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BSI Standards Publication

Cosmetics — Sun protection test methods — In vivo determination of sunscreen UVA protection

(ISO 24442:2011)

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National foreword

This British Standard is the UK implementation of EN ISO 24442:2011.

The UK participation in its preparation was entrusted to Technical Committee CW/217, Cosmetics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN ISO 24442:2011) has been prepared by Technical Committee ISO/TC 217 "Cosmetics" in collaboration with Technical Committee CEN/TC 392 “Cosmetics” the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2012, and conflicting national standards shall be withdrawn at the latest by June 2012.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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Endorsement notice

The text of ISO 24442:2011 has been approved by CEN as a EN ISO 24442:2011 without any modification.

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 24442 was prepared by Technical Committee ISO/TC 217, *Cosmetics*.

Introduction

This International Standard specifies the procedure to determine the Ultraviolet A Protection Factor (UVAPF) of a sunscreen product using the persistent pigment darkening method according to the principles recommended by the Japan Cosmetic Industry Association (JCIA) in 1995[1]. The outcome of this test method can be used to determine the UVA classification of topical sunscreen products according to local regulatory requirements.

Topical sunscreen products are primarily rated and labelled according to their ability to protect against sunburn, using a test method to determine the *in vivo* Sun Protection Factor (see ISO/FDIS 24444). This rating evaluates filtration of sunburn generating radiation across the electromagnetic UV spectrum (290 nm to 400 nm). However, knowledge of the Sun Protection Factor (SPF) rating does not provide explicit information on the magnitude of the protection provided specifically in the UVA range of the spectrum (320 nm to 400 nm), as it is possible to have high SPF products with very modest UVA protection (e.g. SPF 50 with a UVAPF of only 3 to 4). There is demand among medical professionals, as well as knowledgeable consumers, to have fuller information on the UVA protection provided by their sunscreen product, in addition to the SPF, in order to make a more informed choice of product, providing a more balanced and broader-spectrum protection. The UVAPF value of a product provides information on the magnitude of the protection provided explicitly in the UVA portion of the spectrum, independent of the SPF values.

The test method outlined in this International Standard is derived primarily from the UVAPF test methods as developed by the JCIA. Modifications have been made to attempt to harmonize with other methodologies without changing the integrity of the fundamental underlying principles of the test method.

Cosmetics — Sun protection test methods — In vivo determination of sunscreen UVA protection

1 Scope

This International Standard specifies an *in vivo* method for assessment of the UVA protection factor (UVAPF) of topical sunscreen products. This International Standard is applicable to cosmetics, drugs and other products intended to be topically applied to human skin, including any component able to absorb, reflect or scatter UV rays.

It provides a basis for the evaluation of sunscreen products for the protection of human skin against UVA radiation from solar or other light sources.

2 Terms and definitions

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

2.1
ultraviolet radiation
electromagnetic radiation in the range of 290 nm to 400 nm

NOTE UVB = 290 nm to 320 nm; UVA = 320 nm to 400 nm (UVA II = 320 nm to 340 nm; UVA I = 340 nm to 400 nm).

2.2
erythema
reddening of the skin caused by UV radiation

2.3
persistent pigment darkening
PPD
skin darkening that persists more than 2 h after the end of UVA exposure

2.4
minimal persistent pigment darkening dose
MPPDD
lowest Ultraviolet A (UVA) dose that produces the first perceptible unambiguous persistent pigment darkening response with defined borders appearing over most of the field of UVA exposure, observed between 2 h and 24 h after the end of the UVA exposure

NOTE The MPPDD on unprotected skin is referenced as “MPPDDu”, and the MPPDD on sunscreen-protected skin is referenced as “MPPDDp”.

2.5
individual Ultraviolet A protection factor
UVAPFi
ratio of the minimal persistent pigment darkening dose on product-protected skin (MPPDDp) to the minimal persistent pigment darkening dose on unprotected skin (MPPDDu) of the same subject:

$$\mathrm{UVAPFi} = \frac{\mathrm{MPPDDp}}{\mathrm{MPPDDu}}$$

2.6
UVA protection factor of a product
UVAPF
arithmetic mean of all valid individual UVAPFi values obtained from all subjects in the test

2.7
test area
back between the scapula line and the waist

2.8
test site
area of skin to which a test product or reference sunscreen is applied within the test area

NOTE The area used to determine the MPPDDu is also a test site.

2.9
test subsite
skin areas within a test site exposed to UVA radiation

3 Principle

The UVAPF test method is analogous to the test method used to determine the SPF of a sunscreen product. However, it utilizes only the UVA portion of the xenon arc lamp solar simulator of defined and known output to determine the protection provided by sunscreen products on human skin in the UVA portion of the spectrum.

The UVAPF test method uses persistent pigment darkening (PPD) responses of the skin as the end point for evaluating transmitted UVA radiation. The test is restricted to the area of the back of selected human subjects. An area of each subject's skin is exposed to UVA light without any protection and another (different) area is exposed after application of the sunscreen product under test. One further area is exposed after application of a reference UVA sunscreen formulation, which is used to validate the procedure.

To determine the UVAPF, incremental series of UVA exposures are delivered to five or six small subsites on the skin to induce darkening responses. These responses are visually assessed for pigment darkness 2 h to 24 h after UVA exposure, by the judgement of a trained evaluator. The minimal persistent pigment darkening dose (MPPDD) for unprotected skin (MPPDDu) and the MPPDD obtained after application of a sunscreen product (i.e. the MPPDD for product-protected skin, MPPDDp) are determined on the same subject on the same day. An individual sun protection factor (UVAPFi) for each subject tested is calculated as the ratio of MPPDDp/MPPDDu.

4 Test subjects

4.1 Selection of test subjects

4.1.1 General

For subject inclusion and exclusion criteria, refer to Annex A.

4.1.2 Age restriction

Test subjects below age of consent or older than 70 years shall not be included in the UVAPF test panel.

4.1.3 Skin phototype of test subjects

The skin of subjects shall be Fitzpatrick phototype[2] II, III and IV. Alternatively, the colorimetric ITA° value of subjects shall be within the range of 20° and 41°.

