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

Cosmétiques — Méthodes d'essai de protection solaire — Détermination in vivo de la protection UVA d'un produit de protection solaire

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 217, *Cosmetics,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 392, *Cosmetics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 24442:2011), which has been technically revised.

The main changes are as follows:

— this document has been aligned with the revised ISO 24444.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

This document 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 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 [for example SPF 50 with a UVA protection factor (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. Moreover, there is also a demand to prevent UVA-induced darkening of the skin from a cultural point of view even without sunburn. Thus, persistent pigment darkening (PPD) was selected as an endpoint relevant to UVA. Although PPD reflects merely photo-polymerization of melanin monomers[2], it is evaluated as a representative of the biological reactions. 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[3][4][5].

The test method outlined in this document is derived primarily from the UVAPF test methods as developed by the JCIA. Modifications have been made to attempt to be in line with updated International Standards for determination of sun protection factor without changing the integrity of the fundamental underlying principles of the test method.

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

1 Scope

This document specifies a method for the in vivo determination of UVA protection factor (UVAPF) of sunscreen products. It is applicable to products that contain any component able to absorb, reflect or scatter ultraviolet (UV) rays and which are intended to be placed in contact with human skin.

This document provides a basis for the evaluation of sunscreen products for the protection of human skin against UVA radiation induced by solar ultraviolet rays.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 24444, *Cosmetics — Sun protection test methods — In vivo determination of the sun protection factor (SPF)*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
ultraviolet radiation
UVR
electromagnetic radiation in the range of 290 nm to 400 nm

3.1.1
ultraviolet B
UVB
electromagnetic radiation in the range of 290 nm to 320 nm

3.1.2
ultraviolet A
UVA
electromagnetic radiation in the range of 320 nm to 400 nm

Note 1 to entry: UVA II = 320 nm to 340 nm; UVA I = 340 nm to 400 nm.

3.2
erythema
reddening of the skin caused by UV radiation

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3.3
persistent pigment darkening
PPD
skin darkening that persists more than 2 h after the end of UVA exposure

3.4
sunscreen products
products containing any component able to absorb, reflect or scatter UV rays, which are intended to be placed on the surface of human skin with the purpose of protecting against *erythema* (3.2) and other ultraviolet induced damage

3.5
minimal persistent pigment darkening dose
MPPDD
lowest UVA dose that produces the first perceptible unambiguous persistent pigment darkening response with over more than 50 % of UV exposure subsite, observed between 2 h and 24 h after the end of the UVA exposure

3.5.1
$MPPDD_u$
MPPDD on unprotected skin

3.5.1.1
$MPPDD_{iu}$
MPPDD of an individual subject on unprotected skin

3.5.2
$MPPDD_p$
MPPDD on product protected skin

3.5.2.1
$MPPDD_{ip}$
MPPDD of an individual subject on protected skin

3.6
UVA protection factor
UVAPF
ratio of the minimal PPD dose on product protected skin ($MPPDD_p$) to the minimal PPD dose on unprotected skin ($MPPDD_u$) of the same subject:

$$UVAPF = \frac{MPPDD_p}{MPPDD_u}$$

Note 1 to entry: UVAPF is expressed to one decimal place by truncation.

3.6.1
individual UVA protection factor
$UVAPF_i$
ratio of the individual minimal PPD dose on product protected skin ($MPPDD_{ip}$) to the individual minimal PPD dose on unprotected skin ($MPPDD_{iu}$) of the same subject:

$$UVAPF_i = \frac{MPPDD_{ip}}{MPPDD_{iu}}$$

Note 1 to entry: $UVAPF_i$ is expressed to one decimal place by truncation.

3.6.2
product UVAPF
arithmetic mean of all valid individual $UVAPF_i$ values obtained from all subjects in the test

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3.7
test area
area for testing on the back between the scapula line and the waist

Note 1 to entry: Skeletal protrusions and extreme areas of curvature should be avoided.

3.8
test site
area of the skin where a product is applied, or the site used for the determination of the unprotected MPPDD

3.9
exposure sub-sites
areas of skin that are exposed to UV-irradiation within a *test site* (3.8)

3.10
individual typology angle
ITA°
value characterizing the skin colour of the subject as measured by a skin contact reflectance spectrophotometer or skin colorimeter

Note 1 to entry: Refer to Annex E for the detailed requirements of the equipment/measurement.

4 General 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 PPD responses of the skin as the end point for evaluating transmitted UVA radiation.

The test shall be restricted to the area of the back of selected human subjects.

A section of each subject's skin is exposed to UVA radiation without any protection while another (different) section is exposed after application of the sunscreen product under test. One further section is exposed after application of an UVAPF reference sunscreen formulation, which is used for validation of the procedure.

To determine the UVAPF, incremental series of PPD responses are induced on a number of small sub-sites on the skin. These responses are visually assessed for presence of PPD 2 h to 24 h after UVA radiation, by the judgment of a trained and competent evaluator.

The $MPPDD_{iu}$ and the $MPPDD_{ip}$ shall be determined on the same subject on the same day. An $UVAPF_i$ for each subject tested is calculated as the ratio of $MPPDD_{ip}$ divided by $MPPDD_{iu}$, as in the formula given in 3.6.

The UVAPF is the arithmetic mean of all valid $UVAPF_i$ results from each subject in the test expressed to one decimal place.

5 Test subjects

5.1 Selection of the test subjects

5.1.1 General

There are strict requirements governing the inclusion and non-inclusion of test subjects which should be adhered to. The criteria shall be set out in Annex A.

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5.1.2 Skin colour of the test subjects

Test subjects included in the UVAPF test shall have an ITA° value between 18° and 43° by colorimetric methods (see Annexes A and E) and be untanned on the test area.

A trained and competent scientist or technician should examine each subject to ensure that there is no condition which can put the subject at risk and that the outcome of the test cannot be compromised by adverse skin conditions such as sun damage, pigmentation marks and previous history of abnormal response to the sun (see Annex A).

The test sites intended for UV exposure shall be free from blemishes and hair, and have an even colour tone with no variation in ITA° greater than 5° from each other or the $MPPDD_u$ test area.

5.1.3 Age restriction

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

5.1.4 Frequency of participation in tests

Subjects may participate in a test provided that at least 8 weeks have elapsed since they participated in a previous UV exposure study (i.e. SPF, UVAPF, photoallergy, phototoxicity test), and all skin tanned marks from that previous test have cleared from the test sites on the back and are no longer visible.

5.1.5 Ethics and consent

All testing shall be done in accordance with ethical principles, such as the Declaration of Helsinki[7].

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

5.2 Number of test subjects

The minimum number of valid $UVAPF_i$ results shall be 10 and the maximum number of valid $UVAPF_i$ results shall be 20. In order to achieve between 10 and 20 valid results, a maximum of five individual invalid results may be excluded from the calculation of the mean UVAPF. Consequently, the actual number of test subjects used will fall between a minimum of 10 and a maximum of 25 subjects (i.e. a maximum of 20 valid results plus 5 rejected invalid results). In case a screening had been performed to assess a provisional UVAPF (see A.2.2), the 2 to 3 subjects from this preliminary test can be included among the total test subjects if they comply with all other requirements for a valid test result.

Results may only be declared invalid and excluded from the calculation of the mean UVAPF according to 9.7.5 or because of non-conformity with the related protocol.

In order to determine the number of test subjects, the 95 % confidence interval (95 % CI) on the mean UVAPF shall be taken into account. A minimum of 10 subjects shall be tested. The test shall be considered valid for the first 10 subjects if the resulting range of the 95 % CI of the mean UVAPF shall be within ±17 % of the mean UVAPF. If it is not within ±17 % of the mean UVAPF, the number of subjects shall be increased stepwise from the minimum number of 10 until the 95 % CI statistical criterion is met (up to a maximum of 20 valid results from a maximum of 25 subjects tested). If the statistical criterion has not been met after 20 valid results from a maximum of 25 subjects, then the test shall be rejected. For details on statistical definitions, sequential procedure and calculations, refer to Annex D.

6 Apparatus and materials— Source of ultraviolet radiation

6.1 General

The artificial light source used shall comply with the source spectral specifications as described in 6.2 and Annex B. A xenon arc solar simulator with appropriate filters shall be used.

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6.2 Quality of ultraviolet radiation

6.2.1 The solar UV simulator shall emit a continuous spectrum with no gaps or extreme peaks of emission in the UV region. The output from the solar UV simulator shall be stable, uniform across the whole output beam and suitably filtered to create a spectral quality that complies with the required acceptance limits (see Table 1).

6.2.2 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 and 1 500 nm (visible light and infrared). 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 (see Table 1).

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

6.3 Total irradiance (UV, visible and near infrared rays)

If total irradiance is too intense, an excessive feeling of heat or pain may be induced in the irradiated skin of subjects and heat induced erythema may result. Therefore, total irradiance shall not exceed 1 600 W/m^2[8]. When total irradiance is <1 600 W/m^2, it shall still be confirmed, prior to conducting an UVAPF test, that the irradiance to be used (UV, visible and near-infrared rays) will not induce an excessive feeling of heat in the skin. The output of the solar simulator shall be measured with a broad spectrum sensor (capable of measuring between 280 nm and 1 600 nm) calibrated against a standard reference source over the range of 280 nm to 1 600 nm. Alternatively, the source may be measured with a calibrated spectroradiometer over this same wavelength range to determine the total irradiance.

6.4 Uniformity of beam

6.4.1 General

Uniformity of the beam shall be measured periodically depending on the solar simulator type using either UV sensitive film or UV sensor methods (see 6.4.2 and 6.4.3). Solar simulators with large beams (>1,3 cm diameter) or with multiple output ports shall be measured at least every 6 months, or when any modifications are made to the lamp optical components, or when non-uniform PPD spots are seen in test subsites. Solar simulators with a single output port beam (≤1,3 cm diameter) shall be measured at least every 1 month, or when any modifications are made to the lamp optical components, or when non-uniform PPD spots are seen in test subsites.

Uniformity measurements may be conducted using UV sensitive paper that darkens with exposure, or by using a UV sensor that is smaller in active area compared to the beam size by a ratio of at least 1:4.8 with sufficient measurements to cover more than 75 % of the beam area.

Measurements are to be made using the orientation of the source output as used for subject exposures.

