TECHNICAL REPORT

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Cosmetics — Sun protection test methods — Review and evaluation of methods to assess the photoprotection of sun protection products

Cosmétiques — Méthodes d'essai de protection solaire — Revue systématique et évaluation des méthodes usuelles de mesure de la protection solaire fournie par les produits de protection solaire

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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.

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.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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/TR 26369 was prepared by Technical Committee ISO/TC 217, *Cosmetics*.

Cosmetics — Sun protection test methods — Review and evaluation of methods to assess the photoprotection of sun protection products

1 Scope

This Technical Report reviews and evaluates the methods which are currently used to assess, for regulatory or self-regulatory purposes, the photoprotection of sun protection products applied on the human body.

It is applicable to SPF and UVA protection, and both *in vivo* and *in vitro* methods.

This Technical Report does not include the aspects of labelling in a wide sense.

2 Terms and definitions

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

2.1
ultraviolet
UV
electromagnetic radiation with a wavelength shorter than that of visible light, but longer than soft X-rays and so named because the spectrum consists of electromagnetic waves with frequencies higher than those that humans identify as the color violet (purple)

NOTE In this Technical Report the following wavelengths are considered: UVA: 320 nm to 400 nm; UVB: 290 nm to 320 nm.

2.2
sun protection factor
SPF
⟨of a sunscreen⟩ laboratory measurement to assess the effectiveness of sunscreens against UV erythema

NOTE 1 The higher the SPF, the more protection a sunscreen offers.

NOTE 2 The SPF is a ratio between the ultraviolet dose required to produce minimal erythema reaction (redness) in protected skin (skin with sunscreen) compared to unprotected skin (skin without any sunscreen).

3 Principle

This systematic review and evaluation of the methods are conducted for development of those ISO Standards which assess the photoprotection provided by sun protection products applied on the human body. It will serve as a technical/scientific framework to identify the most suitable methods for standardization.

The key parameters and elements are listed in Tables 1 to 6 in order to enable an easy comparison of the methods.

4 Sun protection test methods

4.1 SPF *in vivo*

The SPF *in vivo* methods currently used are given in Table 1.

4.2 SPF *in vitro*

The SPF *in vitro* methods based on transmittance evolved from the Diffey proposal and new methods based on measurement of free radicals or use of skin biopsies are given in Tables 2 and 3. The relevant parameters of methods based on transmittance are given in Table 4.

4.3 UVA *in vivo*

The methods reviewed by ISO/TC 217 are given in Tables 5 and 6.

Table 1 — SPF *in vivo* methods currently used

Parameters	International 2006 [1] [a]	FDA 1999 [2]	Australia 1998 [3]
UV definition (UVB, UVA)	UVB: 290 nm to 320 nm UVA: 320 nm to 400 nm UVAII 320 nm to 340 nm UVAI 340 nm to 400 nm	UVB: 290 nm to 320 nm UVA: 320 nm to 400 nm	Solar UVR: 290 nm to 400 nm UVB: 290 nm to 320 nm UVA: 320 nm to 400 nm
Volunteers selection			
Ethical considerations	Helsinki, national regulations, medical status	Not defined	Medical questionnaire
Age limitation	Yes, excluded below age of consent	Not defined	Not defined
Informed consent	Yes, with signatures	Yes	Yes
Exclusion criteria	Pregnant, lactating women Photosensitizing medication Dermatological problems, history of abnormal response to sun Tanning beds No sun damage, marks, blemishes or nevi	Skin disease, abnormal responses to UV, phototoxic or photo-allergic response, medication (topical or systemic) known to produce abnormal sunlight responses Sunburn, scars, active dermal lesions and uneven skin tones on the areas to be tested	Abnormal response to medication, UV radiation, allergies to topically applied cosmetics Phototoxic or photosensitizing medication
Test subjects			
Skin phototype and skin colour	Fitzpatrick skin type (s) I, II, III or skin colour (ITA° value > 28° very fair, fair-skin and intermediate skin colour) and untanned on the test area	Phototypes I, II, III Fair skin colour	Phototypes I, II, III Fair skin colour
Test area	Back, between scapula line and waist Skeletal protrusions and extreme areas of curvature should be avoided	The back between the beltline and the shoulder blade (scapulae) and lateral to the midline	Back, clean dry skin, without any suntan or sunburn, active dermal lesions, excessive hair, uneven skin tones
Time, interval between two tests	No less than 2 months, sufficient interval for reversal of skin tanning until the site is clear	Not defined	Not defined

[a] The numbers in brackets refer to the Bibliographic references.

