Official Journal L 239

of the European Communities

Volume 22 22 September 1979

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Acts whose titles are printed in light type are those relating to day-to-day management of agricultural matters, and are generally valid for a limited period.

The titles of all other Acts are printed in bold type and preceded by an asterisk.

II

(Acts whose publication is not obligatory)

COMMISSION

COMMISSION DIRECTIVE

of 20 July 1979

adapting to technical progress Council Directive 71/127/EEC on the approximation of the laws of the Member States relating to the rear-view mirrors of motor vehicles

(79/795/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 70/156/EEC of 6 February 1970 on the approximation of the laws of the Member States relating to the type-approval of motor vehicles and their trailers (1) , as last amended by Directive $78/547/EEC$ (2), and in particular Articles 11, 12 and 13 thereof,

Having regard to Council Directive 71/127/EEC of ¹ March 1971 on the approximation of the laws of the Member States relating to the rear-view mirrors of motor vehicles (3) ,

Whereas, in the light of the experience gained and in view of the present state of technology, it is now possible to make the relevant provisions fuller, more stringent and better adapted to actual test conditions;

Whereas Council Directive 71/127/EEC provides that specifications on external rear-view mirrors adjustable from the driving position are to be drawn up as soon as technological development permits;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Committee on the Adaptation to Technical Progress of Directives for the Removal of Technical Barriers to Trade in the Motor Vehicles Sector,

HAS ADOPTED THIS DIRECTIVE:

Article ¹

Directive 71/127/EEC is hereby amended as follows:

1. The last subparagraph of Article $3(2)$ is amended to read as follows:

'There shall be failure to conform to the approved type, within the meaning of the first subparagraph, where the requirements of item 2 of Annex I are not observed.'

2. Article 7 is replaced by the following:

'1. With effect from 1 February 1980, no Member State may, on grounds relating to rear-view mirrors:

- (a) refuse, in respect of a type of motor vehicle, to grant EEC type-approval, to issue the document referred to in the last indent of Article 10 (1) of Directive 70/156/EEC, or to grant national type-approval, or
	- prohibit the entry into service of the vehicles,

if the rear-view mirrors of this type of vehicle or of these vehicles comply with the provisions of this Directive;

 (b) — refuse, in respect of a type of rear-view (3) OJ No L 68, 22. 3. 1971, p. 1. mirror, to grant EEC component

⁽¹⁾ OJ No L 42, 23. 2. 1970, p. 1.

⁽²⁾ OJ No L 168, 26. 6. 1978, p. 39.

type-approval or national type-approval, if these rear-view mirrors comply with the provisions of this Directive, or

- prohibit the placing on the market of rear-view mirrors which bear the EEC component type-approval mark laid down in this Directive.
- 2. With effect from 1 October 1981 a Member State:
- (a) shall not issue the document referred to in the last indent of Article 10 (1) of Directive 70/156/EEC in respect of a type of vehicle of which the rear-view mirrors do not comply with the provisions of this Directive,
	- may refuse to grant national type-approval in respect of a type of vehicle of which the rear-view mirrors do not comply with the provisions of this Directive;
- (b) shall not grant EEC component type-approval in respect of a type of rear-view mirror if the latter does not comply with the provisions of this Directive,
	- may refuse to grant national component type-approval in respect of a type of rear-view mirror if the latter does not comply with the provisions of this Directive.

3 . With effect from ¹ October 1984 Member States: For the Commission **For the Commission**

— may prohibit the entry into service of vehicles of which the rear-view mirrors do not comply with the provisions of this Directive,

- may prohibit the placing on the market of rear-view mirrors which do not bear the EEC component type-approval mark laid down in this Directive.'
- 3. Annexes I, II and III are replaced by Annexes I, II, III and IV to this Directive.

Article 2

Member States shall bring into force the provisions necessary to comply with this Directive not later than ¹ February 1980. They shall forthwith inform the Commission thereof.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 20 July 1979.

Étienne DAVIGNON Member of the Commission

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ANNEX I

1. DEFINITIONS

- 1.1 . 'Rear-view mirror' means any device intended to give, within the field of vision defined in item 3.4, a clear view to the rear, excluding complex optical systems such as periscopes.
- 1.2. 'Interior rear-view mirror' means a device as defined in item 1.1 which can be fitted in the passenger compartment of a vehicle.
- 1.3 . 'Exterior rear-view mirror' means a device as defined in item 1.1 which can be mounted on the external surface of a vehicle.
- 1.4. 'Additional rear-view mirror' means a rear-view mirror other than a device of the type defined in item 1.1 which can be fitted to the inside or outside of the vehicle provided that it complies with the provisions of item 2 other than 2.1.1, 2.2 and 2.3.4.
- 1.5 . 'Rear-view mirror type' means devices which do not differ in respect of the following essential characteristics:
- 1.5.1 . the dimensions and radius of curvature of the rear-view mirror's reflecting surface:
- 1.5.2 . the design, shape or materials of the rear-view mirrors, including the connection with the bodywork.
- 1.6 . 'Class of rear-view mirrors' means all devices having one or more common characteristics or functions. Interior rear-view mirrors are grouped in Class I. Additional interior rear-view mirrors are grouped in Class Is. Exterior rear-view mirrors are grouped in Classes II and III. Additional exterior rear-view mirrors are grouped in Classes lis and Ills.
- 1.7. 'r' means the average of the radii of curvature measured over the reflecting surface, in accordance with the method described in item 2 of Appendix ¹ to this Annex.
- 1.8. **The principal radii of curvature at one point on the reflecting surface** (r_i) **means the** values obtained with the apparatus defined in Appendix 1, measured on the arc of the reflecting surface passing through the centre of the mirror parallel to the segment b, as defined in item 2.2.2.1, and on the arc perpendicular to this segment.
- 1.9. 'The radius of curvature at one point on the reflecting surface (r_p) ' means the arithmetical average of the principal radii of curvature r_1 and r'_1 , i. e.:

$$
r_p = \frac{r_i + r'_i}{2}
$$

- 1.10 . 'Centre of the mirror' means the centroid of the visible area of the reflecting surface.
- 1.11 . 'The radius of curvature of the constituent parts of the rear-view mirror' means the radius 'c' of the arc of the circle which most closely approximates to the curved form of the part in question.
- 1.12. 'The driver's ocular points' means two points 65 mm apart and 635 mm vertically above point R of the driver's seat as defined in Annex IV. The straight line joining these points runs perpendicular to the vertical longitudinal median plane of the vehicle. The centre of the segment joining the two ocular points is in a vertical longitudinal plane which must pass through the centre of the driver's designated seating position, as specified by the vehicle manufacturer.
- 1.13 . 'Ambinocular vision' means the total field of vision obtained by the superimposition of the monocular fields of the right eye and the left eye (see diagram below).

- \mathbf{D} ambinocular angle of vision $=$
- 1.14. . 'Type of vehicle as regards rear-view mirrors' means motor vehicles which are identical in respect of the following basic features:
- 1.14.1 . the bodywork features which reduce the field of vision;
- 1.14.2. the coordinates of point R;
- 1.14.3 . the prescribed positions and types of rear-view mirror.
- 1.15. 'Vehicles of categories M_1 , M_2 , M_3 , N_1 , N_2 and N_3 ' means those defined in Annex I to Directive 70/156/EEC.
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2. PROVISIONS RELATING TO EEC COMPONENT TYPE-APPROVAL OF REAR-VIEW MIRRORS

2.1 . General specifications

- 2.1.1. All rear-view mirrors must be adjustable.
- 2.1.2. The edge of the reflecting surface must be enclosed in a holder which, on its perimeter, must have a value 'c' \geq 2.5 mm at all points and in all directions. If the reflecting surface projects beyond the holder, the radius of curvature 'c' on the edge of the projecting part must be not less than 2-5 mm and the reflecting surface must return into the holder under a force of 50 N applied to the point of greatest projection, relative to the holder, in a horizontal direction approximately parallel to the longitudinal median plane of the vehicle.
- 2.1.3 . When the rear-view mirror is mounted on a plane surface, all parts, irrespective of the adjustment position of the device, including those parts remaining attached to the support after the test provided for in 2.4.2, which are in potential, static contact with a sphere either 165 mm in diameter in the case of an interior rear-view mirror or 100 mm in diameter in the case of an exterior rear-view mirror, must have a radius of curvature 'c' of not less than 2-5 mm.
- 2.1.3.1 . Edges of fixing holes or recesses of which the diameter or longest diagonal is less than 12 mm are exempt from the radius requirements of item 2.1.3 provided that they are blunted.
- 2.1.4. The attachment device on the vehicle must be so designed that a cylinder with a 50 mm radius, having as its axis the axis, or one of the axes, of pivot or rotation which ensure deflection of the rear-view mirror in the direction of impact concerned, passes through at least part of the surface to which the device is attached.
- 2.1.5 . The parts of exterior rear-view mirrors referred to in items 2.1.2 and 2.1.3 which are made of a material with a Shore A hardness not exceeding 60 are exempt from the relevant provisions.

1000

r

1000

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r

 $1 +$

 $1 +$

2.3 . Reflecting surface and coefficients of reflection

2.3.1. The reflecting surface of a rear-view mirror must be either flat or spherically convex.

III M_1 and N_1 13

- 2.3.2. Differences between the radii of curvature
- 2.3.2.1. The difference between r_i or r'_i and r_p at each reference point must not exceed 0-15 r.
- 2.3.2.2. The difference between any of the radii of curvature $(r_{p1}, r_{p2},$ and $r_{p3})$ and r must not exceed 0-15 r.
- 2.3.2.3 . When r is not less than 3 000 mm, the value of 015 r quoted in items 2.3.2.1 and 2.3.2.2 is replaced by 0-25 r.
- 2.3.3 . The value of 'r' must be not less than:
	- ¹ 800 mm for Class II rear-view mirrors,
	- ¹ 200 mm for Class I and III rear-view mirrors.
- 2.3.4. The value of the normal coefficient of reflection, as determined according to the method described in Appendix 2 to this Annex, must be not less than 40 %. If the mirror has two positions ('day' and 'night'), the 'day' position must allow the colours of the signals used for road traffic to be recognized. The value of the normal coefficient of reflection in the 'night' position must be not less than 4 %.
- 2.3.5 . The reflecting surface must retain the characteristics laid down in item 2.3.4 in spite of prolonged exposure to adverse weather conditions in normal use.

2.4. Tests

2.4.1. The reaction of rear-view mirrors to impact and bending on the holder fixed to the stem or support shall be tested in the manner described in items 2.4.2 and 2.4.3 . \bar{z}

2.4.1.1 . The test provided for in item 2.4.2 shall not be required in the case of any Class II or lis exterior rear-view mirror of which no part is less than 2 m from the ground, regardless of the adjustment position, when the vehicle is under a load corresponding to its maximum technically permissible weight.

> In such cases the manufacturer is required to provide a description stipulating that the rear-view mirror must be mounted in such a way that none of its parts, in any of the possible adjustment positions, is less than 2 m above the ground when the vehicle is under a load corresponding to its maximum technically permissible weight.

> Where advantage is taken of this derogation, the arm shall be indelibly marked with the symbol $\frac{2m}{2m}$ and the type-approval certificate shall be endorsed to this effect.

