International Standard

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Cosmetics — Measurement of the sunscreen efficacy by diffuse reflectance spectroscopy

Cosmétiques — Mesurage de l'efficacité des produits de protection solaire par spectroscopie de réflectance diffuse

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

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Foreword

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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).

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Introduction

Exposure to solar ultraviolet radiation (UVR) is the main environmental source of acute and chronic damage to human skin. Skin cancer is the most prevalent form of cancer of the body and is primarily driven by exposure to sunlight. Protection against exposure to solar UVB and UVA radiation is, therefore, an important public health issue. The use of topically applied sunscreens is a critical part of holistic programs of consumer UVR protection, including the use of appropriate clothing, hats and minimizing exposure to the sun.

The sun protection factor (SPF) has historically been measured by an in vivo method (see ISO 24444) to communicate the magnitude of the protection provided by sunscreens from sunburning UVR. Other test methods have been developed and provided to assess the breadth and magnitude of the protection in the UVA portion of the sun's spectrum (see ISO 24442 and ISO 24443).

This test method given in this document is an alternative to ISO 24443 and ISO 24444 methods.

Invasive methods based on tests conducted on human beings are ethically problematic, time-consuming and very costly. Therefore, it has long been desired to develop alternative methods to assess both the magnitude and breadth of protection afforded by sunscreens that do not require invasive procedures and that reliably provide equivalent testing sensitivity and accuracy as the existing invasive in vivo testing methods.

The hybrid diffuse reflectance spectroscopy method described herein, provides a non-invasive optical assessment of the protection provided by topically applied sunscreen products as measured in situ on human skin as used by consumers, without requiring physiological responses and causing no physical harm to the test subject. By combining full spectrum in vitro spectroscopic measurements of the sunscreen, with optical measurements of the sunscreen transmission in the UVA on human skin, a hybrid spectrum is derived that provides full assessment of both magnitude and breadth of sunscreen protection in both the UVB and UVA regions of the sun's spectrum, correlating closely with in vivo SPF, in vitro UVA-PF and critical wavelength test results demonstrating equivalence of this test method against ISO 24444 and ISO 24443 methods.

Cosmetics — Measurement of the sunscreen efficacy by diffuse reflectance spectroscopy

1 Scope

This document provides a procedure to characterize the sun protection factor (SPF), UVA protection factor (UVA-PF) and critical wavelength (CW) protection of sunscreen products without requiring biological responses. The test method is applicable for emulsions and single-phase products. The method has not been evaluated for use with powder forms sunscreen products.

This document gives specifications to enable determination of the absolute spectral absorbance characteristics of a sunscreen product on skin to estimate sunburn and UVA protection. 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.

2 Normative references

There are no normative references in this document.

3 Terms, definitions and symbols

3.1 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/

3.1.1
absorbance
A
measure of the energy blocked, either by optical absorption or by physical scattering/reflection

3.1.2
absorbance spectrum
$A(\lambda)$
sunscreen optical absorbance at wavelength λ

Note 1 to entry: Logarithm to the base 10 of the reciprocal of the spectral transmittance τ(λ). $A(\lambda) = -[\log_{10} \tau(\lambda)]$.

3.1.3
absorbance by diffuse reflectance spectroscopy
absorbance by DRS
$A_{DRS}(\lambda)$
absorbance spectrum calculated from DRS as a function of wavelength λ

Note 1 to entry: The absorbance spectrum relevant to this document is 320 nm to 400 nm.

3.1.4
absorbance after hybridization
$A_{HDRS}(\lambda)$
final absorbance spectrum calculated from the hybridized signals as a function of wavelength λ after correction for photo-degradation

Note 1 to entry: The final absorbance spectrum is 290 nm to 400 nm

3.1.5
calibration factor
C_{cal}
correction applied to a measured quantity value to compensate for a known systematic effect

3.1.6
in vitro UV absorbance spectrum pre irradiation
in vitro absorbance before UV exposure (pre irradiation)
$A_{vt0}(\lambda)$
arithmetic mean in vitro absorbance spectrum of a sunscreen product measured before UV exposure

Note 1 to entry: The absorbance spectrum is 290 nm to 400 nm.

