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Kozmetika - Določevanje zaščitnega faktorja UVA in vitro (ISO/DIS 24443:2020)

Determination of sunscreen UVA photoprotection in vitro (ISO/DIS 24443:2020)

In vitro Bestimmung des UVA-Schutzes von Sonnenschutzmitteln (ISO/DIS 24443:2020)

Détermination in vitro de la photoprotection UVA (ISO/DIS 24443:2020)

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Détermination in vitro de la photoprotection UVA

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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).

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

This second edition cancels and replaces the first edition (ISO 24443:2012), which has been technically revised.

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.

Introduction

This International Standard specifies the procedure to determine the Ultraviolet A Protection Factor (UVAPF) of a sunscreen product using the in vitro UVAPF according to the principles recommended by COLIPA in 2011. 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 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. The UVAPF value of a product provides information on the magnitude of the protection provided explicitly in the UVA portion of the spectrum, independent of the SPF values.

The test method outlined in this International Standard is derived primarily from the in vitro UVAPF test method as developed by COLIPA.

Determination of sunscreen UVA photoprotection in vitro

1 Scope

This document specifies an *in vitro* procedure to characterize the UVA protection of sunscreen products. Specifications are proposed to enable determination of the spectral absorbance characteristics of UVA protection in a reproducible manner.

In order to determine relevant UVA protection parameters, the method has been created to provide an UV spectral absorbance curve from which a number of calculations and evaluations can be undertaken. Results from this measurement procedure can be used for other computations, as required by local regulatory authorities. These include calculation of the Ultraviolet-A protection factor (UVA-PF) [correlating with *in vivo* UVA-PF from the persistent pigment darkening (PPD) testing procedure], critical wavelength and UVA absorbance proportionality. These computations are optional and relate to local sunscreen product labelling requirements. This method relies on the use of static *in vivo* SPF results for scaling the UV absorbance curve.

This document is not applicable to powder products such as pressed powder and loose powder products.

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 http://www.electropedia.org/

3.1
***in vitro* UVA protection factor**
UVA-PF
in vitro UVA protection factor of a sun protection product against UVA radiation, which can be derived mathematically with *in vitro* spectral modelling

3.2
***in vitro* protection factor**
$SPF_{in\ vitro}$, protection factor of a sun protection product against erythema-inducing radiation calculated with spectral modelling

3.3
λc
critical wavelength, calculated from spectral data. It corresponds to the wavelength at which the area under the absorbance curve represents 90 % of the total area under the curve in the UV region

3.4
action spectrum for erythema [1] [2].
E(λ)
relative effects of individual spectral bands of an exposure source for an erythema response

3.5
action spectrum for PPD [3] [4].
P(λ)
relative effects of individual spectral bands of an exposure source for a persistent pigment response

3.6
monochromatic absorbance
A_λ
sunscreen absorbance at wavelength, λ, related to the sunscreen transmittance, T_λ, by

$$A\lambda = -\log(T_\lambda)$$

where transmittance, T_λ, is the fraction of incident irradiance transmitted by the sunscreen film

3.7
irradiance
I
fluence rate per unit area, expressed in W/m^2, for a defined range of wavelengths

EXAMPLE From 290 nm to 400 nm for UVA + UV-B irradiance; from 320 nm to 400 nm for UVA irradiance.

3.8
spectral irradiance for SPF testing or PPD testing
I(λ)
irradiance per unit wavelength, I(λ), expressed in $W/m^2/nm$

3.9
spectrophotometer
equipment for measuring the reflection or transmission properties of a material as a function of wavelength limited to ultraviolet, visible and short infrared ranges in this document

3.10
spectroradiometer
device designed to measure the spectral density of illuminants

3.11
radiometer
device for measuring the radiant flux (power) of electromagnetic radiation

3.12
reference product
reference sunscreen product used to validate the testing procedure

3.13
solar simulator
Equipment used to simulate the solar irradiance and spectrum.

3.14
substrate plate
Material to which the test product is to be applied.