2.4.2. Impact test

2.4.2.1. Description of the test rig.

2.4.2.1.1 . The test rig consists of a pendulum capable of swinging about two horizontal axes at right angles to each other, one of which is perpendicular to the plane containing the 'release' trajectory of the pendulum.

> The end of the pendulum comprises a hammer formed by a rigid sphere with a diameter of 165 ± 1 mm and having a 5 mm-thick rubber covering of Shore hardness A 50.

> A device is provided which permits determination of the maximum angle assumed by the arm in the plane of release.

> A support firmly fixed to the structure of the pendulum serves to hold the specimens in compliance with the impact requirements specified in item 2.4.2.2.6.

> Figure ¹ below gives the dimensions of the test rig and the special design specifications.

Figure ^l

- 2.4.2.1.2. The centre of percussion of the pendulum coincides with the centre of the sphere which forms the hammer. It is at a distance '1' from the axis of oscillation in the release plane which is equal to 1 ± 5 mm. The reduced mass of the pendulum is m = 6.8 ± 0.05 kg (the relationship of 'm to the total mass 'm' of the pendulum and to the distance 'd' between the centre of gravity of the pendulum and its axis of rotation is expressed in the equation: $m_o = m - \frac{d}{1}$).
- 2.4.2.2. Description of the test.
- 2.4.2.2.1 . The procedure used to clamp the rear-view mirror to the support shall be that recommended by the manufacturer of the device or, where appropriate, by the vehicle manufacturer.
- 2.4.2.2.2. Positioning of the rear-view mirror for the test.
- 2.4.2.2.2.1 . Rear-view mirrors shall be so positioned on the pendulum impact rig such that the axes which are horizontal and vertical when the rear-view mirror is installed on a vehicle in accordance with the demander's mounting instructions are in a similar position.
- 2.4.2.2.2.2. When a rear-view mirror is adjustable with respect to the base, the test position shall be that in which any pivoting device is least likely to operate, within the limits of adjustment provided by the demander.
- 2.4.2.2.2.3 . When the rear-view mirror has a device for adjusting its distance from the base, the device must be set in the position in which the distance between the holder and the base is shortest.
- 2.4.2.2.2.4. When the reflecting surface is mobile in the holder, it shall be so adjusted that the upper corner which is furthest from the vehicle is in the position of greatest projection relative to the holder.
- 2.4.2.2.3. Except in the case of test 2 for interior rear-view mirrors (see item 2.4.2.2.6.1), when the pendulum is in a vertical position the horizontal and longitudinal vertical planes passing through the centre of the hammer shall pass through the centre of the mirror as defined in item 1.10 . The longitudinal direction of oscillation of the pendulum shall be parallel to the longitudinal median plane of the vehicle.
- 2.4.2.2.4. When, under the conditions governing adjustment laid down in items 2.4.2.2.1 and 2.4.2.2.2, parts of the rear-view mirror limit the return of the hammer, the point of impact must be displaced in a direction perpendicular to the axis of rotation or pivoting in question.

This displacement must be no greater than is strictly necessary for the execution of the test; it must be limited in such a way that:

- either the sphere delimiting the hammer remains at least tangential to the cylinder as defined in paragraph 2.1.4,
- or the point of contact with the hammer is located at least 10 mm from the periphery of the reflecting surface.
- 2.4.2.2.5 . The test consists in allowing the hammer to fall from a height corresponding to a pendulum angle of 60° from the vertical so that the hammer strikes the rear-view mirror at the moment when the pendulum reaches the vertical position.
- 2.4.2.2.6 . The rear-view mirrors are subjected to impact under the following different conditions:
- 2.4.2.2.6.1 . Interior rear-view mirrors
	- $-$ Test 1 The point of impact shall be as defined in item 2.4.2.2.3. The impact must be such that the hammer strikes the rear-view mirror on the reflecting surface side.
	- Test 2 Point of impact on the edge of the housing, so that the impact produced makes an angle of 45° with the plane of the mirror and is situated in the horizontal plane passing through the centre of the mirror. The impact must occur on the reflecting surface side.
- 2.4.2.2.6.2. Exterior rear-view mirrors
	- Test ¹ The point of impact shall be as defined in item 2.4.2.2.3 or 2.4.2.2.4. The impact must be such that the hammer strikes the rear-view mirror on the reflecting surface side.
	- Test 2 The point of impact shall be as defined in item 2.4.2.2.3 or 2.4.2.2.4. The impact must be such that the hammer strikes the rear-view mirror on the side opposite to the reflecting surface.

2.4.3 . Bending test on the holder fixed to the stem

2.4.3.1 . Description of the test

The holder is placed horizontally in a device in such a way that the adjustment parts of the mounting can be clamped securely. In the direction of the greatest dimension of the holder, the end nearest to the point of fixing on the adjustment part is immobilized by means of a fixed stop 15 mm wide covering the entire width of the holder.

At the other end, a stop identical to the one described above is placed on the holder so that the specified test load can be applied to it (Figure 2).

The end of the holder opposite that at which the force is applied may be clamped instead of simply blocked, as shown in Figure 2.

Example of bending-test apparatus for rear-view mirror holders

- 22. 9. 79 Official Journal of the European Communities No L 239/9 the fragments of glass still adhere to the back of the holder or to a surface firmly attached to the holder; partial separation of the glass from its backing is admissible provided this does not exceed 2-5 mm on either side of the cracks. It is permissible for small splinters to become detached from the surface of the glass at the point of impact; the mirror is made of safety glass. 2.5.3.1. 2.5.3.2. 2.6. 2.6.1. $2.6.1.1.$ 2.6.1.2. 2.6.1.2.1 , 2.6.1.2.2, 2.6.1.2.3 . 2.6.2. 2.6.2.1 . EEC component type-approval conditions and marking Application for EEC component type-approval Application for EEC component type-approval for a type of rear-view mirror shall be made by the holder of the trade mark or name, or by his authorized representative. For each type of rear-view mirror the application shall be accompanied by: a technical description, specifying in particular the type(s) of vehicle for which the rear-view mirror is intended; sufficiently detailed drawings for identification of the rear-view mirror, together with instructions for mounting: the drawings must show the proposed position of the EEC component type-approval mark; four rear-view mirrors: three for use in the tests and one to be retained by the laboratory for any further examination that might subsequently prove necessary. Additional specimens may be called for at the request of the laboratory. EEC component type-approval mark The EEC component type-approval mark shall consist of a rectangle surrounding the lower case letter 'e' followed by the distinguishing letter(s) or number of the Member State which has granted the component type-approval:
	- ¹ for Germany,
	- 2 for France,
	-
	- 3 for Italy,
4 for the N for the Netherlands,
	- 6 for Belgium, .
	- 11 for the United Kingdom,
	- 13 for Luxembourg,
	- 18 for Denmark,
	- IRL for Ireland.

It must also include in the vicinity of the rectangle the EEC component type-approval number.

This number shall consist of the component type-approval number shown on the certificate completed for the type (see Annex II), preceded by two figures indicating the sequence number of the latest amendment to Council Directive 71/127/EEC on the date EEC component type-approval was granted. The amendment sequence number and the component type-approval number shown on the certificate shall be separated by an asterisk. In this Directive the sequence number is 01.

 $2.6.2.2.$ The abovementioned type-approval mark (symbol and number) shall be indelibly inscribed on an essential part of the rear-view mirror in such a way as to be clearly visible even after the rear-view mirror has been mounted on a vehicle.

Examples of EEC component type-approval marks (1)

 $*1471$ $\frac{1}{3}$

The article bearing the EEC component type-approval mark shown above is a Class I mirror (interior rear-view mirror) which has been type-approved in Germany (e 1) under number $01 * 1471$.

(') The numbers in the diagram are for guidance only.

 $a \geq 6$ mm

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The article bearing the EEC component type-approval mark shown above is a Class lis mirror (additional exterior rear-view mirror) which has been type-approved in France (e 2) under the number 01 * 387.

 $\frac{a}{3}$

- 3.1.1.1 . The conditions laid down in item 3.1.1 must be maintained when the vehicle is moving at speeds of up to 80 % of its maximum design speed, but not exceeding 150 km/h.
- 3.1.2. Exterior rear-view mirrors fitted on vehicles of categories M_2 , M_3 , N_2 and N_3 must be Class II mirrors and those fitted on vehicles of categories M_1 and N_1 must be Class II or Class III mirrors.

3.2. Number and position

- 3.2.1 . Rear-view mirrors must be so placed that the driver, when sitting on the driving seat in a normal driving position, has a clear view of the road to the rear of the vehicle.
- 3.2.2. All vehicles of categories M_1 and N_1 must be fitted with both an interior and an exterior rear-view mirror. The latter must be fitted on the left side of the vehicle in Member States with right-hand rule of the road and on the right side of the vehicle in Member States with left-hand rule of the road.

^(*) The numbers in the diagram are for guidance only.

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onto a vertical plane perpendicular to the longitudinal median plane of the vehicle. 3.4.3 . Left-hand exterior rear-view mirror for vehicles driven on the right of the road and right-hand exterior rear-view mirror for vehicles driven on the left of the road: 3.4.3.1 . The field of vision must be such that the driver can see at least a 2-50 m-wide, flat, horizontal portion of the road, which is bounded on the right (in the case of vehicles driven on the right), or on the left (in the case of vehicles driven on the left) by the plane which is parallel to the median longitudinal vertical plane passing through the outermost point of the vehicle on the left (in the case of vehicles driven on the right), or on the right (in the case of vehicles driven on the left) and extends from 10 m behind the driver's ocular points to the horizon (Figure 4). 3.4.4. Right-hand exterior rear-view mirror for vehicles driven on the right and left-hand exterior rear-view mirror for vehicles driven on the left. 3.4.4.1 . The field of vision must be such that the driver can see at least a 3-50 m-wide, flat, horizontal portion of the road, which is bounded on the left (in the case of vehicles driven on the right), or on the right (in the case of vehicles driven on the left) by a plane parallel to the median longitudinal vertical plane of the vehicle and passing through the outermost point of the vehicle on the right (in the case of vehicles driven on the right) or

that they do not obscure more than 15% of the prescribed field of vision when projected

on the left (in the case of vehicles driven on the left) and which extends from 30 m behind the driver's ocular points to the horizon.

- 3.4.4.2. In addition, the road must be visible to the driver over a width of 0.75 m, from a point 4 m behind the vertical plane passing through the driver's ocular points (Figure 4).
- 3.4.5 . Obstructions

In the determination of the fields of vision specified above, no account is taken of obstructions caused by door handles, outline marker lights, direction indicators, the extremities of rear bumpers and obstructions due to the bodywork similar to those caused by the abovementioned elements.

3.4.6 . Test procedure

The field of vision shall be determined by placing powerful light sources at the ocular points and examining the light reflected on a vertical monitoring screen. Other, equivalent methods may be used.

Appendix ¹

PROCEDURE FOR DETERMINING THE RADIUS OF CURVATURE 'r' OF A MIRROR'S REFLECTION SURFACE

MEASUREMENTS 1 .

Equipment 1.1 .

The 'spherometer' described in Figure ¹ is used.

Measuring points 1.2.

