INTERNATIONAL
RECOMMENDATION

OIML R 14

Edition 1995 (E)

Polarimetric saccharimeters graduated in accordance with the ICUMSA International Sugar Scale

Saccharimètres polarimétriques gradués selon
l'Échelle Internationale de Sucre de l'ICUMSA

OIML R 14 Edition 1995 (E)

ORGANISATION INTERNATIONALE
DE MÉTROLOGIE LÉGALE

INTERNATIONAL ORGANIZATION
OF LEGAL METROLOGY

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Foreword

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This publication – reference OIML R 14, edition 1995 (E) – was developped by the OIML subcommittee TC 17/SC 2 S*accharimetry*. It was approved for final publication by the International Committee of Legal Metrology in 1994 and will be submitted to the International Conference of Legal Metrology in 1996 for formal sanction. It supersedes the previous edition dated 1978.

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POLARIMETRIC SACCHARIMETERS
graduated in accordance with
the ICUMSA International Sugar Scale

1 Scope

Polarimetric saccharimeters are instruments which measure the relationship between the optical rotation caused by an aqueous solution of a sample and that caused by a pure sucrose solution of prescribed concentration, using the same polarized light.

This Recommendation applies to polarimetric saccharimeters that are graduated in sugar degrees (°Z) in accordance with the ICUMSA International Sugar Scale (see Annex A) and that belong to the visual or photo-electric type, including:

a) saccharimeters with a rotating polarizer or analyser of which the angle of rotation can be measured and which are illuminated by a monochromatic light source of known wavelength which must be between 540 nm and 633 nm;

b) saccharimeters with quartz wedge compensation and illuminated either by a monochromatic light source of known wavelength which must be between 540 nm and 633 nm, or by the light of an incandescent lamp filtered in such a way that the distribution of the spectral energy from the lamp, arising from the degree of spectral filtration by the filter and from the spectral sensitivity of the detector (eye or photo-multiplier), permits the use of radiation of effective wavelength 587 nm;

c) saccharimeters which operate according to a principle of measurement other than those given in (a) and (b).

2 General

2.1 Visual saccharimeters must be of the half-shadow type, but the method of obtaining this half-shadow must not risk inducing systematic errors.

The half-shadow angle can be fixed or variable; if fixed, it must be between 5° and 10°; if variable, it must be adjustable between at least 0° and 10° and be adjustable to each whole number of degrees.

2.2 Calcite polarizers with inclined faces are not authorized.

3 Scale

3.1 The scale on saccharimeters must be graduated in international sugar degrees (°Z).

3.1.1 The scale must be linear(*); the range of measurement may be from − 30 °Z to + 120 °Z or only a part of this range; the reference temperature must be 20 °C.

(*) A linear scale is sufficient in practice although the optical rotation is not strictly proportional to the sugar concentration; in fact, the deviation from the proportionality does not exceed 0.01 °Z.

3.1.2 Whatever the length of the specific polarimeter tube of a saccharimeter, the scale of which is graduated up to 100 °Z, this scale must be such that a normal sugar solution (see Annex A, subclause A.2) gives an indication of 100 °Z in this tube.

For saccharimeters where the scale is graduated only up to x °Z ($x < 100$), the length of the specific tube must be such that a solution of concentration x % of the normal solution gives an indication of x °Z in this tube.

3.1.3 In all cases, saccharimeters must be graduated so that 100 °Z on the scale corresponds to a normal sugar solution (26 g/100 cm^3).

However, when the instrument is in use, if there is not 26 g of sample to be studied in 100 cm^3 of solution, the °Z value of the sample is determined:

a) either by multiplying the value read by the quotient of the normal concentration (26 g/100 cm^3) and of the concentration of the solution examined (also expressed in g/100 cm^3); this calculation may be made directly by the saccharimeter by an appropriate setting;

b) or by using a tube whose length is obtained by multiplying the specific tube length for the instrument by the factor indicated in (a) above.

If the sample to be studied is not completely soluble in water, there may be variations in volume which, with different samples, may result in deviations. Details for certain products are given in the methods of operation.

3.2 In all cases, it must be possible to calibrate the saccharimeters.

3.2.1 Except in the case mentioned in 3.2.2, it must be possible to test and calibrate the scale by means of quartz plates for saccharimetric control, or by means of other suitable standards(*).