4 Principle

The test is based on the assessment of UV-transmittance through a thin film of sunscreen sample spread on a roughened substrate, before and after exposure to a controlled dose of radiation from a defined UV exposure source.

Because of the several variables that cannot be controlled with typical thin film spectroscopic techniques, each set of sunscreen transmission data is mathematically adjusted so that the in vitro SPF data yield the same measured in vivo SPF value that was determined by in vivo testing. As *in vivo* method could raise ethical consideration, any alternative SPF method, published as an ISO method, may be used.

Samples are then exposed to a specific measured dose of UV radiation to account for the photostability characteristics of the test product.

The resulting spectral absorbance data have been shown to be a useful representation of both the width and height of the UVA protection characteristics of the sunscreen product being tested. The mathematical modelling procedure has been empirically derived to correlate with human *in vivo* (persistent pigment darkening) test results.

5 Apparatus

5.1 UV spectrophotometer specifications

The UV spectrophotometer wavelength range shall span the primary waveband of 290 nm to 400 nm. The wavelength increment step shall be 1 nm.

A UV spectrophotometer that does not have a monochromator after the test sample should employ a fluorescence rejection filter.

The UV spectrophotometer input optics should be designed for diffuse illumination and/or diffuse collection of the transmitted irradiance through the roughened polymethylmethacrylate (PMMA) substrate, with and without the sunscreen layer spread on its surface.

The size of the diameter of the entrance port of the UV spectrophotometer probe shall be smaller than the size of the light spot to be measured at the sample level (in order to account for stray light).

The area of each reading site should be at least 0,5 cm^2 in order to reduce the variability between readings and to compensate for the lack of uniformity in the product layer.

The wavelength should be accurate to within 1 nm, as checked using a holmium-doped filter (see Annex A). The ability of an instrument to accurately measure absorbance is limited by the sensitivity of the instrument. The minimum required dynamic range for this methodology is 2,2 absorbance units as determined according to Annex A.

The maximum measured absorbance should be within the dynamic range of the device used. If the test measurements yield absorbance curves that exceed the determined upper limit of the UV spectrophotometer, the product should be re-tested using an instrument with increased sensitivity and dynamic range.

The lamp in the UV spectrophotometer that is used to measure the transmittance shall emit continuous radiation over the range of 290 nm to 400 nm, and the level of irradiance should be sufficiently low, so that the photostability of the product is not unduly challenged (a xenon lamp is a convenient solution).

Therefore, the UV dose during one measurement cycle should not exceed 0,2 J/cm^2.

NOTE A UV spectrophotometer is used to measure the absorbance properties of the sunscreen on the test plates. A spectroradiometer is used to measure the spectral energy distribution and intensity of the UV exposure source or the UV spectrophotometer during the absorbance measurement of the sunscreen on the test plate. Coupled with an UV source, the radiometer can give similar results to a spectrophotometer.

5.2 Calibration of the UV spectrophotometer

The UV spectrophotometer shall be validated every month by measurements of reference materials.

A three-fold test is required, as described in Annex A:

— dynamic range of the UV spectrophotometer;

— linearity test of the UV spectrophotometer;

— wavelength accuracy test.

5.3 Calibration of the UV exposure source

The spectral irradiance at the exposure plane of the UV exposure source that is used for irradiation (to take into account any photoinstability) shall be as similar as possible to the irradiance at ground level under a standard zenith sun.[1] As defined by COLIPA,[2] the reference standard sun has a total irradiance of 51.4 to 63.7 W/m2 and a UVA to UVB irradiance ratio of 16.9 to 17.5.

Therefore, the UV irradiance shall be within the following acceptance limits (measured at sample distance).

Table 1 — UV exposure source specifications

UV exposure source specifications as measured with a spectroradiometer	
Total UV irradiance (290 nm to 400 nm)	40 W/m^2 to 200 W/m^2
Irradiance ratio of UVA[a] to UVB[b]	11-22
[a] 320 nm to 400 nm.	
[b] 290 nm to 320 nm.	