3.1.7
in vitro UV absorbance spectrum post irradiation
in vitro absorbance after UV exposure (post irradiation)
$A_{vt1}(\lambda)$
arithmetic mean in vitro absorbance spectrum of a sunscreen product measured after UV exposure

Note 1 to entry: The absorbance spectrum is 290 nm to 400 nm.

3.1.8
hybridization constant
C_{Ai}
scalar factor to adjust an in vitro spectrum A_{vt1} (λ) at each wavelength to the individual A_{DRSi}

3.1.9
critical wavelength
CW
λ_c
wavelength at which the area under the absorbance curve represents 90 % of the total area under the curve in the UV region

3.1.10
dose
D
UVA radiant exposure dose for pre-irradiation of sunscreen products (1,2 x UVA-PF_{DRS} J/cm^2)

3.1.11
wavelength step
$d\lambda$
differential of integration (1 nm)

3.1.12
diffuse reflectance spectroscopy
DRS
technique used to measure the remitted light from skin or skin remittance.

Note 1 to entry: Using this technique, the UVA absorbance spectrum of a sunscreen product applied on skin in vivo can be determined.

Note 2 to entry: The term “light” is used generically to describe electromagnetic radiation from both UV and visible wavelengths of optical spectrum throughout the document. It is differentiated as needed in specific sections of the document.

Note 3 to entry: The UV energy that is measured is not energy reflected from the surface of the skin or the applied sunscreen. The UV energy being measured has passed through the sunscreen, entered the surface of the skin, and been scattered therein. Some of this energy is remitted back to the surface of the skin through the sunscreen a second time and picked up by the DRS optical probe. The term "remittance" is used throughout this document whereas historical use of the term "reflectance" has had precedence in published literature.

3.1.13
erythema action spectrum
E(λ)
relative effects of individual spectral bands of an exposure source causing an erythema response in skin

Note 1 to entry: See Annex E.

3.1.14
hybrid diffuse reflectance spectroscopy
HDRS
method to evaluate the protection provided by a sunscreen product applied on skin in vivo wherein the UVA Protection Factor is measured by DRS and the UVB part of the spectrum by in vitro thin film spectroscopy, and the two spectra are merged to form a hybrid absorbance spectrum

Note 1 to entry: The spectral distributions determined by the two different methods are merged to form the hybrid spectral absorption $A_{\mathrm{HDRS}}(\lambda)$.

3.1.15
hybridization wavelength
HW
λ_{HW}
wavelength at which the in vivo DRS spectrum and the in vitro absorbance spectrum are merged

3.1.16
PPD action spectrum
P(λ)
relative effects of individual spectral bands of an exposure source to cause persistent pigment darkening (PPD)

Note 1 to entry: See Annex E.

3.1.17
sun protection factor by hybrid DRS
$\mathbf{SPF_{HDRS}}$
SPF of a sunscreen product calculated from hybridized UV absorbance spectrum adjusted by spectral ratio of photo-degradation (SRPD) (λ)

3.1.18
spectral ratio of photo-degradation (λ)
$\boldsymbol{S_{\mathrm{RPD}}(\lambda)}$
ratio of the in vitro absorbance spectra (post- and pre-irradiation) representing the photo-degradation of the sunscreen product as function of wavelength

Note 1 to entry: SRPD(λ) spectrum is 290 nm to 400 nm

3.1.19
subsite
area within a test site where the DRS probe is placed to take the individual skin remittance measurement denoted by index j

3.1.20
test site
defined area of the skin to which a test sunscreen material is applied and where DRS measurements are conducted

3.1.21
Student's t value
t
two tail Student's t-test critical value for 0,05, with n-1 degrees of freedom

3.1.22
transmittance spectrum by DRS
$T_{DRS}(\lambda)$
in vivo transmittance spectrum of a sunscreen product calculated from DRS as a function of wavelength λ

Note 1 to entry: The in vivo transmittance spectrum is 320 nm to 400 nm.