3.2.2 Saccharimeters with an angle of rotation less than or equal to 2.5° at the 100 °Z point and a wavelength of 546 nm may be calibrated by means of a sugar solution in pure water(**).

4 Materials

4.1 Saccharimeters must be made of materials resistant to the chemical agents used during sugar measurements.

4.2 Ferro-magnetic materials can be used, provided that they do not influence the optical rotation.

4.3 The quartz in saccharimeters with a quartz compensator must be sufficiently free from homogeneity faults, twinning, striae and inclusions, so that the indicated accuracy of measurement is not affected.

(*) The standards will be considered to be suitable if their characteristics are such that their value remains constant to ± 0.02 % for 5 years.

(**) See *Proceedings of the 16th Session of ICUMSA, Ankara, 1974, Subject 5* for details on the preparation of the sugar solution and its measurement.

4.4 The lenses and glass plates through which the light rays pass (between the polarizer and the analyser) must be sufficiently free from internal strain so that variations in the indications of the instrument, for all normal positions of the polarimeter tube or of the control standard, do not exceed 0.01 °Z at any point of the measuring range (see 8.7).

5 Construction

5.1 The frame and the mounting of saccharimeters must be sufficiently stable so that the measurements are not affected by vibrations and shaking that may occur normally in the working premises.

The construction must permit the optical parts of the instrument to be cleaned easily, particularly with regard to the dirt caused by the sugar solution or by the chemicals used during the preparation of solutions.

The internal optical parts must be inaccessible to unauthorized persons.

5.2 The path of the light rays must be such that they are not masked once the polarimeter tube is put into place, and that they are not reflected by the internal wall of this tube.

That part of the path of the rays in which the polarimeter tube lies must be limited at both ends by a circular diaphragm, unless the path of the light rays is established in such a way that it is limited in a parallel manner in the polarimeter tube(*).

5.3 The setting-up or the methods of adjusting the support of the polarimeter tube and the quartz plates or control standards must be such that the angle formed by the optical axis of the instrument and the axis of the tube or the mounts of the plates or standards is (or can be adjusted to be) less than 0.5°.

5.4 A zero resetting device must be provided on the indicator.

5.5 The operation of the instrument must not cause the temperature of the polarimeter tube to increase by more than 1 °C.

5.6 Visual half-shadow saccharimeters must be such that their eye-pieces allow both focusing on the separation line of the two sections of the optical field, and illumination over all parts of the optical field to appear uniform to the observer in the event of equilibrium of the half-shadow.

5.7 The illumination of saccharimeters must be such that, during the measurement of coloured solutions, whose optical density per centimetre does not exceed the values indicated in 5.10, any variation in the effective wavelength of the light, caused by the solution, cannot affect the results of the measurements by more than 0.02 %.

(*) See e.g. *Anwendung der Magnetorotation in lichtelektrischen Polarimetern*, Hans Wenking, Zeitschrift Instrumentenkunde, Vol.66, No 1 (1958), page 4.

5.8 The wavelength of the light source must be chosen by considering:

- ! the optical rotation of the sugar solutions,
- ! the optical density of the sugar solutions (see 5.10),
- ! the spectral sensitivity of the detector.

5.9 The noise level in photo-electric saccharimeters and the sensitivity of visual saccharimeters must be such that the measurement accuracy specified in clause 6 is assured for all measurements to be made by the instruments.

5.10 As far as photo-electric instruments are concerned, the intensity of the light source must be such that, for the chosen wavelength, the optical rotation produced by a solution, with an optical density per centimetre less than or equal to the ordinate value at the point corresponding to the chosen wave-length on the curve given in Figure 1, can be measured by the instrument without reducing the accuracy of measurement specified in clause 6.

