SLOVENSKI STANDARD
oSIST prEN ISO 24444:2019

01-maj-2019

Kozmetika - Preskusne metode za zaščito pred soncem - Določevanje faktorja zaščite pred soncem (SPF) in vivo (ISO/DIS 24444:2019)

Cosmetics - Sun protection test methods - In vivo determination of the sun protection factor (SPF) (ISO/DIS 24444:2019)

Kosmetik - Untersuchungsverfahren fÃ¼r Sonnenschutzmittel - In-vivo-Bestimmung des Sonnenschutzfaktors (SSF) (ISO/DIS 24444:2019)

Cosmétiques - Méthodes d'essai de protection solaire - Détermination in vivo du facteur de protection solaire (FPS) (ISO/DIS 24444:2019)

Ta slovenski standard je istoveten z: **prEN ISO 24444**

ICS:

71.100.70	Kozmetika. Toaletni pripomočki	Cosmetics. Toiletries

oSIST prEN ISO 24444:2019 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 24444

ISO/TC **217**

Secretariat: **ISIRI**

Voting begins on:
2019-03-20

Voting terminates on:
2019-06-12

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

Cosmétiques — Méthodes d'essai de protection solaire — Détermination in vivo du facteur de protection solaire (FPS)

ICS: 71.100.70

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 24444:2019(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword

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

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

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

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.

ISO 24444 was prepared by Technical Committee ISO/TC 217, *Cosmetics*.

Introduction

The level of sun protection provided by sunscreen products has traditionally been estimated using the sun protection factor or SPF test, which uses the erythemal response of the skin to ultraviolet (UV) radiation. The SPF is a ratio calculated from the energies required to induce a minimum erythemal response with and without sunscreen product applied to the skin of human test subjects. It uses ultraviolet radiation usually from an artificial source.

Different standard methods are available and described in the technical report ISO/TR 26369[1-3]. For this version of ISO 24444, these references have been updated in the Bibliography section.

Since publication of the first version of this Standard, harmonization has been achieved in many member countries. The objective of this updated version is to further improve reproducibility between test sites, so as to obtain the same SPF value.

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

1 Scope

This International Standard specifies a method for the *in vivo* determination of the sun protection factor (SPF) 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 erythema induced by solar ultraviolet rays.

2 Terms and definitions

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

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

2.1.1
ultraviolet B
UVB
electromagnetic radiation in the range of 290 nm to 320 nm

2.1.2
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.

2.1.3
Erythemal effective irradiance E_{er}
radiometric quantity derived by multiplying the spectral irradiance E(λ) of the solar simulator with the erythema action spectrum[4] $s_{er}(\lambda)$ at each wavelength λ and integrating over wavelength range of 290 nm to 400 nm

$$E_{er} = \int_{290}^{400} E(\lambda) s_{er}(\lambda) d\lambda \quad \text{unit: W/m}^2 \text{ (eff.)}$$

2.1.4
Erythemal effective radiant exposure (erythemal dose) H_{er}
radiometric quantity defined as time integral of erythemal effective irradiance $E_{er}(t)$

$$H_{er} = \int_{t} E_{er}(t) dt \quad \text{unit: J/m}^2 \text{ (eff.)}$$

2.2
erythema
reddening of the skin caused by UV radiation

2.3
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

2.4
minimal erythemal dose
MED
lowest erythemal effective radiant exposure (H_{er}) that produces the first perceptible unambiguous erythema with defined borders appearing over more than 50% of UV exposure subsite, 16 h to 24 h after UV exposure. Annex F contains visual references and colourimetric guidance for the acceptable MED appearance

2.4.1
MED_u
MED on unprotected skin.

2.4.1.1
MED_{iu}
MED of an individual subject on unprotected skin

2.4.2
MED_p
MED on product protected skin

2.4.2.1
MED_{ip}
MED of an individual subject on protected skin

2.5
individual sun protection factor
SPF_i
ratio of the individual minimal erythemal dose on product protected skin (MED_{ip}) to the (individual) minimal erythemal dose on unprotected skin (MED_{iu}) of the same subject:

$$SPF_i = \frac{MED_{i(protected)}}{MED_{i(unprotected)}} = \frac{MED_{ip}}{MED_{iu}}$$