- 1.2.1 . The principal radii of curvature shall be measured at three points situated as close as possible to positions at one third, half and two thirds of the distance along the arc of the reflecting surface passing through the centre of the mirror and parallel to segment b, or of the arc passing through the centre of the mirror which is perpendicular to it if this arc is the longer.
- 1.2.2. Where, owing to the size of the mirror, it is impossible to obtain measurements in the directions defined in item 1.8 of Annex I, the technical services responsible for the tests may take measurements at the said point in two perpendicular directions as close as possible to those prescribed above.

2. CALCULATION OF THE RADIUS OF CURVATURE (r)

'r' expressed in mm is calculated from the formula:

$$
r = \frac{r_{p1} + r_{p2} + r_{p3}}{3}
$$

where:

 r_{p1} = the radius of curvature at the first measuring point,

 r_{p2} = the radius of curvature at the second measuring point,

 r_{p3} = the radius of curvature at the third measuring point.

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Appendix 2

TEST METHOD FOR DETERMINING REFLECTIVITY

1. DEFINITIONS

- 1.1. 'CIE standard illuminant A^1 : Colorimetric illuminant, respecting the full radiator at $T_{68} = 2855.6$ K.
- 1.2. 'CIE standard source A¹: Gas-filled tungsten filament lamp operating at a correlated colour temperature of $T_{68} = 2855.6$ K.
- 1.3. 'CIE 1931 standard colorimetric observer' (1): Receptor of radiation whose colorimetric characteristics correspond to the spectral tristimulus values \bar{x} (λ), \bar{y} (λ), \bar{z} (λ) (see table).
- 1.4. 'CIE spectral tristimulus values' (*): Tristimulus values of the spectral components of an equi-energy spectrum in the CIE (XYZ) system.
- 1.5. 'Photopic vision' (J): Vision by the normal eye when it is adapted to levels of luminance of at least several candelas per square metre.

2. APPARATUS

2.1. General

The apparatus shall consist of a light source, a holder tor the test sample, a receiver unit with a photodetector and an indicating meter (see figure 1), and means of eliminating the effects of extraneous light.

The receiver may incorporate a light-integrating sphere to facilitate measuring the reflectance of non-flat (convex) mirrors (see figure 2).

2.2. Spectral characteristics of light source and receiver

The light source shall consist of a CIE standard source A and associated optics to provide a near-collimated light beam. A voltage stabilizer is recommended in order to maintain a fixed lamp voltage during instrument operation.

The receiver shall have a photodetector with a spectral response proportional to the photopic luminosity function of the CIE (1931) standard colorimetric observer (see table). Any other combination of illuminant-filter-receptor giving the overall equivalent of CIE standard illuminant A and photopic vision may be used. When an integrating sphere is used in the receiver, the interior surface of the sphere shall be coated with a matt (diffusive) spectrally non-selective white coating.

2.3 . Geometrical conditions

The angle of the incident beam (θ) should preferably be 0-44 \pm 0-09 rad (25 \pm 5°) from the perpendicular to the test surface and shall not exceed the upper limit of the tolerance (i.e. 0.53 rad or 30°). The axis of the receptor shall make an angle (θ) with this perpendicular equal to that of the incident beam (see figure 1). The incident beam upon arrival at the test surface shall have a diameter of not less than 19 mm (0-75 in). The reflected beam shall not be wider than the sensitive area of the photodetector, shall not cover less than 50 % of such area, and as nearly as possible shall cover the same area segment as used during instrument calibration.

When an integrating sphere is used in the receiver section, the sphere shall have a minimum diameter of 127 mm (5 in). The sample and incident beam apertures in the sphere wall shall be of such a size as to admit the entire incident and reflected light beams. The photodetector shall be so located as not to receive direct light from either the incident or the reflected beam.

^f ¹) Definitions taken from CIE publication 50 (45), International Electronical Vocabulary, Group 45 : Lighting.

2.4. Electrical characteristics of the photodetector-indicator unit

The photodetector output as read on the indicating meter shall be a linear function of the light intensity on the photosensitive area. Means (electrical and/or optical) shall be provided to facilitate zeroing and calibration adjustments. Such means shall not affect the linearity or the spectral characteristics of the instrument. The accuracy of the receptor-indicator unit shall be within \pm 2% of full scale, or \pm 10% of the magnitude of the reading, whichever is the smaller.

2.5 . Sample holder

The mechanism shall be capable of locating the test sample so that the axes of the source arm and receptor intersect at the reflecting surface. The reflecting surface may lie within or at either face of the mirror sample, depending on whether it is a first-surface, second-surface or prismatic 'flip'-type mirror.

3. PROCEDURE

3.1 . Direct calibration method

In the direct calibration method, air is used as the reference standard. This method is applicable for those instruments which are so constructed as to permit calibration at the 100% point by swinging the receiver to a position directly on the axis of the light source (see figure 1).

It may be desired in some cases (such as when measuring low-reflectivity surfaces) to use an intermediate calibration point (between 0 and 100% on the scale) with this method. In these cases, a neutral density filter of known transmittance shall be inserted in the optical path, and the calibration control shall then be adjusted until the meter reads the percentage transmission of the neutral density filter. This filter shall be removed before reflectivity measurements are performed.

3.2. Indirect calibration method

The indirect calibration method is applicable in the case of instruments with fixed source and receiver geometry. A properly calibrated and maintained reflectance standard is required. This reference standard should preferably be a flat mirror with a reflectance value as near as possible to that of the test samples.

3.3 . Flat mirror measurement

The reflectance of flat mirror samples can be measured on instruments employing either the direct or the indirect calibration method. The reflectance value is read directly from the indicating meter.

3.4. Non-flat (convex) mirror measurement

Measurement of the reflectance of non-flat (convex) mirrors requires the use of instruments which incorporate an integrating sphere in the receiver unit (see figure 2). If the instrument indicating meter indicates n_e divisions with a standard mirror of E% reflectance, then, with a mirror of unknown reflectance, n_x divisions will correspond to a reflectance of X%, in accordance with the formula

$$
\zeta = \mathbf{E} \frac{\mathbf{n}_{\mathbf{x}}}{\mathbf{n}_{e}}
$$

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Figure 1. Generalized reflectometer showing geometries for the two calibration methods

Figure 2. Generalized reflectometer incorporating an integrating sphere in the receiver

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(This table is taken from IEC publication 50 (45) (1970))

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(¹) Changed in 1966 (from 3 to 2).

(¹) Abridged table. The values of \bar{y} (λ) = V (λ) are rounded off to four decimal places.

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(1) Delete where inapplicable.

Name of administration

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ANNEX II

MODEL EEC COMPONENT TYPE-APPROVAL CERTIFICATE

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ANNEX III

ANNEX TO THE EEC TYPE-APPROVAL CERTIFICATE FOR A VEHICLE WITH REGARD TO THE INSTALLATION OF REAR-VIEW MIRRORS

(Articles 4 (2) and 10 of Council Directive 70/156/EEC of 6 February 1970 on the approximation of the laws of the Member States relating to the type-approval of motor vehicles and their trailers)

^(*) Where appropriate, state whether the extension of the initial EEC type-approval is the first, second, etc.

 (2) Delete where inapplicable.

- 18 . The following documents, bearing the type-approval number shown above, are annexed to this 'certificate:
	- drawings showing the mountings of the rear-view mirrors,
	- drawings and plans showing the mounting positions and the characteristics of the part of the structure where the rear-view mirrors are mounted,
	- general view from the front, the rear and the passenger compartment showing where the rear-view mirrors are fitted.

These documents must be supplied to the competent authorities of the other Member States at their express request.

ANNEX IV

PROCEDURE FOR DETERMINING THE H POINT AND VERIFYING THE RELATIVE POSITIONS OF THE R AND H POINTS

The relevant parts of Annex III to Directive 77/649/EEC are applicable.

FIRST COMMISSION DIRECTIVE

of 26 July 1979

Laying down Community methods of analysis for testing certain sugars intended for human consumption

(79/78 6/EEC)

THE COMMISSION OF THE EUROPEAN HAS ADOPTED THIS DIRECTIVE: COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 73 /437/EEC of ¹¹ December 1973 on the approximation of the laws of the Member States concerning certain sugars intended for human consumption (1), and in particular Article 11 thereof,

Whereas Article 11 of that Directive lays down that the composition of certain sugars shall be verified by Community methods of analysis;

Whereas it is desirable to adopt an initial series of methods in respect of which studies have been completed;

Whereas the method of determining the colour type for sugar or white sugar and for extra-white sugar, the method of measuring the conductivity ash in extra-white sugar, in sugar solution, in invert sugar solution and in invert sugar syrup, and the method of determining the colour in solution of extra-white sugar and sugar solution are laid down in the Annex to Directive 73/437/EEC;

Whereas, on the other hand, pending the formulation of further Community methods for the determination of reducing sugars, it would be advisable to allow the Member States the option of continuing to authorize the use of the Lane and Eynon method (methods 7 and 8 in Annex II, III.3 and III.4) instead of the Luff-Schoorl method (method 6 in Annex II, III.3 and III.4);

Whereas the methods of analysis provided for in this Directive are in accordance with the opinion of the Standing Committee on Foodstuffs,

1. Member States shall require that the analyses necessary for verification of the criteria set out in Annex I be performed according to the methods described in Annex II to this Directive.

2. Without prejudice to the second subparagraph, the Luff-Schoorl method (Annex II, method 6) shall be used to determine the reducing sugars in the following sugars:

- sugar solution,
- white sugar solution,
- invert sugar solution,
- white invert sugar solution,
- invert sugar syrup,
- glucose syrup,
- dried glucose syrup,
- dextrose monohydrate,
- dextrose anhydrous.

Member States may, however, require the use in their territory of the Lane and Eynon method (Annex II, methods 7 and/or ⁸ as appropriate) to determine the reducing sugars in one or more of the sugars listed above.

3. If a Member State makes use of the option provided for in the second subparagraph of paragraph 2, it shall forthwith inform the Commission and the other Member States thereof.

Article 2

Member States shall bring into force the laws, regulations or administrative provisions necessary to

Article ¹

^(!) OJ No L 356, 27. 12. 1973 , p. 71 .

comply with this Directive not later than 18 months Done at Brussels, 26 July 1979. following its notification. They shall forthwith inform the Commission thereof.

Article 3

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This Directive is addressed to the Member States. Member of the Commission

For the Commission fitienne DAVIGNON

ANNEX I

SCOPE OF THE COMMUNITY METHODS OF ANALYSIS FOR CERTAIN SUGARS INTENDED FOR HUMAN CONSUMPTION

I. Determination of the loss of mass on drying in:

- semi-white sugar
- sugar or white sugar
- extra-white sugar

II. Dry matter determination in:

- II.1. glucose syrup
	- dried glucose syrup $\left\{\n\begin{array}{c}\n\text{using method 2,}\n\end{array}\n\right.$
	- dextrose monohydrate $\qquad \qquad$ Annex II)
	- dextrose anhydrous J
- II.2. sugar solution or white sugar solution
	- invert sugar solution or white invert sugar (using method 3, solution $\left\{\n\begin{array}{c}\n\text{using method 3,}\n\end{array}\n\right.$
	- invert sugar syrup or white invert sugar syrup

III. Measurement of reducing sugars in:

- III.1. semi-white sugar
- III.2. sugar or white sugar
- extra-white sugar III.3. - sugar solution
	- white sugar solution
		- invert sugar solution
		- white invert sugar solution
		-
		- invert sugar syrup
		- white invert sugar syrup
- III.4. glucose syrup
	- dried glucose syrup
	- dextrose monohydrate
	- dextrose anhydrous

IV. Sulphated ash determination in:

- glucose syrup
- dried glucose syrup
- dextrose monohydrate
- dextrose anhydrous

V. Determination of polarization in:

- semi-white sugar
- sugar or white sugar
- extra-white sugar

(using method 1, Annex II)

Annex II)

(using method 4, Annex II) (using method 5, Annex II)

(using method 6 or 7, Annex II)

(using method 6 or 8 , Annex II)

(using method 9, Annex II)

(using method 10, Annex II)

ANNEX II

METHODS OF ANALYSIS TO VERIFY THE COMPOSITION OF CERTAIN SUGARS INTENDED FOR HUMAN CONSUMPTION

INTRODUCTION

1. Preparation of the sample for analysis

Thoroughly mix the sample received at the laboratory.