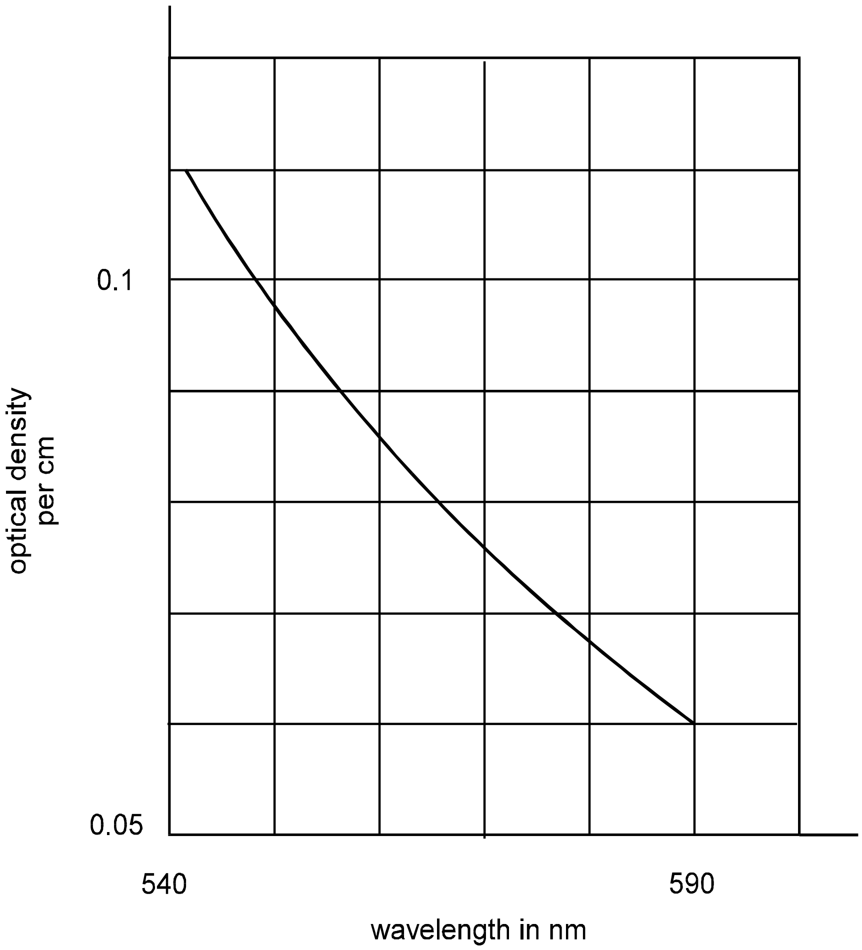

Figure 1

5.11 After the first half-hour of operation, the systematic error due to drift of photo-electric saccharimeters must not exceed 0.05 °Z over 24 hours.

5.12 The definitive indication of automatic saccharimeters must be obtained within a maximum of 30 seconds, whatever the value of this indication.

5.13 Electrically-powered saccharimeters must satisfy the specifications of this Recommendation despite variations of the electric power supply of ± 10 % in voltage and ± 2 % in frequency in relation to their nominal values.

6 Classes of accuracy and measurement uncertainty

6.1 According to the uncertainty affecting the measurements, saccharimeters are divided into three classes of accuracy:

class 0.2 – class 0.1 – class 0.05

6.2 The overall uncertainty affecting any measurement over the scale must not exceed:

a) for saccharimeters of class 0.2 .... ± 0.2 °Z
b) for saccharimeters of class 0.1 .... ± 0.1 °Z
c) for saccharimeters of class 0.05 .... ± 0.05 °Z

calculated according to the *Guide to the expression of uncertainty in measurement*, with a coverage factor k = 2.

7 Inscriptions

Saccharimeters must bear the following inscriptions, in a clearly legible and indelible manner:

! name and address of the manufacturer or his mark,
! serial number,
! legend "ICUMSA International Sugar Scale",
! class of accuracy,
! length of the polarimeter tube or tubes which must be used,
! mass of the sample of the substance, contained in 100 cm^3 of solution, where this mass is not 26 g, and if the instrument scale takes this difference into account, i.e. the instrument indication gives, by simple reading, the value of the sample expressed in °Z (see 3.1.3),
! reference temperature of 20 °C and the wavelength for which the saccharimetric scale is valid.

8 Polarimeter tubes

8.1 General

Polarimeter tubes can be simple or with a circulating solution and, in both cases, with or without a thermostatic sheath. It must be possible to fill them completely without leaving any air bubbles likely to affect the path of the light rays.

In tubes with a removable thermostatic sheath, it must be possible to dismantle and reassemble this sheath easily without affecting the length of the tube by more than the tolerances given in columns 2 and 3 of Table 1 (see 8.5). In tubes with a circulating solution, the entry and exit points of the solution must be as near the ends as possible.

The ends of the tubes must extend from 0.2 mm to 1 mm beyond the supporting collars centering the tube.

