

**Organisation Internationale de Métrologie Légale
(OIML)**

INTERNATIONAL RECOMMENDATION

Second Draft

**Automated Refractometers
Methods and Means of Verification**

Subcommittee: TC17/SC2

Secretariat: Russia

Participating countries:

China, Cuba, France, Germany,
Poland, Russian Federation,
Switzerland, United Kingdom, USA

Observing countries:

Australia, Bulgaria, Czech Rep.,
Egypt, Finland, Japan, Romania,
Serbia and Montenegro, Slovakia,
Slovenia, Spain

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Explanatory Note to Second Draft Recommendation
“Automated Refractometers.
Methods and Means of Verification”

The present Recommendation has been developed according to the working plan of TC17/SC2 “Saccharimetry”. In the Second Draft Recommendation the requirements of the “Directives for the Technical work, p 2 (OIML, 1994)” and the “Guide to expression of uncertainty in measurements” are taken into account. In this Draft some statements of the R 108 “Refractometers for the measurement of the sugar content of fruit juices” have been used too. The statements of the present Recommendation do not influence or abrogate the other Recommendations, in particular, OIML R 124 “Refractometers for the measurement of the sugar content of grape must”

CONTENTS

1	Scope	4
2	Terminology	4
3	Classification	4
4	Units	8
5	Metrological requirements	9
6	Technical requirements	10
7	Verification	10
8	Drawing up of verification results	15
	Annex 1. Technical and metrological characteristics of the most common refractometer types	16
	Annex 2. Wavelengths and the corresponding spectral lines of chemical elements	18
	Annex 3. Refractive index values of refractometric liquids used for verification of refractometers	19
	Annex 4. International Refractive Index Scale of ICUMSA	20
	Annex 5. Mass fraction correction to refractometric tables for sucrose solutions	22
	Annex 6. Terminology (the definitions from VIM)	23
	Annex 7. - Procedure for Preparation of Test Sucrose Water Solutions	24
	Annex 8. Verification Protocol	25

Second Draft Recommendation

“Automated Refractometers. Methods and Means of Verification”

1. SCOPE

1.1. This Recommendation applies to working automated refractometers fed by mains or battery, which have been just manufactured, being in service or after repair.

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

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

1.4. The working automated refractometers on the basis of the interference and goniometric methods for measurement of the composition of liquid media by the difference of refractive indices between a controlled solution and a standard one, as well as specialized refractometers, are not the subject matter of this Recommendation.

2. TERMINOLOGY

2.1. Refractometers

Instruments for measuring the refractive index. If they are provided with another scale or an additional one 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 these refractometers shall be accompanied with a conversion table for refractive index values.

2.2. Working automated refractometers

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

2.3. **Working automated refractometers** may be equipped with a built-in microprocessor displaying the measurement data, as well as be connected to secondary indicating devices, printing units and other auxiliary devices, including a PC.