Remove a sub-sample of at least 200 g and transfer immediately to a clean, dry, moisture-tight vessel fitted with an airtight closure.

2. Reagents and apparatus

In the description ofthe apparatus, reference is made only to special instruments and apparatus or to those calling for special standards.

Wherever mention is made of water, this means distilled water or demineralized water of at least equivalent purity.

All reagents shall be of analytical reagent quality unless otherwise specified.

Wherever reference is made to a reagent solution without further qualification, an aqueous solution is meant.

3. Expression of results

The result referred to in the official analysis report shall be the mean value of at least two satisfactory replicate determinations.

Unless otherwise stated the results shall be expressed as a percentage by mass of the original sample as received at the laboratory.

The number of significant figures in the result so expressed shall be governed by the precision of the method.

METHOD 1

DETERMINATION OF THE LOSS OF MASS ON DRYING

1. Scope and field of application

The method determines the loss of mass on drying in:

- semi -white sugar,
- sugar or white sugar,
- extra-white sugar.

2. Definition

'Loss of mass on drying': the value of the loss of mass on drying as determined by the method specified.

3 . Principle

The loss of mass on drying is determined by drying at a temperature of 103 ± 2 °C.

4. Apparatus

- 4.1. Analytical balance, accurate to within 0.1 mg.
- 4.2. Oven, suitably ventilated, thermostatically controlled, and capable of being maintained at $103 + 2$ °C.
- 4.3. Metal weighing dish, flat-bottomed, resistant to attack by the samples and the conditions of test, diameter at least 100 mm, depth at least 30 mm.

4.4. Desiccator, containing freshly activated silica gel or an equivalent desiccant, with a water content indicator.

5. Procedure

- N.B. : The operations described in sections 5.3 to 5.7 must be performed immediately after opening the sample container.
- 5.1. Dry the dish (4.3) to constant weight in the oven (4.2) at 103 ± 2 °C.
- 5.2 . Allow the dish to cool in the desiccator (4.4) for at least 30 to 35 minutes and then weigh to the nearest 0'1 mg.
- 5.3 . Weigh accurately, to the nearest 0-1 mg, approximately 20 to 30 g of the sample into the dish.
- 5.4. Place the dish in the oven (4.2) at 103 \pm 2 °C for three hours.
- 5.5 . Allow the dish to cool in a desiccator (4.4) and weigh to the nearest 01 mg.
- 5.6. Replace the dish in the oven at 103 ± 2 °C for 30 minutes.

Allow to cool in the desiccator (4.4) and weigh to the nearest 01 mg. Repeat this operation if the difference between two weighings is more than 1 mg. Should an increase in mass occur, the . lowest recorded reading will be used in the calculation.

5.7. Do not exceed four hours total drying time.

6. Expression of results

6.1 . Formula and method of calculation

The loss of mass on drying, as a percentage by mass of the sample, is given by the following formula :

$$
\frac{(\mathrm{m_o} - \mathrm{m_1})}{\mathrm{m_o}} \times 100
$$

where:

 $m₀$ is the initial mass, in grams, of the test portion, $m₁$ is the mass, in grams, of the test portion after drying.

6.2. Repeatability

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0 02 g per 100 g of sample.

METHOD 2

DETERMINATION OF DRY MATTER

Vacuum oven method

1. Scope and field of application

The method determines the dry matter content in:

- glucose syrup,
- dried glucose syrup,
- dextrose monohydrate,
- dextrose anhydrous.

2. Definition

'The dry matter content': the content of dry matter as determined by the method specified.

3 . Principle

The dry matter is determined at a temperature of 70 \pm 1 °C using a vacuum oven at a pressure not exceeding 3-3 kPa (34 mbar). The test portions in the case of glucose syrup or dried glucose syrups, are prepared by mixing with water and kieselguhr before drying.

4. Reagents

4.1. Kieselguhr: place in a Buchner funnel and purify by repeated washings with dilute hydrochloric acid (1 ml of concentrated acid, density at 20 °C = 1.19 g/ml per litre of water). The treatment is complete when the washings remain definitely acid. Wash with water until the pH value of the filtered water is greater than 4. Dry in an oven at 103 ± 2 °C and store in an airtight container.

5. Apparatus

- 5.1. Vacuum drying oven, leak tight, thermostatically controlled and equipped with a thermometer and a vacuum manometer. The oven design must be such that the heat is rapidly transferred to the weighing dishes placed on the shelves.
- 5.2. Air-drying train consisting of a glass tower filled with freshly activated dry silica gel or an equivalent desiccant containing a water content indicator. This tower is mounted in series with a gas scrubber containing concentrated sulphuric acid connected to the air intake of the oven.
- 5.3 . Vaccum pump capable of maintaining the presure in the oven at 3-3 kPa (34 mbar) or less.
- 5.4. Metal weighing dish, flat-bottomed, resistant to attack by the samples and the conditions of test, diameter at least 100 mm, depth at least 300 mm.
- 5.5 . Glass rod of a length such that it cannot completely fall into the container.
- 5.6. Desiccator containing freshly activated dry silica gel, or an equivalent desiccant, with a water content indicator.
- 5.7. Analytical balance accurate to within 0-1 mg.

6. Procedure

6.1 . Pour approximately 30 g of kieselguhr (4.1) into the weighing dish (5.4) equipped with a glass rod (5.5). Place the whole in the oven (5.1) at 70 \pm 1 °C and reduce the pressure to 3.3 kPa (34 mbar) or less.

Dry for at least five hours, drawing a slow stream of air into the oven through the drying train. Check the pressure from time to time and correct it if necessary.

- 6.2 . Restore atmospheric pressure in the oven by cautiously increasing the intake of dry air. Immediately place the dish together with the glass rod in the desiccator (5.6). Allow to cool and then weigh.
- 6.3 . Accurately weigh to the nearest ¹ mg approximately 10 g of the sample to be analyzed into a 100 ml beaker.
- 6.4 . Dilute the test portion with 10 ml of warm water and transfer the solution quantitatively into the weighing dish, using the glass rod (5.5) .
- 6.5 . Place the dish containing the test portion and the glass rod in the oven and reduce the pressure to 3.3 kPa (34 mbar) or less. Dry at 70 \pm 1 °C, allowing a slow stream of dry air to pass through the oven.

The drying operation should proceed for 20 hours; the bulk of the loss should occur towards the end of the first day. It will be necessary to keep the vacuum pump working at a preset pressure and allow a slow stream of dry air to enter the oven so as to maintain a pressure of approximately 3-3 kPa (34 mbar) or less during the night.

- 6.6 . Restore atmospheric pressure in the oven by cautiously increasing the intake of dry air. Immediately place the weighing dish and contents in the desiccator. Allow to cool and then weigh to the nearest ¹ mg.
- 6.7. Continue operation (6.5) for a further four hours. Restore atmospheric pressure in the oven and immediately place the dish in the desiccator. Allow to cool and then weigh. Ascertain whether constant mass has been reached. It is considered that constant mass has been satisfactorily attained if the difference between the two weighings of the same dish does not exceed 2 mg. If the difference is greater, repeat operation 6.7.

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6.8 . For the determination of the dry matter in dextrose anhydrous or dextrose monohydrate samples the use of kieselguhr and water is not required.

7. Expression of results

7.1. Formula and method of calculation

The dry matter content, expressed as a percentage by mass of the sample is given by:

$$
(\mathbf{m}_1 - \mathbf{m}_2) \times \frac{100}{\mathbf{m}_0}
$$

where:

 m_0 = the initial mass, in grams, of the test portion,

 m_1 = the mass, in grams, of the weighing dish plus the kieselguhr, the glass rod and the residue of the test portion after drying,

 m_2 = the mass, in grams, of the weighing dish plus the kieselguhr and the glass rod.

7.2. Repeatability

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 012 g per 100 g of sample.

METHOD 3

DETERMINATION OF TOTAL DRY MATTER

(Refractometric method)

1. Scope and field of application

The method determines the dry-matter content in:

- sugar solution,
- white sugar solution,
- invert sugar solution,
- white invert sugar solution,
- invert sugar syrup,
- white invert sugar syrup.

2. Definition

'Dry matter content': the content of dry matter as determined by the method specified.

3. Principle

The refractive index of a test portion is determined at 20 °C and converted into dry matter content by reference to tables showing the concentration as a function of the refractive index.

4. Apparatus

- 4.1. Refractometer, accurate to four decimal places, provided with a thermometer and a water-circulation pump connected to a water-bath thermostatically controlled at 20 \pm 0.5 °C.
- 4.2. Light source consisting of a sodium vapour lamp.

5. Procedure

5.1 . If any crystals are present in the sample, redissolve them by diluting the sample in the ratio $1:1$ (m/m).

5.2. Measure the refractive index of the sample at 20 $^{\circ}$ C in the refractometer (4.1).

6. Expression and calculation of results

- 6.1. Calculate the dry matter content from the refractive indices for sucrose solutions at 20 °C in the table given and correct for the presence of invert sugars by adding to the result obtained from the tables, 0 022 for every 1% of invert sugar present in the sample as analyzed.
- 6.2. If the sample was diluted to $1:1 \text{ (m/m)}$ with water, the calculated dry matter content must be multiplied by two.

6.3 . Repeatability

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0-2 g dry matter per 100 g of sample.

REFERENCE TABLES

Refractive indices (n) of sucrose solutions at 20 °C (1)

(1) *n* values in these tables are calculated from the equation developed by K. Rosenhauer for ICUMSA, programed and computed by Frank G. Carpenter of UDSA, and published in *Sugar* J. 33, 15-22 (June 1970). Refractive in

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METHOD 4

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MEASUREMENT OF REDUCING SUGARS EXPRESSED AS INVERT SUGARS

(Berlin Institute method)

Scope and field of application

 $1.$

The method determines the reducing sugar content expressed as invert sugar in semi-white sugar. \sim

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2. Definitions

'Reducing sugars expressed as invert sugar': the content of reducing sugars as determined by the method specified.

3. Principle

The sample solution containing reducing sugars is used to reduce a solution of copper II complex. The copper I oxide formed is then oxidized with standard iodine solution, the excess of which is determined by back-titration with standardized sodium thiosulphate solution.