Tubes must be made of materials resistant to chemical cleaning products, whether these products are of high pH or low (e.g. 50 % acetic acid) and their construction must allow manual external and internal cleaning.

8.2 Lengths

The nominal tube lengths must be a minimum of 10 mm at the reference temperature of 20 °C.

According to the accuracy of the adjustment of their true length to the nominal value of this length, the tubes are divided into two classes of accuracy given in columns 2 and 3 of Table 1 which gives, as example, six lengths(*) in current use.

At 20 °C, the measured length of a tube must not differ from its nominal value by more than the permissible deviation given for its class of accuracy. If the difference between the measured length and the nominal length is less than the permissible deviation shown in column 2, this tube need not bear an indication of its measured length. If the difference between the measured length and the nominal length exceeds the permissible deviation shown in column 2 (while remaining less than that given in column 3), this tube must bear an indication of its measured length, given to the accuracy in column 2. When this tube is used, the quotient of its nominal length to its measured length can act as a correction factor for precision measurements.

8.3 Internal tube diameter

The internal tube diameter must not exceed 10 mm. All precautions must be taken to reduce internal reflections (particularly in long, small diameter tubes).

8.4 Refilling of tubes with a circulating solution

Tubes with a circulating solution must have dimensions and a construction such that these tubes can be refilled (total replacement of the contained solution) without the total error of measurement exceeding the maximum permissible error for the class of accuracy of the instrument.

8.5 End faces of the tubes

a) Quality of surface finish

The plane end faces of glass tubes must be of good optical quality. The plane end faces of tubes of materials other than glass must be ground. In all cases, the surface condition of these faces must be such that the mean arithmetic roughness characterising this condition does not exceed in any direction the values shown in column 4 of Table 1.

b) Flatness, parallelism, and perpendicularity to tube axis

The end faces must be plane, parallel to each other, perpendicular to the tube axis within the limits shown in columns 5, 6, and 7 of Table 1.

(*) The values of the characteristics shown in Table 1 correspond to the nominal lengths chosen as examples. For different lengths, the values are calculated by interpolation and rounding.

Table 1 - Characteristics of polarimeter tubes

Nominal length L mm	Admissible deviation ΔL between the measured length and the nominal length		Plane end faces maximum permissible errors			
	ΔL/L = 0.01% ± µm	ΔL/L = 0.2% ± µm	Maximum roughness R_a(*) µm	Flatness µm	Parallelism µm	Perpendicularity to tube axis Minutes of angle
(1)	(2)	(3)	(4)	(5)	(6)	(7)
10	1	20	0.2	1	1	2
20	2	40	0.3	1	2	3
50	5	100	0.4	2	4	4
100	10	200	0.6	3	6	6
200	20	400	0.8	4	8	8
400	40	800	1.0	5	10	10

(*) R_a = Mean arithmetic deviation of the average line of the profile (see ISO R 468-1966).

8.6 Mounting of the tubes

The tubes and their mountings must be such that, when mounted in the corresponding saccharimeter, they do not cause disturbance of the light rays, and the tube axis coincides with the optical axis of the saccharimeter to within 0.5° (see 5.3).

8.7 Cover glasses

The thickness of the cover glasses at the tube ends must be between 1 and 2 mm. Their faces must have a good optical finish and they must be flat to within 0.01 % of the length of the shortest tube with which they are to be used. The parallelism of the two faces must be such that their angle is less than 5'.

These glasses must be sufficiently free from internal strain so that the saccharimeter indication does not vary by more than 0.01 °Z in the following cases:

! when one cover glass of an empty tube is rotated in relation to the other between 0° and 180°,
! when the empty tube with its glasses is rotated around its axis between 0° and 180° (or, if the tube cannot rotate in this way, when only the two glasses are rotated simultaneously).

8.8 Inscriptions

Each tube must bear the following inscriptions, in a clearly legible and indelible manner:

! name and address of the manufacturer or his mark,
! serial number,
! class of accuracy represented by the accuracy of its length (ΔL/L = 0.01 % or ΔL/L = 0.2 %),
! nominal length and, if necessary, measured length (see 8.2).

9 Quartz plates for saccharimetric control

9.1 General

The calibration of the saccharimetric scale with a sugar solution is easily affected by errors due, for example, to the evaporation of the solution, to its instability, and to the considerable effect of temperature on optical rotation. It is therefore recommended that quartz plates be used for the calibration and control of saccharimeters.