6.4.2 Film densitometry

Exposure doses of the UV sensitive film shall be calibrated to achieve film darkening (converted to grey scale) to a density in the mid-range of the scale (on a 0 to 255 range of black to white). A series

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of exposures shall be used to determine the mid-range density exposure using a calibrated scanning measurement device with at least 600 dots per inch (dpi) resolution. Exposures can be modified by use of neutral density filters or exposure times to achieve this level of exposure for uniformity measurements. Areas to be measured shall be the same as those diagrammed below (see Figures 1 and 2). Films are to be scanned for density values, and average values for each area of the beam as outlined above shall be calculated, and beam uniformity calculated as per Formula (1) (see 6.4.4).

6.4.3 UV sensor

Alternatively, a small aperture (quadrant) UV sensor with a mechanical alignment fixture may be used to measure sub-sections of the output beam intensity as outlined below and the beam uniformity calculated as per Formula (1) (see 6.4.4).

6.4.4 Large beam source

When a large-beam UV source is used to simultaneously expose several subsites (i.e. at least two subsites) within an irradiation series by varying the exposure time, the intensity of the beam shall be as uniform as possible. A UV film densitometry method or a UV radiometer method may be used. The minimum number of sample sites of equal area within the beam [Area of Interest (AOI)] to be assessed shall be determined by dividing the area of the beam by 6,45. For example, if the beam is 232 cm^2 in area, then the minimum number of measurements shall be 36.

UV film densitometry method: The UV sensitive film at least as large as the beam shall be exposed by the entire beam so that the entire beam fits inside the borders of the film.

The uniformity shall be ≥90 % as calculated by the Formula (1):

$$U = (1-(max-min)/(\bar{X})) \quad (1)$$

where

U is the uniformity in percentage;

$\bar{X}$ is the average.

If the uniformity is less than 90 %, then optical components should be adjusted or appropriate compensation for different irradiance shall be made in the exposure time on each sub-site.

6.4.5 Small beam source

6.4.5.1 General

For a small beam UV source, which exposes sub-sites individually, the beam intensity uniformity shall be as measured. A UV Sensitive film densitometry method or a UV radiometer method may be used.

6.4.5.2 Single output device

For a single port device, five equal size areas of the beam intensity shall be measured to assess the uniformity within the beam as shown in Figure 1. The uniformity shall be ≥90 % as calculated by Formula (1).

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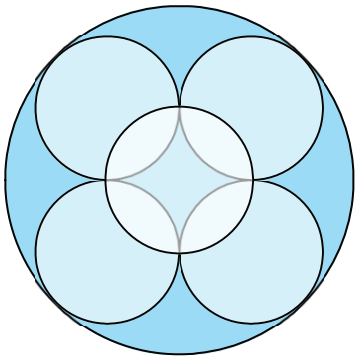

Figure 1 — Single output device

6.4.5.3 Multiple output device

For a multiple port device, the intensity uniformity of each output beam shall be determined by measuring at least 4 circles of equal area of each output beam (see Figure 2), as calculated by Formula (1).

The average uniformity of all beams for the multiport device shall be ≥90 %, with no individual port having uniformity of <85 %.

If the uniformity is less than prescribed, then adjustments to the lamp optical system shall be made to bring the uniformity within the limits above.

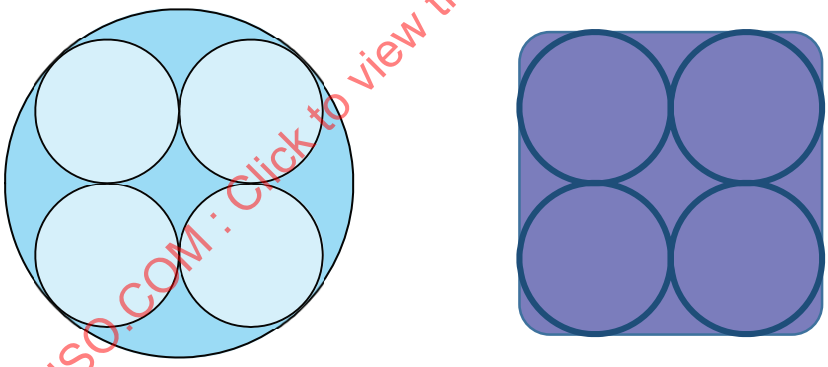

Figure 2 — Multiple output device

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7 Maintenance and monitoring the UV solar simulator output

7.1 Spectroradiometry

There shall be a spectroradiometric check of the spectrum of each solar simulator output port (UVA and UVB) and intensity made by the laboratory at least once every 12 months or after 2 500 h of lamp running time and after changing any significant physical (optical) component (including the bulb) of the solar simulator. The simple use of specific filters is not in itself adequate assurance that the UV output is of the correct quality. This periodical inspection should be conducted by a trained, competent, and suitably qualified person (internal or external) using a spectroradiometer that has been calibrated against a standard lamp that is traceable to a national or an international calibration standard, with a band width of 2 nm or smaller and having a dynamic range of at least 5 decades which is usually met by spectroradiometers equipped with double monochromator. Measurements shall be recorded at 1 nm increments.

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Optical alignment fixtures shall be used to assure accurate radiometer alignment and reproduction of the simulator output at the same optical reference plane measured with the spectroradiometer.

Detailed instructions for ensuring correct lamp output are given in Annex B.

7.2 Radiometry

Prior to making any measurements of the simulator output with a radiometric device, the front surface of the radiometer sensor shall be cleaned with a dry cotton cloth, and the optical tips of the light guides from the xenon source shall be cleaned with alcohol or optical cleaning fluid with lint-free cloth to remove any visible or invisible materials or residual sunscreen.

Before UV exposure of each test site, the UV irradiance shall be measured and recorded with an UVA weighted radiometer cross-calibrated against a spectroradiometric measurement of the solar simulator output as detailed in 7.1. Optical alignment shall be configured to ensure accurate radiometer alignment and reproduction of the simulator output at the same optical reference plane measured with the spectroradiometer. A calibration factor Y for each radiometer shall be determined by Formula (2):

$$Y = \frac{P_s}{P_r} \tag{2}$$

where

Y is the calibration factor for each radiometer;

P_s is UVA irradiance (W/cm^2) of the solar simulator as measured by the spectroradiometer;

P_r is UVA irradiance (W/cm^2) of the solar simulator as measured by the radiometer.

The UV exposure time (in seconds) for a given test shall be calculated using Formula (3):

$$t = \frac{H}{P_s} = \frac{H}{Y * P_r} \tag{3}$$

where

t is the time, in seconds, for the UV exposures for a given test;

H is the desired dose (J/cm^2).

Output intensity should be measured before exposure of each test site in order to ensure the correct intensity is applied for each exposure. Where the solar simulator is capable of continuous monitoring of output intensity, it should be measured during the exposure of the test subjects. The average intensity of the solar simulator as measured by the calibrated radiometer shall be included on the test study report (W/cm^2), as well as the doses (J/cm^2) for the $MPPDD_{iu}$, and $MPPDD_{ip}$ for each subject.

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8 Reference sunscreen formulations

8.1 General

The method is controlled by the use of one of five reference sunscreen formulations to verify the test procedure. Therefore, one of the prescribed reference formulations shall be measured on the same day as products are tested except for P8. Whether a low or high UVAPF reference formulation is to be used depending on the expected UVAPF of the test products. In case of using P8, the reference sunscreen may be measured on the same subject either one day prior or after instead of the same day as products are tested.

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8.2 Reference standard to be used

8.2.1 Preliminary testing: When testing is being done on a preliminary basis, such as for product development investigations, any reference standard listed in Annex C may be used for each subject.

8.2.2 Establishment of UVAPF for product claim: When testing is conducted for the purpose of supporting a label claim of a product intended for market, the following reference standards shall be used for testing with the test product not more than two different reference sunscreen standards in total.

— 4 > UVAPF: S1, S2, P2, P5 or P8;

— 8 > UVAPF ≥ 4: S1, S2, P5 or P8;

— 20 > UVAPF ≥ 8: S2, P5 or P8 reference standard (on at least 5 subjects) and S1 or P2 on the remaining subjects.

— UVAPF ≥ 20: P8 reference standard (on at least 5 subjects) and one of the lower reference standards on the remaining subjects.

Additional subjects may be added as necessary to achieve means for the reference standards that are within the acceptance range.

Assignment of the reference standards to be used on specific subjects shall be randomized.

If S2, P5 or P8 reference standard is used, there is no necessity to also include lower UVAPF reference standard in the test even though there may be lower UVAPF test products. Only one UVAPF reference standard is required on each test subject. However, in case of using P2 or S1, the test should be invalid if mean UVAPF of the test sample exceeds 4 or 8, respectively. Also, if resulted mean UVAPF exceeds 20 under the use of reference standard without using P8 on at least 5 subjects, obtained UVAPF shall be invalid.

Acceptance UVAPF ranges for the reference sunscreens are shown in Annex C. If the mean UVAPF of the reference standard obtained in any test do not fall within their acceptance limits shown in Annex C for that reference standard, then the entire test (i.e. all test products) shall be rejected.

The formulae details and manufacturing instructions for the reference formulations are given in Annex C.

9 Procedure

9.1 Main steps

a) Acclimatization period for the skin.

b) Determination of ITA° on the back of the subject.

c) Delineation of test sites on the back of the subject.

d) Weighing of the product for application to the test site.

e) Application of the product to the test site.

f) Waiting period (15 min to 30 min) before UV exposure to the test site.

g) UV exposure.

h) Waiting period (2 h to 24 h) before MPPDD assessment.

i) MPPDD assessment.

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j) Calculations.

9.2 Test conditions

Product application, UV exposures and MPPDD assessment should be carried out in stable conditions, with the room temperature maintained between (23 ± 3) °C.

9.3 Position of the test subjects

The product shall be applied to subjects in the same position as will be utilized for the irradiation procedure (sitting or prone). Powder and products which may flow (very low viscosity liquids) should be tested in the prone position to prevent the samples from falling off the surface.

9.4 Product application

9.4.1 Overview

The amount of product applied and the uniformity of spreading on the test sites affect the magnitude and variability of the test results. It is therefore very important to follow the recommendations set out in 9.4.2 to 9.4.5.

