	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85
15	-0.29	-0.30	-0.32	-0.33	-0.34	-0.35	-0.36	-0.37	-0.37	-0.38	-0.38	-0.38	-0.38	-0.38	-0.38	-0.38	-0.37	-0.37
16	-0.24	-0.25	-0.26	-0.27	-0.28	-0.28	-0.29	-0.30	-0.30	-0.30	-0.31	-0.31	-0.31	-0.31	-0.31	-0.30	-0.30	-0.30
17	-0.18	-0.19	-0.20	-0.20	-0.21	-0.21	-0.22	-0.22	-0.23	-0.23	-0.23	-0.23	-0.23	-0.23	-0.23	-0.23	-0.23	-0.22
18	-0.12	-0.13	-0.13	-0.14	-0.14	-0.14	-0.15	-0.15	-0.15	-0.15	-0.15	-0.15	-0.15	-0.15	-0.15	-0.15	-0.15	-0.15
19	-0.06	-0.06	-0.07	-0.07	-0.07	-0.07	-0.07	-0.08	-0.08	-0.08	-0.08	-0.08	-0.08	-0.08	-0.08	-0.08	-0.08	-0.07
20	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
21	+0.06	+0.07	+0.07	+0.07	+0.07	+0.07	+0.08	+0.08	+0.08	+0.08	+0.08	+0.08	+0.08	+0.08	+0.08	+0.08	+0.08	+0.07
22	+0.13	+0.14	+0.14	+0.14	+0.15	+0.15	+0.15	+0.15	+0.16	+0.16	+0.16	+0.16	+0.16	+0.16	+0.15	+0.15	+0.15	+0.15
23	+0.20	+0.21	+0.21	+0.22	+0.22	+0.23	+0.23	+0.23	+0.23	+0.24	+0.24	+0.24	+0.24	+0.23	+0.23	+0.23	+0.23	+0.22
24	+0.27	+0.28	+0.29	+0.29	+0.30	+0.30	+0.31	+0.31	+0.31	+0.32	+0.32	+0.32	+0.32	+0.31	+0.31	+0.31	+0.30	+0.30
25	+0.34	+0.35	+0.36	+0.37	+0.38	+0.38	+0.39	+0.39	+0.40	+0.40	+0.40	+0.40	+0.40	+0.39	+0.39	+0.38	+0.38	+0.37
26	+0.42	+0.43	+0.44	+0.45	+0.46	+0.46	+0.47	+0.47	+0.48	+0.48	+0.48	+0.48	+0.48	+0.47	+0.47	+0.46	+0.46	+0.45
27	+0.50	+0.51	+0.52	+0.53	+0.54	+055	+0.55	+0.56	+0.56	+0.56	+0.56	+0.56	+0.56	+0.55	+0.55	+0.54	+0.53	+0.52
28	+0.58	+0.59	+0.60	+0.61	+0.62	+0.63	+0.64	+0.64	+0.64	+0.65	+0.65	+0.64	+0.64	+0.63	+0.63	+0.62	+0.61	+0.60
29	+0.66	+0.67	+0.68	+0.70	+0.71	+0.71	+0.72	+0.73	+0.73	+0.73	+0.73	+0.73	+0.72	+0.72	+0.71	+0.70	+0.69	+0.67
30	+0.74	+0.76	+0.77	+0.78	+0.79	+0.80	+0.81	+0.81	+0.82	+0.82	+081	+0.81	+0.80	+0.80	+0.79	+0.78	+0.76	+0.75
31	+0.83	+0.84	+0.85	+0.87	+0.88	+0.89	+0.89	+0.90	+0.90	+0.90	+0.90	+0.89	+0.89	+0.88	+0.87	+0.86	+0.84	+0.82
32	+0.92	+0.93	+0.94	+0.96	+0.97	+0.98	+0.98	+0.99	+0.99	+0.99	+0.99	+0.98	+0.97	+0.96	+0.95	+0.93	+0.92	+0.90
33	+1.01	+1.02	+1.03	+1.05	+1.06	+1.07	+1.07	+1.08	+1.08	+1.08	+1.07	+1.07	+1.06	+1.04	+1.03	+1.01	+1.00	+0.98
34	+1.10	+1.11	+1.13	+1.14	+1.15	+1.16	+1.16	+1.17	+1.17	+1.16	+1.16	+1.15	+1.14	+1.13	+1.11	+1.09	+1.07	+1.05
35	+1.19	+1.21	+1.22	+1.23	+1.24	+1.25	+1.25	+1.26	+1.26	+1.25	+1.25	+1.24	+1.23	+1.21	+1.19	+1.17	+1.15	+1.13
36	+1.29	+1.30	+1.31	+1.33	+1.34	+1.34	+1.35	+1.35	+1.35	+1.34	+1.34	+1.33	+1.31	+1.29	+1.28	+1.25	+1.23	+1.20
37	+1.39	+1.40	+1.41	+1.42	+1.43	+1.44	+1.44	+1.44	+1.44	+1.43	+1.43	+1.41	+1.40	+1.38	+1.36	+1.33	+1.31	+1.28
38	+1.49	+1.50	+1.51	+1.52	+1.53	+1.53	+1.54	+1.54	+1.53	+1.53	+1.52	+1.50	+1.48	+1.46	+1.44	+1.42	+1.39	+1.36
39	+1.59	+1.60	+1.61	+1.62	+1.63	+1.63	+1.63	+1.63	+1.63	+1.62	+1.61	+1.59	+1.57	+1.55	+1.52	+1.50	+1.47	+1.43
40	+1.69	+1.70	+1.71	+1.72	+1.73	+1.73	+1.73	+1.73	+1.72	+1.71	+1.70	+1.68	+1.66	+1.63	+1.61	+1.58	+1.54	+1.51

