SLOVENSKI STANDARD
oSIST prEN ISO 24442:2021

01-april-2021

Kozmetika - Preskusne metode za zaščito pred soncem - Določevanje zaščitnega faktorja UVA in vivo (ISO/DIS 24442:2021)

Cosmetics - Sun protection test methods - In vivo determination of sunscreen UVA protection (ISO/DIS 24442:2021)

Kosmetik - Prüfverfahren für Sonnenschutzmittel - In-vivo-Bestimmung des UVA-Sonnenschutzes (ISO/DIS 24442:2021)

Cosmétique -- Méthodes d'évaluation de la protection solaire -- Détermination in vivo de la protection UVA (ISO/DIS 24442:2021)

Ta slovenski standard je istoveten z: prEN ISO 24442

ICS:

71.100.70	Kozmetika. Toaletni pripomočki	Cosmetics. Toiletries

oSIST prEN ISO 24442:2021 en,fr,de

DRAFT INTERNATIONAL STANDARD
ISO/DIS 24442

ISO/TC **217** Secretariat: **ISIRI**

Voting begins on: **2021-02-22** Voting terminates on: **2021-05-17**

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

Cosmétique — Méthodes d'évaluation de la protection solaire — Détermination in vivo de la protection UVA

ICS: 71.100.70

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 24442:2021(E)

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Foreword

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

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).

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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

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

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

The main changes compared to the previous edition are as follows:

— Revision in accordance with revision of relevant ISO Standard, ISO 24444.

Introduction

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

Topical sunscreen products are primarily rated and labelled according to their ability to protect against sunburn, using a test method to determine the *in vivo* Sun Protection Factor (see ISO 24444). This rating evaluates filtration of sunburn generating radiation across the electromagnetic UV spectrum (290 nm to 400 nm). However, knowledge of the Sun Protection Factor (SPF) rating does not provide explicit information on the magnitude of the protection provided specifically in the UVA range of the spectrum (320 nm to 400 nm), as it is possible to have high SPF products with very modest UVA protection (e.g. SPF 50 with a 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 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 International Standard 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.

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

1 Scope

This International Standard specifies a method for the *in vivo* determination of UVA protection factor (UVAPF) of sunscreen products. This International standard 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.

It 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 24442:2012, *Cosmetics — Sun protection test method — In vivo determination of sunscreen UVA protection*[6]

ISO 24444:2019, *Cosmetics — Sun protection test methods — In vivo determination of the sun protection factor (SPF)*[7]

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 http://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

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 and other ultraviolet induced damage

3.5
minimal persistent pigment darkening dose
MPPDD
lowest Ultraviolet A (UVA) dose that produces the first perceptible unambiguous persistent pigment darkening response with defined borders appearing over most of the field of UVA exposure, observed between 2 h and 24 h after the end of the UVA exposure

3.5.1
$MPPDD_u$
MPPDD on unprotected skin

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

3.5.2
$MPPDD_p$
MPPDD on product protected skin

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

3.6
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.

3.7
UVA protection factor of a product: UVAPF
arithmetic mean of all valid individual $UVAPF_i$ values obtained from all subjects in the test

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

3.8
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.

3.9
test site
area of the skin where a product is applied or the site used for the determination of the unprotected MPPDD

3.10
exposure sub-sites
areas of skin that are exposed to UV-irradiation within a test site

3.11
individual typology angle
ITA°
value characterizing the skin colour of the subject as measured by a skin contact reflectance spectrophotometer or skin colourimeter

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 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 $MPPDD_{ip}$ divide by $MPPDD_{iu}$, as in the formula given in item 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.

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 colourimetric methods (see Annex 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 might 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 the Declaration of Helsinki[8] and national regulations regarding human studies should also be taken into account.

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. For the test to be considered valid for the first 10 subjects, the resulting range of the 95 % CI of the mean shall be within ± 17 %. 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).

Results may only be declared invalid and excluded from the calculation of the mean UVAPF according to 9.5.3 or because of non-compliance 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.

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 (visible light and infrared) and 1 500 nm. 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 (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 1500 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 [9]. 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 and 1 600 nm) calibrated against a standard reference source over the range of 280 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 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 6.4.4.3).

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 6.4.4.3).

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).

6.4.4.1 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.

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

$$\text{Uniformity } \% = (1-(\text{max}-\text{min}) / (\text{average}))\% \quad (1)$$

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

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.

6.4.5.1 Single output devise

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).

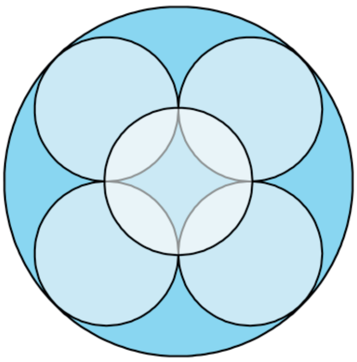

Figure 1 — Single output device

6.4.5.2 Multiple output devise

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 above, then adjustments to the lamp optical system shall be made to bring the uniformity within the limits above.

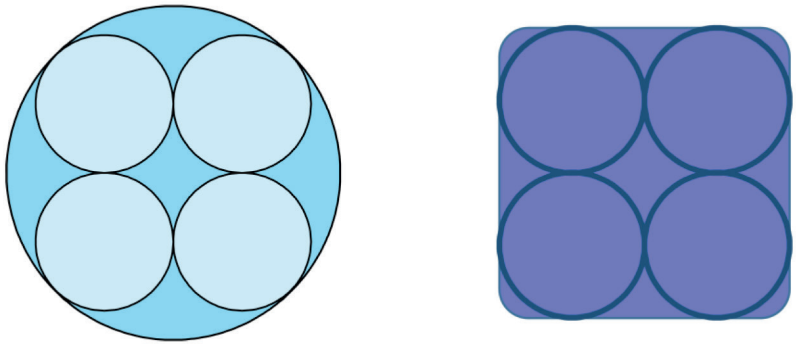

Figure 2 — Multiple output device

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.

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