3.1.23
UVA protection factor by DRS
UVA-PF_{DRS}
initial UVA protection factor of a sunscreen product calculated using the measured in vivo absorbance spectrum from DRS before correction for photo-degradation

3.1.24
UVA protection factor by HDRS
UVA-PF_{HDRS}
UVA protection factor of a sunscreen product calculated from hybridized UV absorbance spectrum adjusted by SRPD(λ)

3.2 Symbols

I_u	irradiance of remitted UVA from unprotected skin with polychromatic DRS measurement device
I_p	irradiance of remitted UVA from sunscreen-treated skin with polychromatic DRS measurement device
i	index for individual subject
ITA°	individual typology angle
$I_{rad,UVA}$	calibrated UVA irradiance
j	index for individual test subsite
k	index for individual PMMA plate (in vitro measurement)
l	number of subsite measurements on a PMMA plate
m	index for individual spot of in vitro measurement
n	number of context dependent elements (these elements can be the subjects, the spots on a PMMA plate or the valid test results)
$R_p(\lambda)$	irradiance of remittance spectrum (320 nm to 400 nm) of product-treated skin
$R_u(\lambda)$	irradiance of remittance spectrum (320 nm to 400 nm) of unprotected skin
s_i	scalar multiplier for scaling in vitro spectra for an individual
$S(\lambda)$	spectral irradiance of the light source used to expose the plates
stdev, σ	standard deviation of the ln transformed $UVA\text{-}PF_{HDRSi}$ values or the ln transformed SPF_{HDRSi} values (context dependent)
$T_{vt0}(\lambda)$	in vitro transmittance spectrum (290 nm to 400 nm) before UV-exposure

$T_{vt1}(\lambda)$	in vitro transmittance spectrum (290 nm to 400 nm) after UV-exposure
$UVA\text{-}PF_{DRS}$	initial UVA protection factor of a sunscreen product calculated using the measured in vivo absorbance spectrum from DRS before correction for photo-degradation
$UVA\text{-}PF_{HDRS}$	UVA protection factor of a sunscreen product calculated from hybridized UV absorbance spectrum adjusted by SRPD
$UVA\text{-}PF_{vt0}$	in vitro UVA Protection Factor of a sunscreen product calculated using the absorbance spectrum A_{vt0}
$UVA\text{-}PF_{vt1}$	in vitro UVA Protection Factor of a sunscreen product calculated using the absorbance spectrum A_{vt1}
vt	index for in vitro
λ_c	critical wavelength (including calibration factor)
$\lambda_{c'}$	raw critical wavelength
λ_{HW}	hybridization wavelength

4 Principle

This method provides a hybrid (in vitro and in vivo) testing procedure to characterize UV protection provided by sun care preparations. The primary outputs of this test procedure are measures of the spectral absorbance characteristics of a sunscreen product. Different approaches to generate hybridized absorbance spectra are available, i.e. monochromatic as well as polychromatic measurement techniques.

The UVA-PF can be predicted by diffuse reflectance spectroscopy (DRS) measuring the UVA absorbance of skin (320 nm to 400 nm) and has been shown to correlate with in vivo assessment using ISO 24442 (see also References [5] and [6]), as well as UVA-PFs using ISO24443 (see References [7] to [13]. Because of the high UVB absorbance characteristics of the stratum corneum and epidermis, the human skin does not remit enough UVB radiation for absorbance measurements. Therefore, the spectral absorbance 'shape' in the UVB region must be assessed separately by in vitro thin film transmittance spectroscopy. To account for sunscreen products photo-instability of the sunscreen under evaluation, the same approach used in ISO 24443 is applied. The in vitro thin film sunscreen sample is subjected to a controlled dose of simulated sunlight radiation to determine the shape of the spectrum after UV exposure which is used to adjust the hybrid diffuse reflectance spectroscopy (HDRS) absorbance spectrum.

In order to obtain a full UV absorbance spectrum, the in vitro absorbance is scaled to match the DRS absorbance values and then the in vitro UVB portion is mathematically attached to the UVA portion from the DRS technique. This HDRS absorbance spectrum is then used to calculate the UVA-PF, SPF and critical wavelength (CW) of the sunscreen products being tested[10],[11].

Samples submitted for testing should not have a SPF or UVA-PF target or other protection category description.

5 Apparatus and test method

5.1 In vitro UV spectrophotometer

The in vitro UV spectrophotometer shall follow the specifications and calibration procedure as described in Annex B.