4.1.4 Frequency of participation in tests

Since a sufficient interval after a previous test is needed in order to allow for reversal of skin tanning resulting from that previous test, a test site that has been exposed to UV should not be used in a subsequent test before two months have elapsed and the site is free of any sign of previous pigmentation marks.

4.1.5 Consent

Informed, written (signature) consent shall be obtained from all test subjects.

4.1.6 Ethical aspect

All testing shall be done in accordance with the Declaration of Helsinki and national regulations regarding human studies, if any.

4.2 Number of subjects

The test subjects shall be required to provide a minimum of 10 valid UVAPFi values and a maximum of 20 valid results. A maximum of five individual invalid results may be excluded from the calculation of the mean UVAPF, but each exclusion shall be justified according to 10.3.3 or other non-compliance with protocol. Consequently, the total number of subjects will be between a minimum of 10 and a maximum of 25 subjects.

In order to determine the number of subjects, the 95 % confidence interval (CI) shall be taken into account. The 95 % confidence interval should lie within $\pm$17 % of the mean UVAPF, and a minimum of 10 subjects is required. Otherwise, the number of subjects is increased stepwise from 10 until the statistical criterion is met (up to a maximum of 20 valid results or a maximum of 25 subjects tested). If this statistical criterion is not reached after 20 valid results from a maximum of 25 subjects, then the entire test is rejected, and a new test shall be initiated. For details of statistical definitions, sequential testing procedure and calculations, refer to Annex D.

5 Reference sunscreen formulae

The method is controlled by the use of a reference sunscreen formulation to verify the test procedure. If all test samples have an expected UVAPF value below 12, a control sample S1 sunscreen formula (see Annex C) may be used in every study to confirm the reliability of the results obtained for the test samples. The mean UVAPF value of the control sample is 4,4. The results of the UVAPF of the S1 control shall lie between 3,8 and 5,0 or the test is invalid and shall be repeated.

Reference S2 sunscreen formula[4] (see Annex C) shall be used in a study if any test sample has an expected UVAPF of 12 or above. The mean UVAPF for the reference sample S2 is 12,7. The test results of the reference S2 UVAPF shall lie between 10,7 and 14,7 or the test is invalid and shall be repeated. The reference S2 may be used to validate any product test.

Only one reference sunscreen formula is required for each test.

6 UVA source

6.1 Spectral characteristics

The source of UVA radiation shall be a xenon arc lamp solar simulator (of which the spectrum encompasses primarily UVA radiation from 320 nm to 400 nm) with a continuous spectrum. Typical sources used for this testing are multiport or single-port solar simulators fitted with optical cut-off filters to eliminate wavelengths below 320 nm (UVB) and between 400 nm (visible light and infrared) and 1 500 nm, and which yield the performance specifications given in Table 1.

The maximum level of visible and infrared (IR) radiation in the source beam shall be less than 5 % of the total source output. The amount of UVA I radiation shall be between 80 % and 92 % of the total UVA output (UVA I/UVA = 80 % to 92 %), and the amount of UVA II (320 nm to 340 nm) shall be between 8 % and 20 % of the total UVA irradiance (UVA II/UVA = 8 % to 20 %). There shall be less than 0,1 % of UVB contained in the source beam.

The spectrum shall be measured by an expert using a spectroradiometer that is traceable to a recognized standard lamp source.

Table 1 — Performance specifications

Spectral range	Measured
<320 nm (UVB)	<0,1 % of total UV
320 nm to 340 nm (UVA II)	8 % to 20 % of total UVA
340 nm to 400 nm (UVA I)	80 % to 92 % of total UVA
400 nm to 1 500 nm (visible and near-IR)	<5 % of total output of the source

6.2 Maintenance and monitoring the UV solar simulator output

6.2.1 Radiometry

Before UV exposure of each test site, the UV irradiance should be checked with a radiometer calibrated against a spectroradiometric measurement of the solar simulator output.

6.2.2 Spectroradiometry

It is recommended that a complete spectroradiometric check (UVA and UVB) of output spectrum and intensity be made by the laboratory at least once every 18 months, or after 3 000 h of lamp running time, and after changing any significant physical (optical) component of the solar simulator. It is strongly recommended that an independent expert conduct this periodical inspection.

The simple use of specific filters is not in itself adequate assurance that the UV output is of the correct quality. Detailed instructions for ensuring correct lamp output are given in Annex B.

6.3 Beam size and uniformity

6.3.1 General

Beam size for each exposure subsite shall be at least 0,5 cm^2. The intensity of the beam shall be as uniform as possible.

6.3.2 Large beam sources

When a large-beam lamp is used to simultaneously expose several subsites, the minimum beam irradiance, at any UV exposure site, shall be no more than 10 % lower than the maximum beam irradiance at any UV exposure site. If the variation exceeds 10 %, then appropriate compensation for different irradiance should be made in the exposure time on each UV exposure site.

6.3.3 Small beam sources

For a small beam UV source, an uneven skin darkening (such as a half-moon shape) indicates that the irradiance is not uniform and the delivery system shall be realigned or corrected.

6.4 Total irradiance (UV, visible and near-infrared rays)

The test conductor shall confirm that the total irradiance shall not exceed 1 600 W/m^2 (reciprocity has been tested over the range of 370 W/m^2 to 1 440 W/m^2)[3].

When total irradiance is strong, an excessive feeling of heat or pain might be induced in the irradiated skin of subjects. Therefore, it shall be confirmed that the maximum irradiance that will be used (UV, visible and near-infrared rays) will not induce an excessive feeling of heat in the skin prior to conducting a UVAPF test.