4. Reagents

- 4.1. Copper II solution (Muller's solution)
- 4.1.1. Dissolve 35 g of copper II sulphate, pentahydrate (CuSO₄.5H₂O) in 400 ml of boiling water. Allow to cool.
- 4.1.2. Dissolve 173 g of sodium potassium tartrate tetrahydrate (Rochelle salt or Seignette salt; $KNaC_4H_4O_6 \cdot 4H_2O$) and 68 g of anhydrous sodium carbonate in 500 ml of boiling water. Allow to cool.
- 4.1.3 . Transfer both solutions (4.1.1 and 4.1.2) to a one litre volumetric flask and make up to one litre with water. Add 2 g of activated carbon, shake, allow to stand for several hours and filter through thick filter paper or a membrane filter.

If small amounts of copper I oxide appear during storage, the solution should be re-filtered.

- 4.2. Acetic acid solution 5 mol/litre.
- 4.3 . Iodine solution 0-01665 mol/litre (i.e. 0-0333 N, 4-2258 g/litre).
- 4.4. Sodium tbiosulphate solution 0-0333 mol/litre.
- 4.5. Starch solution: to one litre of boiling water add a mixture of 5 g of soluble starch slurried in 30 ml of water. Boil for three minutes, allow to cool and add, if required, 10 mg of mercury II iodide as a preservative.

5. Apparatus

- 5.1. Conical flask, 300 ml; precision burettes and pipettes.
- 5.2. Water-bath, boiling.
- 6. Procedure
- 6.1 . Weigh a portion of the sample (10 g or less) containing not more than 30 mg of invert sugar in a 300 ml conical flask and dissolve in about 100 ml of water.

Pipette 10 ml of the copper II solution (4.1), into the flask containing the sample solution. Mix the contents of the flask by swirling and place it in the boiling water-bath (5.2) for exactly 10 minutes.

The level of the solution in the conical flask should be at least 20 mm below the level of the water in the water-bath. Cool the flask rapidly in a stream of cold running water. During this operation the solution should not be stirred otherwise atmospheric oxygen will reoxidize some precipitated copper ^I oxide.

Add ⁵ ml of ⁵ mol/litre acetic acid (4.2) by pipette without shaking and immediately add an excess (between 20 and 40 ml) of the iodine solution 0-01665 mol/litre (4.3) from a burette.

Stir to dissolve the copper precipitate. Titrate the excess iodine against the sodium thiosulphate solution 0.0333 mol/litre (4.4) using the starch solution (4.5) as indicator. The indicator is added towards the end of the titration.

- 6.2. Carry out a blank test with water. This is to be carried out with each new copper II solution (4.4). The titration shall not exceed 0-1 ml.
- 6.3 . Carry out a control test under cold conditions with the sugar solution. Allow to stand at room temperature for 10 minutes to permit any reducing agents such as sulphur dioxide which may by present to react.
- 7. Expression of results.
- 7.1 . Formula and method of calculation

Volume of iodine consumed = ml 0-01665 mol/litre iodine added in excess minus ml 0-0333 mol/litre sodium thiosulphate used in titration.

The volume (in ml) of 0.01665 ml/litre iodine consumed is corrected by subtracting:

7.1.1. The number of ml consumed in the blank test carried out with water (6.2).

- 7.1.2. The number of ml consumed in the cold test with the sugar solution (6.3).
- 7.1.3 . A value of 2-0 ml for every 10 g of sucrose present in the aliquot used, or a proportionate quantity where the sample contains less than 10 g sucrose (correction for sucrose).

After these corrections are made each ml of iodine solution (4.3) which has reacted corresponds to ¹ mg of of invert sugar.

The invert sugar contents, as a percentage of the sample, is given by the formula:

$$
\frac{V_1}{10 \times m_o}
$$

where:

 V_1 = the number of ml of iodine solution (4.3) after correction,

 m_0 = the mass, in grams, of the sample used.

7.2. Repeatability

The difference betweeh the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0 02 g per 100 g of sample.

METHOD 5

MEASUREMENT OF REDUCING SUGARS EXPRESSED AS INVERT SUGAR

(Knight and Allen method)

1. Scope and field of application

The method determines the reducing sugar content expressed as invert sugar in:

- sugar or white sugar,
- extra white sugar.

2. Definition

'Reducing sugars expressed as invert sugar': the content of reducing sugars as determined by the method specified.

3. Principle

Copper II reagent is added in excess to the sample solution, reduced and the unreduced portion is back-titrated with EDTA solution.

4. Reagents

- 4.1 . Ethylene diamine tetra-acetic acid solution (disodium salt) (EDTA) 0-0025 mol/litre: dissolve 0-930 g of EDTA in water and make up to one litre with water.
- 4.2. Murexide indicator solution: add 0-25 g of murexide to 50 ml of water and mix with 20 ml of a 0-2 g /100 ml aqueous solution of methylene blue.
- 4.3 . Alkaline copper reagent: dissolve 25 g of anhydrous sodium carbonate and 25 g of potassium sodium tartrate tetrahydrate in about 600 ml of water containing 40 ml of 1-0 mol/litre sodium hydroxide. Dissolve 6.0 g of copper II sulphate pentahydrate (CuSO₄.5H₂O) in about 100 ml of water, and add to the tartrate solution. Dilute to one litre with water.

 $N.B.:$ the solution has a limited life (one week).

4.4. Standard invert sugar solution: dissolve 23-750 g of pure sucrose (4.5) in about 120 ml of water in a 250 ml graduated flask, add 9 ml of hydrochloric acid ($\zeta = 1.16$) and allow to stand for eight days at room temperature. Make the solution up to 250 ml and check for completion of hydrolysis by a polarimeter or saccharimeter reading in a 200 mm tube. This should be — $11-80^\circ \pm 0.05^\circ$ (see Note 8). Pipette 200 ml of this solution into a 2 000 ml graduated flask. Dilute with water and while shaking (to avoid excessive local alkalinity) add 71-4 ml of sodium hydroxide solution (1 mol/litre) in which 4 g of benzoic acid has been dissolved. Make up to 2 000 ml to give a 1 $g/100$ ml solution of invert sugar. This solution should be approximately pH 3.

This stable stock solution should only be diluted immediately before use.

- 4.5. Pure sucrose: sample of pure sucrose with an invert sugar content not greater than 0.001 g/100 g.
- 5. Apparatus
- 5.1. Test tubes, 150×20 mm.
- 5.2. White porcelain dish.
- 5.3. Analytical balance, accurate to within 0.1 mg.

6. Procedure

- 6.1 . Dissolve 5 g of sugar sample in 5 ml of cold water in the test tube (5.1). Add 2-0 ml of the copper reagent (4.3) and mix. Immerse the tube in a boiling water bath for five minutes and then cool in cold water.
- 6.2. Transfer quantitatively the solution in the test tube to the white porcelain dish (5.2) using as little water as possible, add three drops of indicator (4.2) and titrate with EDTA solution (4.1). V_0 is the number of ml of EDTA used in the titration.

Just before the end-point the colour of the solution changes from green through grey to purple at the end-point. The purple colour will disappear slowly because of oxidation of copper ^I oxide to copper II oxide at a rate dependent on the concentration of reduced copper present. The end-point of the titration shall therefore be approached fairly rapidly.

6.3 . Construct a calibration graph by adding known amounts of invert sugar (as solution 4.4 appropriately diluted) to 5 g of pure sucrose (4.5) and add sufficient cold water so that a total of 5 ml of solution is added. Plot the titration volumes (in ml) against the percentage of invert sugar added to the 5 g of sucrose: the resultant graph is a straight line over the range 0-001 to 0-019 g/100 g invert sugar/100 g sample.

7. Expression of results

7.1. Method of calculation

Read on the calibration curve the percentage of invert sugar corresponding to the value V_0 ml of EDTA determined when analyzing the sample.

- 7.2. When a concentration greater than 0-017 g invert sugar/100 g sample is expected in the sample to be analyzed, the sample size taken in Procedure (6.1) must be appropriately reduced but the analysis sample made up to 5 g with pure sucrose (4.5).
- 7.3 . Repeatability

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same, analyst, under the same conditions, shall not exceed 0-005 g per 100 g of sample.

8. Note

Divide by 2-889 to convert °S to polarmetric degrees of arc (precision tubes of 200 mm; light source consisting of a sodium vapour lamp; the instrument must be installed in a room where the temperature may be maintained close to 20 °C).

METHOD 6

DETERMINATION OF REDUCING SUGARS EXPRESSED AS INVERT SUGAR OR DEXTROSE EQUIVALENT

(Luff-Schoorl method)

1. Scope and field of application

The method determines:

- 1.1. The reducing sugars content expressed as invert sugar in:
	- sugar solution,
	- white sugar solution,
	- invert sugar solution,
	- white invert sugar solution,
	- invert sugar syrup,
	- white invert sugar syrup.
- 1.2. The reducing sugar content, expressed and calculated (on the dry matter) as the dextrose equivalent in:
	- glucose syrup,
	- dried glucose syrup
- 1.3. The reducing sugar content expressed as D-glucose in:
	- dextrose monohydrate,
	- dextrose anhydrous
- 2. Definition

'Reducing sugars expressed as invert sugars, D-glucose or dextrose equivalent': the content of reducing sugars expressed or calculated as invert sugar, D-glucose or dextrose equivalent as determined by the method specified.

3. Principle

The reducing sugars in the sample (clarified if necessary) are heated to boiling point under standardized conditions with a copper II solution, which is partially reduced to copper I. The excess copper II is subsequently determined iodometrically.

4. Reagents

- 4.1. Carrez solution I: dissolve 21.95 g of zinc acetate dihydrate $(Zn(CH_3COO)_2.2H_2O)$ (or 24 g of zinc acetate trihydrate $(Zn (CH_3COO)_2 3H_2 O)$ and 3 ml of glacial acetic acid in water and make up to 100 ml with water.
- 4.2. Carrez solution II: dissolve 10-6 g of potassium hexacyanoferrate II trihydrate K_4 [Fe(CN)₆]. $3H₂O$ in water and make up to 100 ml with water.
- 4.3. Luff-Schoorl reagent: prepare the following solutions:
- 4.3.1 . Copper II sulphate solution: dissolve 25 g of iron-free copper II sulphate pentahydrate $(CuSO₄.5H₂O)$ in 100 ml water.
- 4.3.2. Citric acid solution: dissolve 50 g of citric acid monohydrate $(C_6H_8O_7.H_2O)$ in 50 ml of water.
- 4.3.3 . Sodium carbonate solution: dissolve 143-8 g of anhydrous sodium carbonate in about 300 ml of warm water and allow to cool.
- 4.3.4. Add the citric acid solution (4.3.2) to the sodium carbonate solution (4.3.3) in a one litre volumetric flask with gentle swirling. Swirl until effervescence ceases and then add the copper II sulphate solution $(4.3.1)$ and make up to 1 000 ml with water. Allow the solution to stand overnight and then filter if necessary. Check the molarity of the reagent thus obtained by the method described in 6.1 (Cu 0.1 mol/litre; $Na₂CO₃$ 1 mol/litre).
- 4.4. Sodium thiosulphate solution, 01 mol/litre.
- 4.5. Starch solution: to one litre of boiling water add a mixture of 5 g of soluble starch slurried in 30 ml of water. Boil for three minutes, allow to cool and add, if required, 10 mg of mercury II iodide as a preservative.
- 4.6. _ Sulphuric acid, 3 mol/litre.
- 4.7. Potassium iodide solution, 30% (m/v).
- 4.8. Pumice chips, boiled in hydrochloric acid, washed free of acid with water and then dried.
- 4.9. Isopentanol
- 4.10. Sodium hydroxide, 0-1 mol/litre.
- 4.11. Hydrochloric acid, 0.1 mol/litre.
- 4.12. Phenolphthalein solution, 1% (m/v) in ethanol.