Table 1 (*continued*)

Parameters	International 2006 [1]	FDA 1999 [2]	Australia 1998 [3]
Source of UV radiation			
Solar simulator Filtration	Continuous emission spectrum with no gaps or extreme peaks Stable output Xenon Arc lamp recommended with dichroic mirror and WG320 + UG11/1 mm	Continuous emission spectrum 290 nm to 400 nm, similar to sunlight at sea level, 10° zenith angle < 1 % energy < 290 nm ⩽ 5 % energy > 400 nm Stable output after appropriate warm-up time	Xenon arc is preferred No peak in UVB; continuation in the UVA WG320 filter, dichroic mirror or heat absorbing filter
Acceptance limits %RCEE UVA2/UVA1	% RCEE defined in different bands W.L. range: RCEE%: ⩽ 290 nm < 0,1 % 290 nm to 300 nm 1,0 % to 8,0 % 290 nm to 310 nm 49,0 % to 65,0 % 290 nm to 320 nm 85,0 % to 90,0 % 290 nm to 330 nm 91,5 % to 95,5 % 290 nm to 340 nm 94 % to 97 % 290 nm to 400 nm 99,9 % to 100 % UVAII ⩾ 20 % UVAI ⩾ 60 % of the total UV irradiance to ensure that appropriate amounts of UVA radiation are included	Not defined	< 0,01 % < 290 nm “Red” & “blue” acceptance limits (± 4 nm): graph
Irradiance uniformity	As uniform as possible, no more than 10 % for large beam	Within 10 %	Uniformity of spot appearance (no half-moon shape)
Total irradiance	Lower than 160 mW/cm^2	Not defined	Not defined

Table 1 (*continued*)

Parameters	International 2006 [1]	FDA 1999 [2]	Australia 1998 [3]
(Spectro) radiometry			
Checking of UV source emission spectrum by spectroradiometry	Spectroradiometric check at least once a year by an independent expert or each time a significant physical (optical) component is changed Colipa guidelines “monitoring of UV light sources”	Measured periodically with an accurately-calibrated spectroradiometer system or equivalent instrument	Not defined
Radiometry	Before exposure of each test site, checking with a calibrated radiometer	Not defined	Before and after each test series, variations kept to a minimum; UV monitor response restricted to UV range recommended
Test site description			
Mode of delineation	Skin marker and/or template made from a non-absorbent material	Outlined with ink	Means which do not interfere with the test or harm the subject
Application surface	Between 30 cm^2 and 60 cm^2	Minimum 50 cm^2, e.g. 5 × 10 cm	Minimum of 30 cm^2, maximum not defined
Space between test sites	Minimum distance of 1 cm	Not defined	Not defined
Test site pre-treatment	Possible with dry cotton pad	Not defined	Warm water and toweling

Table 1 (*continued*)

Parameters	International 2006 [1]	FDA 1999 [2]	Australia 1998 [3]
Product quantity and application			
Quantity applied	2 mg/cm^2 ± 2,5 % Sensitivity of the balance, at least 0,1 mg Method of weighing by loss	2 mg/cm^2	2 ± 0,1 mg/cm^2
Position of volunteers	Position in a way to ensure that the complete amount of test product is evenly applied and remains on skin, seated or prone position, excepted for powder products tested only in prone position	No indication, same position as delineation?	Not defined
Mode of delivery	Lotion, liquid, milk, cream, spray: syringe/pipette droplets on the whole test site Spreading time in the range of 20 s to 50 s, low pressure of application Powders: spatula, finger, Applicator puff. + water CD-ROM for application procedure training for emulsions and powders	Volumetric syringe Pastes and ointments shall be weighed	Weighing boat or weighed syringe Spreading according to the sponsor instructions Product film lightly and evenly applied with uniform thickness Validation of the method by the test facility
Room temperature, air conditioning	Room temperature between 18 °C and 26 °C	Not defined	Air-conditioned, 20 °C and 25 °C
Drying time	15 min to 30 min	At least 15 min	At least 15 min
Finger cot	If appropriate	Yes	Yes recommended, other appropriate means may be used
Randomization	Yes	Yes	Not defined
Blinded application	Not defined	Yes	Not defined

Table 1 (*continued*)

Parameters	International 2006 [1]	FDA 1999 [2]	Australia 1998 [3]
UV exposures			
Position of volunteers	Position shall be the same for product applications, for UV exposure and for MED assessment	Upright or prone position	Seated or prone position
Exposure sub-site surface	At least 0,5 cm^2, recommended 1 cm^2 Distance between sub-sites at least 0.8 cm	$\geqslant$ 1 cm^2	Approximately 1 cm^2 Distance between sub-sites at least 1 cm and 1 cm from any edge of the test site
Number of sub-sites	Minimum of 5 for MEDu and MEDp	5 for the unprotected area 7 for the protected areas	Minimum of 5 for MEDu and MEDp
Provisional individual MEDu	The day prior to the product testing, determined again on the same day as the test sunscreens or estimation of the MEDu by colorimetry (ITA°)	Usually the day prior to testing a product Determined again on the same day as the test sunscreens	Prediction by experienced tester or provisional MEDu the day before
Progression of UV dose	Geometric progression of either ($1,25^n$) or ($1,12^n$) for the unprotected area. For the protected areas, a minimum of five sub-sites centered on the expected SPF × MEDu shall be exposed with a geometric progression of either ($1,25^n$) or ($1,12^n$) A maximum progression of 1,12 must be used for expected SPF > 25	Geometric progression ($1,25^n$) for the unprotected area For the protected areas geometric series of five exposure where the middle exposure is placed to yield the expected SPF plus two other exposures placed around the middle exposure According to the expected SPF (X) SPF < 8: 0,64, 0,8, 0,9, 1,1 .1, 1.25, 1,56X SPF 8 to 15: 0,69, .83, 0,91, 1, 1,09, 1,2, 1.44X SPF > 15: 0,76, 0,87, 0,93,1, 1,07,1,15, 1,32X	Unprotected MED re-determined with a dose range of ca 0,6 to 1,5 provisional MEDu For protected skin the dose range is multiplied by the expected SPF Increments between sub-sites no more than 1,25 $\leqslant$ 1.118 for SPF $\geqslant$ 25
Randomized UV exposure	Not defined	Yes if only one product is being tested	Not defined
Product removal	Products may be removed gently using a cotton pad and mild lotion	Not defined	Not defined
Ambient conditions	18 °C to 26 °C	Not defined	Not defined