7 Product application quantity and procedure

7.1 General

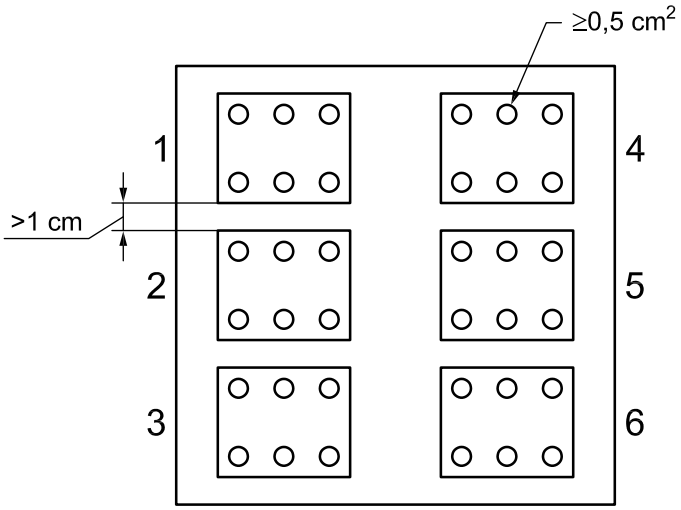
The application of the products should be made by a trained technician. The room temperature shall be between 18 °C and 26 °C. The use of a finger cot is optional but recommended. If employed, a new finger cot shall be used for each new application of product and should not be pre-saturated with the test product. When a naked finger is used, the finger shall be cleaned between product applications. All products should be homogeneous and should be shaken if necessary, before weighing, to ensure uniform dispersion.

7.2 Position of the subject

Product shall be applied to subjects in the same position as will be utilized for the irradiation procedure (sitting or prone). Powder samples should be tested in the prone position to prevent the samples from falling off the surface of the skin.

7.3 Defining test sites

Areas of at least 30 cm^2 to at most 60 cm^2, located between the scapula line and the waist, shall be delineated using a template and a special skin marker with a distance of at least 1 cm between each test site. The number of sites shall be restricted to no more than six. The location of the products on the sites and the unprotected site shall be randomized on the subject's back (see Figure 1). Before application of the test product or standard formula, the test site can be cleaned with a clean, dry cotton pad or equivalent material.



Keys

1 product 1
2 reference sunscreen
3 product 2
4 product 4
5 MPPDDu (no topical application)
6 product 3

Figure 1 — Example of arrangement of test sites

7.4 Application procedure

7.4.1 Product application technique — Liquid-type products, e.g. lotions, milks, creams, sticks, sprays

7.4.1.1 The amount of product to be applied is to be weighed in a syringe or pipette or, alternatively, in another device such as a watch glass or weigh boat (container). A method of weighing by loss should be used.

7.4.1.2 Care shall be taken to prevent evaporative loss of volatile components when the product is being weighed and before application on the skin. It is important that the total quantity of weighed product be transferred to the product application site.

7.4.1.3 The product is then dispensed in small droplets (approximately 15 per 30 cm^2, 30 per 60 cm^2) over the whole test site at a dose of 2,00 $mg/cm^2 \pm 0,05$ mg/cm^2 delivered.

7.4.1.4 The product should be gently spread with a finger cot or a clean finger using circular and then linear movements (up and down), without excessive pressure. The time used to spread the product on the test site shall be from 20 s to not more than 50 s.

7.4.1.5 If necessary, in case of uneven application, application may be repeated on a new test site.

7.4.2 Product application technique — Powders

7.4.2.1 Purified water or another suitable solvent that has no other UV protection properties may be applied on the skin before the powder application to help the sample adhere to the application site.

7.4.2.2 Aliquots of powder should be transferred to the skin in a grid-like manner, using a spatula or finger.

7.4.2.3 The accumulated powder is tapped and then spread over the whole test site using a finger with or without a finger cot.

7.4.2.4 Alternatively, the tip of a preloaded cosmetic application puff may be used instead of a finger. In this case, it is important to verify that 2,00 $mg/cm^2 \pm 0,05$ mg/cm^2 of test powder remains on the skin after spreading by weighing the application puff.

8 Determination of minimal persistent pigment darkening doses (MPPDD)

8.1 UV exposure timing and subject position

Exposure of the test site to the sequence of UV doses shall start no sooner than 15 min and no more than 30 min after the application of the product(s). The position of the subjects during the whole exposure period shall be the same as when the product was applied. Any extraneous exposure of the test sites to UV light (artificial or natural) shall be avoided during this period and for a period of 24 h after exposure.

8.2 Determination of the minimal pigmenting dose on the unprotected test sites (MPPDDu) using a multiple-beam solar simulator

8.2.1 When a multiple-beam solar simulator is used, UVA radiation is conducted onto multiple UVA exposure sites (typically six), each of which receives an independent dose of radiation of identical spectrum, but with different intensity. The individual UVA exposure sites are typically between 8 mm and 10 mm in diameter.

8.2.2 Measure and adjust the UVA irradiance of each optical beam to obtain a geometric progression of 25 % (0,64, 0,8, 1, 1,25, 1,56, 1,95) using a radiometer calibrated with the UVA source.

8.2.3 Determine the exposure time needed to achieve the range of doses to be typically used for the determination of the MPPDDu: 8 J/cm^2, 10 J/cm^2, 12,5 J/cm^2, 15,6 J/cm^2, 19,3 J/cm^2, and 24,4 J/cm^2 (depending on the radiometer used). Other (higher or lower) exposure doses and times may be based on prior data of an individual subject or ITA° testing prediction for that subject (see Annex A).

EXAMPLE

— UVA irradiance at third highest intensity optical fibre = 50 mW/cm^2;

— MPPDDu (estimated) = 12,5 J/cm^2;

— Exposure time for MPPDDu = 12,5 J/cm^2/50 mW/cm^2 = 250 s.

8.2.4 Expose the unprotected test subsites using the predetermined time for the unprotected-site MPPDDu determination.

8.3 Determination of the MPPDDu using a single-beam output solar simulator

8.3.1 When using a single beam output solar simulator, there is only one beam to be measured with the radiometer calibrated with the solar simulator to determine UVA irradiance.

8.3.2 Measure the UVA irradiance of the one output beam at the plane of the skin exposure with the calibrated radiometer.

8.3.3 Determine the exposure times needed to achieve the range of doses generally used for the determination of the MPPDDu, which is approximately 8 J/cm^2, 10 J/cm^2, 12,5 J/cm^2, 15,6 J/cm^2, 19,3 J/cm^2, and 24,4 J/cm^2. Other (higher or lower) exposure doses and times may be used based on prior data of an individual subject or ITA° testing prediction for that subject.