5. Apparatus

- 5.1. Conical flask, 300 ml, fitted with a reflux condenser.
- 5.2. Stop-watch.

6. Procedure

- 6.1. Standardization of the Luff-Schoorl reagent (4.3)
- 6.1.1 . To 25 ml of Luff-Schoorl reagent (4.3) add ³ g of potassium iodide and 25 ml of ³ mol/litre sulphuric acid (4.6).

Titrate with 0.1 mol/litre sodium thiosulphate (4.4) using starch solution (4.5) as indicator added towards the end of the titration. If the volume of 0.1 mol/litre sodium thiosulphate used is not 25 ml the reagent must be made up afresh.

- 6.1.2. Pipette 10 ml of the reagent into a 100 ml volumetric flask and dilute to volume with water.
	- Pipette 10 ml of the diluted reagent into 25 ml of 0.1 mol/litre hydrochloric acid (4.11) in a conical flask and heat for one hour in a boiling water-bath. Cool, make up to the original volume with freshly boiled water and titrate with 0-1 mol/litre sodium hydroxide (4.10) using phenolphthalein (4.12) as indicator.

The volume of 0-1 mol/litre sodium hydroxide (4.10) used must be between 5-5 and 6-5 ml.

6.1.3 . Titrate 10 ml of the diluted reagent (6.1.2) with 0-1 mol/litre hydrochloric acid (4.11) using phenolphthalein (4.12) as indicator. The end-point is characterized by the disappearance of the violet colour.

The volume of 01 mol/litre hydrochloric acid (4.11) used must be between 6-0 and 7-5 ml.

- 6.1.4. The pH of the Luff-Schoorl reagent must be between 9-3 and 9-4 at 20 °C.
- 6.2. Preparation of the solution
- 6.2.1 . Accurately weigh, to the nearest ¹ mg, 5 g of the sample and transfer quantitatively to a 250 ml volumetric flask, with 200 ml water. Clarify, if necessary, by adding 5 ml of Carrez solution I (4.1) followed by 5 ml of Carrez solution II (4.2). Mix after each addition. Make up to 250 ml with water. Mix well. Filter if necessary.
- 6.2.2. Dilute the solution (6.2.1) so that 25 ml of the solution contains not less than 15 mg and not more than 60 mg of reducing sugars expressed as glucose.
- 6.3 . Titration by the Luff-Schoorl method

Pipette 25 ml of Luff-Schoorl reagent (4.3) into a 300 ml conical flask (5.1). Pipette 25 ml of the sugar solution (6.2.2) into the conical flask and introduce two pumice chips (4.8). Fit a reflux condenser to the conical flask (5.1) and immediately place the apparatus on an asbestos wire gauze over a Bunsen flame. The gauze shall have a hole cut in the asbestos part of the same diameter as the base of the flask. Heat the liquid to boiling point over a period of about two minutes and simmer gently for exactly 10 minutes. Cool immediately in cold water and after five minutes titrate as follows:

Add 10 ml of potassium iodide solution (4.7) then immediately add with caution (because of effervescence) 25 ml of 3 mol/litre sulphuric acid (4.6). Titrate with 01 mol/litre sodium thiosulphate solution (4.4) until the solution is almost colourless, then add a few ml of starch solution (4.5) as indicator and continue titrating until the blue colour disappears.

Carry out a control test, using 25 ml of water in place of the 25 ml of sugar solution (6.2.2).

7. Expression of results

7.1. Formula and method of calculation

From the table below, find (interpolating if necessary) the weight of glucose or of invert sugar in mg corresponding to the difference between the two titration readings, expressed in ml of 0-1 mol/litre sodium thiosulphate.

Express the result in terms of invert sugar or D-glucose as percentage (m/m) of the dry matter.

7.2. Repeatability

The difference between the results of two titrations when carried out simultaneously or in rapid succession on the same sample by the same analyst, under the same conditions, shall not exceed 0-2 ml.

8. Note

A small volume of isopentanol (4-9) may be added before acidifying with sulphuric acid to reduce foaming.

Table of values according to Luff-Schoorl reagent

METHOD 7

MEASUREMENT OF REDUCING SUGARS EXPRESSED AS INVERT SUGAR (Lane and Eynon constant volume modification)

1. Scope and field of application

The method determines the reducing sugars, expressed as invert sugar, in:

- sugar solution,
- white sugar solution,
- invert sugar solution,
- white invert sugar solution,
- invert sugar syrup,
- white invert sugar syrup.

2. Definition

'Reducing sugars expressed as invert sugar': the content of reducing sugars as determined by the method specified.

3. Principle

The sample solution is titrated at the boiling point against a specified volume of Fehling's solution, using methylene blue as internal indicator.

4. Reagents

- 4.1. Fehling's solution:
- 4.1.1 . Solution A:

Dissolve 69.3 g of copper II sulphate pentahydrate ($CuSO₄$.5H₂O) in water and make up to ¹ 000 ml .

4.1.2. Solution B:

Dissolve 346.0 g of double sodium potassium tartrate tetrahydrate (KNaC₄H₄O₆.4H₂O) with 100 0 g of sodium hydroxide in water and make up to ¹ 000 ml. The clear solution should be decanted from a sediment that may form from time to time.

Note:

These two solutions should be stored in brown or amber bottles.

- 4.2. Sodium hydroxide solution, 1 mol/litre.
- 4.3 . Standard invert sugar solution: dissolve 23-750 g of pure sucrose in about 120 ml of water in a 250 ml graduated flask, add 9 ml of hydrochloric acid ($\zeta = 1.16$) and allow to stand for eight days at room temperature. Make the solution up to 250 ml and check for completion of hydrolysis by a polarimeter or saccharimeter reading in a 200 mm tube. This should be — $11·80° \pm 0·05°$ S (see note 8). Pipette 200 ml of this solution into a 2 000 ml graduated flask. Dilute with water and while shaking (to avoid excessive local alkalinity) add 71-4 ml of sodium hydroxide solution (1 mol/litre) (4.2) in which 4 g of benzoic acid has been dissolved. Make up to 2 000 ml to give a 1 g/100 ml solution of invert sugar. This solution should be a pH of approximately 3.

This stable stock solution should only be diluted immediately before use.

To make up the 0-25 g/ 100 ml invert sugar solution, fill a 250 ml graduated flask to the mark with the stock ¹ g/100 ml invert solution at 20 °C. Wash the contents of this flask into a ¹ 000 ml graduated flask and dilute to the mark with water again at 20 °C.

- 4.4. Methylene blue solution, ¹ g/100 ml.
- 5. Apparatus
- 5.1 . Narrow-necked laboratory boiling flasks, 500 ml.
- 5.2. Burette, 50 ml, with tap and offset tip, graduated to 0-05 ml.
- 5.3 . Pipettes graduated at 20, 25 and 50 ml.
- 5.4. One mark volumetric flasks, 250, ¹ 000 and 2 000 ml.
- 5.5 . A heating device, suitable for maintaining boiling according to the conditions described in 6.1 , permitting the observation of the end-point colour change without the necessity of removing the boiling flask (5.1) from the source of heat.
- 5.6. Stop-watch, indicating to within at least one second.

6 . Procedure

 \mathcal{L}

- 6.1 . Standardization of Fehling's solution
- 6.1.1 . Pipette 50 ml of solution B (4.1.2) and then 50 ml of solution A (4.1.1) into a clean dry beaker and mix well.
- 6.1.2 . Rinse and fill the burette with 0-25 % (0-25 g/100 ml) standard invert sugar solution (4.3).
	- 6.1.3. Pipette a 20 ml aliquot of the mixed solutions A and B $(6.1.1)$ into a 500 ml boiling flask (5.1) . Add 15 ml of water to the flask. Run in, from the burette, 39 ml of the invert sugar solution, add a small quantity of anti-bumping granules and mix the contents of the flask by gentle swirling.
	- 6.1.4 . Heat the flask and contents till boiling and allow to boil for exactly two minutes; the flask must not be removed from the heat source during the course of the rest of the procedure, or allowed to cease boiling.

Add three or four drops of methylene blue solution (4.4) at the end of the two-minute boiling period: the solution should be a definite blue colour.

- 6.1.5 . Continue the standardization by adding, from the burette, the standard invert sugar solution in small increments, initially of 0.2 ml; then 0.1 ml and finally in single drops until the end-point is reached. This is indicated by the disappearance of the blue colour imparted by the methylene blue. The solution has then assumed the reddish colour associated with a suspension of copper I oxide.
- 6.1.6 . The end-point should be reached at the end of three minutes from when the solution started to boil. The final titre, V_0 , shall be between 39.0 and 41.0 ml. If V_0 lies outside these limits, adjust the copper concentration of Fehling's solution A (4.1.1) and repeat the standardization process.
- 6.2. Preparation of sample solutions

The concentration of the sample test solution should be such that it contains between 250 and 400 mg invert sugar per 100 ml.

- 6.3 . Preliminary test
- 6.3.1 . A preliminary test must be carried out to ensure that the quantity of water to be added to the 20 ml of mixed solutions A and B is sufficient to ensure that a final volume after titration of 75 ml is obtained.

The same procedure as described in 6.1.4 is carried out except that the sample solution is used instead of the standard invert sugar solution, i.e. 25 ml of the sample solution is run into the flask from the burette. 15 ml of water is added, and the solution is allowed to boil for two minutes and then titrated until the end-point is reached as described in 6.1,5 .

6.3.2. If, after the addition of the methylene blue solution, the reddish colour persists, the sample solution used is too concentrated. In this case, the test is discarded but repeated using a less concentrated sample solution.

If more than 50 ml of sample solution are required to obtain the reddish colour, a more concentrated solution of the sample must be used.

Calculate the quantity of water to be added by subtracting the volumes of mixed Fehling's solution (20 ml) and of the sample solution from 75 ml.

6.4. Final analysis of sample solution

- 6.4.1 . Pipette into the boiling flask 20 ml of mixed Fehling's solution and the quantity of water determined as in 6.3.
- 6.4.2 . Add, from the burette, the observed titre of the sample solution (as determined in 6.3) less ¹ ml. Add some anti-bumping granules, mix the contents of the flask by swirling, boil the flask and contents and titrate as previously (6.3). The end-point should be reached one minute from the time of addition of the methylene blue solution. Final titre = V_1 .

7. Expression of results

7.1 . Formula and method of calculation

The reducing sugars content of the sample calculation as invert sugar, is given by:

 $%$ reducing sugars (as invert sugar $=$

$$
\frac{V_0 \times 25 \times f}{C_0 \times V_1}
$$

where:

- $C =$ the concentration of the sample test solution in g per 100 ml.
	- V_0 = the volume in ml of the standard invert solution used in the standardization titration,
	- V_1 = the volume in ml of the sample test solution used in the accurate analysis in 6.4.2,
	- $f =$ the correction factor to take account of the sucrose concentration in the sample test solution. Values are shown in the table below:

Corrections for varying sucrose contents of the sample test solution may be calculated from the table by interpolation.