9.4.2 General

The test sites intended for UV exposure shall be free from blemishes and hair, and have an even colour tone with no variation in ITA° greater than 5° from each other or the $MPPDD_u$ test area. When necessary, hair shall be shaved more than three days prior to the test, but not thereafter. If necessary, hair may be clipped or cut with scissors on the test day.

The minimum total area for a test site for product application shall be 30 cm^2 and the maximum shall be 60 cm^2.

The positions of the test products and reference sunscreen test sites shall be distributed randomly on the backs of subjects over the whole test group in order to reduce error arising from anatomical differences in skin. The unprotected test site used to determine $MPPDD_u$ shall be randomized as one of the test sites across the test area and across subjects.

There shall be a minimum distance of 1 cm between the borders of adjacent test sites.

Before product application, the test area may be cleaned by using a dry cotton pad or equivalent.

The test sites shall be delineated by a method which does not interfere with the test or harm the subject such as skin marker and/or a template made from non-absorbent material. The skin marker shall be indelible so as to be discernible at the time of MPPDD evaluations 2 h to 24 h post UV-exposure.

9.4.3 Amount of product applied

The amount of test product and reference sunscreen formulation applied to the skin after spreading shall be (2,00 ± 0,05) mg/cm^2.

The balance used to weigh the products should be capable of weighing to the nearest 0,1 mg.

All products shall be shaken to be homogeneous before weighing, to ensure uniform dispersion.

When handling the product during weighing or before application to the skin, take appropriate measures to prevent evaporative loss of the volatile components. It is important that the total quantity of weighed product is transferred to the product application site. The amount of product to be applied shall be weighed in a syringe or in another device such as a watch glass. A method of weighing by loss is required.

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9.4.4 Mode of delivery

9.4.4.1 General

It is recommended to practice application and spreading of the test materials on a subject (not in the test) to determine the best procedure to obtain uniform product spreading. Once the best practice is determined, it should be unchanged for all the subjects during the test. The use of a finger cot (i.e. latex, nitrile, etc.) shall be required except in cases when use of a finger cot interferes with even application of the product. A new finger cot shall be used for each new application of product and shall not be pre-saturated with the test product. When a naked finger is used a maximum of 2,1 mg/cm^2 (additional 5 %) shall be applied to the test area to account for the additional area of the application finger, and the finger shall be cleaned between product applications with an alcohol wipe.

9.4.4.2 Product application technique

The application technique to be used is dependent on the product type.

Table 2 — Recommended application method by dosage form

Form	Recommended application method
Lotion	Method A
Cream	Method A
Oil	Method A
Liquid	Method A
Gel	Method A
Stick	Method B
Balm	Method B
Aerosol spray	Degas then Method A
Pump spray	Method A
Roll on	Method A or B
Powder	Method C
Foaming formulations	Method D

Method A: Fluid products

To aid uniform coverage, droplets (at least 15 per 30 cm^2, 30 per 60 cm^2) of the product should be deposited within the test site using a syringe/pipette at one time, then spread over the whole test site, first with circular movements to gather the droplets and second in horizontal and vertical directions using light pressure as shown in Figure 3. It is recommended that during the whole process, the application finger stays in contact with the skin.

Spray products provided in a pressurized container shall first be degassed by puncturing a very small pinhole in the container to relieve all of the pressure. Degassing shall be done with appropriate safety precautions by securing the can within a ventilated safety hood with appropriate personnel safety equipment. The degassed can shall be allowed to rest for 24 h at room temperature when the product shall be decanted into a separate closed container with minimal headspace to minimize evaporation.

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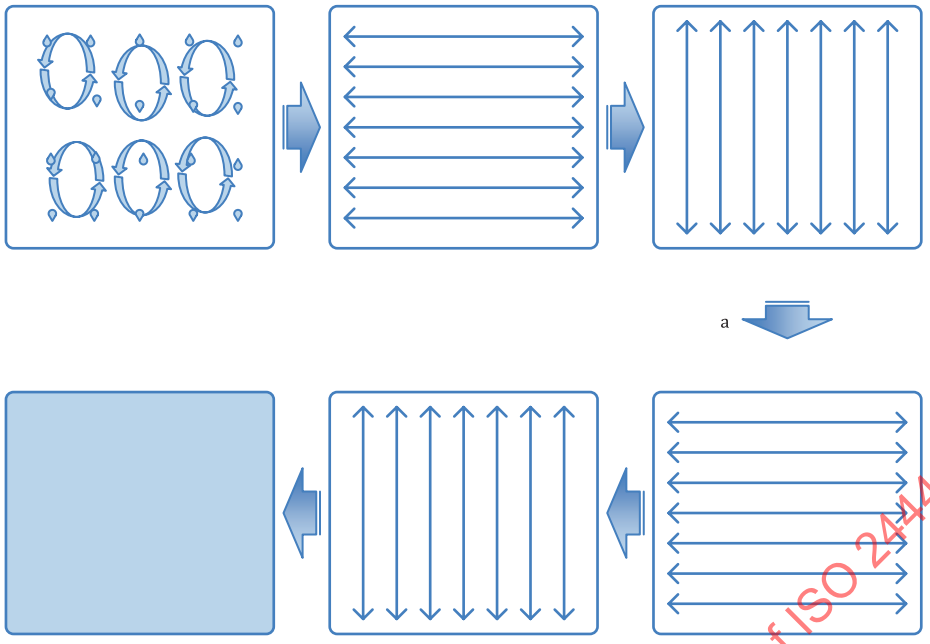

a Optional.

Figure 3 — Application techniques for Methods A and B

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Method B: Non-flowing viscous liquids and semi-solids

Test product should be measured into a weigh boat and applied by finger in multiple areas of the test site, first with circular movements to gather the material and second in horizontal and vertical directions using light pressure as in Figure 3. It is recommended that during the whole process, the application finger stays in contact with the skin.

Method C: Powders

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

The accumulated powder shall be tapped and then spread over the whole test site using a finger with or without a finger cot. Alternatively, the tip of a pre-loaded cosmetic applicator puff may be used instead of a finger. In this case, it is important to verify that (2,00 ± 0,05) mg/cm^2 of test powder product remains on the skin after spreading, by weighing the powder remaining on the tip of the applicator puff.

Purified water or another suitable solvent that has no UV protection properties may be applied on the skin before the powder application to help the sample adhere to the application site. Water or solvent should not transform the powder into a paste and thus influence its UVAPF value.

Method D: Foaming formulations

For samples which are presented as foams and where the contents cannot be extracted or dispensed other than as a foam, the test product should be measured into a weighing boat and then the sample allowed to degas or deaerate until they can be easily applied to the skin. Application will be subsequently accomplished following Method B.

9.4.4.3 Spreading

Spreading time should be in the range of (35 ± 15) s depending on the surface and ease of spreading of the product. Volatile liquids should be spread without delay.

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9.4.5 Evaluation of application uniformity

After application is completed, and before commencement of the UV exposure doses, the application shall be checked with an ultraviolet-A "Woods" lamp with at least 6 W of power, that is capable to visualize the uniformity of the application. If noticeable non-uniformity or streaking of the product is noted, the test site shall be rejected and may not be used for the test. If another test site is available, a new application may be attempted.

9.4.6 Drying time between application and UV exposure

Exposure of the first test site to the sequence of UV doses shall start 15 min to 30 min after the product is 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.

9.4.7 Exposure sub-sites

Where a template is used to demarcate the exposure sub-sites (such as large-beam UV solar simulator), the template should be of non-absorbent material.

The minimum area of each exposure sub-site shall be 0,5 cm^2.

The minimum distance between borders of each exposure sub-site (spots) shall be at least 0,8 cm.

The distance between any exposure sub-site and any edge of the test site shall be at least 1 cm.

The minimum number of exposure sub-sites used shall be five for unprotected MPPDD ($MPPDD_u$) and five for protected MPPDD ($MPPDD_p$).

9.5 UV exposure

9.5.1 Provisional $MPPDD_{iu}$

Before starting the main test, it may be necessary to determine a provisional $MPPDD_{iu}$ in order to centre the UV dose ranges for the exposures of $MPPDD_{iu}$ and $MPDD_{ip}$. A provisional $MPPDD_{iu}$ is a pre-test in which the $MPPDD_{iu}$ of a subject should be determined prior to establishing the test. This is performed by applying a preliminary series of UV exposures up to one week before the test. Maximum total duration of a test should not exceed 3 months. The provisional $MPPDD_{iu}$ may be determined by using the UV dose range starting 8 J/cm^2 up to 24,4 J/cm^2. The maximum dose shall not initially exceed 25 J/cm^2 in order to protect a subject from overexposure to UV light.

9.5.2 Estimated $MPPDD_{iu}$

If the provisional $MPPDD_{iu}$ has not been determined before the product test day, the $MPPDD_{iu}$ from previous record may be used for the estimated $MPPDD_{iu}$ for the measurement. However, since MPPDD is not correlated with ITA° unlike MED, colorimetric technique (ITA°) without UV exposure shall not be applied for UVAPF measurement.

For each subject, the unprotected $MPPDD_{iu}$ shall be determined on the same day as the test product protected $MPPDD_{ip}$.

9.5.3 Incremental progression of UV dose

9.5.3.1 For the unprotected site, the range of UV doses applied should be predicted using the provisional $MPPDD_{iu}$ or the subject's previous record of $MPPDD_{iu}$. Provisional $MPPDD_{iu}$ shall be obtained if information regarding subject's response to UVA is not available. A minimum of five sub-sites centred on or close to the provisional/estimated $MPPDD_{iu}$ shall be exposed with incremental UV doses using a recommended geometric progression of 1,25 ×. Other geometric progressions of less than 1,25× may be used (such as 1,2; 1,15; 1,12) but shall be consistent throughout the test (same

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progression used for unprotected and protected sites). Exposure times may be rounded to the closest integer seconds. Determine the exposure time needed to achieve the range of doses to be typically used for the determination of the $MPPDD_u$: 8 J/cm^2, 10 J/cm^2, 12,5 J/cm^2, 15,6 J/cm^2, 19,5 J/cm^2, and 24,4 J/cm^2 (depending on the radiometer used). In case of UVA irradiance of 50 mW/cm^2, exposure time for $MPPDD_u$ of 12,5 J/cm^2 is 250 s. Other (higher or lower) exposure doses and times may be based on prior data of an individual subject.