2.4. Basic metrological terms, used in this Recommendation, are given in Annex 6 (informative).

3. CLASSIFICATION

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

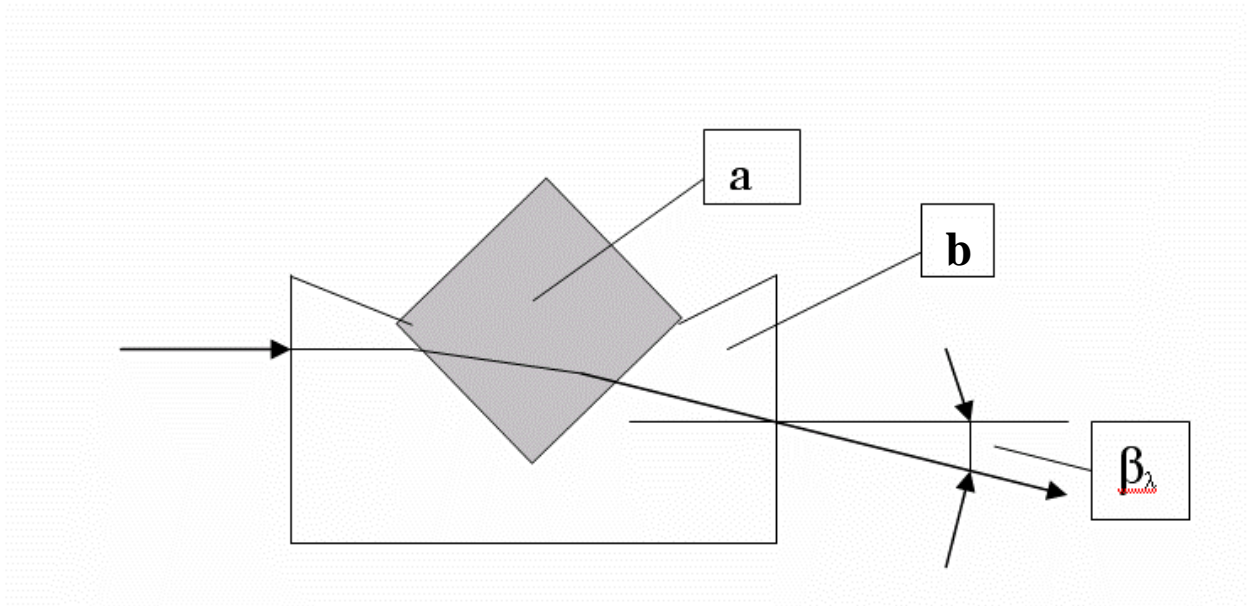


Figure 1

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

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

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 transparency of the material and radiation intensity of the source.

The angle between the active faces of the sample shall be $90^\circ \pm 1'$.

3.2.2. Refractometers measuring the refractive index by the total internal reflection method (TIR refractometers), which are based on determination of the critical angle of total internal reflection, the light being reflected from the boundary of the test sample, and are in contact with the measuring prism having a higher refractive index than the test sample, or with grazing incidence of the beam on this boundary.

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 emergence from the measuring prism is measured to determine the refractive index $n(\lambda)$ (a).

A Pulfrich refractometer uses the line spectrum light. Replaceable measuring prisms with the prism angle $\varepsilon=90^\circ$ (Fig. 2) as well as the prism angle $\varepsilon=60^\circ$ are applied.

