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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

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Foreword

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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 24442 was prepared by Technical Committee ISO/TC 217, *Cosmetics*.

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/FDIS 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. 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 UVAPF test methods as developed by the JCIA. Modifications have been made to attempt to harmonize with other methodologies 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 an *in vivo* method for assessment of the UVA protection factor (UVAPF) of topical sunscreen products. This International Standard is applicable to cosmetics, drugs and other products intended to be topically applied to human skin, including any component able to absorb, reflect or scatter UV rays.

It provides a basis for the evaluation of sunscreen products for the protection of human skin against UVA radiation from solar or other light sources.

2 Terms and definitions

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

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

NOTE UVB = 290 nm to 320 nm; UVA = 320 nm to 400 nm (UVA II = 320 nm to 340 nm; UVA I = 340 nm to 400 nm).

2.2
erythema
reddening of the skin caused by UV radiation

2.3
persistent pigment darkening
PPD
skin darkening that persists more than 2 h after the end of UVA exposure

2.4
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

NOTE The MPPDD on unprotected skin is referenced as “MPPDDu”, and the MPPDD on sunscreen-protected skin is referenced as “MPPDDp”.

2.5
individual Ultraviolet A protection factor
UVAPFi
ratio of the minimal persistent pigment darkening dose on product-protected skin (MPPDDp) to the minimal persistent pigment darkening dose on unprotected skin (MPPDDu) of the same subject:

$$\mathrm{UVAPFi} = \frac{\mathrm{MPPDDp}}{\mathrm{MPPDDu}}$$

2.6
UVA protection factor of a product
UVAPF
arithmetic mean of all valid individual UVAPFi values obtained from all subjects in the test

2.7
test area
back between the scapula line and the waist

2.8
test site
area of skin to which a test product or reference sunscreen is applied within the test area

NOTE The area used to determine the MPPDDu is also a test site.

2.9
test subsite
skin areas within a test site exposed to UVA radiation

3 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 persistent pigment darkening (PPD) responses of the skin as the end point for evaluating transmitted UVA radiation. The test is restricted to the area of the back of selected human subjects. An area of each subject's skin is exposed to UVA light without any protection and another (different) area is exposed after application of the sunscreen product under test. One further area is exposed after application of a reference UVA sunscreen formulation, which is used to validate the procedure.

To determine the UVAPF, incremental series of UVA exposures are delivered to five or six small subsites on the skin to induce darkening responses. These responses are visually assessed for pigment darkness 2 h to 24 h after UVA exposure, by the judgement of a trained evaluator. The minimal persistent pigment darkening dose (MPPDD) for unprotected skin (MPPDDu) and the MPPDD obtained after application of a sunscreen product (i.e. the MPPDD for product-protected skin, MPPDDp) are determined on the same subject on the same day. An individual sun protection factor (UVAPFi) for each subject tested is calculated as the ratio of MPPDDp/MPPDDu.

4 Test subjects

4.1 Selection of test subjects

4.1.1 General

For subject inclusion and exclusion criteria, refer to Annex A.

4.1.2 Age restriction

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

4.1.3 Skin phototype of test subjects

The skin of subjects shall be Fitzpatrick phototype[2] II, III and IV. Alternatively, the colorimetric ITA° value of subjects shall be within the range of 20° and 41°.

4.1.4 Frequency of participation in tests

Since a sufficient interval after a previous test is needed in order to allow for reversal of skin tanning resulting from that previous test, a test site that has been exposed to UV should not be used in a subsequent test before two months have elapsed and the site is free of any sign of previous pigmentation marks.

4.1.5 Consent

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

4.1.6 Ethical aspect

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

4.2 Number of subjects

The test subjects shall be required to provide a minimum of 10 valid UVAPFi values and a maximum of 20 valid results. A maximum of five individual invalid results may be excluded from the calculation of the mean UVAPF, but each exclusion shall be justified according to 10.3.3 or other non-compliance with protocol. Consequently, the total number of subjects will be between a minimum of 10 and a maximum of 25 subjects.