EXAMPLE

— UVA irradiance = 50 mW/cm^2

— Exposure times for series are:

  — 8 J/cm^2/50 mW/cm^2 = 160 s;

  — 10 J/cm^2/50 mW/cm^2 = 200 s;

  — 12,5 Jcm^2/50 mW/cm^2 = 250 s;

  — 15,6 J/cm^2/50 mW/cm^2 = 312 s;

  — 19,3 J/cm^2/50 W/cm^2 = 386 s;

  — 24,4 J/cm^2/50 mW/cm^2 = 488 s.

8.3.4 Expose the unprotected area subsites sequentially with the times for the unprotected-site MPPDDu determination.

8.4 Determination of the MPPDDp using a multiple-beam solar simulator

8.4.1 Before the exposure of each test site, measure and adjust the UVA irradiance of each optical fibre to obtain a geometric progression of 25 % (0,64, 0,8, 1, 1,25, 1,56, 1,95) using a radiometer calibrated with the solar simulator.

8.4.2 Determine the exposure time needed to achieve the range of doses to be used for the determination of the MPPDDp.

8.4.3 Multiply the MPPDDu of the subject (determined in a pretest or predicted) by the expected UVAPF value of the test product (supplied by the sponsor or estimated from *in vitro* UVAPF testing) to determine the estimated MPPDDp of the product. Divide the estimated MPPDDp by the UVA irradiance of the third highest intensity optical fibre to determine the exposure time for the sequence of doses for the test product.

EXAMPLE

— UVA irradiance at third highest intensity optical fibre = 50 mW/cm^2;

— MPPDDu = 10 J/cm^2;

— Estimated test product UVAPF = 10;

— Estimated MPPDDp = 10 J/cm^2 × 10 = 100 J/cm^2;

— Exposure time for MPPDDp = 100 J/cm^2/50 mW/cm^2 = 2 000 s.

8.4.4 After completion of the exposure of all the protected test sites, proceed to conducting the exposure of the unprotected MPPDDu site as determined in 8.2.

8.5 Determination of the MPPDDp on the protected test site with a single-beam solar simulator

8.5.1 Measure the UVA irradiance of the one output beam at the plane of the skin exposure with the calibrated radiometer before starting the exposures within each test site.

8.5.2 Determine the exposure times needed to achieve the range of doses for irradiation of the sunscreen-protected sites as given in 8.5.3.

8.5.3 Multiply the MPPDDu of the subject (determined in a pretest or predicted) by the expected UVAPF value of the test product (supplied by the sponsor or estimated from *in vitro* UVAPF testing) to determine the estimated MPPDDp of the product. Divide the estimated MPPDDp by the UVA irradiance of the third highest exposure dose in the series to determine the exposure time. The two lower doses, and two (or three) higher doses, shall be each 1,25 times lower or higher than the preceding dose.

EXAMPLE

— MPPDDu = 10 J/cm^2;

— Expected UVAPF for test product = 10;

— MPPDDu expected = 10 × 10 J/cm^2 = 100 J/cm^2;

— UVA irradiance = 50 mW/cm^2;

— Estimated MPPDDp exposure time = 100 J/cm^2/50mW/cm^2 = 2 000 s;

— Exposure sequence should be:

  — 1 280 s, 1 600 s, 2 000 s, 2 500 s, 3 125 s, and optionally 3 906 s.

8.5.4 After completion of the exposure of all the protected test sites, proceed to conducting the exposure of the unprotected MPPDDu site as described in 8.3.

9 Product removal

After UV exposure, control and test products may be removed gently, using a cotton or cellulose pad with a neutral lotion to eliminate traces of pigments or coloured products that could interfere with the pigmentation evaluation.

10 MPPDD assessment procedure

10.1 Observation time for responses of MPPDDs

The MPPDD responses shall be assessed between 2 h and 24 h after completion of the exposure of the last UVA exposure sites. Observations of protected (product) and unprotected test sites shall be conducted at the same relative time point after the end of the exposures (i.e. if at 4 h, it is 4 h after completion of exposures for the unprotected site and 4 h after completion of the protected site, not actually at the same time). Observations shall all be made on the same day.

10.2 Position of subjects for MPPDD observations

The position of the subject should be the same for product application, for UV exposure and for MPPDD assessment.

10.3 MPPDD evaluation

10.3.1 Observation conditions

Visual evaluation should be performed in a blinded manner by a qualified observer under standardized, sufficient and uniform illumination conditions (white lamps, industry type, delivering at least 450 lx over the examination plane), with the subject in the same position that was used for the product application and UVA exposures.

In order to ensure observer blinding, the observer shall not be the same individual that applied the product or administered the UVA exposures. Observation documents shall be coded so that the identity of the location and identity of MPPDDu, the UVA treatment doses, the test products, and the control product are not revealed.

10.3.2 MPPDD determinations

The minimal persistent pigment darkening dose (MPPDD) corresponds to the lowest UVA dose within each test site causing the first perceptible unambiguous persistent pigment darkening response appearing over most of the field of UVA exposure, with defined borders (see 2.4).

10.3.3 Data rejection

Data from a test site may be rejected only on the basis of one or more of the following criteria:

a) there is no pigmenting response on any UVA exposure subsites;

b) all subsites have a pigmented response;

c) there are random pigmenting responses that do not follow the logical sequence of the test (randomly absent responses);

d) the test subject is non-compliant or becomes ill, or does not shield the test area from sunlight after exposures;

e) a technical error occurs during UVA exposure.

11 Calculations of the UVAPF and statistics

11.1 Calculation of the individual UVAPF (UVAPFi) for each test product for each subject

The UVAPFi is calculated for each test product for each volunteer as the ratio of the minimal UVA dose necessary to induce the defined pigmentation response (see 2.4) on the MPPDDp and the minimal UVA dose necessary to induce the MPPDDu:

$$\text{UVAPFi} = \frac{\text{MPPDDp}}{\text{MPPDDu}}$$

where

MPPDD(u,p) is the minimal persistent pigment darkening dose.