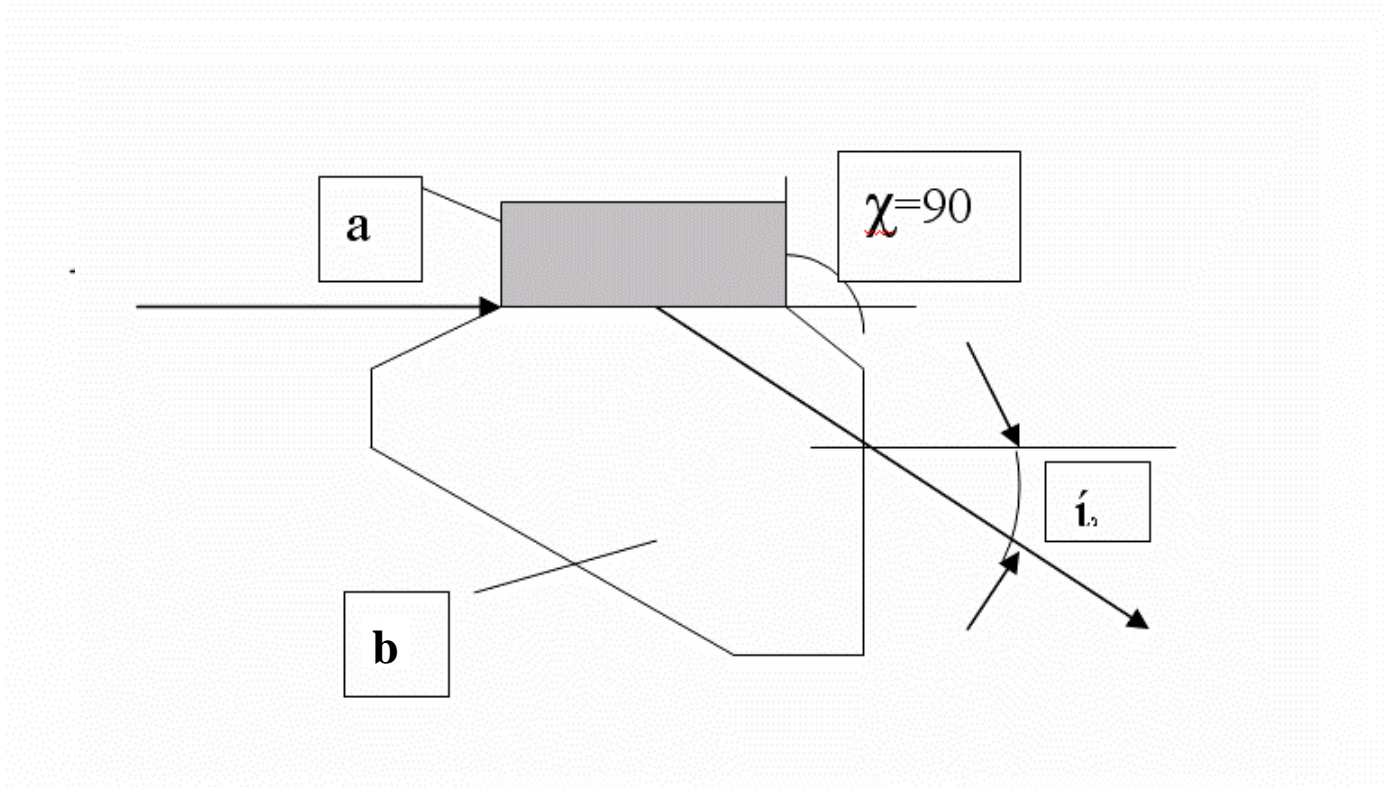


Figure 2

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

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

where: N_λ is the refractive index of the measuring prism (b) for the wavelength λ .

3.2.3.1.2. The test sample (a) shall be in the form of a rectangular plate with the minimum dimensions $15 \times 15 \times 4$ mm.

The angle between the active faces shall be $90^\circ \pm 10'$. 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 the maximum local deviation of 0,5 fringe.

The surfaces of the active faces shall be polished. The roughness parameter is $R_z \leq 0,050 \mu\text{m}$.

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

The immersion liquid used for lapping the sample shall have the 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 the prism angle $\varphi=60^\circ$ (Fig. 3).