5.2 In vitro substrate/plate

The substrate/plate is the material to which the test product is applied for the in vitro part of this method. Polymethylmethacrylate (PMMA) plates with one rough side of the substrate shall be used and prepared as specified in Annex D.

5.3 In vivo diffuse reflectance spectrometers (DRS) specifications

Common elements for the monochromatic and polychromatic DRS systems include the following.

5.3.1 Optical light source

A short arc xenon bulb emitting continuous radiation over the range of 290 nm to 400 nm is recommended. A maximum exposure dose of 10 J/m^2 eff dose shall not to be exceeded for any measurement subsite. The maximum exposure irradiance at skin surface shall be less than 5 mW/cm^2. Calibration of radiometers for this evaluation shall be done in accordance with Annex C. The spectral irradiance of the illuminating source shall be evaluated once per year to validate that the maximum exposure irradiance and dose are not exceeded during a subsite measurement.

5.3.2 DRS illumination/Collection fibres

A UV grade fused silica bifurcated fibre probe comprised of a fibre arrangement as described in Annex I, with approximately 1,5 m common probe length and two 0,5 m short arms (one for excitation and one for emission) is recommended. The area of the common optical probe shall be less than 1,2 cm^2. The common bundle shall have ≥ 800 individual fibres with a ratio of illuminating fibres to collection fibres between 45:55 and 55:45.

Annular fibre optic bundles: the centre illuminating fibre bundle shall have a 200 µm spacer between it and the surrounding collection fibres.

Randomized fibre optic bundles: ≥ 95 % of the illuminating fibres shall be adjacent to a collection fibre with a minimum spacing between the centres of adjacent fibres of 280 µm. See Annex I for the fibre configuration.

5.3.3 Detector system

A bi-alkali photo multiplier cathode detector (PMT) is recommended. To obtain a better signal to noise ratio it is recommended that the detector be cooled (i.e. -20 °C). The PMT temperature is recommended to be approximately 40 °C lower than room temperature.

5.3.4 Sensitivity requirements

A linear response detection shall be at least 5 decades (100 000:1), (6 decades (1 000 000:1) are recommended) in the range of 290 nm to 400 nm. Usually, this can be achieved by a double monochromator spectrophotometer with a good stray light rejection and an appropriated, cooled PMT. The chosen voltage of the PMT (gain) should allow a high sensitivity at lower wavelengths (<320 nm) and avoid an overload of the PMT at higher wavelengths (>370 nm).

5.3.5 Monochromatic DRS system monochromators

Monochromators used for excitation or emission can be single or double monochromators with a wavelength accuracy of ±0,1 nm. The ratio of stray light (at a distance from the peak wavelength that is 10 x the bandwidth at half maximum of the laser line peak irradiance), to the peak irradiance of a laser line shall be less than 5 x 10^{-5}. Furthermore, installed filters shall be used to block any visible light from entering the photomultiplier detector. The system shall have the specifications as described in 5.3.1 to 5.3.4.

5.3.6 Polychromatic DRS system

In vivo polychromatic DRS measurements shall be conducted using a light source with spectral output as described in Annex E and a PMT detector system with a response spectrum similar to the human persistent

pigment darkening (PPD) action spectrum as described in Annex E. Any differences between the PMT detector system X spectral output of the source and the human PPD action spectrum X spectral output of the source shall be corrected with a spectral mismatch calculation routine. The system shall have the specifications as described in 5.3.1 to 5.3.4.

A visible light ("black glass") blocking filter is recommended to be included before a broad-spectrum photo multiplier cathode detector to eliminate measurement of visible fluorescence using the polychromatic DRS system and to shape the action spectrum of the detector to be similar to the skin's PPD action spectrum as described in Annex E.

5.4 Monitoring the DRS systems

5.4.1 Monochromatic system

Wavelength accuracy shall be checked regularly either with a holmium oxide filter (according to B.2) or with a low-pressure mercury, "cold quartz" or equivalent lamp following usual calibration procedures.

A periodic inspection of the DRS wavelength accuracy and fibre output irradiance at least once per year shall be conducted using calibrated equipment by a trained, competent and suitably qualified person (internal or external). The optical fibre bundle shall be inspected at least once per year to validate compliance with 5.3.2 and to check for broken fibres.