11.2 Calculation of the mean UVAPF

The UVAPF value of a test product is determined as the arithmetic mean of the individual volunteer UVAPFi values using a minimum of 10 subjects with valid results. Additional subjects may be used, as required by local regulations or to meet the statistical test requirements below.

11.3 Statistical test

The 95 % confidence interval shall lie within ±17 % of the mean UVAPF; a minimum of 10 subjects is required. Otherwise, the number of subjects is increased stepwise from 10 until the statistical criterion is met (up to a maximum of 20 valid results or a maximum of 25 subjects tested). If this statistical criterion is not reached after 20 valid results from a maximum of 25 subjects, the entire test is rejected and a new test shall be initiated. For details of statistical definitions, sequential testing procedure and calculations, refer to Annex E.

11.4 Test rejection for failure of meeting the statistical test for the reference sunscreen

If the mean value of the reference sunscreen (S1 or S2) is not in the expected range, the entire test shall be rejected.

12 Test report

The test report shall contain the following information:

a) product identifier, code and expected UVAPF;

b) subject information (number, name or identification code, skin phototype or ITA° value);

c) identification and characterization of the UVA source with intensity in units of energy/unit area;

d) individual MPPDDu and MPPDDp for unprotected skin and test-product-protected skin and the reference sunscreen-protected skin;

e) individual UVAPFi values expressed to one decimal place, including all valid data and rejected data for the test product and for the reference sunscreen;

f) mean UVAPF values, standard deviation on the mean and confidence interval of 95 %;

g) protocol deviations (if any);

h) identification of the technician who conducted the test, listed by subject;

i) date of the test.

Annex A
(normative)

Selection criteria for the test subjects

A.1 Skin phototypes

Subjects should be selected using Fitzpatrick skin phototype or ITA° value.

The skin phototype of subjects shall be Fitzpatrick type II, III or IV. Alternatively, the colorimetric ITA° value of subjects shall be within the range of 20° to 41°.

The Fitzpatrick skin phototype definitions are based on the first 30 min to 45 min of sun exposure after a winter season without sun exposure, with the following results:

— Type I: always burns easily, never tans;

— Type II: always burns easily, tans minimally;

— Type III: burns moderately, tans gradually;

— Type IV: burns minimally, always tans well;

— Type V: rarely burns, tans profusely;

— Type VI: never burns; deeply pigmented.

Colorimetric ITA values and skin colour categories are defined by the colorimetric descriptors of Chardon et al[5] using the CIE (1976) $L^*a^*b^*$ colour space.

Table A.1 — ITA° values for skin colours

Skin colours	ITA° value ranges
Very light	>55°
Light	>41° to 55°
Intermediate	>28° to 41°
Tan (or matt)	>10° to 28°
Brown	> −30° to 10°
Black	≤ −30°

ITA° = {Arc Tangent [(L^* - 50)/b^*]} 180/3,141 6.

A.2 Medical and ethical considerations

It is recommended that new subjects first be interviewed by a health professional to establish their medical status and suitability for inclusion in the subject panel. It should be verified that there is no condition (such as abnormal response to the sun) which would put the subject at risk. Subjects shall be also presented with the list of exclusion criteria below and attest that they do not have any of the listed conditions (without having to identify which one). Before participating in a study, it shall be verified that the subjects' medical status has not changed since the original interview. A trained scientist or technician shall also check each subject visually before qualifying them to participate in a study in order to ensure that their skin colour within the test sites is uniform, without pigmentation marks, nevi, and that no sunburn (erythema) is present on the test area.

Human subjects should be adequately informed of the aims and potential risk (direct or secondary effects) of the study and any discomfort they might experience. Each subject shall give a written agreement to participate in UVAPF tests (free informed written consent is mandatory prior to entering the study, according to the general Declaration of Helsinki).

When there is some doubt on the expected UVAPF of the test product, a screening should first be performed. To protect the volunteers, it is recommended that the test be started with a lower MPPDDp value which is increased progressively.

A.3 Exclusion criteria

All exclusion criteria shall be checked before testing.

The following conditions shall automatically exclude a subject from the test group:

— children and persons below the age of consent or older than 70 years;

— pregnant or lactating women;

— subjects using medication with photo-sensitizing potential;

— subjects that are immunosuppressed, such as HIV-positive patients or transplant patients;

— subjects with a family history of skin cancer;

— subjects receiving chemotherapy or radiotherapy;

— subjects using anti-inflammatory medication;

— subjects with dermatological conditions;

— subjects with a history of abnormal response to the sun;

— subjects accustomed to using tanning beds;

— subjects who have had UV exposure on the back area in the four weeks prior to UVAPF testing.

A.4 Frequency of subject participation (interval between two tests)

The proposed test sites on the backs of test subjects shall not have been used in any sun product testing within the previous two months.

A.5 Quality of the test site

The test sites shall have:

— no pigmentation spots from previous tests;

— no evidence of UV exposure (sunburn or tanning);

— no scars or active dermal lesions;

— uniform colour, without nevi, blemishes or solar lentigo, and without excessive hair.

Annex B
(normative)

Definition of the source of UVA radiation

B.1 Purpose

The aim of these specifications is to define practical criteria for testing the spectral compliance of the source of UVA radiation used for UVAPF testing. The artificial light source used shall comply with the source spectral specifications as described below.

B.2 Quality of radiation

The source shall emit a continuous spectrum with no gaps or extreme peaks of emission in the UVA region. A xenon arc source with appropriate filters is required. The output from the UVA source shall be stable, uniform across the whole output beam (particularly important for a single large beam) and suitably filtered to create a spectral quality that complies with the required acceptance limits (see Table B.1).

B.3 Total irradiance (UV, visible and near-infrared rays)

When total irradiance is strong, an excessive feeling of heat or pain might occasionally be induced in the irradiated skin of subjects. Therefore, prior to conducting a UVAPF test, it shall be confirmed that the maximum irradiance that will be used (UV, visible and near-infrared rays) will not induce an excessive feeling of heat in the skin. For that reason, total irradiance shall not exceed 1 600 W/m^2 (reciprocity has been tested over the range of 370 W/m^2 to 1 440 W/m^2)[3].