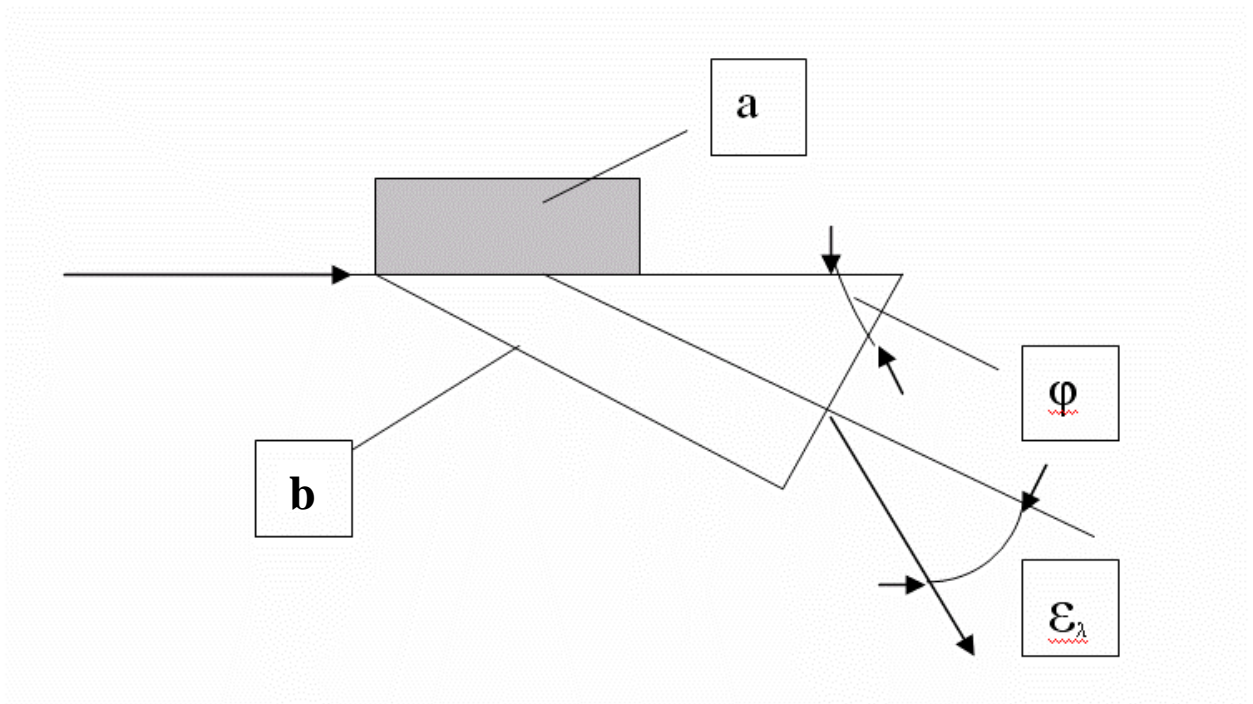


Figure 3

The Abbe refractometer is also equipped with an illuminating prism and an additional system of compensating prisms that allow for measuring in the white light (day light or electric light).

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

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

where φ is the measuring prism refraction angle;

N_{λ} is the measuring prism refractive index;

ε_{λ} is the critical angle of the beam entrance.

3.2.3.3. Dipping refractometers.

A dipping refractometer is one of the most accurate refractometers based on the total internal reflection (TIR) method.

The measuring prism of a dipping refractometer has a shape of an obliquely cut cylinder and is immersed into a test liquid during measurement.

The boundary of light and shade is sharper due to moving away the illumination prism that improves the measurement accuracy. The refractometer is also equipped with replaceable thermostated Abbe prisms.

3.2.3.4. Refractometers with broken total internal reflection (BTIR).

Refractometers measuring the refractive index by the BTIR method based on 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 ration of the velocity of light in vacuum “ c ” to the velocity of light in the medium “ v ” and is called an 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 air in laboratory environment under standard conditions.

The standard conditions for measurement of relative refractive indices are:

- temperature, $T = (20 \pm 2)$ °C;
- atmospheric pressure, $P = (101325 \pm 1000)$ Pa;
- relative humidity, $f = (50 \pm 30)$ %.

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

- for sodium green line Hg ($\lambda = 546,1$ nm) – 1,0002726;
- for Na doublet yellow line ($\lambda = 589,3$ nm) – 1,0002719;
- for He/Ne laser red line ($\lambda = 632,99$ nm) – 1,0002712.

The ratio 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.

Dependence of the refractive index n on temperature $t = 20$ °C is designated, for example, as n^{20} .

Dependence of the refractive index n on wave length is designated, for example, as n_D , where

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

D_1 is 589,6 nm;

D_2 is 589,0 nm.

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

Note: Modern instrument often apply LEDs with the wavelength close to the sodium D-line (e.g., $\lambda = 590$ 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.

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5.2. The maximum permissible error (MPE) in a certain range of influencing quantities shall be specified in the operation manual for refractometers of a certain type.

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 (informative Annex 2).

