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Cosmetics — Sun protection test methods — Water immersion procedure for determining water resistance

Cosmétiques — Méthodes d'essai de protection solaire — Mode opératoire d'immersion dans l'eau pour la détermination de la résistance à l'eau

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Foreword

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

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.

Cosmetics — Sun protection test methods — Water immersion procedure for determining water resistance

1 Scope

This document specifies a procedure of water immersion for the in vivo determination of the water resistance of sunscreen products.

This document is applicable to products intended to be placed in contact with human skin including any component able to absorb, reflect or scatter UV rays and which, in addition, are designed to be less readily removed from the skin by water and/or during water immersion. It is intended to be read in conjunction with ISO 24444.

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 24444:2019, *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
simulated swim test device
spa, whirlpool or similar device designed for water immersion

Note 1 to entry: For the purposes of this document, the simulated swim test device shall be in accordance with Annex A.

3.2
individual water resistance sun protection factor
individual water resistance SPF
SPF_{iwr}
SPF determined after the water immersion step on each subject

Note 1 to entry: SPF_{iwr} is calculated by a simple division of MED_{ipi} by MED_{iui}.

3.3
static sun protection factor
static SPF
SPF without water resistance challenge

Note 1 to entry: This is determined in accordance with ISO 24444.

3.4
post-water immersion sun protection factor
post-water immersion SPF
arithmetic mean of all valid SPF_{iwr} (3.2) values for the study

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

3.5
water resistance sun protection factor
water resistance SPF
SPF_{wr}
arithmetic mean of all valid SPF_{iwr} (3.2) values

4 Principle

4.1 Main steps

All of the test steps set out in ISO 24444 apply. Clause 5 provides an additional step to be inserted in the test sequence described in ISO 24444:2019, 9.4. This water immersion step commences after the test products have been applied, following the dry-down period given in ISO 24444:2019, 9.4.10, and prior to the exposure to simulated sunlight given in ISO 24444:2019, 9.4.11 to 9.4.15. In all other respects, the principles and procedures of ISO 24444 apply. A tabulation of steps is set out in Table 1.

Table 1 — Main steps

Test requirements	ISO 24444	This document
Terms and definitions	applicable	applicable
General principle	applicable	applicable
Test subjects	applicable	not applicable
Apparatus and materials	applicable	not applicable
Reference sunscreens	applicable	applicable
Test conditions	applicable	applicable
Water immersion procedure	> > > > > > > > >	applicable
Reversion to ISO 24444		< < < < < < < < <
Procedure for UV exposure	applicable	not applicable
Subsequent procedure	applicable	not applicable

4.2 General principle

A controlled amount of sunscreen product(s) is applied to an area of each subject's skin, restricted to the back between the scapula line and the waist. The sunscreen test sample is challenged in situ when the test subject undertakes a period of water immersion in a simulated swim test device for a designated period of time. Following a further drying period, an area without any protection and the protected area are exposed to ultraviolet light using the same procedure as the static SPF test described in ISO 24444.

5 Test criteria

5.1 Selection of the test subjects

Participants are enrolled for the study in accordance with the criteria described in ISO 24444. At least 10 subjects shall be selected.

Since not all volunteers for static SPF testing are prepared to participate in water resistance testing, specific informed consent shall be obtained. The Declaration of Helsinki[3] is relevant to testing using

human subjects. Informed, written (signature) consent shall be obtained from all test subjects. The consent should include specific consent to participate in water resistance testing, including the length of time, since the temperature of the water is likely to become chilled during testing.

5.2 Test area

As per the requirements of ISO 24444, the individual product test sites and the unprotected test site shall be delineated within the region between the scapula line and the waist. Additionally, the test sites shall be configured such that they will be fully immersed when the test subject is located comfortably in the simulated swim test device.

5.3 Product application

The product shall be applied in accordance with the procedure set out in ISO 24444.

6 Water immersion procedure

6.1 Room conditions

The temperature of the swim immersion room shall be maintained in the range of 20 °C to 26 °C.

6.2 Water quality

The simulated swim test device shall be filled with standardized water as described in Annex B.

The temperature of the water shall be maintained at (30 ± 2) °C for the duration of the test period.

6.3 Immersion sequencing cycle

The following sequence of immersion and a rest period shall be followed:

— 20 min of immersion of the test subjects with water circulated for the full period;

— 5 min to 20 min drying time with no towelling permitted between immersion periods.

For 40 min water resistance, repeat this sequence two times.

For 80 min water resistance, repeat this sequence four times.

For extended water resistance times, repeat this process as appropriate.

At the conclusion of the final immersion period, allow the test subjects to dry in the air (with no towelling off of the test sites) for at least 15 min. No water droplets shall be visible and additional drying time can be required prior to initiating any exposure, as described in ISO 24444:2019, 9.4.10.

6.4 Positioning of test subjects

The positioning of test subjects needs to take into account the various shapes of spa baths.

The positioning of the subject in the water immersion device shall be such that it minimizes the possibility of the subject touching the test areas on the back with the spa sides or another test subject, and is comfortable for the subject.

The subjects shall sit in a position such that water jets cannot impinge directly on the test sites.

The randomization of test sites shall be in accordance with Annex D.

6.5 Drying after immersion

After the completion of the final immersion session, the test areas shall be allowed to dry until no water droplets are visible and for at least 15 mins prior to initiating any UV exposures.