In broad-beam UV-sources, spectra from different locations under the beam shall be recorded over at least 5 different locations (a location is defined for each plate) in order to account for uniformity.

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

(1-(max-min)/(average))% ≥ 90% *

*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 plate.

The UV exposure source device should have the ability to maintain samples within the range of 27 °C to 32 °C, with intra laboratory temperature ± 2 °C. It is important that the temperature of the sample itself on the plate shall be measured and not just the surrounding air temperature. Therefore, the measurement of the temperature shall be on plate level.

To maintain samples at required temperature, a filter system that particularly reduces Infrared radiation shall be used to achieve the specified temperature range. Cooling trays for the sample plates or ventilators shall be used to maintain a temperature lower than 32 °C and warming devices to maintain samples at or above 27 °C.

Measurement to be made using a sensor that is traceable to a national or an international calibration standard, within the range of use.

5.4 Monitoring of the UV exposure source

The emission of the UV exposure source used for exposure shall be checked for compliance with the given acceptance limits by a suitably qualified expert (at least) every 12 months, or 2500 hours of lamp running time. The inspection should be conducted with a spectroradiometer that has been calibrated against a standard lamp that is traceable to a national or an international calibration standard provided

by an accredited ISO17025 laboratory. In addition to the spectroradiometric inspection, the intensity of the UV exposure source used for exposure shall be checked prior to each use.

This can be done using either a spectroradiometer or a radiometer with sensitivity in the UVA, calibrated for the same UV exposure source spectrum used for the exposure step of the procedure, applying the coefficient of calibration to adjust for variance between the UVA radiometer and the reference spectroradiometer.

5.5 Calibration of the UVA radiometer used to monitor the test sample irradiation

If a UVA radiometer is used, this device shall have been suitably calibrated. This requires that it be calibrated to the spectroradiometer used to measure the exposure source (as during annual solar simulator calibration).

Calibration shall be conducted in terms of UVA irradiance (320 nm to 400 nm) and shall be at the same level at which the test plates are exposed. Once calibrated with the spectroradiometer, the UVA radiometer may be used to determine the UV doses to be used during the exposure procedure on a day-to-day basis. Annex B provides the step-by-step calibration procedure.

5.6 Substrate/plate

The substrate/plate is the material to which the test product is to be applied. For this method, PMMA plates with one rough side of the substrate are to be used and are commercially available. The size of the substrate should be chosen such that the application area is not less than 16 cm^2.

The specifications and preparation of this type of plate[9] are described in Annex D.

6 Test method

6.1 Outline of the test procedure

6.1.1 Conduct the calibration and validation of the test equipment, including the UV spectrophotometer used for transmission/absorbance measurements and the UVA radiometer (or spectroradiometer) used to measure the UV exposure source, and verify the transmission properties of the test plates as described in Annex D.

6.1.2 Conduct blank measurements of a glycerin-treated or Vaseline-treated plate for the reference "blank", which will be used in the subsequent absorbance measurements.

6.1.3 Conduct *in vitro* absorbance measurements of the sunscreen product spread on a PMMA plate, prior to any UV irradiation. Acquire the initial mAf spectrum with $A_0(\lambda)$ data.

6.1.4 Conduct the mathematical adjustment of the initial UV absorbance spectrum using coefficient "C" (see the calculation in 6.7.2) to achieve an *in vitro* SPF (no UV dose) equal to the measured static *in vivo* SPF. Initial UVA-PF_0 is calculated using $A_0(\lambda)$ and C. A single UV exposure dose, *D*, is calculated, equal to 1,2 × UVA-PF_0 in J/cm^2, for each plate

6.1.5 Conduct UV exposure of the same sample as in 6.1.3, according to the calculated UV exposure dose *D*.

6.1.6 Measure the *in vitro* absorbance of the sunscreen product after UV exposure. Acquire the second UV spectrum with A(λ) data.