5.4.2 Polychromatic system

The illumination beam of the polychromatic DRS system shall be checked periodically to assure conformance to the specifications described for the UVA radiation source in Annex E. A spectroradiometric inspection of the spectrum shall be conducted at least once per year by a trained, competent, and suitably qualified person (internal or external) using a system calibrated to a traceable national or international calibration standard lamp. The optical bundle shall be inspected at least once per year to validate compliance with 5.3.2 and to check for broken fibres.

5.5 Test method

5.5.1 General

DRS measurements and product application assessment are recommended to be carried out in stable conditions, with the room temperature maintained between (23 ± 3) °C.

5.5.2 Subject exclusion criteria

Exclusion criteria shall be checked before testing.

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

a) children or persons below the locally legal age of consent;

b) subjects with systemic dermatological conditions in the test area (including dysplastic nevi);

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

d) subjects with average individual typology Angle (ITA°) <28°;

e) subjects having UV-exposures applied to the test sites, [i.e. SPF (ISO 24444, UVA-PF (ISO 24442], photo-allergy or photo-toxicity tests, or sun-tanning) within the past 8 weeks and having pigmentation marks or erythema in the test sites.

5.5.3 Skin colour of the test subjects

Test subjects shall have an ITA° value ≥ 28° as determined by colorimetric methods with the same acceptance criteria for number of subjects in each of the three ITA° bands (28°to 40°, 41°to 55°, and ≥56° as stipulated in ISO 24444:2019, 5.1.2). The average of the subjects making up a test panel shall have an ITA° between 41° and 55°. When possible, subjects with ITA°s in each of the three ITA° bands, 28° to 40°, 41° to 55°, and >56° (ITA°value shall be truncated with no significant digits). Where this is not possible, there shall be at least three individuals in each of two of the three ITA° bands described in the previous sentence.

The test sites intended for DRS measurements shall be free from blemishes and hair and have an even colour tone with no variation in ITA° greater than 5° from each other with < 5° difference in ITA° within a given test site. Hair may be shaved up to 3 days prior to the test date, but not thereafter. If necessary, hair may be clipped or cut with scissors on the test date.

5.5.4 Frequency of participation in tests

Subjects may participate in a HDRS-test at most once per seven days (to ensure clearance of applied sunscreen).

5.5.5 Number of test subjects

Valid results from at least 10 subjects is required. A maximum number of valid results shall be 20. In order to achieve between 10 and 20 valid results, a maximum of five individual results may be excluded from the calculation of the mean values based on statistical outlier analysis (see Annex F).

5.5.6 Ethics and consent

All testing shall be done in accordance with the Declaration of Helsinki[14]. Informed, written (signature) consent shall be obtained from all test subjects and retained.

5.5.7 Study preparations

All equipment to be used for measuring and exposing the samples shall be turned on to warm up for at least 20 min prior to initiating measurement procedures or according with manufacturer instructions.

Devices used to apply a measured amount of product to the skin (e.g. micropipettes, syringes, weigh boats, etc.) shall deliver 2,00 mg/cm^2 ± 0,05 mg/cm^2 of the sunscreen. A finger cot shall be used for spreading the sunscreen on the skin for all products except in cases when use of a finger cot interferes with even application of the product. Sunscreen formulation application should follow procedures as described in Annex J. 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.

5.5.8 Unprotected skin remittance measurement

5.5.8.1 General

Test sites for sunscreen application are to be chosen wherein the skin colour is uniform, without pigmentation marks or mottled pigmentation, sun tanned areas, scars, or other skin lesions. Test sites shall be placed on the back according to Annex J. The test sites shall be at least 30 cm^2 in area (e.g. 5 cm x 6 cm) and the maximum shall be 60 cm^2. The corners of the test sites shall be marked with permanent marker or skin marker or a stamp template with non-absorb material. The identity code of the test site can be marked on the skin.

Measurements can be performed by directly placing the DRS optical probe on the subsite within the test site (constant and light pressure). Measurements may be made anywhere within test site as long as the measurement sites do not overlap. The subsequent steps are related to monochromatic and polychromatic DRS measurement and product application.