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

2.6
sun protection factor of a product: SPF
arithmetic mean of all valid individual SPFi values obtained from all subjects in the test

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

2.7
test area
on the back between the scapula line and the waist. Skeletal protrusions and extreme areas of curvature should be avoided

2.8
test site
area of the skin where a product is applied or the site used for the determination of the unprotected MED

2.9
exposure sub-sites
areas of skin that are exposed to UV-irradiation

2.10
individual typology angle
ITA°
value characterizing the skin colour of the subject as measured by a skin contact reflectance spectrophotometer or skin colourimeter. Refer to Annex E for the detailed requirements of the equipment / measurement

3 General principle

The SPF test method is a laboratory method that utilizes a xenon arc lamp solar simulator (or equivalent) of defined and known output to determine the protection provided by sunscreen products on human skin against erythema induced by solar ultraviolet rays.

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 ultraviolet light without any protection and another (different) section is exposed after application of the sunscreen product under test. One further section is exposed after application of an SPF reference sunscreen formulation which is used for validation of the procedure.

To determine the sun protection factor, incremental series of delayed erythemal responses are induced on a number of small sub-sites on the skin. These responses are visually assessed for presence of erythema 16 to 24 hours after UV radiation, by the judgment of a trained and competent evaluator.

The individual minimal erythemal dose for unprotected skin (MED_{iu}) and the individual MED obtained after application of a sunscreen product (MED_{ip}) shall be determined on the same subject on the same day. An individual sun protection factor (SPF_i) for each subject tested is calculated as the ratio of individual MED on product protected skin divided by the individual MED on unprotected skin, as in the equation of item 2.5.

The sun protection factor for the product (SPF) is the arithmetic mean of all valid SPF_i results from each subject in the test expressed to one decimal place.

4 Test subjects

4.1 Selection of the test subjects

4.1.1 General

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

4.1.2 Skin colour of the test subjects

Test subjects included in the SPF test shall have an ITA° value of at least 28° by colourimetric methods (see Annexes A and E) and be untanned on the test area.

The average of the subjects making up a test panel shall have an ITA° between 41° and 55°, and there shall be at least one subject from each ITA° band 28-40°, 41-55°, and >56°.

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 MEDu test area.

4.1.3 Age restriction

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

4.1.4 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, UVA-PF, 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.

4.1.5 Ethics and consent

All testing shall be done in accordance with the Declaration of Helsinki[7] and national regulations regarding human studies, if any.

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

4.2 Number of test subjects

The minimum number of valid SPFi results shall be 10 and the maximum number of valid SPFi 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 SPF. 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 SPF according to 8.6.4 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 SPF 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 SPF shall be within ±17 % of the mean SPF. If it s not within ±17 % of the mean SPF, 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.

5 Apparatus and materials

5.1 Source of ultraviolet radiation

5.1.1 General

The artificial light source used shall comply with the source spectral specifications as described in 5.1.2 and Annex B. A xenon arc solar simulator with appropriate filters shall be used.

5.1.2 Quality of ultraviolet radiation

5.1.2.1 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 B.1).

5.1.2.2 To ensure that appropriate amounts of UVA radiation are included in the spectrum of the solar UV simulator, the total radiometric proportion of the UVA II (320 nm to 340 nm) irradiance of the

simulator shall be ≥ 20 % of the total UV (290 nm to 400 nm) irradiance. Additionally, the UVA I region (340 nm to 400 nm) irradiance shall be ≥ 60 % of the total UV irradiance.

5.1.2.3 The source spectral specification is described in terms of cumulative erythemal effective irradiance by successive wavelength bands from < 290 nm up to 400 nm. The erythemal effective irradiance of each wavelength band is expressed as a percentage of the total erythemal effective irradiance from < 290 nm to 400 nm, or as the percentage relative cumulative erythemal effectiveness (% RCEE). The definition and calculation of % RCEE values is described in Annex B and the acceptance limits are given in Table B.1.