9.5.3.2 For the product protected sites, the UV doses delivered are defined by the expected $MPPDD_{ip}$, which is the multiple of the expected UVAPF of the test product (as determined by the test sponsor or previous data) and the provisional or estimated $MPPDD_{iu}$ for the subject. A minimum of five sub-sites centred on or close to the expected $MPPDD_{ip}$ shall be exposed with incremental UV doses using a recommended geometric progression of 1,25×. Other geometric progressions may be used (such as 1,2, 1,15 or 1,12). Smaller geometric progressions (such as 1,12) may be used but shall also be consistent throughout the exposure procedure (same progression used for unprotected and protected sites).

The expected value of the UVAPF may be changed during the testing of the product between test subjects as requested by the test sponsor or the laboratory management to prevent test failures or overexposure of subjects.

9.6 Product removal

After UV exposures, reference and test products may be gently removed using an appropriate means.

9.7 Procedure for MPPDD assessment

9.7.1 General

The $MPPDD_{iu}$, the $MPPDD_{ip}$ for test product and the $MPPDD_{ip}$ for the reference sunscreen formulation shall all be determined on the same day.

9.7.2 Time of assessment of MPPDD

The MPPDD(s) shall be assessed 2 h to 24 h after UV exposure as measured from the end of the last exposure period. During the time interval between UV exposure and MPPDD assessment, the subject shall avoid any extra UV exposure (artificial UV light or sunlight) to the exposed area. Any additional UV exposure (natural or artificial) to the test area of an individual will invalidate the data from that individual and that data shall be rejected from the test results and not count against the total allowable rejected subjects. 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.

The MPPDDs shall be assessed visually. The observer's eyesight should have been checked for normal colour vision. A yearly check of acuity of vision is recommended.

Visual assessment should be performed in sufficient and uniform illumination. At least 450 lux in the plane parallel with the back of the test subject is required using a lamp with a colour temperature of 6 500°K.

The determination of MPPDD(s) shall be carried out in a room with matt, neutral wall colours.

PPD responses shall be observed in a “blind” manner. The observers of PPD responses on any subjects shall not be the same persons as the ones who performed product application and exposure. The observers shall be not aware of the test design (randomization of test sites) on that subject.

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9.7.3 Grading scale for the $MPPDD_i$s

Unprotected UV exposed sites and protected UV exposed sites should be graded with the same reference scale and same visual references as shown in Annex F:

— 0: no PPD present;

— 0,5: ambiguous PPD, and/or no clear border, and/or not filling more than 50 % of the exposure subsite;

— 1: Perceptible unambiguous PPD with defined borders filling more than 50 % of the exposure subsite (MPPDD if it is the lowest exposure dose with grade 1);

— 2: Moderate to intense PPD.

9.7.4 Erythema responses

The responses observed at the exposure sites may be pink/red (erythema), grey/brown (pigmentation) or mixture of both. If the exposure site has pink/red colouration, the surface of the skin shall be lightly pressed with a glass slide to determine if there is erythema present. If erythema is present, there will be a slight blanching of the any redness of the skin with the pressure and colour will return after removal of the pressure. The site shall basically be scored as having unambiguous PPD over more than 50 % of the exposure sub-site. This method evaluates PPD as a representative skin reaction to UVA. In this context, erythema responses (with no PPD) under this condition are also considered as a qualifying response to UVA.

9.7.5 Data rejection criteria

Test data are deemed invalid and shall be rejected under the circumstances given in Table 3.

Table 3 — Data rejection criteria

Observation	$MPPDD_{iu}$	$MPPDD_{ip}$	Reference standard
No grade of at least 1 for any exposed sub sites[a]	Data for subject is rejected Does not count against total allowable rejected number of subjects	Data for test product is rejected Does not count against total allowable rejected number of subjects	Data for subject is rejected Failure counts against allowable rejected number of subjects
All test subsites show PPD of at least grade 1[b]	Data for subject is rejected Does not count against number of total allowable rejections	Data for test product is rejected Counts against number of total allowable rejections	Data for subject is rejected Counts against number of total allowable rejections

Observations definitions:

[a] No Grade of PPD of at least 1 for any exposed sub-site: All exposed sub-sites have grades of 0, or 0,5, and no qualifying MPPDD (Grade 1) is observed.

[b] All test sub-sites show PPD of at least Grade 1: No sites have grades of 0 or 0,5, and a MPPDD response cannot be established.

[c] PPD response(s) is (are) absent for exposures higher than the determined MPPDD (randomly absent): A Grade of 0 is observed at an exposure dose higher than the determined MPPDD, (randomly absent or illogical sequence).

[d] Non-compliance of the subject: Subject does not follow instructions during or after the treatment or UV exposures that could affect the outcome of the test (wipes sunscreen treated areas during application or exposures, medicates with anti-inflammatory drugs, exposes treatment areas to UV light (sunlight or other UV source), irritates treated area, etc.).

[e] Technical failure: Failure of equipment or procedures during the treatment phases of the procedure (for example: incorrect lamp intensity or fluctuations, incorrect exposure times, incorrect site application of sunscreen, and similar reasons) that would jeopardize the integrity of the treatments and conclusions.

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Table 3 *(continued)*

Observation	$MPPDD_{iu}$	$MPPDD_{ip}$	Reference standard
PPD response(s) is (are) absent for exposures higher than the determined MPPDD (randomly absent)[c]	Data for subject is rejected Does not count against number of total allowable rejections	Data for test product is rejected Counts against number of total allowable rejections	Data for subject is rejected Counts against number of total allowable rejections
Non-conformity of the subject[d] OR Technical failure[e]	Data for subject is rejected Does not count against number of total allowable rejections	Data for subject is rejected Does not count against number of total allowable rejections	Data for subject is rejected Does not count against number of total allowable rejections

Observations definitions:

[a] No Grade of PPD of at least 1 for any exposed sub-site: All exposed sub-sites have grades of 0, or 0,5, and no qualifying MPPDD (Grade 1) is observed.

[b] All test sub-sites show PPD of at least Grade 1: No sites have grades of 0 or 0,5, and a MPPDD response cannot be established.

[c] PPD response(s) is (are) absent for exposures higher than the determined MPPDD (randomly absent): A Grade of 0 is observed at an exposure dose higher than the determined MPPDD, (randomly absent or illogical sequence).

[d] Non-compliance of the subject: Subject does not follow instructions during or after the treatment or UV exposures that could affect the outcome of the test (wipes sunscreen treated areas during application or exposures, medicates with anti-inflammatory drugs, exposes treatment areas to UV light (sunlight or other UV source), irritates treated area, etc.).

[e] Technical failure: Failure of equipment or procedures during the treatment phases of the procedure (for example: incorrect lamp intensity or fluctuations, incorrect exposure times, incorrect site application of sunscreen, and similar reasons) that would jeopardize the integrity of the treatments and conclusions.

9.7.6 Test failure criteria

If data have to be rejected for the test product on more than five subjects, then the whole test for that product shall be invalid and shall be rejected.

If data have to be rejected for the Reference Sunscreen on more than five subjects, then the whole test shall be invalid and shall be rejected.

9.7.7 Expression of MPPDDs

MPPDDs shall be expressed in terms of energy J/cm^2 (integers).

All irradiance measurements shall use a radiometer previously cross calibrated against a spectroradiometric measurement weighted with PPD action spectrum, or a spectroradiometer measurement weighted with PPD action spectrum to determine the J/cm^2.

10 Calculation of the UVA protection factor and statistics

10.1 Calculation of the individual UVAPF ($UVAPF_i$)

The $UVAPF_i$ of both the reference sunscreen and the product under test for each subject shall be calculated as shown in Formula (4) and expressed to one decimal place by truncation.

$$UVAPF_i = \frac{MPPDD_{ip}}{MPPDD_{iu}} \tag{4}$$

where

$MPPDD_{ip}$ is the MPPDD of sunscreen protected skin for an individual;

$MPPDD_{iu}$ is the MPPDD of unprotected skin for an individual.

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10.2 Calculation of product UVAPF

The UVAPF result for the test product and for the reference sunscreen formulation shall be calculated as the arithmetic mean of all valid individual UVAPFi values.

The minimum number of valid $UVAPF_i$ values shall be ten and the maximum number of valid $UVAPF_i$ values twenty. A maximum of five results may be excluded from the calculation of the mean UVAPF, but each exclusion shall be justified according to 9.7.5 or if protocol non-conformance has occurred. A sixth invalid result automatically invalidates the whole test for that test product and no UVAPF can be calculated for it.

UVAPF shall be expressed to one decimal place by truncation.

10.3 Statistical criterion

The statistical criterion for the test product UVAPF measurements is that the 95 % confidence interval on the mean UVAPF measured shall comply with the ±17 % CI criteria of the measured mean UVAPF. This only applies test products.

Consequently, the actual number of subjects tested shall be determined as the number (minimum ten) required to produce a mean test product UVAPF with a 95 % CI which falls within a range of ±17 % of the measured mean UVAPF for the tested product.

For the reference sunscreens used on test panelists, the average value for each of the reference sunscreens shall fall within their respective acceptance ranges specified in Annex C. No further statistical requirement is needed for the reference sunscreen.

A minimum of ten valid results is only sufficient if the statistical criterion is fulfilled and the means of the reference sunscreens fall within their respective acceptance ranges. If not, the number of subjects shall be increased from ten until the statistical criteria are met up to a maximum of twenty valid results.

The full statistical procedure for this calculation is described in Annex D.

10.4 Validation of the test

The mean UVAPF of the reference sunscreen formulation used in the test shall fall within the acceptance limits shown in Annex C.

11 Test report

11.1 Overview

The test report shall contain at least the following information.

11.2 General information

a) Identification of the testing laboratory.

b) A reference to this test method.

c) Product identifier and expected UVAPF.

d) Any instructions in accordance with this document given by the sponsor for the application of the product (i.e. fingercot or not, latex or nitrile, pretreatment for powders, etc.).

e) Any specific instructions not in accordance with this document given by the sponsor for the application of the product. For example, special preparation of sample prior to application, such as recombining 2 phase systems.