B.4 Uniformity of beam

B.4.1 When a large-beam UV source is used to simultaneously expose several subsites within an irradiation series by varying the exposure time, the intensity of the beam shall be as uniform as possible. The minimum beam irradiance, at any point, shall be no more than 10 % lower than the maximum beam irradiance at any point. If the variation exceeds 10 %, then appropriate compensation for different irradiance shall be made in the exposure time on each UV exposure subsite.

B.4.2 For a small-beam UV source, the intensity of the beam shall be as uniform as possible. An uneven darkening response in unprotected skin (such as a half-moon shape) indicates that the irradiance is not uniform and the delivery system shall be realigned or corrected.

B.5 Maintenance and monitoring of the UV solar simulator output

B.5.1 Radiometry

Before UVA exposure of each test site, the UVA irradiance shall be checked with a radiometer calibrated against a spectroradiometric measurement of the UVA source.

B.5.2 Spectroradiometry

It is recommended that a complete spectroradiometric check (UVA,UVB, visible and infrared) of the output spectrum and intensity be made at least once every 18 m or after 3 000 h of running time and each time a significant physical (optical) component is changed. It is strongly recommended that an experienced expert conduct this periodical inspection and certify that the lamp complies with the output as specified in Table B.1.

The simple use of specified filters is not in itself adequate assurance that the UV output is of the correct quality. All ports of the light source shall be measured independently to assure compliance.

Table B.1 — Percentage acceptance limits for the UVA solar simulator output

Spectral range	Measured	
	Lower limit	Upper limit
<320 nm (UVB)		<0,1 % of total UV
320 nm to 340 nm (UVA II)	8 %	20 % of total UVA
340 nm to 400 nm (UVA I)	80 %	92 % of total UVA
400 nm to 1 500 nm (visible + near-IR)	<5 % of total source irradiance	

Annex C
(normative)

UVAPF reference sunscreen S1

C.1 Mean UVAPF and acceptance limits for reference sunscreen formulations

Mean UVAPF and acceptance limits for reference sunscreen formulations are given in Table C.1.

Table C.1 — Mean UVAPF and acceptance limits for reference sunscreen formulations

Reference sunscreen formulation	Mean UVAPF	Acceptance limits	
		Lower limit	Upper limit
S1	4,4	3,8	5,0
S2	12,7	10,7	14,7

C.2 UVAPF reference formula S1

Table C.2 gives the UVAPF reference formula S1.

Table C.2 — UVAPF reference formula S1

Ingredients	% mass of total mass
Phase 1 (aqueous)	
Purified water	57,13
Dipropylene glycol	5,0
Potassium hydroxide	0,12
Trisodium EDTA	0,05
Phenoxyethanol	0,3
Phase 2 (oil)	
Stearic acid	3,0
Glyceryl stearate (S.E.)	3,0
Cetearyl alcohol	5,0
Petrolatum soft yellow	3,0
Glyceryl tri(2-ethylhexanoate)	15,0
Ethylhexyl methoxycinnamate	3,0
Butyl methoxydibenzoylmethane	5,0
Ethylparaben	0,2
Methylparaben	0,2

C.3 Manufacturing process

C.3.1 Combine the phase 1 aqueous materials given in Table C.2 and mix until completely uniform and dissolved. Commence heating to 70 °C.

C.3.2 In a separate vessel, combine the phase 2 oil materials. Heat to 70 °C and mix until uniform and molten.

C.3.3 Combine phases by adding the phase 2 materials to the phase 1 materials and mix until uniform.

C.3.4 Homogenize at 4 000 r/min for between 3 min and 5 min.

C.3.5 Continue mixing with an overhead stirrer as the batch cools.

C.4 Physicochemical data

C.4.1 Appearance: white cream.

C.4.2 pH: 6,5 ± 0,5 (diluted 1 part to 5 parts freshly distilled water).

C.4.3 Viscosity (20 °C): 50 000 cP to 80 000 cP using Brookfield Spindle D[1)] at 10 r/min.

C.4.4 Density (20 °C): 0,95 g/cm^3 ± 0,05 g/cm^3.

C.5 Storage and expiry

The formulation shall be stored for 12 months from the date of manufacture at 20 °C, in a vessel protected from light.

C.6 Analytical data

C.6.1 Principle

The formulation is sampled gravimetrically and dissolved in ethanol (in which the analytes are soluble). The solution is filtrated and chromatographed on a microparticulate silica gel column using a mixture of water and ethanol as the mobile phase. The concentrations of the analytes in the sample are determined by quantification against a mixed external standard solution of analyte raw materials.

NOTE See Reference [11].

C.6.2 Chemical reagents

C.6.2.1 Absolute ethanol (HPLC grade).

C.6.2.2 Ultrapure water (HPLC grade).

C.6.2.3 Phosphoric acid, 85 % analytical purity.

C.6.2.4 Ethylhexyl methoxycinnamate.

1) Brookfield Spindle D is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

C.6.2.5 Butyl methoxydibenzoylmethane.

C.6.3 High-pressure liquid chromatography apparatus

C.6.3.1 Injector, with an injection volume of 10,0 µl.

C.6.3.2 Column, e.g. Waters Symmetry Shield C18[2)], 5 µm, with a length of 150 mm, inner diameter of 4,6 mm and a flow rate of 1,2 ml/min.

C.6.3.3 Eluent A, ultrapure water acidified with H_3PO_4 (67µl/l).

C.6.3.4 Eluent B, absolute ethanol (HPLC grade).

C.6.3.5 Detector, of type UV wavelength, 312 nm.

C.6.3.6 Data, i.e. quantification of peak area.

C.6.4 Method

C.6.4.1 Using an analytical balance, weigh approximately 50 mg of formulation to the nearest 0,1 mg into a 25 ml volumetric flask.

C.6.4.2 Dilute to volume with ethanol.

C.6.4.3 Shake with a vortex and, in case of a non-liquid formulation, sonicate with an ultrasonic bath until homogenization is achieved.

C.6.4.4 Filter through a 0,45 µm PVDF disc filter.

C.6.4.5 To prepare the working standard, weigh 100 mg of ethylhexyl methoxycinnamate (C.6.2.4) and 50 mg of butyl methoxydibenzoylmethane (C.6.2.5), then dilute with ethanol to volume in a 100 ml volumetric flask.