5.1.3 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 1600 W/m^2. When total irradiance is < 1600 W/m^2, it shall still be confirmed, prior to conducting an SPF 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 1600nm) calibrated against a standard reference source over the range of 280 to 1600nm. Alternatively, the source may be measured with a calibrated spectroradiometer over this same wavelength range to determine the total irradiance.

5.1.4 Uniformity of beam

Uniformity of the beam shall be measured periodically depending on the solar simulator type using either UV sensitive film or UV sensor methods (see 5.1.4.3 and 5.1.4.4). 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 erythema 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 erythema 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.

5.1.4.1 Large beam source: When a large-beam UV source is used to simultaneously expose several sub-sites (i.e. at least two sub-sites) 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).

5.1.4.1.1 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. See 5.1.4.3 for further details.

5.1.4.1.2 UV Radiometer method: A UV radiometer sensor may be used to sample the beam intensity at multiple sites. Measurements shall be made at equally distributed points

5.1.4.1.3 The uniformity shall be ≥ 90% as calculated by the equation:

(1-(max-min)/(average))% **≥** 90% (tentative pending data)

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.

5.1.4.2 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.

5.1.4.2.1 Single output device: For a single output 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 the equation:

(1-(max-min)/(average))% ≥ 90%

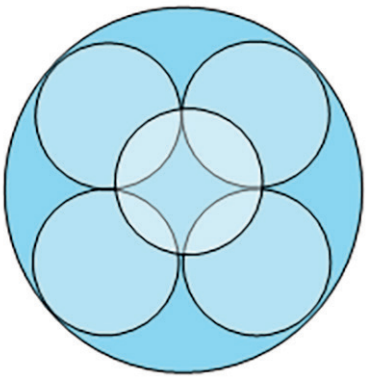

Figure 1

5.1.4.2.2 Multiple output device: For a multiple output 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 the equation:

Uniformity % = (1-(max-min)/(average))%

The average uniformity of all beams for the multiple output device shall be ≥ 90%

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.

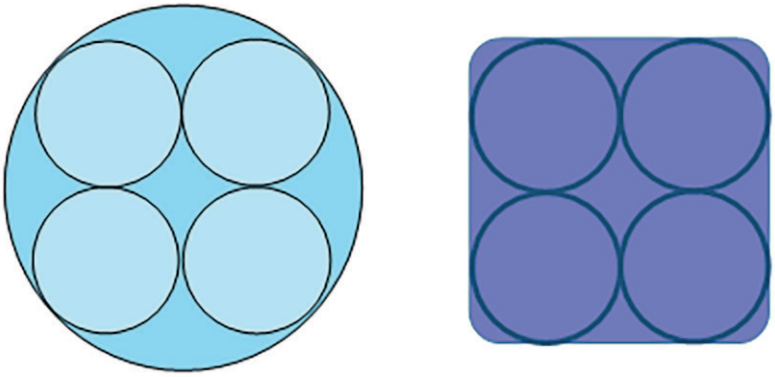

Figure 2

5.1.4.3 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 – 256 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 diagramed above. 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 5.1.4.1.3, 5.1.4.2.1 and 5.1.4.2.2.

5.1.4.4 UV Sensor:

Alternatively, a small aperture (quadrant) UV sensor with an mechanical alignment fixture may be used to measure sub-sections of the output beam intensity as outlined above and the beam uniformity calculated as per 5.4.1.2.1 and 5.4.1.2.2.

6 Maintenance and monitoring the UV solar simulator output

6.1 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 2500h 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 with a band width of 2nm 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.

6.2 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 erythema weighted radiometer cross-calibrated against a spectroradiometric measurement of the solar simulator output as detailed in 6.1. 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 dividing the erythema effective irradiance E_{er} (W/m^2 erythemal weighted) of the solar simulator as measured by the spectroradiometer (6.1) by the erythema weighted irradiance E_{er} (W/m^2) of the solar simulator as measured by the radiometer.

$$Y = \frac{E_{er(\text{spectroradiometer})}}{E_{er(\text{radiometer})}}$$