In the visible spectral region the light monochromatization shall be realized basically for the spectral lines: C ($\lambda_c = 656,3$ nm), C' ($\lambda_{c'} = 643,8$ nm), D ($\lambda_D = 589,3$ nm), d ($\lambda_d = 587,6$ nm), e ($\lambda_e = 546,1$ nm), F ($\lambda_F = 486,1$ nm), F' ($\lambda_{F'} = 480,0$ nm).

5.4. When refractometers are intended for control of optical glasses, measurements shall be taken basically for the following spectral lines: F' and C' (cadmium lamp), e (mercury lamp) and d (helium discharge tube).

5.5. When refractometers are designed for measurement of the refractive indices of liquids, measurements shall be taken basically 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 ($\lambda = 589,8$ nm), the refractive index values can be covered to n_D using the dispersion formula, if necessary. The mass fraction ($\%_{\text{mass}}$) shall be corrected therewith for taking into account dispersion and its dependence on mass fraction of a test liquid sample.

5.6. When refractometers are intended for simultaneous measurements of the refractive indices of solids and liquids, measurement shall be taken for the spectral lines indicated in 5.4 and 5.5.

Note: 1. When refractometers operate outside the visible spectral region, the radiation lines indicated in Table 2 (informative Annex 2), as well as LEDs, are used. The wavelengths may be different therewith from those indicated in 5.4.

2. It is permitted to apply a continuous spectrum source with interference filters or a monochromator (particularly, in the spectral region more than $1,5 \mu\text{m}$).

5.7. For all refractometers indicated in Table 1 (informative Annex 1), if the temperature of a test medium deviates from the standard value $20 \text{ }^\circ\text{C}$, the main normalizing parameters shall be maintained on condition that the temperature correction is applied, which value shall be indicated in the specifications for refractometers of certain type.

6. TECHNICAL REQUIREMENTS

6.1. 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-preventing coat (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. A measuring prism or a cell, its mandrel and other parts being in contact with the environment shall be made of materials that are stable towards this environment, as well as to acids, alkaline compounds and organic solvents.

6.5. Planes of a measuring prism and an illumination prism shall be closely fitting to each other providing for an optical contact and barring from running-off of a test liquid.

6.6. The design of compensator prisms shall provide for measurement results of the refractive index with a deviation of not more than a half of the maximum permissible error irrespective of the compensator.

6.6.1. The resolution of the automated digital refractometers shall not exceed $1 \cdot 10^{-4}$ or $0.1 \%_{\text{mass}}$ for the refractive index or the mass concentration, respectively.

6.6.2. The working temperature range shall be specified in the operation manual. The scale interval of applied thermometric sensors is $0.5 \text{ }^{\circ}\text{C}$; $0.2 \text{ }^{\circ}\text{C}$; $0.1 \text{ }^{\circ}\text{C}$ or less.

6.7. Thermostatic chambers shall be hermetically sealed and be equipped with a built-in thermometer or have an opening for installation of a thermometer.

6.8. Refractometers with external power sources (mains) shall have a safe bonding point.

6.9. Plane-parallel plates, prisms and refractometric liquids shall be applied for verification of refractometers.

6.10. Refractometers shall operate continuously not less than 8 hours, the warm-up time being not more than 10 % of the continuous operation time. In switching the ranges the warm-up time shall be not more than 5 % of the continuous operation time.

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

6.12. The warranty assurance of automated refractometers shall be minimum 18 months after putting into operation.

7. VERIFICATION

7.1. Conditions of verification and preparation to it.

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

7.1.2. Verification of refractometers using refractometric and immersion liquids shall be carried out in a room with active exhaust ventilation.

7.1.3. A refractometer shall be installed so that there is sufficient space for heat irradiation and air circulation, away from a conditioner or central heating at the distance at least 1,5 m.