Note:

The approximate sucrose concentration may be found by subtraction of the dissolved solids concentration due to the invert sugar (estimated for the purposes of this calculation f as 1-0), from the total dissolved solids concentration, expressed as sucrose, obtained from the refractive index of the solution using method three of this document.

7.2. Repeatability

The difference between the results of two determinations, carried out simultaneously or in rapid succession on the same sample by the same analyst under the same conditions, shall not exceed 1-0 % of their arithmetic mean.

8. Note

Divide by 2-889 to convert °S to polarmetric degrees of arc (precision tubes of 200 mm; light source consisting of a sodium vapour lamp; the instrument must be installed in a room where the temperature may be maintained close to 20 °C).

METHOD 8

DETERMINATION OF DEXTROSE EQUIVALENT

(Lane and Eynon constant)

1. Scope and field of application

This method determines the dextrose equivalent of:

- glucose syrup,
- dried glucose syrup,
- dextrose monohydrate,
- dextrose anhydrous.

2. Definition

- 2.1 . 'Reducing power': the reducing sugar content, determined by the method specified, expressed in terms of anhydrous dextrose (D-glucose) and calculated as a percentage by mass of the sample.
- 2.2. 'Dextrose equivalent': the reducing power, calculated as a percentage by mass of the dry matter in the sample.

3. Principle

The test solution is titrated at the boiling point against a specified volume of mixed Fehling's solution, under strictly specified conditions, using methylene blue as an internal indicator.

4. Reagents

- 4.1. Fehling's solution:
- 4.1.1. Solution A:

Dissolve 69.3 g of copper II sulphate pentahydrate (CuSO₄.5H₂O) in water and make up to volume in a ¹ 000 ml volumetric f ask.

4.1.2 . Solution B:

Dissolve 346-0 g of sodium potassium tartrate tetrahydrate (KNaC₄H₄O₆,4H₂O) and 100 g of sodium hydroxide in water. Make ip to volume in a ¹ 000 ml volumetric flask. Decant the clear solution from any sediment that may from time to time form.

Note:

These two solutions (4.1.1 and 4.1.2) should be stored in brown or amber bottles.

4.1.3 . Preparation of the mixed Fehling's solution

Pipette 50 ml of solution B (4.1.2) and then 50 ml of solution A (4.1.1) into a clean dry beaker and mix well.

Note:

Mixed Fehling's solution shall not be stored but made up afresh every day and standardized (6.1) .

4.2. Anhydrous dextrose (D-glucose) $(C_6H_{12}O_6)$

This material shall be dried before use for four hours in a vacuum oven at 100 ± 1 °C or less, and an internal pressure of approximately 10 kPa (103 mbar).

4.3 , Standard dextrose solution, 0-600 g/100 ml

Weigh, to the nearest 0-1 mg, 0-6 g of anhydrous dextrose (4.2), dissolve it in water, transfer the solution quantitatively into a 100 ril volumetric flask (5.4) , dilute to the mark and mix. This solution shall be freshly prepared on each day of use.

4.4. Methylene blue solution, 0-1 g/100 ml Dissolve 0-1 g of methylene blue in 100 ml water.

5. Apparatus

- 5.1. Narrow necked laboratory boiling jlasks, 250 ml.
- 5.2 . Burette, 50 ml, with tap and offset tip, graduated to 0-05 ml.
- 5.3. One mark pipettes, 25 ml and 50 ml.
- 5.4. One mark volumetric flasks, 100 and 500 ml.
- 5.5. A heating device, suitable for main aining boiling according to the conditions described in 6.1 , permitting the observation of the end-point colour change without the necessity of removing the boiling flask (5.1) from the source of heat (see 6.1, note 3).
- 5.6 . A stop-watch, indicating to at least the nearest second.

6 . Procedure

- 6.1 . Standardization of the Fehling's solution
- 6.1.1 . Pipette 25 ml of Fehling's solution (4.1.3) into a clean, dry boiling flask (5.1).
- 6.1.2. Fill the burette (5.2) with standard dextrose solution (4.3) and adjust the meniscus to the zero mark.
- 6.1.3 . Run into the boiling flask (5.1) from the burette 18 ml of standard dextrose solution (4.3). Swirl the flask to mix contents.
- 6.1.4. Place the boiling flask on the heating device (5.5) , previously adjusted so that boiling commences in 120 ± 15 seconds.

The heating device shall not be further adjusted during the whole of the titration (see note 1).

6.1.5 . When boiling commences, start the stop-watch from zero.

6.1.6 . Boil the contents of the flask for 120 seconds, as timed by the stop-watch. Add ¹ ml of methylene blue solution (4.4) towards the end of this period.

6.1.7. After boiling has continued for 120 seconds (by the stop-watch) start adding standard dextrose solution to the boiling flask (5.1) from the burette (6.1.2) in 0.5 ml increments until the colour of the methylene blue is discharged (see notes 2 and 3).

- 6.1.8 . Repeat 6.1.1 and 6.1.2.
- 6.1.9 . Run into the boiling flask (5.1) from the burette a volume of standard dextrose solution equal to $(X-0.3)$ ml.
- 6.1.10 Repeat 6.1.4, 6.1.5 and 6.1.6.
- 6.1.11 . After boiling has continued for 120 seconds (by the stop-watch), start adding standard dextrose solution to the boiling flask (5.1) from the burette, initially in 0.2 ml increments and finally dropwise, until the colour of the methylene blue is just discharged.

Towards the end of this action the time between successive additions of standard dextrose solution shall be 10 to 15 seconds.

These additions shall be completed within 60 seconds, making the total time to boiling no longer than 180 seconds.

A third titration with a slightly larger, appropriately adjusted, initial addition of standard dextrose solution $(6.1.9)$ may be necessary to achieve this.

- 6.1.12. Note the volume $(V_0 \text{ ml})$ of standard dextrose solution used up to the end-point of the final titration (see note 4).
- 6.1.13. V_0 shall be between 19 \cdot 0 and 21 \cdot 0 ml standard dextrose solution (4.3).

If V_0 lies outside these limits, adjust the concentration of the Fehling's solution A (4.1.1) appropriately and repeat the standardization process.

6.1.14. For the day-to-day standardization of the mixed Fehling's solution, as V_0 is known with accuracy, a single titration only is necessary, using an initial addition of $(V_0 - 0.5)$ ml standard dextrose solution.

Note 1:

This ensures that once boiling has commenced the evolution of steam is brisk and continuous throughout the whole of the titration process, thus preventing to the maximum possible extent the entrance of air into the titration flask with consquent re-oxidation of its contents.

Note 2:

The disappearance of the colour of the methylene blue is best seen by looking at the upper layers and the meniscus of the contents of the titration flask, as these will be relatively free from the precipiated, red copper I oxide. The colour disappearance is more easily seen when indirect lighting is used. A white screen behind the titration flask is helpful.

Note 3 :

The burette should be isolated as much as possible from the source of heat during the determination.

Note 4:

As there is always a personal factor involved, each operator shall carry out his own standardization titration and use his own value of V_0 in the calculation (7.1).

6.2 . Preliminary examination of the prepared sample

- 6.2.1 . Unless the reducing power (2.1) of the prepared sample is known approximately, it is necessary to carry out a preliminary examination in order to obtain an approximate figure for it so that the mass of the test portion (6.3) can be calculated. This examination is carried out as follows:
- 6.2.2. Prepare a 2% m/v solution of the sample, 'Z' having an estimated value.
- 6.2.3 . As 6.1.2, using the sample solution (6.2.2) in place of the standard dextrose solution.

6.2.4. As 6.1.1 .

6.2.5 . As 6.1.3 , using 10-0 ml sample solution instead of 18-0 ml standard dextrose solution.

6.2.6. As 6.1.4.

6.2.7. Heat the contents of the flask to boiling. Add ¹ ml methylene blue solution (4.4).

Note the total volume of standard dextrose solution added up to and including the penultimate 0-5 ml increment (X ml).

- 6.2.8 . Immediately boiling has started, start the stop-watch (5.6) from zero and commence adding sample solution to the flask from the burette in 1.0 ml increments at intervals of approximately 10 seconds until the blue colour of the methylene blue is discharged. Note the total volume of sample solution added up to and including the penultimate increment $(Y$ ml $)$.
- 6.2.9. 'Y' must not exceed 50 ml. If it does, increase the concentration of the sample solution and repeat the titration.
- 6.2.10. The approximate reducing power of the prepared sample in percent by mass is given by:

6.3. Test portion

Weigh out, to the nearest 0-1 mg, a mass of the prepared sample (mg) which contains between 2-85 and 3-15 g reducing sugars, expressed as anhydrous dextrose (D-glucose) using in the calculation either known approximate figure for the reducing power (2.1) or the approximate figure obtained in 6.2.10.

6.4. Test solution

Dissolve the test portion in water and make up to 500 ml in a volumetric flask.

- 6.5 . Determination
- 6.5.1. As 6.1.1.
- 6.5.2. Fill the burette (5.2) with test solution (6.4) and adjust the meniscus to the zero mark.
- 6.5.3 . Run into the boiling flask from the burette 18-5 ml test solution. Swirl the flask to mix the contents.
- 6.5.4. As 6.1.4.
- 6.5.5 . As 6.1.5 .
- 6.5.6 . As 6.1.6 .
- 6.5.7. As 6.1.7, using test solution in place of standard dextrose solution.
- 6.5.8 . As 6.1.8 .
- 6.5.9 . As 6.1.9, using test solution in place of standard dextrose solution.
- 6.5.10. As 6.1.10.

6.5.1 ¹ . As 6.1.11 , using test solution in place of standard dextrose solution.

6.5.12. Note the volume (V_1) of test solution used up to the end-point of the final titration.

6.5.13. V_1 shall be between 19 \cdot 0 and 21 \cdot 0 ml test solution.

If V_1 lies outside these limits, adjust the concentration of the test solution appropriately and repeat 6.5.1 to 6.5.12.

6.5.14. Carry out two determinations on the same test solution.

6.6 . Dry matter content

Determine the dry matter content of the prepared sample by method 2.

- 7. Expression of results
- 7.1. Formulae and method of calculation
- 7.1.1 . Reducing power

The reducing power, calculated as a percentage by mass of the prepared sample, is given by:

$$
\frac{300 \times V_0}{V_1 \times M}
$$

where:

- V_0 = the volume, in ml, of the standard dextrose solution (4.3) used in the standardization titration (6.1) ,
- V_1 = the volume, in ml, of the test solution (6.4) used in the determination titration (6.5),
- $M =$ the mass, in grams, of the test portion (6.3) used to make 500 ml test solution.

7.1.2. Dextrose equivalent

The dextrose equivalent, calculated as a percentage by mass of the dry matter in the prepared sample, is given by:

$$
\frac{RP \times 100}{D}
$$

where:

 RP = the reducing power, calculated as a percent by mass of the prepared sample (7.1.1),

 $D =$ the dry matter content of the prepared sample in percent by mass.

- 7.1.3 . Take as the result the arithmetic mean of the two determinations provided that the requirement concerning repeatability (7.2) is satisfied.
- 7.2. Repeatability

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 10 % of their arithmetic mean.