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f) The geometric progression used for the individual MPPDD.

g) Commencement and ending dates of the test.

h) Identification of the reference sunscreen used and evidence of conformance with the acceptance range for this sunscreen according to the limits described in C.1.

i) A reference to latest calibration and statement of conformity document (date and provider) of solar simulators used in the test.

j) Mean UVAPF value expressed in one decimal place (truncated), standard deviation on the mean, and 95 % CI as a number and as a %, and 17 % of the mean UVAPF.

k) Protocol deviations if any.

11.3 Data in tabular for each test subject

Annex G provides a template for reporting the following data:

a) the subject number in sequence;

b) identification by subject, of the technicians who applied, exposed, and evaluated responses during the test;

c) subject ITA° value;

d) the dates of UV exposure for each subject;

e) identification by code number, of each subject;

f) the intensity of the solar simulator output in W/cm^2. In the case of a multiport device, this should be the value for the highest intensity port;

g) seconds of exposure for the $MPPDD_{iu}$, $MPPDD_{ip}$ for test sample, and $MPPDD_{ip}$ for reference standard;

h) individual MPPDD for unprotected skin, test product protected skin and reference sunscreen protected skin, reported as J/cm^2 (no decimal);

i) individual $UVAPF_i$ values expressed in one decimal place (truncated), including all valid data and rejected data for the test product and for the reference sunscreen;

j) indication if the UVAPF$_i$ value was rejected (Y or N?).

11.4 Statistics for the test products

After completion of at least 10 valid test subjects, calculations and statistics as described in Annex D and at least a statement that the test study complies with the statistical validations:

a) mean UVAPF of the test product truncated to one decimal place;

b) standard deviation (s) calculation;

c) confidence interval (CI) calculation;

d) CI (%);

e) 95 % CI;

f) 17 % of the mean UVAPF calculation.

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Annex A
(normative)

Selection criteria for the test subjects

A.1 Rationale

Experience has shown that utilization of skin phototyping is problematic, and unable to adequately distinguish appropriate subjects for UVAPF testing and for estimating skin sensitivity to PPD. Although the ITA° value does not predict $MPPDD_u$ value correctly, it has been successfully used for screening test subjects for UVAPF testing for many years by many different laboratories. Their experience has been utilized to establish the limits of useful range of ITA° values. This document references only the use of ITA° values for qualifying subjects for the UVAPF test.

A.2 Selection criteria for the test subjects

A.2.1 Skin colour

Subjects shall be selected using the colorimetric ITA° value. The skin of subjects shall have a colorimetric ITA° range of subjects of between 18° and 43°.

Colorimetric ITA° values and skin colour categories are defined by the colorimetric descriptors of Chardon et al[10]. using the CIE (1976) $L^*a^*b^*$ colour space.

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

where

$$ITA° = \{\text{arc tangent } [(L^* - 50)/b^*]\}180/3{,}141\ 6.$$

A.2.2 Medical and ethical considerations

Subjects should be adequately informed of the aims and potential risk (direct or secondary effects) of the study and any discomfort they may experience. Each subject shall give a written agreement to participate in UVAPF tests.

It is recommended that new subjects first be interviewed by a health professional to establish their medical status and suitability prior to inclusion in the subject panel.

Subjects should be checked visually by a trained and competent scientist or technician before participating in a study. Their skin colour shall be uniform over the whole test area without pigmentation, nevi, or the like and no sunburn (erythema) shall be present on the test area.

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When there is some doubt on the provisional UVAPF value of the test product, a screening should first be performed. In order to protect the subjects a lower UVAPF value should be used on two or three subjects and increased progressively on the other subjects. Data from these tests may be included in the final results provided they comply with all other requirements for a valid test result.

UVAPF measurements should be designed to minimize any harmful, long-lasting effects on human test subjects. Tests shall be performed by trained and competent personnel in order to avoid any damage to the skin of the test subjects involved in the test.

A.2.3 Non-inclusion criteria

All non-inclusion criteria shall be checked before testing.

The following conditions shall automatically disallow inclusion of a subject in the test group:

a) children and persons below the locally legal age of consent or >70 years;

b) pregnant or lactating women;

c) subjects using medication with photo-sensitizing potential;

d) subjects using anti-inflammatory medication;

e) subjects with systemic dermatological conditions (including dysplastic nevi);

f) subjects with a history of abnormal response to the sun or a history of skin cancer;

g) subjects who have used tanning beds in the previous eight weeks prior to UVAPF testing;

h) subjects having had sun exposure on the back area in the previous eight weeks prior to UVAPF testing;

i) subjects having marks, blemishes or nevi in the test area;

j) subjects presenting with existing sun damage in the test area;

k) subjects having excessive hair in the area on the test on the day of testing (hair may be shaved up to 3 days prior to the test day, or may be clipped or cut with scissors on the test day.);

l) subjects having skeletal protrusions and extreme areas of curvature in the test area.

A.2.4 Frequency of subject participation (interval between two tests)

Subjects may participate in a test provided that at least 8 weeks have elapsed since they participated in a previous UV exposure study (i.e. SPF, UVAPF, photoallergy, phototoxicity test), and all skin tanned marks from that previous test have cleared from the test sites on the back and are no longer visible.

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Annex B
(normative)

Definition of the source of UVA radiation

B.1 General

The aim of these specifications is to define practical criteria for testing the spectral conformity of the source of UVA radiation used for UVAPF testing. The artificial light source used shall be in accordance with the source spectral specifications as described in this annex.

B.2 Rationale for specifications

B.2.1 Sun UV spectra and UV range

UV rays in natural sunlight covers 290 nm to 400 nm of UV range and are responsible for most of the sun's damaging effects on skin. Measured solar spectra have been published taking into account different geographical latitudes and altitudes, and variations due to year, season, time of day and ozone content. However, as the purpose of this method is to evaluate the effect of UVA on the skin, the definition of the spectrum of the UV solar simulator is limited to the UVA-wavelengths, i.e. from 320 nm to 400 nm.

Wavelengths below this range (<320 nm) should be excluded in order to avoid any effects caused by UVB, whilst those above this range (>400 nm) may cause undesirable side effects (particularly thermal effects) and should be removed using appropriate devices.

B.2.2 PPD induction wavelengths

The PPD induced by sunlight UV appears after relatively short time upon receiving sunlight. The reaction is considered at least partially by way of polymerization of melanin monomers[2]. The action spectrum for PPD is not limited to UVA range, however inclusion of UVB hinders PPD by stronger reaction of erythema. The purpose of this evaluation method is to estimate protection efficacy against various UVA damages other than PPD by way of measuring PPD reaction as a representative. For this reason, UVB shall be removed from the light source by appropriate filters.

B.2.3 Solar simulator and filtration

A lamp that produces a continuous spectrum can readily be adapted to fulfil the UVA acceptance limits for the output between 320 nm and 400 nm by using specific optical filters. To ensure uniformity in spectral shape in UVAPF testing, UV solar simulators utilizing a xenon arc lamp, shall be filtered with a dichroic UV filter to minimize IR radiation, and UV shaping filters such as WG335 and UG11 or equivalent filters.

The simple use of the recommended filters is not, in itself, an adequate assurance that the UV output is of the correct quality and so the spectral output shall be confirmed by spectroradiometric measurement.

B.2.4 UVA radiation specification

The limits prescribed for UVA are shown in Table 1 in 6.2.2. The light source shall be monitored periodically in B.3.1.1.

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B.3 Mode of operation

B.3.1 Quality of the UV solar simulator output

B.3.1.1 Spectroradiometric measurements

The output spectrum of the UV solar simulator, including all filters and optical components, shall be measured with a spectroradiometer or spectrograph. The spectroradiometer should be fitted with a double monochromator and its bandwidth should be ≤ 2 nm (1 nm is recommended) in order to ensure that all energies are represented in an amplitude range of at least 5 decades. Measurements shall be made in steps not exceeding the bandwidth.

The instrument shall have been calibrated against standard light sources for its response to spectral irradiances, for its wavelength accuracy (for example mercury lamp) and for linearity of signal responses at all wavelengths over an irradiance range covering the actual source measurement range.

The units of source irradiance should be in actual spectral energy ($W/m^2 \cdot nm$, $mW/cm^2 \cdot nm$).

B.3.1.2 Radiometric measurements

The UV irradiance of the solar simulator is controlled with a radiometer that has been previously cross-calibrated for this source spectrum against the spectroradiometric measurement (see B.3.1.1).

A UV dose is the result of multiplying the UV source irradiance by the exposure duration. When a large-beam UV solar simulator is used, allowing simultaneous exposure of several sub-sites by varying the exposure time, the uniformity in beam irradiance should be as high as possible. This uniformity can be measured with the radiometer. The range of irradiance variation over all the exposure sub-sites should be less than 10 %. If the variation exceeds 10 %, then appropriate compensation for different irradiance levels should be made in the exposure time on each sub-site. Solar simulators with light guides or multiple small beams, exposing all sub-sites for the same duration but with varied irradiance values should be checked to ensure that each beam or guide generates uniform PPD responses.

A warm-up time of at least 20 min shall be allowed for the UV solar simulator to stabilize before starting exposures. This is to ensure a consistent irradiance over the whole exposure period.

B.3.2 Evaluating conformity

For each reference waveband, the UVA radiation of the source shall comply with those specified in Table 1. All values shall lie within the acceptance limits. If the UV solar simulator spectrum is outside the limits in any of the wavebands, then the filtration should be adjusted to comply with the spectral output specifications.

In addition, the solar simulator spectrum shall include less than 0,1 % of total UV below 320 nm and, to ensure that the solar simulator contains the correct balance of UVA I:UVA II, the output from the lamp system should contain 80 % to 92 % of UVA I (340 nm to 400 nm) and ≥8 % to 20 % of UVA II (320 nm to 340 nm).

The total irradiance of the source shall be measured.

B.3.3 Adjusting UV solar simulator output

If the output spectrum of the UV solar simulator needs to be adjusted to fit the acceptance specifications, this may be achieved either by checking the xenon lamp's elapsed life and replacing it if necessary, or by adapting the spectral shaping filters within the UV solar simulator, particularly the thickness of the short cut-off filter.

If the total irradiance of the UV solar simulator exceeds 1 600 W/m^2, the irradiance can usually be reduced by lowering the electrical current supplying the xenon lamp, provided that the current remains

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in the normal operational stability range. If total irradiance is adjusted in this way, then the quality of the emission spectrum should be checked again to ensure that the acceptance specifications are met.