Annex 6
(Informative)

Terminology

(Definitions from the VIM, 3rd edition, final draft 2006-08-01)

Error of indication

Difference of indication of a measuring system and the true value of the measurand

Maximum permissible error (MPE)

Extreme value of the measurement error, with respect to a known reference quantity value, permitted by specifications or regulations for a given measurement, measuring instrument, or measuring system

Uncertainty of measurement

Parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

Reference material (RM)

Material, sufficiently homogeneous and stable regarding one or more properties, used in calibration, in assignment of a value to another material or in quality assurance

Certified reference material (CRM)

Reference material, accompanied by documentation issued by an authoritative body and referring to valid procedures used to obtain a specified property value with uncertainty and traceability

Annex 7
(Informative)

Procedure for the preparation of sucrose-water test solutions

1 Preparation of the initial solution (No. 1)

Initial solution No. 1 in the amount of 1 000 ml is prepared from 20 g of chemically pure sucrose and distilled water. The solution is mixed thoroughly and certified by the difference between the refractive indices (Δn) relative to distilled water using a laboratory interference refractometer.

The prepared solution is kept in a closed glass container in the dark. The storage life of the solution does not exceed two weeks.

Before usage the solution is certified using an interference refractometer with a maximum permissible error of $\pm 3 \cdot 10^{-5}$

2. Preparation of test solutions from initial solution No. 1

Test solutions are prepared by diluting initial solution No. 1 with distilled water. A minimum of three reference sucrose-water solutions are prepared, which have the concentrations 5 %, 10 % and 15 % respectively.

Depending on the difference between the refractive indices of the test solution and the distilled water, the volume of water to be added to initial solution No. 1 is calculated by the formula:

$$V_{w_i} = \left(1 - \frac{\Delta n_{t_i}}{\Delta n_{t_I}}\right) V_{v_i}$$

where:

V_{w_i} is the distilled water volume required for preparation of the test solution (ml);

Δn_{t_i} is the difference between the refractive indices of the prepared test solution with the chosen sucrose content by mass and distilled water;

Δn_{t_I} is the difference of the refractive indices of initial solution No. 1 to distilled water, measured before preparation of the test solution;

V_{v_i} is the prepared test solution volume (ml) with a minimum volume of 100 ml;

i is the prepared test solution number.

The prepared test solution is certified by the difference between the refractive indices using an interference refractometer with a maximum permissible error of $\pm 3 \cdot 10^{-5}$.

The required test solution can be prepared using another test solution with a higher difference of refractive index.

Volumetric flasks, graduated cylinders and burettes should be used for the preparation of solutions.

The test solution is prepared in a 500 ml volumetric glassware (a cylinder or a flask). The required amount of initial solution No. 1 or utilized test solution is measured off by means of a graduated cylinder, then poured out into the glassware, and distilled water is added.

The glassware for the preparation and storage of solutions should be washed and dried beforehand. The storage life of solutions does not exceed two weeks.

The glassware prepared and used for verification solutions should bear an inscription with the number of the solution, the refractive index difference (concentration) during their certification before usage, as well as the expiry date.

Annex 8
(Informative)

Verification report

Refractometer

Serial number

Inventory number

Manufacturer

Owner of refractometer

Date of testing

Conditions of verification performing:

-ambient temperature	^{o}C
-atmospheric pressure	kPa
-relative humidity	%

Verification results:

1. Results of visual examination
2. Results of testing:

2.1 Measurement range_____

2.2 Check of an initial value of the scale, $\%_{mass}$ (Brix)_____

2.3 Check of software

2.4 Check of operation of temperature stabilizing device

3. Results of determining metrological characteristics:

3.1 Thermometer readouts_____ ^{o}C

3.2 Average value of the refractive index, $n_{D\,av}$ _____

3.3 Refractometer error, E_____

3.4 Standard uncertainty, type A, u_A_____

3.5. Standard uncertainty, type B, u_B_____

3.6 Total standard uncertainty, u_C_____

3.7 Expanded uncertainty, $U\ (k = 2)$_____

3.8 Conformity to 7.5.9_____

3.9 Conformity to 7.5.10_____

Conclusion

Person(s) responsible for the verification:

Signature(s)_____

Date_____

Title(s)_____