7.1.4. Refractometers with line supply (mains) shall be verified in 30 minutes after turning on.

7.1.5. A refractometer shall not be exposed to direct sun rays.

7.1.6. During verification a refractometer shall not be subject to vibration, shaking or shocks, as well as to external electric and magnetic fields, which may influence its operation, based on the requirements to the rooms where verification is carried out.

7.1.7. Permissible concentrations of interfering and aggressive components in ambient air in a room where verification is carried out shall not exceed the values specified in the national normative documents.

7.1.8. Before verification a refractometer shall be kept in a laboratory at the temperature from +18 °C to +22 °C during at least 12 hours.

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

7.1.10. The verification means shall be prepared in accordance of their instruction manuals.

7.2. Performance of verification.

7.2.1. External examination.

During external examination one shall determine the following:

- completeness of a refractometer in accordance with the requirements of an instruction manual;
- absence of defects preventing from 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 a manufacturer's name or trade mark, a type and a serial number.

7.2.2. Refractometers not meeting the above requirements are not subject to further verification.

7.3. Testing.

7.3.1. During testing the operation of a refractometer shall be checked in accordance with the operation manual using standard means of verification having certificates or verification reports.

7.3.2. The following points shall be ascertained during testing:

- correspondence of the working range of a refractometer to the one specified in the operation manual. (It is checked by a single measurement of the refractive index of the initial point of the working range, e.g., n_D^{20} near 1.3 and the end point of the working range, e.g., n_D^{20} near 1.7 using the verification means indicated in clause 7.4.)

Note: The refractometer working range claimed for verification may depend on measurement problems and may be lower than the underlying construction capabilities, verification being performed in the claimed working range.

- coincidence 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});
- work of the refractometer software and correctness of the displayed information;
- operation of a device for measurement and stabilization of the measuring prism temperature (readings shall correspond to $(20 \pm 0,1)^\circ\text{C}$).

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 1,47 - 1,94 and trihedral acute-angled and rectangular glass prisms in the measuring range 1,25 - 1,45.

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

- liquid samples: refractometric liquids in the measuring range 1,3 – 1,7.

Note: 1. The list of refractometric liquids recommended for verification of refractometers with the nominal refractive index values is given as an example in Table 3 (Recommended Annex 3).

2. The verification means shall be approved by the national and international metrology organizations as certified reference materials (CRM's) and be accompanied with a certificate containing the names, the values of refractive indices relative to air, the 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 expiry date.

7.4.1. Verification by means of solid samples is carried out in the following way.

A drop of immersion liquid with the diameter of about 1 mm is put on the polished work surface of a well-cleaned standard plate or prism. The following liquids shall 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 upon the measuring prism of a refractometer so that the immersion liquid could be equally distributed, and is grinded to obtain an optical contact. The refractive index is measured. Measurements are repeated five times. The standard plate or prism is removed therewith 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.

Doubly distilled water with the conductivity $(1,0 \dots 1,1) \cdot 10^{-6} \Omega^{-1}\text{cm}^{-1}$ is used to verify the lower limit.

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

The other points on the refractometer scale are verified by 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 of constant values of their refractive indices, low volatility, non-toxicity, non-hygroscopicity and 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 shall 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 volume of ampoules is determined by the manufacturer reasoning from their sufficiency for verification of a refractometer at the minimum flow-rate of a liquid sample within the limits of 0.2 - 1 ml.

7.4.3. Verification by liquid samples using refractometric liquids is carried out in the following way.

7.4.3.1. To apply 0,2 – 1 ml of doubly distilled water on the surface of the measuring prism or to pour it into the measuring cell of the refractometer and to wait for the temperature to be set within the required values, and to measure the refractive index.

7.4.3.2. Remove the sample of doubly distilled water from the surface of the measuring prism or cell. Put again the other 0.2 - 1 ml of doubly distilled water on the surface of the measuring prism or cell and take readings after the required temperature has been set. Measurements are taken five times, the measuring sample being put or removed from the surface of the measuring prism or cell. The average refractive index is calculated from the five values obtained.