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Annex C
(normative)

UVAPF reference sunscreens formulations

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	Applicable range of UVAPF	Mean UVAPF	Acceptance limits	
			Lower limit	Upper limit
S1	<8	4,4	3,8	5,0
S2	<20	12,7	10,7	14,7
P2	<4	2,7	2,3	3,2
P5	<20	13,4	11,3	15,3
P8	Whole range	27,5	23,4	31,7

C.2 S1 UVAPF Reference standard

C.2.1 General

Table C.2 gives the UVAPF reference formula S1.

Table C.2 — UVAPF reference formula S1

Ingredients	% mass of total mass
Phase 1	
Purified water	57,13
Dipropylene glycol	5,0
Potassium hydroxide	0,12
Trisodium EDTA	0,05
Phenoxyethanol	0,3
Phase 2	
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

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C.2.2 Manufacturing process

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

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

c) Combine phases by adding the phase 2 materials to the phase 1 materials and mix until uniform.

d) Homogenize at 4 000 r/min for between 3 min and 5 min.

e) Continue mixing with an overhead stirrer as the batch cools.

C.2.3 Physicochemical data

a) Appearance: white cream.

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

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

d) Density (20 °C): 0,95 g/cm3 ± 0,05 g/cm^3.

C.2.4 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.2.5 Analytical data

C.2.5.1 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 [3].

C.2.5.2 Chemical reagents

a) Absolute ethanol (HPLC grade).

b) Ultrapure water (HPLC grade).

c) Phosphoric acid, 85 % analytical purity.

d) Ethylhexyl methoxycinnamate.

e) Butyl methoxydibenzoylmethane.

C.2.5.3 High-performance liquid chromatography apparatus

a) Injector, with an injection volume of 10,0 µl.

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.

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b) Column, for example 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) Eluent A, ultrapure water acidified with H_3PO_4 (67 µl/l).

d) Eluent B, absolute ethanol (HPLC grade).

e) Detector, of type UV wavelength, 312 nm.

f) Data, quantification of peak area.

C.2.5.4 Method

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

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

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

d) Dilute to volume with ethanol.

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

f) Filter through a 0,45 µm PVDF disc filter.

g) Analyse the standard and mixed working standard by reverse-phase HPLC.

C.2.5.5 Quality control

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

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

C.2.5.6 Calculations

Analyte percentage is calculated using Formula (C.1):

$$\frac{M \times h \times 2,5}{P \times H} \tag{C.1}$$

where

M is the weight in mg of analyte;

P is the weight in mg of sample;

h is the area of analyte peak for sample;

H is the area of analyte peak for standardization.

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.

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C.2.5.7 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.3 S2 UVAPF Reference standard[9]

C.3.1 General

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	
Propylene glycol	1,00
Xanthan gum	0,60
Carbomer	0,15
Disodium EDTA	0,08
Phase 2	
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)	Appropriate amount
Ethylparaben (and)	Appropriate amount
Butylparaben (and)	Appropriate amount
Propylparaben	Appropriate amount
Phase 3	
Cyclopentasiloxane	2,00
Triethanolamine	0,225

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C.3.2 Manufacturing process

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

2) Add phase 2 materials slowly to phase 1 materials while stirring phase 1.

3) Cool to 40 °C while stirring.

4) Add phase 3 materials to phase 1 and 2 materials while stirring.

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5) Compensate water loss and homogenize.

C.3.3 Physicochemical data

a) Colour: white to slightly yellow.

b) pH: 6,5 ± 0,5

c) Density: 0,96 g/cm^3 to 1 g/cm^3.

d) 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.3.4 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.3.5 Analytical data

C.3.5.1 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 [9].

C.3.5.2 Chemical reagents

a) Absolute ethanol (HPLC grade).

b) Phosphoric acid, 85 % analytical purity.

c) Ethylhexyl methoxycinnamate.

d) Butyl methoxydibenzoylmethane.

e) Octocrylene.

f) Bis-ethylhexyloxyphenol-methoxyphenyl triazine.

g) 1,4-Dioxane (HPLC Grade).

C.3.5.3 High-performance liquid chromatography apparatus

a) Injector, with an injection volume of 10,0 µl.

b) Column, for example 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) Eluent A, ultrapure water acidified with H_3PO_4 (67 µl/l).

d) Eluent B, absolute ethanol (HPLC grade).

e) Detector, of type UV wavelength, 312 nm.

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.

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f) Data, quantification of peak area.

C.3.5.4 Method

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

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

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

d) Dilute to volume with ethanol.

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

f) Filter through a 0,45 µm PVDF disc filter.

g) Analyse the sample and mixed working standard by reverse-phase HPLC.

C.3.5.5 Quality control

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

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

C.3.5.6 Calculations

The analyte percentage is calculated using Formula (C.1).

C.3.5.7 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 chromatography peaks in the sample placebo or working solvent.

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C.4 P2 UVAPF Reference standard

C.4.1 General

Table C.4 gives the UVAPF reference formula P2.

Table C.4 — Formula for standard formulation P2

Ingredients	% mass of total mass
Phase 1	
lanolin	4,5
theobroma cacao (cocoa) seed butter	2,0

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Table C.4 *(continued)*

Ingredients	% mass of total mass
glyceryl monostearate SE	3,0
stearic acid	2,0
ethylhexyldimethyl PABA (CAS 21245-02-3) (2-ethylhexyl-4-(dimethylamino)-benzoate)	7,0
benzophenone-3 (CAS 131-57-7)	3,0
Phase 2	
water	71,6
sorbitol (liquid 70 %)	5,0
triethanolamine (99 %)	1,0
methylparaben	0,3
propylparaben	0,1
Phase 3	
benzyl alcohol	0,5

C.4.2 Manufacturing process

1) Melt the ingredients of Phase 1 and mix using a propeller agitator at 77 °C to 82 °C until uniform.

2) Mix Phase 2 using a propeller agitator, at 77 °C to 82 °C.

3) Add the batch of step 1 to the batch of step 2 and mix until smooth and uniform; slowly cool the batch to 49 °C to 54 °C.

4) Add benzyl alcohol of phase 3 to the batch of step 3; mix until uniform and continue to cool batch to 35 °C to 41 °C.

5) Compensate for water loss and homogenize, avoiding air entrapment; cool batch to 27 °C to 32 °C.

C.4.3 Physicochemical data

a) Appearance: white/yellowish fluid emulsion.

b) pH: 8,0 ± 0,5.

c) Viscosity (20 °C): range of values: 19 000 mPa·s to 33 000 mPa·s.

[Brookfield® [4)] rotating viscometer, RV type, helipath type, spindle B, speed 10 r/min (0,167 s^{-1}), rotation time 60 s].

NOTE The values provided above are specific to the material used.

d) Density (20 °C): 0,970 g/cm^3 ± 0,05 g/cm^3.

C.4.4 Storage and expiration

Store the reference material at 20 °C in a vessel protected from light. The package label shall include an expiration date provided by the manufacturer specifications.

4) The RV type Brookfield rotating viscometer is the trade name of a product supplied by Brookfield Engineering Laboratories. This information is given for the convenience of users of document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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C.4.5 Analytical data

C.4.5.1 Principle

The formulation shall be sampled gravimetrically and dissolved in methanol, in which the analytes are soluble. The solution shall be diluted with HPLC mobile phase and analysed by reverse phase HPLC.

The concentrations of the analytes in the sample are determined by quantification against a mixed external standard solution of analyte raw materials.

C.4.5.2 Chemical reagents

a) Benzophenone-3, production raw material, various suppliers.

b) Ethylhexyldimethyl PABA, production raw material, various suppliers.

c) Methanol, HPLC grade.

d) Water, fresh distilled.

e) Glacial acetic acid, of high purity.

f) Solution, with mass fractions of 85 % methanol and 1 % acetic acid.

Add 10 ml of glacial acetic acid to 850 ml of methanol and make up to 1 000 ml with water. Filter under vacuum through a 0,45 µm PTFE membrane filter.

C.4.5.3 High-performance liquid chromatography apparatus

Injector:	Injection volume	10,0 µl
Column:	Type	reverse phase C8 5 µm 4,6 mm × 250 mm or equivalent
	Mobile phase	solution in accordance with C.4.5.2 f)
	Flowrate	1,5 ml/min
Detector:	Type	UV
	Wavelength	308 nm [or 254 nm for fixed wavelength detection (less sensitive, less specific)]
Data:	Quantification	peak area

C.4.5.4 Method

a) Mixed standard: Accurately weigh 30 mg of benzophenone-3 and 70 mg of octyl dimethyl PABA into a 100 ml volumetric flask and dissolve in and make to volume with methanol. Mix well.

b) Mixed working standard: Pipette 5 ml of mixed standard into a 50 ml volumetric flask and make to volume with solution.

c) Using an analytical balance, weigh approximately 1 g of formulation, to the nearest 0,1 mg, into a 50 ml volumetric flask.

d) Add methanol to dissolve the sample and make up to volume.

e) Ultrasonicate the flask for 5 min and shake to completely mix the sample.

f) Pipette 1 ml into a 10 ml graduated tube and make up to volume with HPLC mobile phase.

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g) Analyse the sample and mixed working standard by reverse phase HPLC.

C.4.5.5 Quality control

a) Analyse a sample of HPLC mobile phase and a placebo, if available, prepared in accordance with the method, by reverse phase HPLC, to confirm the absence of interfering chromatographic peaks.

b) Analyse three mixed working standards [see C.4.5.4 b)] by reverse phase HPLC and calculate the coefficient of variation of the analyte peak areas.

C.4.5.6 Calculations

The analyte is calculated using Formula (C.2)

$$A_{\text{pmf}} = \frac{A}{A_{\text{std}}} \times \frac{C}{1\,000} \times \frac{50}{m} \tag{C.2}$$

where

A_{pmf} is the percent mass fraction of the analyte;

A is the peak area in the sample extract;

C is the mass concentration of analyte in the working standard in milligrams per litre;

A_{std} is the analyte peak area in the working standard;

m is the mass of the sample expressed in grams.