9.2 Sugar values of quartz control plates

9.2.1 A quartz control plate is called normal when it produces the same optical rotation as the normal sugar solution, in the reference conditions set out in Annex A, subclause A.2. Such a plate has a sugar value of 100 °Z for $\lambda = 546.2271$ nm.

9.2.2 A quartz control plate, for monochromatic light of wavelength λ other than 546.2271 nm, will have a sugar value of 100 °Z, if the rotation which it produces is the one resulting from the definitions given in Annex A, subclause A.3 (the spectral region used being limited to the interval from 540 nm to 633 nm).

9.2.3 A quartz control plate for a quartz wedge saccharimeter illuminated through a filter by the non-monochromatic light of an incandescent lamp (effective wavelength 587 nm, see 1.1.b) will have a sugar value of 100 °Z, if the values of optical rotation which it produces are(*):

for mercury: $\alpha_{546.2271\ \mathrm{nm}} = 40.704°$
for sodium: $\alpha_{589.4400\ \mathrm{nm}} = 34.629°$
for He-Ne laser: $\alpha_{632.9914\ \mathrm{nm}} = 29.792°$

9.3 Quality of the quartz

9.3.1 The quality of the quartz plates used must be such that:

- when placed between crossed polarizers, this quartz allows one to obtain total extinction in intense white light with the aid of a quartz compensator,
- when observed visually, this quartz is optically homogeneous and free from twinning, striae and inclusions,
- the sugar value of the plate does not vary by more than 0.005 °Z when the plate is rotated in its plane.

9.4 Form and dimensions

9.4.1 The quartz plates must be in the form of a right circular cylinder with a diameter of 16.0 mm ± 1.0 mm. They must have plane, parallel faces, cut perpendicular to the optic axis of the quartz. The circular edges must be chamfered to a maximum of 0.2 mm.

(*) The sugar value of quartz control plates for saccharimeters with quartz wedge compensation in non-monochromatic light is easily and accurately determined by measuring the optical rotation in monochromatic light (see F. Bates and R. F. Jackson, Bulletin of the Bureau of Standards 13 (1916); R. Bünnagel, PTB - Mitt. Nr 5, 1966).

9.4.2 Quartz plates where the optical rotation for λ = 546 nm is less than 10° must consist of a plate of right-handed quartz and a plate of left-handed quartz. The thickness of each plate must not be less than 0.4 mm and the sum of the thicknesses must not exceed 1.6 mm.

9.5 Tolerances on form and dimensions

9.5.1 Flatness of the faces

It must be possible to contain each face between two imaginary parallel planes 0.5 µm apart, at the maximum.

9.5.2 Parallelism of the faces

If the length of the tube is not less than 20 mm, the thickness of the plate must not vary by more than 0.3 µm over its whole area. For the tube lengths less than 20 mm, this variation must not exceed 0.15 µm.

9.5.3 Optic axis error

The angle formed by the optic axis of the quartz and the perpendicular to one of the faces must be less than 10'.

9.6 Mounting of the quartz plates

9.6.1 The single or double quartz plates must be mounted, free from strain, in a tubular mount with circular flanges at the ends.

9.6.2 The free internal diameter of the tubular mount must be at least equal to 10 mm.

9.6.3 The diameter of the end flanges must be suitable for supporting the polarimeter tubes in order to allow coincidence of the optic axis of the saccharimeter and the axis of the mounting.

The permitted machining tolerances on these diameters must be such that the possible difference between the diameters cannot produce an angle error of more than 10' between the abovementioned axes.

9.6.4 The angle formed by the axis of the mount and the perpendicular to one of the faces of the plate must not exceed 10'.

9.6.5 The play of the plate within its mount must be less than or equal to 0.2 mm in the direction of the plane of the faces and contained within 5 µm and 30 µm in the direction of the axis. For lengths less than 20 mm, the play must be contained between 5 µm and 20 µm in the direction of the axis.

9.7 Inscriptions

One of the faces of the mount or its tubular part must bear the following inscriptions, in a clearly legible and indelible manner:

- name and address of the manufacturer or his mark,
- sugar value, in °Z, of the quartz control plate for a tube length of 200 mm,
- wavelength at which this plate must be used, given to five significant figures,
- year and serial number corresponding to the information on the calibration certificate, when a certificate is issued with the quartz control plate.