6.6 Reversion to ISO 24444 procedure

Upon completion of the water immersion procedure, complete the solar simulator exposure steps in accordance with ISO 24444:2019, 9.4.11 to 9.4.15.

7 Water quality and condition

The simulated swim test device shall be filled with standardized water as described in Annex B.

The temperature of the water shall be maintained at (30 ± 2) °C for the duration of the test period.

The temperature of the swim immersion room shall be maintained in the range of 20 °C to 26 °C.

8 Procedural validation

8.1 General

The reference sunscreen shall be the product described as P2 high SPF reference formula in ISO 24444:2019, Annex C. This reference sunscreen has been shown to be water resistant.

It is not required to use the reference sunscreen to check the utility and consistency of the immersion procedure for each test subject. However, a compliant post-water immersion SPF mean value shall be established by the laboratory on a frequency of every 200 subjects or every 2 months (whichever is sooner), either by:

a) the inclusion of additional subjects in water resistance SPF studies, on whom P2 shall be tested, to provide at least 10 results to calculate a post-water immersion SPF mean value; for the purposes of calculation, the rolling average of the last 10 subjects shall be used; or

b) performing a full water resistance SPF test using P2 as the test product.

The post-water immersion SPF mean value for P2 (from either of the approaches above) shall fall between the values specified in Annex C.

8.2 Calculation of the individual water resistance SPF (SPF_{iwr})

Individual water resistance retention shall be calculated for each individual subject where concurrent static SPF data also exist. The procedure for the calculation of individual water resistance retention is set out in ISO 18861[1)].

The SPF_{iwr} of the reference sunscreen and the product under test for each subject is calculated as shown in Formula (1):

$$SPF_{iwr} = \frac{MED_{ps}}{MED_{up}} = \frac{MED_{ipi}}{MED_{iui}} \quad (1)$$

where

1) To be published.

MED_{ps} is the MED on protected skin;

MED_{up} is the MED on unprotected skin;

MED_{ipi} is the individual MED for the sunscreen-protected site after immersion;

MED_{iui} is the individual MED for the unprotected site.

8.3 Calculation of the water resistance SPF (SPF_{wr})

The water resistance SPF (SPF_{wr}) result for the test product and for the reference sunscreen formulation is calculated as the arithmetic mean of all valid individual SPF_{iwr} values.

The minimum number of valid SPF_{iwr} values shall be 10 and the maximum number of valid SPF_{iwr} values shall be 20. A maximum of 5 results may be excluded from the calculation of SPF_{wr}, but each exclusion shall be justified. A sixth invalid result automatically invalidates the whole test for that test product and no SPF_{wr} can be calculated for it.

8.4 Statistical criterion

The statistical criterion shall be the same as those applied to the static SPF as described in ISO 24444:2019, Clause 10. This is applicable to test products and the reference sunscreen product.

A minimum of 10 valid results is only sufficient if the statistical criterion is fulfilled, otherwise the number of subjects is increased from 10 until the statistical criterion is met, up to a maximum of 20 valid results.

The full statistical procedure for this calculation is described in ISO 24444:2019, Annex D.

9 Test report — post-water immersion SPF

The test report shall contain all of the information as required by ISO 24444 and, in addition, the following:

— the period of time of the post-water immersion SPF test;

— the temperature of the simulated swim test device and the environment;

— the test conditions as designated in Annex B;

— the SPF_{iwr} values obtained for each test subject and reported as post-water immersion SPF;

— the SPF_{wr} obtained;

— the SPF_{iwr} for P2 and the SPF_{wr} value for the last 10 measurements (i.e. the moving average).

Annex A
(normative)

Simulated swim test device design

The device shall be of a design that permits efficient maintenance and sanitization.

The device shall be capable of maintaining water temperature within the limits defined in Clause 7.

The dimensions of the device shall be such that the test areas are immersed in water and shall accommodate test subjects in such a way that the test areas on the subject's back cannot be touched by the device or other subjects.

The dimensions shall be such that the test areas are not in the direct path of the jets.

It is recommended that the device should be emptied and refilled every day during testing activity.

The linear flow rate of the simulated swim rest device shall be 0,02 m/s to 0,05 m/s when measured at the point where the test subject's back is intended to be located during the test but without the test subject in the device. The flow rate shall be measured with a flow meter with appropriate sensitivity and of a type which is waterproof.

Annex B
(normative)

Standardized water requirements

Generally, the water to be used starts as potable water. The water used for each immersion test shall conform to this annex. All measurements should be conducted before the test volunteer(s) enter(s) the water.

Conductivity shall be equal to or higher than 500 microSiemens (mS).

Conductivity may be increased by the addition of sodium chloride.

Water pH shall fall between pH 6,5 to 7,5 citric acid. If necessary, sodium hydrogen sulfate shall be used to adjust the pH downward and sodium bicarbonate shall be used to adjust the pH upward.

Local health and safety requirements can apply to the quality of the water. Water hardness may be determined using data supplied by water authorities or by analytical determination.

Annex C
(normative)

Standard reference sunscreen

The formula, manufacturing instructions, stability and physiochemical specifications for the reference product are given in ISO 24444:2019, Annex C.

The values obtained (n = 10 at least) shall fall within the limits given in Table C.1.

Table C.1 — Expected post-immersion values and water resistance percentage

Immersion time	Acceptance range	
	Lower limit	Upper limit
40 min	9,0	15,0
Mean WR%	50,0	85,0