C.4.5.7 Acceptance criteria

The analytical results are acceptable if the following are achieved:

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

— recovery value shall be 100 % ± 5 % for all actives;

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

C.5 P5 UVAPF Reference standard

C.5.1 General

Table C.5 gives the formula for standard formulation P5.

Table C.5 — Formula for standard formulation P5

Ingredients	% mass of total mass
Phase A1	
Water	39,35
Disodium EDTA	0,05
Methylparaben	0,35
Chlorphenesin	0,20
Phenoxyethanol	0,70
Phase A2	
Glycerin	5,00

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Table C.5 *(continued)*

Ingredients	% mass of total mass
Phase B1	
Xanthan Gum	0,01
Butyl Methoxydibenzoylmethane	3,00
Octocrylene	10,00
Octyl Salicylate	5,00
Benzophenone-3	5,00
Phase B2	
PPG-2 Myristyl Ether Propionate	2,00
Octyldodecyl Neopentanoate	2,00
Butyloctyl Salicylate	8,00
PVP/Eicosene Copolymer	1,30
Phase B3	
Polyglyceryl-3 Methyl Glucose Distearate	2,00
Cetyl Alcohol	0,50
Stearic Acid	1,00
Butylparaben	0,03
Phase C	
Cyclopentasiloxane	3,00
Acrylates/C10-30 Alkyl Acrylates Crosspolymer	0,20
Phase D	
Water	1,00
Triethanolamine (99 %)	0,06
Phase E	
Water	10,00
Potassium Cetyl Phosphate	0,25

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C.5.2 Manufacturing process

a) Combine A1 into main kettle. Heat and mix to 80 °C.

b) While contents in main kettle are heating, premix A2. Add A2 to main kettle when temperature is 75 °C.

c) Combine ingredients of B1 in side kettle #1. Mix and heat to 80 °C. Maintain heat and mix in until homogenous.

d) Combine ingredients of B2 inside kettle #2. Mix and heat to 80 °C. Maintain heating and mixing until homogenous. Add B2 ingredients into kettle with B1 ingredients. Mix well.

e) Combine ingredients in B3 in side kettle #3. Heat and mix to 80 °C. Maintain heating and mixing until homogenous. Add ingredients in B3 into kettle #1 with ingredients of B1/B2. Mix well.

f) Add ingredients from kettle #1 containing B1/B2/B3 into the main kettle containing A1/A2. Start homogenization. Maintain temperature and mixing for 10 min to 15 min.

g) Begin cooling to room temperature while maintaining homogenization.

h) When mixture has cooled to 60 °C, add premix of ingredients of C into the main kettle. Mix until uniform.

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i) When temperature reaches 35 °C to 40 °C, add ingredients in D premixture into the main kettle. Mix until uniform.

j) Also while the temperature is between 35 °C and 40 °C, add ingredients in E premixture into the main kettle. Mix until uniform.

k) Continue cooling to room temperature.

C.5.3 Physiochemical data

a) Color: white/slightly off-white

b) Odor: characteristic

c) Appearance: smooth lotion

d) pH: 5,5 ± 0,5

e) Viscosity (20 °C): 77,000 cPs ± 10 % (Brookfield LV with heliopath, spindle F, 12 r/min, reading after 60 s)

f) Specific gravity (20 °C): 1,00 g/cm^3 ± 0,05 g/cm^3

C.5.4 Storage and expiry

Twelve months at 20 °C from the fabrication date, in a vessel protected from light. Store the reference material at 20 °C in a vessel protected from light. The package label shall include an expiration date provided by the manufacturer specifications.

C.5.5 Analytical method

UV filters present can be measured using EN 16344[11] analytical method.

C.5.6 Acceptance criteria

The analytical results are acceptable if the following are achieved:

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

— recovery value is 100 % / 5 % for all actives.

C.6 P8 UVAPF Reference Standard

C.6.1 General

Table C.6 gives the formula for standard formulation P8.

Table C.6 — Formula for standard formulation P8

Ingredients	% mass of total mass
Phase 1 (Oil phase)	
Ceteareth-12	1,00
C12-15 Alkyl Benzoate	7,00
Isopropyl Palmitate	5,00
Ethylhexyl methoxycinnamate	5,00
Bis-Ethylhexyloxyphenol Methoxypheyl Triazine	3,00
Ethylhexyl Salicylate	3,00

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Table C.6 *(continued)*

Ingredients	% mass of total mass
Phase 2 (Water phase)	
Water	47,30
Disodium EDTA	0,20
Chlorphenesin	0,30
Phenoxyethanol	1,00
Phase 3	
Water and Acrylates/Beheneth-25 Methacrylates Copolymer (23-28 % acrylates/behenyth-25 methacrylate copolymer)	1,20
Phase 4	
Water and Sodium Hydroxide (30 % NaOH)	adjust to pH = 7
Phase 5	
Cyclohexasiloxane, Cyclopentasiloxane	6,0
Phase 6	
Methylene Bis-Benzotriazolyl Tetramethylbutylphenol (nano), water,-decyl glucoside, propylene glycol, xanthan gum (50 % MBBT)	20,00

C.6.2 Manufacturing process

a) Heat Phase 1 and Phase 2 in separate kettles up to 80 °C. Mix each phase until uniform.

b) Under mixer, add Phase 1 at 80 °C into Phase 2 at 80 °C.

c) Add immediately Phase 3 under homogenizer. Mix until homogeneous.

d) Adjust pH to 7 with Phase 4. Mix with homogenizer until homogeneous.

e) Cool down to 60 °C and add Phase 5. Mix until homogeneous.

f) Cool down to room temperature and add Phase 6 under stirrer. Mix until homogeneous.

g) Adjust for water loss and homogenize, avoiding air entrapment.

C.6.3 Physicochemical data

a) Appearance: White cream.

b) pH value (25 °C): 7,1 ± 0,3.

c) Viscosity: 12 000 mPas–1 to 15 000 $mPas^{-1}$ using Brookfield DVIII Ultra, Spindle RV-5 at 10 r/min.

d) Density: 0,97 g/cm^3 to 1 g/cm^3.

C.6.4 Storage and expiry

Store the reference material at 20 °C in a vessel protected from light. The package label shall include an expiration date provided by the manufacturer specifications, and a statement.

C.6.5 Analytical method

UV filters present can be measured using EN 16344[12].

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C.6.6 Acceptance criteria

The analytical results are acceptable if the following are achieved:

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

— recovery value is 100 % / 5 % for all actives.

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Annex D
(normative)

Calculations and statistics

D.1 General formulae

D.1.1 Individual UVA protection factor ($UVAPF_i$)

The $UVAPF_i$ of each product on each subject shall be calculated from the individual MPPDD on unprotected skin ($MPPDD_{iu}$) and the individual MED on product protected skin (MED_{ip}) according to Formula (D.1):

$$UVAPF_i = MPPDD_{ip} / MPPDD_{iu} \quad (D.1)$$

D.1.2 UVA protection factor (UVAPF)

The UVAPF of the product shall be the arithmetic mean of the individual $UVAPF_i$ values obtained from the total number, n, of subjects with valid results, expressed to one decimal point, as shown in Formula (D.2):

$$UVAPF = (\sum UVAPF_i) / n \quad (D.2)$$

Its standard deviation, s, is given by Formula (D.3):

$$s = \sqrt{[(\sum(UVAPF_i)2 - ((\sum UVAPF_i)2 / n) / (n - 1)]} \quad (D.3)$$

D.1.3 95 % confidence interval

The 95 % confidence interval (95 %CI) for the mean UVAPF shall be expressed by Formula (D.4):

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

where c is calculated as shown in Formula (D.5) and (D.6):

$$c = (t) \times SEM = \frac{t \times s}{\sqrt{n}}$$

$$c = \frac{t \times s}{\sqrt{n}} \quad (D.5)$$

$$CI[\%] = 100 \times c / UVAPF \quad (D.6)$$

where

SEM is the standard error of the mean;

n is the total number of subjects used;

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

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Table D.1 — Student-*t* distribution

n	10	11	12	13	14	15	16	17	18	19	20
t	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, t can be modelled by: $t = 2{,}03 + \frac{12{,}7}{n^{1{,}75}}$ (for $n \geq 4$).

D.2 Experimental calculation procedure

D.2.1 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 UVA protection factors ($UVAPF_i$) for the product on each subject are then calculated according to Formula (D.1).

From these individual $UVAPF_i$ values, a provisional mean sun protection factor for the initial n' subjects ($UVAPF_{n'}$) is calculated according to Formula (D.2), together with a provisional 95 % confidence interval (95 $\%CI_{n'}$) using Formula (D.4), (D.5) and (D.6) and Table D.1, i.e.:

$$UVAPF_{n'} = \Sigma UVAPF_i / n'$$

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

where $c_{n'}$ is calculated as:

$$c_{n'} = \frac{t_{n'} \times s_{n'}}{\sqrt{n}}$$

and where $s_{n'}$ is the standard deviation from the first n' subjects calculated according to Formula (D.7) and (D.8):

$$s_{n'} = \sqrt{[(\sum(UVAPF_i)^2 - ((\sum UVAPF_i)^2 / n')) / (n' - 1)]} \quad (D.7)$$

$$CI_{n'}[\%] = 100 \times c_{n'} / UVAPF_{n'} \quad (D.8)$$

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 ≤ 17 % of the mean provisional UVAPF.

If this criterion is not fulfilled after twenty valid subjects, then the entire test shall be repeated.

D.2.2 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 Formula (D.9) and rounded up to the nearest integer:

$$n' = \left(\frac{t_{n'} \times s_{n'}}{c_{n'}} \right)^2 \quad (D.9)$$

where

$t_{n'}$ is the t statistic from Table D.1, with n' results;

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

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$c_{n'}$ is 17 % of mean $UVAPF_{n'}$, representing the required confidence interval.

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

$n^* = (2{,}262\ s_n' / 0{,}17\ UVAPF_n')^2$

i.e.

$n^* = (13{,}30\ s_n' / UVAPF_n')^2$

D.3 Examples

D.3.1 Example 1

Table D.2 is an example of a table gathering data, calculations and results. When data are entered in spreadsheet software, all calculations can be performed automatically.