In order to determine the number of subjects, the 95 % confidence interval (CI) shall be taken into account. The 95 % confidence interval should lie within ±17 % of the mean UVAPF, and a minimum of 10 subjects is required. Otherwise, the number of subjects is increased stepwise from 10 until the statistical criterion is met (up to a maximum of 20 valid results or a maximum of 25 subjects tested). If this statistical criterion is not reached after 20 valid results from a maximum of 25 subjects, then the entire test is rejected, and a new test shall be initiated. For details of statistical definitions, sequential testing procedure and calculations, refer to Annex D.

5 Reference sunscreen formulae

The method is controlled by the use of a reference sunscreen formulation to verify the test procedure. If all test samples have an expected UVAPF value below 12, a control sample S1 sunscreen formula (see Annex C) may be used in every study to confirm the reliability of the results obtained for the test samples. The mean UVAPF value of the control sample is 4,4. The results of the UVAPF of the S1 control shall lie between 3,8 and 5,0 or the test is invalid and shall be repeated.

Reference S2 sunscreen formula[4] (see Annex C) shall be used in a study if any test sample has an expected UVAPF of 12 or above. The mean UVAPF for the reference sample S2 is 12,7. The test results of the reference S2 UVAPF shall lie between 10,7 and 14,7 or the test is invalid and shall be repeated. The reference S2 may be used to validate any product test.

Only one reference sunscreen formula is required for each test.

6 UVA source

6.1 Spectral characteristics

The source of UVA radiation shall be a xenon arc lamp solar simulator (of which the spectrum encompasses primarily UVA radiation from 320 nm to 400 nm) with a continuous spectrum. 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, and which yield the performance specifications given in Table 1.

The maximum level of visible and infrared (IR) radiation in the source beam shall be less than 5 % of the total source output. 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.

The spectrum shall be measured by an expert using a spectroradiometer that is traceable to a recognized standard lamp source.

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

6.2 Maintenance and monitoring the UV solar simulator output

6.2.1 Radiometry

Before UV exposure of each test site, the UV irradiance should be checked with a radiometer calibrated against a spectroradiometric measurement of the solar simulator output.

6.2.2 Spectroradiometry

It is recommended that a complete spectroradiometric check (UVA and UVB) of output spectrum and intensity be made by the laboratory at least once every 18 months, or after 3 000 h of lamp running time, and after changing any significant physical (optical) component of the solar simulator. It is strongly recommended that an independent expert conduct this periodical inspection.

The simple use of specific filters is not in itself adequate assurance that the UV output is of the correct quality. Detailed instructions for ensuring correct lamp output are given in Annex B.

6.3 Beam size and uniformity

6.3.1 General

Beam size for each exposure subsite shall be at least 0,5 cm^2. The intensity of the beam shall be as uniform as possible.

6.3.2 Large beam sources

When a large-beam lamp is used to simultaneously expose several subsites, the minimum beam irradiance, at any UV exposure site, shall be no more than 10 % lower than the maximum beam irradiance at any UV exposure site. If the variation exceeds 10 %, then appropriate compensation for different irradiance should be made in the exposure time on each UV exposure site.

6.3.3 Small beam sources

For a small beam UV source, an uneven skin darkening (such as a half-moon shape) indicates that the irradiance is not uniform and the delivery system shall be realigned or corrected.

6.4 Total irradiance (UV, visible and near-infrared rays)

The test conductor shall confirm that the total irradiance shall not exceed 1 600 W/m^2 (reciprocity has been tested over the range of 370 W/m^2 to 1 440 W/m^2)[3].

When total irradiance is strong, an excessive feeling of heat or pain might be induced in the irradiated skin of subjects. Therefore, it shall be confirmed that the maximum irradiance that will be used (UV, visible and near-infrared rays) will not induce an excessive feeling of heat in the skin prior to conducting a UVAPF test.