10 Liability to metrological controls

When, in any country, polarimetric saccharimeters are submitted to State metrological controls, these controls must include, in accordance with the internal legislation of that country, all or some of the following operations.

10.1 Pattern approval

10.1.1 Each pattern of a polarimetric saccharimeter from each manufacturer is submitted to the pattern approval procedure.

10.1.2 Without special authorisation, no modification may be made to an approved pattern.

10.2 Initial verification

New, repaired or readjusted polarimetric saccharimeters must undergo the initial verification tests.

10.3 Periodic verifications

Periodic verifications will be made to ensure that polarimetric saccharimeters in service retain their metrological properties.

10.4 Control procedures will be fixed in accordance with the national regulations of each country.

11 Metrological control marks

Metrological control marks and, if necessary, information on calibrations, will be applied on a visible part (where they do not interfere with the use of the instrument) of saccharimeters, polarimeter tubes, and quartz control plate mounts.

ANNEX A

ICUMSA INTERNATIONAL SUGAR SCALE

A.1 The ICUMSA International Sugar Scale fixes the basis of a polarimetric method allowing determination of the relationship between the optical rotation caused by an aqueous solution of a sample and that caused by a pure sucrose solution of prescribed concentration using the same polarized light.This scale is determined and graduated in accordance with the following requirements.

A.2 The 100 °Z point of the International Sugar Scale is fixed by the optical rotation α undergone by the polarized light of the green line of the mercury isotope 198 ($\lambda = 546.2271$ nm in vacuum), when passing through a 200.000 mm length of sucrose solution in pure water, kept at a temperature of 20.00 °C, and containing 26.0160 g of pure sucrose weighed in vacuum per 100.000 cm^3 of solution (normal sugar solution).

A mass of 26.0160 g of sucrose corresponds to 26.000 g when this sucrose is weighed in air by means of weights with a density of 8 000 $\mathrm{kg/m}^3$ in air, at a standard pressure of 101 325 Pa, at a temperature of 20 °C and a relative humidity of 50 %, the density of this air therefore being 1.2 $\mathrm{kg/m}^3$ (see International Recommendation OIML R 33).

The 0 °Z point is fixed by the indication given by the saccharimeter for pure water.

In the range from 0 °Z to 100 °Z, the scale graduation is linear because the optical rotation is practically proportional to the sucrose concentration of the solution.

In the given conditions, 100 °Z corresponds to an angle of optical rotation(*):

$$\alpha_{546.2271\,\mathrm{nm}}^{20.00\,^\circ\mathrm{C}} = (40.777 \pm 0.001)^\circ$$

A.3 For light of wavelengths other than that of the green line of the mercury isotope 198 (546.2271 nm), the 100 °Z point is defined by the optical rotation which is given (under the same conditions as those given in A.2) by the formula:

$$\frac{\alpha_\lambda}{\alpha_{546.2271\,\mathrm{nm}}} = a + \frac{b}{\lambda^2} + \frac{c}{\lambda^4} + \frac{d}{\lambda^8}$$

where $a = -\,1.7982 \times 10^{-3}$ $b = +\,2.765318 \times 10^{5}$
$c = +\,6.55736 \times 10^{9}$ $d = +\,1.03825 \times 10^{19}$
λ being in nm the wavelength in vacuum of the light used.

A.4 For the yellow light of spectrally purified sodium, the wavelength $\lambda_{\mathrm{vacuum}} = 589.4400$ nm is taken as the mean optical centre of gravity.

For 100 °Z, A.2 and A.3 give a rotation value(*) of:

$$\alpha_{589.4400\,\mathrm{nm}}^{20.00\,^\circ\mathrm{C}} = (34.626 \pm 0.001)^\circ$$

A.5 For the red light of the He-Ne laser with $\lambda_{\mathrm{vacuum}} = 632.9914$ nm, the 100 °Z point from A.2 and A.3 results in a rotation value(*) of:

$$\alpha_{632.9914\,\mathrm{nm}}^{20.00\,^\circ\mathrm{C}} = (29.751 \pm 0.001)^\circ$$

(*) These rotation values were accepted at the 1986 ICUMSA meeting and have been valid since 1 July 1988.