Table D.2 shows the results for product EX1 with expected UVAPF 10. After ten subjects had been exposed, the results were:

— $UVAPF_{n'} = 10{,}6$;

— $s_{n'} = 1{,}8$;

— $c_{n'} = 1{,}7$;

— 95 %$CI_{n'} = 9{,}7$ to 13,1;

— $CI_{n'}$[%] = 11,3 %.

Since the $CI_{n'}$[%] was smaller than 17 %, no further testing was necessary and the final UVAPF of the product EX1 was:

— UVAPF = 10,6 with CI[%] = 11,3 %.

D.3.2 Example 2

Table D.3 shows the results for product EX2 with expected UVAPF 20. After ten subjects had been exposed, the results were:

— $UVAPF_{n'} = 21{,}3$;

— $s_{n'} = 6{,}1$;

— $c_{n'} = 4{,}34$;

— 95 %$CI_{n'} = 17{,}0$ to 25,6;

$CI_{n'}$ [%] = 20,4 %.

The relative variation of the results was higher than in Example 1 and the statistical criterion was not met ($CI_{n'}$ [%] was greater than 17 %). The test had to be continued and the likely total number, n, of subjects necessary was calculated as shown in Formula (D.10):

$$n^* = (t_{n'} \times s_{n'} / C_{n'})^2 = (2{,}262 \times 6{,}1 / 4{,}34)^2 = 14 \quad \text{(D.10)}$$

Therefore, five subjects were added and the newly calculated provisional results were:

— UVAPF15 = 21,2;

— $s15 = 6{,}2$;

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— $c15$ = 3,5 with n = 15 and t15 = 2,145;

— 95 %CI15 = 17,7 to 24,7;

— CI [%]15 = 16,3 %.

The criterion was met after the fifteenth subject ($CI_{n'}$ [%] smaller than 17 % of the mean UVAPF) and the final UVAPF of product EX2 was:

— UVAPF = 21,2 with CI[%] = 16,3 %

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Table D.2 — Example of calculation with 10 subjects (expected UVAPF10)

ISO 24442 (2022) Test Method																		
Laboratory:																		
Test Product Description:					**Dose Increments:**							**Start Date:**			**End Date:**			**UV source:**
	TEST			**SIM**	**TEST SUBJECTS**							**RESULTS**						**CONCLUSION:**
Subj.	Expo-sure	Ap-plied	Read	SS output (highest)	Sub-ject	Skin	$MPPDD_u$		$MPPDD_p$		$UVAP\text{-}F_i$	Reject? Y? or N?	$UVAP\text{-}F_{n'}$	$s_{n'}$	$c_{n'}$	$CI_{n'}$[%]	n	
N°	date	by	by	mW/cm²	code	ITA°	sec-onds	J/cm²	sec-onds	J/cm²					0,17	($100.c_{n'}$/UVAP-$F_{n'}$)		$CI_{n'}$[%] ≤ 17 % ?
1								10		125	12,5		—	—	—	—	—	—
2								12,5		156	12,5							—
3								8		80	10,0							—
4								10		100	10,0							—
5								15,6		156	10,0							—
6								10		80	8,0							—
7								12,5		125	8,0							—
8								12,5		125	10,0							—
9								8		100	12,5							—
10								10		125	12,5		**10,6**	1,8	**1,29**	11,3 %	8	Complies
FINAL RESULT:					**Mean UVAPF =**		**10,6**			***s* =**	**1,8**		***c* =**	**1,19**	**CI[%] =**	**11,3 %**	**95 % CI:**	**9,41-11,8 (*n* = 10)**

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Table D.3 — Example of calculation with 15 subjects (expected UVAPF20)

ISO 24442 (2022) Test Method																		
Laboratory:																		
Test Product Description:					**Dose Increments:**								**Start Date:**		**End Date:**			**UV source:**
	TEST			**SIM**	**TEST SUBJECTS**								**RESULTS**					**CONCLUSION:**
Subj.	Exposure	Applied	Read	SS Output (highest)	Subject	Skin	$MPPDD_u$		$MPPDD_p$		UVAP-F_i	Rejected?	UVAP-$F_{n'}$	$s_{n'}$	$c_{n'}$	$CI_{n'}$[%]	n	
N°	date	by	by	mW/cm²	code	ITA°	seconds	J/cm²	seconds	J/m²		Y? or N?			0,17	(100.$c_{n'}$/ $SPF_{n'}$)		$CI_{n'}$[%] =< 17 % ?
1								10		200	20,0		—	—	—	—	—	—
2								12,5		313	25,0							—
3								8		200	25,0							—
4								10		128	12,8							—
5								15,6		390	25,0							—
6								10		250	25,0							—
7								12,5		200	16,0							—
8								12,5		160	12,8							—
9								8		250	31,3							—
10								10		200	20,0		**21,3**	6,1	**4,34**	20,34 %	14	Does not comply
11								15,6		200	12,8		**20,5**	6,3	**4,23**	20,6 %	17	Does not comply
12								19,5		609	31,2		**21,4**	6,7	**4,28**	20,0 %	18	Does not comply
13								12,5		313	25,0		**21,7**	6,5	**3,94**	18,2 %	16	Does not comply
14								15,6		250	16,0		**21,3**	6,5	**3,73**	17,5 %	16	Does not comply
15								10		200	20,0		**21,2**	6,2	**3,45**	16,3 %	15	Complies
FINAL RESULT:					**Mean UVAPF=21,2**					***s* =**	**6,2**		***c* =**	**3,5**	**CI[%] =**	**16,3 %**	**95 % CI:**	**95 %CI: 17,7 – 24,7 (*n* = 15)**

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Annex E
(normative)

Colorimetric determination of skin colour typing

E.1 General

The International Commission on Illumination (CIE) normalized tristimulus colorimetry and spectrocolorimetry, using the *L*a*b*/L*CH* colour spaces, have long been internationally accepted and validated. They are routinely used to analyse colours in a way that is strictly correlated with human vision[12].

Skin colour, as characterized by the individual typology angle (ITA°), appears to be used when selecting subjects (see Annex A).

The traditional Fitzpatrick phototype classification is based on the subject's experience of his/her own sensitivity to actinic erythema and the ability to tan after a first sun exposure. However, this classification provides a subjective and unchanging indication of the skin's sensitivity to UV, which does not take into account the level of melanisation of the subject's skin. This can lead to misinterpretation of the UV-sensitivity of the subject and to the use of inappropriate UV doses when determining the MED or MPPDD.

Measuring skin colour in the *L*a*b** system as defined by the CIE, allows the melanotic status of the skin, at the time of testing, to be taken into account.

E.2 Apparatus

E.2.1 The measurement equipment is a skin contact reflectance spectrophotometer and/or colorimeter with a view of at least 8 mm diameter which utilizes the *L*a*b** colour space and complies with CIE recommendations.

E.2.2 A lamp for illumination of the back of the subject shall have a colour temperature of 6 500 K.

E.3 Mode of operation

E.3.1 For reliable colour measurements on skin, allow subjects to rest for at least 10 min with the skin uncovered, until elimination of contact or stress-related redness and marks.

E.3.2 During measurements, care should be taken to apply the cone aperture of the reflectance colorimeter sensing head so that it just makes contact with the skin, without any pressure. This is critical as undue pressure may cause a "blanching" effect in the skin which may lead to seriously inaccurate measurements. An ergonomic position should be adopted and preliminary training may be necessary, until a standard deviation smaller than 0,2 (0,1 typically) on the *L**, *a** or *b** co-ordinate can be obtained by repeated triplicate measurements on the same skin area[12].

E.4 Skin colour typing

E.4.1 For this purpose absolute *L**, *a** and *b** values should be recorded. The reflectance colorimeter aperture should not be reduced by use of any form of diaphragm.

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E.4.2 Calibrate the skin contact reflectance spectrophotometer and/or colorimeter as per manufacturer's instructions. Perform $L^*a^*b^*$ colorimetric measurements on the test sites where products will be applied and exposed later in the test. Calculate the mean L^*, a^*, b^* value (at least three measurements on each test site of each subject).

E.4.3 Calculate the individual typology angle, ITA°, on the mean L^* and b^* values using Formula (E.1):

$$\text{ITA}^\circ = \left\{\text{arc tangent}\ \frac{(L^*-50)}{b^*}\right\}\frac{180}{\pi} \tag{E.1}$$

where the arc tangent is expressed in radians. Round the value off and express ITA° to the nearest integer.

E.4.4 The difference in average ITA° values between test sites used on a subject shall be less than 5.

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Annex F
(informative)

Visual guidance for PPD grading— Visual appearance of PPD

The acceptance or rejection of qualifying PPD reactions for determination of the MPPDD is critical to uniform determination of the UVAPF of a sunscreen product. The grading scale for the MPPDD is described.

IMPORTANT — When reading this document on a computer, ensure that both the computer screen colour (white point) and the screen intensity are adjusted so that the tan calibration scale, as shown in Figure F.1 can be visually discriminated between left and right. Refer to setting instructions specific to your computer operating system.

Figure F.1 — Colour swatch for calibration

Visual appearance of PPD should be performed in sufficient and uniform illumination. At least 450 lux in the plane parallel with the back of the test subject should be provided by a lamp with a continuous emission in the visible spectrum with a color temperature of 6 500 K. Incandescent light bulbs or Light Emitting Diode (LED) lamps are recommended and can be found in this colour temperature range. Fluorescent lamps may not be used as the sole source for the visual assessment.

The determination of MPPD shall be carried out in a room with matte, neutral wall colours.

PPD responses shall be observed in a "blind" manner (with exception of the provisional MPPD). The observers of PPD responses on any subjects shall not be the same persons as the ones who perform product application and exposure. The observers shall not be aware of the test design (randomization of the test sites) on that subject.

The grading scale for UV exposed test subsites shall be:

— **0**: no PPD present

— **0,5**: ambiguous PPD, and/or no clear border, and/or not filling more than 50 % of the exposure subsite

— **1**: Perceptible unambiguous PPD with defined borders filling more than 50 % of the exposure subsite (MPPDD if it is the lowest exposure dose with grade 1)

— **2**: Moderate to intense PPD

Evaluation shall be done on each subsite individually using the definition of MPPDD. The evaluation has to be done on the presence or absence of the PPD and not on the intensity. An illogical progression but with PPD (≥ Grade 0,5) present does not have to be discarded. Examples are given for guidance.

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