METHOD 9

DETERMINATION OF SULPHATED ASH

- 1. Scope and field of application
	- The method determines the sulphated ash content in:
	- glucose syrup,
	- dried glucose syrup,
	- dextrose monohydrate,
	- dextrose anhydrous.

2. Definition

'Sulphated ash content': the content of sulphated ash as determined by the method specified.

3. Principle

The residual mass of a test portion is determined after incineration in an oxidizing atmosphere at 525 °C in the presence of sulphuric acid and calculated as a percentage by mass of the sample.

4. Reagents

4.1. Sulphuric acid, dilute solution: slowly and cautiously add 100 ml of concentrated sulphuric acid (density at 20 °C = 1.84 g/ml; 96% m/m) to 300 ml water with stirring and cooling.

5. Apparatus

- 5.1. Electric muffle furnace, equipped with a pyrometer and capable of operating at a temperature of $525 + 25$ °C.
- 5.2. Analytical balance, accurate to 0.1 mg.
- 5.3 . Ashing crucibles, platinum or quartz, of suitable capacity.
- 5.4. Desiccator, containing freshly activated silica gel or an equivalent desiccant with a water content indicator.

6. Procedure

Heat a crucible (5.3) to the ashing temperature, cool in a desiccator and weigh. Accurately weigh, to the nearest 01 mg, 5 g of glucose syrup or dried glucose syrup, or about 10 g of dextrose monohydrate or dextrose anhydrous into the crucible.

Add 5 ml of sulphuric acid solution (4.1) (see note 8.1) and carefully heat the sample in the crucible over a flame or on a hotplate until it is completely carbonized. This carbonization process, during which vapours are burnt off from the sample (see note 8.2), should be carried out in a fume cupboard.

Place the crucible (5.3) in the muffle furnace (5.1) heated to $525 \pm 25^{\circ}$ C until a white ash is obtained. This normally takes two hours (see note 8.3).

Allow the sample to cool for about 30 minutes in a desiccator (5.4) and then weigh.

7. Expression

7.1 . Formula and method of calculation

The sulphated ash content expressed as a percentage by mass of the sample to be analyzed is given by:

$$
S = \frac{m_1}{m_0} \times 100
$$

where:

 m_1 = the mass, in grams, of the ash,

 m_0 = the mass, in grams, of the test portion.

7.2. Repeatability

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 2% of their arithmetic mean.

8. Notes

- 8.1. The sulphuric acid is added in small quantities to prevent excessive foaming.
- 8.2. Every relevant precaution must be taken during the first carbonization to prevent losses of sample or of ash through excessive swelling of the sample.
- 8.3 . If the sample is difficult to ash completely (i.e. black particles remain) the crucible should be removed from the muffle furnace and the residue moistened, after cooling, with a few drops of water before being returned to the furnace.

METHOD 10

DETERMINATION OF POLARIZATION

$1.$ Scope and field of application

The method determines the polarization in:

- semi-white sugar,
- sugar or white sugar,
- extra-white sugar.

2. Definition

The polarization is the rotation of the polarized light plane by a sugar solution with 26 g of sugar per 100 ml contained in a tube of 200 mm in length.

3 . Principle

The polarization is determined by using a saccharimeter or a polarimeter according to the conditions described in the following method.

4. Reagents

4.1. Clarification agent: basic lead acetate solution.

Add 560 g of dry basic lead acetate to about ¹ 000 ml of freshly boiled water. Boil the mixture for 30 minutes and then leave it to stand overnight.

Decant the supernatant liquid and dilute with freshly boiled water to obtain a solution of density of 1-25 g/ml, at 20 °C.

Protect this solution from contact with air.

4.2. Diethyl ether

5. Apparatus

5.1. Saccharimeter graduated for the normal weight of 26 g of sucrose, or polarimeter

This instrument must be installed in a room where the temperature may be maintained close to 20 °C. Calibrate the instrument against standard quartz plates.

- 5.2. Light source, consisting of a sodium vapour lamp.
- 5.3. Precision polarimeter tubes, length 200 mm, error not exceeding \pm 0.02 mm.
- 5.4. Analytical balance, accurate to within 0-1 mg,
- 5.5 . Individually calibrated 100 ml volumetric flasks stoppered. Flasks with a real capacity in the range 100.00 ± 0.01 ml may be used without correction. Flasks with a capacity outside those limits must be used with an appropriate correction to adjust the capacity to 100 ml.
- 5.6. Water-bath, controlled thermostatically at 20 \pm 0.1 °C.

6. Procedure

6.1. Preparation of the solution

Weigh as quickly as possible 26 ± 0.002 g of the sample and transfer it quantitatively into a 100 ml volumetric flask (5.5) with approximately 60 ml of water.

Dissolve by swirling but without heating.

Where clarification is necessary, add 0.5 ml of lead acetate reagent (4.1).

Mix the solution by rotating the flask and wash the flask walls, until the volume is such that the meniscus is about 10 mm below the calibration mark.

Place the flask in the water-bath controlled (5.6) at 20 \pm 0.1 °C until the temperature of the sugar solution is constant.

Eliminate any bubbles formed at the surface of the liquid with a drop of diethyl ether (4.2).

Make up to volume with water.

Stopper and mix thoroughly by inverting the flask at least three times.

Allow to stand for five minutes.

6.2. Polarization

Maintain the temperature at 20 ± 1 °C for all subsequent operations.

- 6.2.1 . Obtain the zero correction of the apparatus.
- 6.2.2 . Filter the sample through a filter paper. Discard the first 10 ml of the filtrate. Collect the next 50 ml of the filtrate.
- 6.2.3. Wash the polarimeter tube by rinsing twice with the sample solution to be examined (6.2.2).

6.2.4. Fill the tube carefully at 20 \pm 0.1 °C with the sample solution to be examined.

Remove all air bubbles when sliding the end-plate into position. Place the filled tube in the cradle of the instrument.

6.2.5 . Read the rotation to within 0-05 °S or 0-02 angular degrees. Repeat a further four times. Take the mean of the five readings.

7. Expression of results

7.1 . Formula and method of calculation

The results are expressed in degrees S to the nearest 0.1 °S. To convert the angular degrees into degrees S, the following formula is used:

$^{\circ}$ S = degree of arc \times 2.889

7.2. Repeatability

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, and each representing the mean of five readings, must not exceed 0-1 °S.

FIRST COMMISSION DIRECTIVE

of 10 August 1979

amending the Annex to Council Directive 77/101/EEC on the marketing of straight feedingstuffs

(79/797/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

HAS ADOPTED THIS DIRECTIVE:

Article ¹

The Annex to Directive 77/101/EEC is replaced by the Annex hereto.

Article 2

The Member States shall bring into force by ¹ January 1981 the laws, regulations or administrative provisions necessary to comply with this Directive.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 10 August 1979.

For the Commission Finn GUNDELACH Vice-President

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 77/101/EEC of 23 November 1976 on the marketing of straight feedingstuffs (1), as last amended by Council Directive 79/372/EEC (2), and in particular Article 10 thereof,

Whereas, in view of progress made in science and technology, the Annex to the aforementioned Directive must be amended;

Whereas certain of the general provisions of the Annex must be clarified and supplemented so that the Directive can be correctly implemented;

Whereas a number of adjustments to the special provisions laid down in respect of certain straight feedingstuffs are also required;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Standing Committee for Feedingstuffs,

⁽¹⁾ OJ No L 32, 3. 2. 1977, p. 1.

⁽²⁾ OJ No L 86, 6. 4. 1979, p. 29.

ANNEX

PART A

GENERAL PROVISIONS

1. Designation

- 1.1 . If the straight feedingstuff has undergone a process which is not indicated by name, there must always be added to the name of the product, particulars of the process used, the method by which it was obtained, and, if applicable, the type of presentation, e.g. 'pressed', 'rolled', 'crushed', 'ground', ' milled'.
- 1.2. In the case of the straight feedingstuffs listed in items 2.1.1 to 2.1.3 of Part B, it may be laid down that the name must be supplemented by particulars of the type or types of wheat used: common wheat, durum wheat or common wheat and durum wheat.
- 1.3 . In the case of the straight feedingstuffs listed in items 2.9.2 and 3.2.8 of Part B, it may be laid down that the name must be accompanied by particulars of the vegetable or animal species from which the product is derived.

2. Compulsory declarations and requirements

- 2.1. The levels indicated or to be declared as specified in Part B refer to:
	- the weight of the straight feedingstuff as such, for the purpose of columns 4 and 5,
	- the weight of dry matter contained in the straight feedingstuff, for the purpose of column 6, with the exception of moisture content and items 2.6.5, 2.6.6, 2.9.2, 3.2.8 and 3.3.2.

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- 2.2. Where the products referred to in column 2 of Part B of the Annex are used to denature or bind straight feedingstuffs, the following information must be given:
	- denaturing agents: nature and quantity of the products used,
	- binding agents: nature of the products used.

In the case of binding agents, the quantity of the products used may not exceed 3% of the total weight.

- 2.3 . Without prejudice to the provisions laid down in Article 3 and within the composition requirements, the botanical purity of the products and by-products listed in columns ¹ and 2 of Part B must not be less than 95% unless a different content is specified in column 6.
- 2.4. Considered as being botanical impurities are:
	- (a) natural but innocuous impurities (e.g. straw and straw waste, seeds of other cultivated species or of weeds);
	- (b) harmless residues of other oil seeds or oleaginous fruit derived from a previous manufacturing process, the level of which docs not exceed 0 5% .
- 2.5 . Where, on official analysis puruant to Article 12 of the Directive, the composition of a straight feedingstuff is found to depart from the declared composition in a manner such as to reduce its value, the following minimum tolerances are permitted:
	- (a) for crude protein, total sugars, reducing sugars and sucrose, lactose and glucose (dextrose):
		- two units for declared contents of 20% or more,
		- 10% of the declared content for declared contents of less than 20%,
		- $-$ 0.5 unit for declared contents of less than 5%;
	- (b) for starch and inulin:
		- three units for declared contents of 30% or more,
		- 10% of the declared content for declared contents of less than 30%,
		- one unit for declared contents of less than 10 % ;
	- (c) for crude oils and fats and crude fibre:
		- 1-5 units for declared contents of 15 % or more,
		- $-$ 10 % of the declared content for declared contents of less than 15%,
		- $-$ 0.5 unit for declared contents of less than 5% ;
- (d) for moisture, crude ash, total phosphorus, sodium, calcium carbonate, calcium, magnesium, acid index, and matter insoluble in light petroleum:
	- one unit for declared contents (values) of 10% (10) or more, as appropriate,
	- 10% of the declared content (value) for declared contents of less than 10% (10), as appropriate,
	- $-$ 0.2 unit for declared contents (values) of less than 2% (2), as appropriate;
- (e) for ash insoluble in hydrochloric acid and chlorides expressed as NaCl:
	- 10% of the declared content for declared contents of 3% or more,
	- $-$ 0.3 unit for declared contents of less than 3%;
- (f) for carotene, vitamin A and Xanthophyll:
	- 30% of the declared content;
- (g) for methionine, lysine and volatile nitrogenous bases:
	- 20% of the declared content.
- 2.6. Without prejudice to the provisions laid down -in Article 3 and within the composition requirements, the content of ash insoluble in hydrochloric acid (HC1) listed in Part B must not exceed 2% unless a different content is specified in column 6.

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