Table 1 (*continued*)

Parameters	International 2006 [1]	FDA 1999 [2]	Australia 1998 [3]
Response description			
Definition of response	The MED is the lowest UV dose that produces the first perceptible unambiguous erythema with defined borders appearing over most of the field, 16 h to 24 h after UV exposure	The MED is the quantity of erythema-effective energy required to produce the first perceptible unambiguous redness reaction with clearly defined borders at 22 h to 24 h post-exposure	Minimum quantity of radiant energy to produce a perceptible reddening of human skin. The first subsite to show a minimal redness perceptible to the eye, (with normal vision)
Units	J/m^2 or mJ/cm^2 or MED units or time (seconds) if the flux is constant throughout the test	J/m^2	Energy or time (if flux constant)
Time of assessment	16 h to 24 h post-exposure MEDu and MEDp on same day	22 h to 24 h post-exposure	16 h to 24 h post-exposure
Conditions of observation	Sufficient and uniform illumination: at least 450 lx	Illumination: tungsten or warm white fluorescent light bulb 450 lx to 550 lx (at the test site)	Full daylight, or a tungsten filament light providing adequate illumination; matt neutral wall colours
Position of volunteers	Same position used for the UV exposure	Same position used for the UV exposure	Seated or prone position
Biological endpoint	Erythema	Erythema	Erythema
Evaluator	Normal colour and acuity vision different from the person who applied the sunscreen or administered the UV doses	Different from the person who applied the sunscreen or administered the UV doses	Normal vision, colour vision checked
Data rejection criteria on individual test site	All sites visible or no site visible, responses on the treated sites randomly absent Rejection of the subject if MEDu or MEDp of standard product not determined	All sites visible or no site visible, responses on the treated sites randomly absent (indication of uneven spreading)	If the result obtained using the reference product on a subject varies by > 25 % of the average value of that test series the results of the subject are excluded If more than two subjects return SPFs for the test product which vary by > 25 % of the mean SPF, a new sample should obtained

Table 1 (*continued*)

Parameters	International 2006 [1]	FDA 1999 [2]	Australia 1998 [3]
Reference sunscreen formulations			
Reference sunscreen formulations used	Expected SPF < SPF 20 P2 or P3 or P7 Expected SPF ⩾ SPF 20 P2 or P3 The same has to be tested on every subject in the same series of at least ten subjects	Homosalate 8 % SPF 4,47 (S.D.: 1,279)	On each test subject either: *Homosalate 8 % SPF 4,47 *P3 SPF 15,5 or values derived from the laboratory's historical record on its test results
Acceptance limits (ranges)	Mean SPF ± 2 SE P2: 16,6 (14,2 to 19,0) P3: 16,2 (13,8 to 18,7) P7: 5,1 (4,4 to 5,9)	The SPF must fall within the range 4,47 ± 1,279 and the 95 % CI of the mean SPF must contain the value four	* Homosalate 8 % SPF ± 2 SD ∈ [4 to 5] * P3 SPF ± 2 SD ∈ [12.5 to 18.5]
Calculation and results			
Number of test subjects	Minimum of ten, maximum of twenty five	No more than twenty five, at least twenty valid data	Minimum of ten, maximum not defined
Calculation of mean SPF	Arithmetic mean, minimum of ten valid results and a maximum of twenty shall be used for the calculation of SPF A maximum of five results may be excluded from the calculation of the mean SPF; each exclusion has to be justified	Mean, SD, t value at 5 % with $n - 1$, SEM	Arithmetical mean, expressed to one decimal point
Statistical criterion	95 % confidence interval should fall within the range of ± 17 % of the mean SPF A minimum of ten valid results is only sufficient if the criterion is fulfilled, otherwise the number of subjects is increased stepwise from ten until the statistical criterion is met up to a maximum of twenty valid results	No	SEM ⩽ 7 % of mean SPF for valid result