7.4.3.3. Measure one after another the refractive indices of some other standard refractometric liquids (CRM) chosen from Table 3 (Recommended Annex 3), performing operations from clauses 7.4.3.1 and 7.4.3.2.

7.4.3.4. Before each measurement the planes of the prisms or the cell shall be cleaned with distilled water or ethyl alcohol, wiped with gauze or cotton wool and dried. The time interval between measurements shall be at least 60 seconds including the time of cleaning and drying of the surfaces of the prisms or the cell after each measurement.

7.4.4. Refractometers with scales numbered in the weight values of dry substance can be also verified using fresh sucrose-water solution in accordance with the Table for sucrose-water solutions (Informative Annex 4) of the standard (ICUMSA Specification and Standard SPS - 3 2000). The corrections should be applied if the temperature deviates from 20 °C, using Table 5 (Informative Annex 5). The procedure for preparation of sucrose-water solutions is given in Informative Annex 7. The measurement procedure corresponds to clauses 7.4.3.1 and 7.4.3.2, and the further processing of measurement results corresponds to clause 7.5.

7.5. Processing of measurement results. (Budget of uncertainties)

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

7.5.2. The initial data for determination of the deviations of the refractometer measurement results from the measurand values are the results of multiple measurements ($i=5$).

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

$$E = n_{D_0} - n_{D_{av}} \quad (4),$$

where n_{D_0} is the refractive index value of the standard verification mean (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 are the following:

- refractive index uncertainty of the standard verification mean determined in the process of its certification;
- random scatter of the measurement results;
- uncertainty of temperature maintenance, measurement temperature of the measuring prism and temperature dependence of the refractive index of the standard liquid sample (CRM) (if the CRM of liquid is applied).

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

$$u_A = \sqrt{\frac{1}{m(m-1)} \cdot \sum_{i=1}^m (n_{D_i} - n_{D_{av}})^2} \quad (5),$$

where: $m = 5$ (number of measurements)

$n_{D_{av}}$ is the average value of the refractive indices of the standard verification mean obtained during measurement using the refractometer;

7.5.6. Type B standard uncertainty:

$$u_B = \sqrt{u_{CRM}^2 + u_{reading}^2 + u_t^2}, \quad (6)$$

where: u_{CRM} is the standard uncertainty $u(k=1)$ of the applied 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 by the formula:

$$u_c = \sqrt{u_A^2 + u_B^2} \quad (7)$$

7.5.8. The expanded uncertainty $U(k=2)$ is calculated by the formula:

$$U(k=2) = 2 \cdot u_c \quad (8)$$

7.5.9. The refractometer error (E) calculated by the formula in clause 7.5.3 shall be smaller or equal to the maximum permissible error (MPE) at each control point. The maximum permissible error is given in the operation manual of the refractometer.

$$|n_{D_0} - n_{D_{av}}| \leq MPE \quad (9)$$

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

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

8. DRAWING UP OF VERIFICATION RESULTS

8.1. The verification results are entered into the protocol.

8.2. The verification results are considered to be positive if the requirements of clause 7 are met and the received values of the refractometer measurement results do not exceed the MPE specified in the operation manual.

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

8.4. Seals with the verification mark stamps are put in those places that prevent from entering the adjustment components of the refractometer.

8.5. If according to the verification results the refractometer is rejected as defective, the stamp of the verification mark canceled and the certificate of verification is abrogated.

8.6. If the results of verification of refractometers are negative after their production, the refractometers are returned to the manufacturer to remedy the defects with an opportunity to repeat verification.