C.6.4.6 To prepare the mixed working standard, take 5 ml of each solution in a 50 ml volumetric flask and complete with ethanol.

C.6.4.7 Analyse the standard and mixed working standard by reverse-phase HPLC.

C.6.5 Quality control

C.6.5.1 Analyse a sample of HPLC mobile phase and a placebo, if available, prepared according to C.6.4 by reverse-phase HPLC in order to confirm the absence of interfering chromatographic peaks.

C.6.5.2 Inject a standard solution three times by reverse-phase HPLC and calculate the coefficient of variation of the analysis peak areas.

2) Waters Symmetry Shield C 18 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

C.6.6 Calculations

Analyte percentage is calculated using the following formula:

$$\frac{M \times h \times 2,5}{P \times H}$$

where

M is the mass of analyte, expressed in mg;

P is the mass of sample, expressed in mg;

h is the area of analyte peak of the sample;

H is the area of analyte peak from standardization.

C.6.7 Acceptance criteria

The analytical results are acceptable if the following conditions are achieved:

— the standard coefficient of variation is ≤2,5 %;

— recovery value is 95 % to 105 % of the formula amount;

— there are no interfering chromatographic peaks in the sample placebo or the working solvent.

C.7 Formula and preparation for standard formulation S2

Table C.3 gives the formula for standard formulation S2.

Table C.3 — Formula for standard formulation S2

Ingredients	% mass of total mass
Phase 1 (aqueous)	
Water	62,445
Propylene glycol	1,00
Xanthan gum	0,60
Carbomer	0,15
Disodium EDTA	0,08
Phase 2 (oil)	
Octocrylene	3,00
Butyl methoxydibenzoylmethane	5,00
Ethylhexyl methoxycinnamate	3,00
Bis-ethylhexyloxyphenol-methoxyphenyl triazine	2,00
Cetyl alcohol	1,00
Steareth-21	2,50
Steareth-2	3,00
Dicaprylyl carbonate	6,50
Decyl cocoate	6,50
Phenoxyethanol (and)	1,00
Methylparaben (and)	
Ethylparaben (and)	
Butylparaben (and)	
Propylparaben	
Phase 3	
Cyclopentasiloxane	2,00
Triethanolamine	0,225

C.7.1 Manufacturing process

C.7.1.1 Heat phase 1 and phase 2 materials separately until a temperature of 75 °C is reached.

C.7.1.2 Add phase 2 materials slowly to phase 1 materials while stirring phase 1.

C.7.1.3 Cool to 40 °C while stirring.

C.7.1.4 Add phase 3 materials to phase 1 and 2 materials while stirring.

C.7.1.5 Compensate water loss and homogenize.

C.7.2 Specifications

C.7.2.1 Colour: white to slightly yellow.

C.7.2.2 Density: 0,96 g/cm^3 to 1 g/cm^3.

C.7.2.3 Viscosity: 7 000 cP to 12 000 cP using Brookfield DV-II Helipath Mobile Spindleset B[3)] at 20 r/min for 60 s.

C.7.3 Storage and expiry

The formulation shall be stored for 13 months from the date of manufacture, at 20 °C, in a vessel protected from light.

C.7.4 Analytical data

C.7.4.1 Principle

The formulation is sampled gravimetrically and dissolved in ethanol (in which the analytes are soluble). Solution is filtrated and chromatographed on a microparticulate silica gel column, using a mixture of water and ethanol as the mobile phase. The concentrations of the analytes in the sample are determined by quantification against a mixed external standard solution of analyte raw materials.

NOTE See Reference [6].

C.7.4.2 Chemical reagents

C.7.4.2.1 Absolute ethanol (HPLC grade).

C.7.4.2.2 Phosphoric acid, 85 % analytical purity.

C.7.4.2.3 Ethylhexyl methoxycinnamate.

C.7.4.2.4 Butyl methoxydibenzoylmethane.

C.7.4.2.5 Octocrylene.

C.7.4.2.6 Bis-ethylhexyloxyphenol-methoxyphenyl triazine.

C.7.4.2.7 1,4-Dioxane (HPLC Grade).

C.7.4.3 Working standard

C.7.4.3.1 To prepare the working standard, weigh 100 mg of octocrylene (C.7.4.2.5) and ethylhexyl methoxycinnamate (C.7.4.2.3) and 50 mg of butyl methoxydibenzoylmethane (C.7.4.2.4), then dilute with ethanol to volume in a 100 ml volumetric flask. Weigh 200 mg of bis-ethylhexyloxyphenol-methoxyphenyl triazine (C.7.4.2.6) and dilute with 1,4-dioxanne (C.7.4.2.7) to volume in a 100 ml volumetric flask.

C.7.4.4 Mixed working standard

C.7.4.4.1 To prepare the mixed working standard, take 5 ml of each solution in a 50 ml volumetric flask. Complete with ethanol.

C.7.5 High-performance liquid chromatography apparatus

C.7.5.1 Injector, with an injection volume of 10,0 µl.

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3) Brookfield DV-II Helipath Mobile Spindleset B is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

C.7.5.2 **Column**, e.g. Waters Symmetry Shield C 18, 5 µm, with a length of 150 mm, inner diameter of 4,6 mm and a flow rate of 1,2 ml/min.

C.7.5.3 **Eluent A**, ultrapure water acidified with H_3PO_4 (67 µl/l).

C.7.5.4 **Eluent B**, absolute ethanol (HPLC grade).

C.7.5.5 **Detector**, of type UV wavelength, 312 nm.

C.7.5.6 **Data**, i.e. quantification of peak area.

C.7.6 Method

C.7.6.1 Using an analytical balance, weigh approximately 50 mg of formulation to the nearest 0,1 mg, into a 25 ml volumetric flask.

C.7.6.2 Dilute to volume with ethanol.

C.7.6.3 Shake with a vortex and, in the case of a non-liquid formulation, sonicate with an ultrasonic bath until homogenized.

C.7.6.4 Filter through a 0,45 µm PVDF disc filter.