7 Product application quantity and procedure

7.1 General

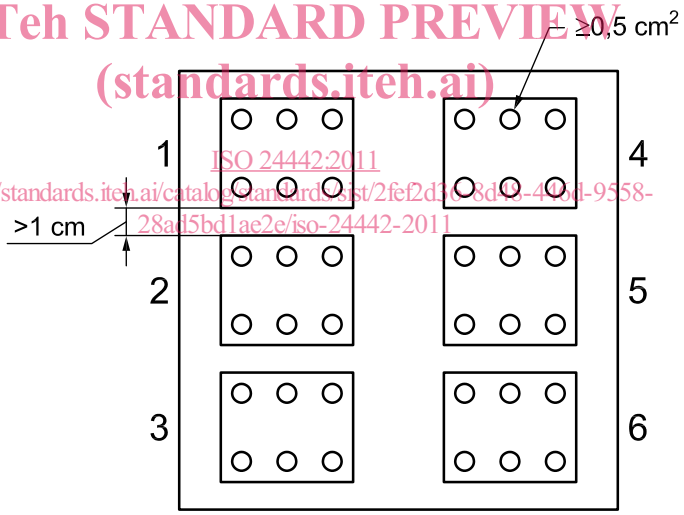
The application of the products should be made by a trained technician. The room temperature shall be between 18 °C and 26 °C. The use of a finger cot is optional but recommended. If employed, a new finger cot shall be used for each new application of product and should not be pre-saturated with the test product. When a naked finger is used, the finger shall be cleaned between product applications. All products should be homogeneous and should be shaken if necessary, before weighing, to ensure uniform dispersion.

7.2 Position of the subject

Product shall be applied to subjects in the same position as will be utilized for the irradiation procedure (sitting or prone). Powder samples should be tested in the prone position to prevent the samples from falling off the surface of the skin.

7.3 Defining test sites

Areas of at least 30 cm^2 to at most 60 cm^2, located between the scapula line and the waist, shall be delineated using a template and a special skin marker with a distance of at least 1 cm between each test site. The number of sites shall be restricted to no more than six. The location of the products on the sites and the unprotected site shall be randomized on the subject's back (see Figure 1). Before application of the test product or standard formula, the test site can be cleaned with a clean, dry cotton pad or equivalent material.



Keys

1 product 1
2 reference sunscreen
3 product 2
4 product 4
5 MPPDDu (no topical application)
6 product 3

Figure 1 — Example of arrangement of test sites

7.4 Application procedure

7.4.1 Product application technique — Liquid-type products, e.g. lotions, milks, creams, sticks, sprays

7.4.1.1 The amount of product to be applied is to be weighed in a syringe or pipette or, alternatively, in another device such as a watch glass or weigh boat (container). A method of weighing by loss should be used.

7.4.1.2 Care shall be taken to prevent evaporative loss of volatile components when the product is being weighed and before application on the skin. It is important that the total quantity of weighed product be transferred to the product application site.

7.4.1.3 The product is then dispensed in small droplets (approximately 15 per 30 cm^2, 30 per 60 cm^2) over the whole test site at a dose of 2,00 $mg/cm^2 \pm 0,05$ mg/cm^2 delivered.

7.4.1.4 The product should be gently spread with a finger cot or a clean finger using circular and then linear movements (up and down), without excessive pressure. The time used to spread the product on the test site shall be from 20 s to not more than 50 s.

7.4.1.5 If necessary, in case of uneven application, application may be repeated on a new test site.

7.4.2 Product application technique — Powders

7.4.2.1 Purified water or another suitable solvent that has no other UV protection properties may be applied on the skin before the powder application to help the sample adhere to the application site.

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

7.4.2.3 The accumulated powder is tapped and then spread over the whole test site using a finger with or without a finger cot.

7.4.2.4 Alternatively, the tip of a preloaded cosmetic application puff may be used instead of a finger. In this case, it is important to verify that 2,00 $mg/cm^2 \pm 0,05$ mg/cm^2 of test powder remains on the skin after spreading by weighing the application puff.

8 Determination of minimal persistent pigment darkening doses (MPPDD)

8.1 UV exposure timing and subject position

Exposure of the test site to the sequence of UV doses shall start no sooner than 15 min and no more than 30 min after the application of the product(s). The position of the subjects during the whole exposure period shall be the same as when the product was 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.

8.2 Determination of the minimal pigmenting dose on the unprotected test sites (MPPDDu) using a multiple-beam solar simulator

8.2.1 When a multiple-beam solar simulator is used, UVA radiation is conducted onto multiple UVA exposure sites (typically six), each of which receives an independent dose of radiation of identical spectrum, but with different intensity. The individual UVA exposure sites are typically between 8 mm and 10 mm in diameter.

8.2.2 Measure and adjust the UVA irradiance of each optical beam to obtain a geometric progression of 25 % (0,64, 0,8, 1, 1,25, 1,56, 1,95) using a radiometer calibrated with the UVA source.