Technical and Metrological Characteristics of the Most Common Refractometer Types

Table 1

Type and model of a refractometer	Refractive index measurement range	Standard total measurement uncertainty		Purpose and prevailing scope
		refractive index u_{c_n}	dispersion u_{cD_n}	
1. 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
2. TIR refractometers: - 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

3. 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
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Wavelengths and the corresponding spectral lines of chemical elements

Table 2

Wavelength λ, nm	Wavelength designation	Chemical element
365,0 ₁	<i>i</i>	Hg
404,6 ₆	<i>h</i>	Hg
435,8 ₃	<i>g</i>	Hg
479,9 ₉	<i>F'</i>	Cd
486,1 ₃	<i>F</i>	H
546,0 ₇	<i>e</i>	Hg
587,5 ₆	<i>d</i>	He
589,2 ₉	<i>D</i>	Na
632,9 ₉	-	He/Ne (laser)
643,8 ₅	<i>C'</i>	Cd
656,2 ₈	<i>C</i>	H
694,3	-	Cr+Al ₂ O ₃ (laser)
706,5 ₂	<i>r</i>	He
852,1 ₁	<i>s</i>	Cs
1013,9 ₈	<i>t</i>	Hg
1060,0	-	Nd (laser)
1128,6 ₆	-	Hg
1153,0	-	He/Ne (laser)
3392,2	-	He/Ne (laser)
1395,1	-	Hg
10600,0	-	CO ₂ (laser)

Refractive Index Values of Refractometric Liquids (CRMs) Used for Verification of Refractometers

Table 3

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
doubly distilled water	1,3330

Note:

More accurate refractive index values of refractometric liquids relative to air at the temperature 20 °C and their temperature coefficients shall be indicated in passports or certificates 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 shall not exceed therewith the value $\pm (2 - 3) \cdot 10^{-5}$.

International Refractive Index Scale of ICUMSA (1974)
for pure sucrose solutions at 20°C and 589 nm
 This Table gives values of refractive index against air with sucrose mass fraction

Annex 4
(informative)

Table 4

n_D^{20} 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	1.344765	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
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

Table 4 (continued)

International Refractive Index Scale of ICUMSA (1974)
for pure sucrose solutions at 20°C and 589 nm

This Table gives values of refractive index against air with sucrose mass fraction

Sucrose g/100 g	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
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
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	1.454893	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

This Table gives mass fraction corrections to refractometric
Tables for sucrose solutions at 589 nm for temperatures
different from 20°C

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	+0.16	+0.16	+0.16	+0.16	+0.16	+0.16	+0.16	+0.16	+0.15	+0.15	+0.15
23	+0.20	+0.21	+0.21	+0.22	+0.22	+0.23	+0.23	+0.23	+0.24	+0.24	+0.24	+0.24	+0.24	+0.24	+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	+0.32	+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	+0.40	+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	+0.48	+0.47	+0.46	+0.46	+0.45
27	+0.50	+0.51	+0.52	+0.53	+0.54	+0.55	+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	+0.81	+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

Terminology

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

Error of indication

difference of indication of a measuring system and 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 authoritative body and referring to valid procedures used to obtain a specified property value with uncertainty and traceability

Procedure for Preparation of Test Sucrose-Water Solutions

1. Preparation of initial solution (No.1)

Initial solution No.1 in the amount of 1000 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 a interference refractometer with the uncertainty $\pm 3 \cdot 10^{-5}$

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

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

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

$$V_{w_i} = \left(1 - \frac{\Delta n_{t_i}}{\Delta n_{t_1}} \right) V_{v_i}$$

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_1} is the difference of the refractive indices of initial solution No.1 relative to distilled water measured before preparation of the test solution;

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

i is the prepared test solution number.

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

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

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

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

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

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

Verification Protocol

Refractometer
 Serial number
 Inventory number
 Manufacturer
 Owner of refractometer
 Date of testing

Conditions of verification performing:

-ambient temperature °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 _____ °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 Results of checking the fulfillment of the requirements of item 7.5.9 _____
 - 3.9 Results of checking the fulfillment of the requirements of item 7.5.10 _____

Conclusion**Person(s) responsible for the verification:**

Signature(s) _____

Date _____

Title(s) _____