C.7.6.5 Analyse the sample and mixed working standard by reverse-phase HPLC.

C.7.7 Quality control

C.7.7.1 Analyse a sample of HPLC mobile phase and a placebo, if available, prepared according to C.7.6, by reverse-phase HPLC, in order to confirm the absence of interfering chromatographic peaks.

C.7.7.2 Inject the standard solution three times by reverse-phase HPLC and calculate the coefficient of variation of the analysis peak areas.

C.7.8 Calculations

The analyte percentage is calculated using the following formula:

$$\frac{M \times h \times 2,5}{P \times H}$$

where

M is the mass of standard material, expressed in mg;

P is the mass of the sample, expressed in mg;

h is the area of analyte peak for the sample;

H is the area of analyte peak for the standard material (each).

C.7.9 Acceptance criteria

The analytical results are acceptable if the following conditions are achieved:

— the standard coefficient of variation is ≤2,5 %;

— recovery value is 95 % to 105 % of the formula amount;

— there are no interfering chromatographic peaks in the sample placebo or working solvent.

Annex D
(normative)

Statistics and calculations

D.1 Individual Ultraviolet A protection factor (UVAPFi)

The individual UVAPFi of each product on each subject is calculated from the individual MPPDD on unprotected skin (MPPDDu) and the individual MPPDD on product-protected skin (MPPDDp) according to Equation (D.1):

$$\text{UVAPFi} = \frac{\text{MPPDDp}}{\text{MPPDDu}} \tag{D.1}$$

D.2 Product Ultraviolet A protection factor

The UVAPF of the product is the arithmetical mean of the individual UVAPFi values obtained from the total number, n, of subjects used, expressed to one decimal point:

$$\text{UVAPF} = \frac{\sum \text{UVAPFi}}{n} \tag{D.2}$$

Its standard deviation is given by Equation (D.3):

$$s = \frac{\sqrt{\left\{\left(\sum (\text{FPUVAi})^2 - \left[\sum (\text{FPUVAi})^2 / n\right]\right)\right\}}}{(n-1)} \tag{D.3}$$

D.3 95 % confidence interval

The 95 % confidence interval (95 % CI) for the mean UVAPF is expressed by Equation (D.4):

$$95\ \%\ \text{CI} = (\text{UVAPF} - c) \text{ to } (\text{UVAPF} + c) \tag{D.4}$$

c is calculated as: $c = (t \text{ value}) \times \text{SEM} = (t \text{ value}) \times s/\sqrt{n}$

$$c = t \times s/\sqrt{n} \tag{D.5}$$

$$\text{CI}\ [\%] = 100 \times c/\text{UVAPF} \tag{D.6}$$

where

SEM is the standard error of the mean;

n is the total number of subjects used;

t is the t value from the “two-sided” Student-t distribution of Table D.1, at a probability level $p = 0{,}05$ and with degrees of freedom $v = (n - 1)$.

Table D.1 — Two-sided Student-*t* distribution table

n	10	11	12	13	14	15	16	17	18	19	20
t value	2,262	2,228	2,201	2,179	2,160	2,145	2,131	2,120	2,110	2,101	2,093

NOTE For spreadsheet calculation, the t value can be modelled by: $t = 2{,}03 + 12{,}7/n^{1{,}75}$ (for $n \geq 4$).

D.4 Experimental calculation procedure

D.4.1 Sequential procedure

An UVAPF test is begun by testing the product on an initial panel of n' subjects (n' shall be at least 10). The individual sun protection factors (UVAPFi) for the product on each subject are then calculated according to Equation (D.1).

From these individual UVAPFi values, a provisional mean UVA protection factor for the initial n' subjects ($UVAPF_{n'}$) is calculated according to Equation (D.2), together with a provisional 95 % confidence interval (95 % $CI_{n'}$) using Equations (D.4), (D.5) and (D.6) and Student-t table (Table D.1), i.e.:

$$UVAPF_{n'} = \Sigma UVAPFi/n' \tag{D.7}$$

$$95\ \%\ CI_{n'} = UVAPF_{n'} - c_{n'} \text{ to } UVAPF_{n'} + c_{n'} \tag{D.8}$$

$$c_{n'} \text{ is calculated as } c_{n'} = t_{n'} \times s_{n'}/\sqrt{n'} \tag{D.9}$$

where $s_{n'}$ is the standard deviation from the first n′ subjects calculated according to Equation (D.3):

$$s_{n'} = \frac{\sqrt{\left\{\left(\sum (FPUVAi)^2 - \left[\sum (FPUVAi)^2 / n\right]\right)\right\}}}{(n-1)} \tag{D.10}$$

$$CI_{n'}[\%]\ \ 100 \times c_{n'}/UVAPF_{n'} \tag{D.11}$$

If the calculated provisional $CI_{n'}$[%] is greater than 17 % of the provisional mean $UVAPF_{n'}$ value, then testing of the product shall continue on additional subjects until the provisional $CI_{n'}$[%] is less than 17 % of the mean provisional UVAPF. If this criterion is not fulfilled after 20 valid subjects, then the entire test shall be repeated.

D.4.2 Predicted number of subjects, n^*

If the $CI_{n'}$[%] on the provisional $UVAPF_{n'}$ is greater than 0,17 $UVAPF_{n'}$, then the predicted likely total number of subjects, n^*, necessary to meet the statistical criterion can be estimated according to the following formula and rounded up to the nearest integer:

$$n^* = (t_{n'} \times s_{n'}/C_{n'})^2 \tag{D.12}$$

where

$t_{n'}$ is the *t* statistic from Student-*t* table or Equation (D.1), with *n′* results;

$s_{n'}$ is the best estimate of population standard deviation (i.e. from the n′ results);

$C_{n'}$ is 17 % of the mean UVAPFn′, representing the required confidence interval.

EXAMPLE When n^* is calculated after the first 10 data, then:

$$n^* = (2{,}262\ s_{n'}/0{,}17\ \mathrm{UVAPF}_{n'})^2 \quad \text{(D.13)}$$

i.e.

$$n^* = (13{,}30\ s_{n'}/\mathrm{UVAPF}_{n'})^2 \quad \text{(D.14)}$$

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