
**Cosmetics — Sun protection test
methods — Percentage of water
resistance**

*Cosmétiques — Méthodes d'essai de protection solaire — Pourcentage
de résistance à l'eau*

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Published in Switzerland

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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).

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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*.

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

The protection which cosmetic products containing organic or inorganic sunscreens provide against sunburn is neither absolute nor permanent.

One of the many factors that can have an effect on the level of protection given by these products is water contact. UV absorbers in the formulation can leach out or be physically removed by the washing action in the sea or swimming pool.

In order to make the sun products more effective, manufacturers have developed formulations which are more substantive to the skin during water immersion. These products have been variously labelled as water resistant or very water resistant.

In order to substantiate these product efficacy claims, a number of methods has been developed and published: including a method promulgated in the United States of America FDA monograph on OTC sunscreen drug products (Federal Register/ Vol. 58, No 90). Standard methods have also been published in Australia/New Zealand (AS/NZS 2640) and in the Republic of South Africa (SABS 1557).

The method for conditions required for water resistance SPF test exists as an International Standard, i.e. ISO 16217, and requires a sun protection factor to be measured following a defined water immersion procedure.

This document describes the procedure for water resistant percentage calculation, based on water immersion procedure described in ISO 16217, which is the ratio between before and after bath SPF.

The SPF measurement procedure is that described by ISO 24444.

All references to the sun protection factor (SPF) test method herein, relate to ISO 24444.

The reader should ensure that the latest version of ISO 24444 sun protection factor (SPF) test method is followed.

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Cosmetics — Sun protection test methods — Percentage of water resistance

1 Scope

This document specifies a procedure for evaluating the water resistance retention percentage, by comparing the sun protection factor (SPF) before water immersion (hereafter referred to as the “static” SPF) and after a fixed period of water immersion (hereafter referred to as the “wet” SPF).

2 Normative references

The following document is 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 16217:2020, *Cosmetics — Sun protection test methods — Water immersion procedure for determining water resistance*

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

[SOURCE: ISO 16217:2020, 3.1]

3.2

standardized water

water for use in the *simulated swim test device* (3.1)

3.3

static sun protection factor

static SPF

SPF_{is}

SPF without water resistance challenge

3.4

SPF before water immersion

arithmetic mean of all valid SPF_{is} (3.3) values for the study, expressed to one decimal by truncation

3.5

individual water resistance sun protection factor individual water resistance SPF

SPF_{iwr}

SPF determined after the water immersion step on each subject

3.6

SPF post water immersion

arithmetic mean of all valid SPF_{iwr} (3.5) values for the study, expressed to one decimal by truncation

4 Test method

4.1 Outline of the method

The principle is to compare the sun protection factor (SPF) for a sunscreen product after a period of immersion in water with the static SPF without immersion in water.

4.2 Test subject selection

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

The Declaration of Helsinki^[1] 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 length of time — temperature of the water — likelihood of becoming chilled during testing.

4.3 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. Test product application to test sites should be randomized on each individual subject and over the whole test panel, as described in ISO 24444.

4.4 Product application

The product shall be applied according to the procedure set out in ISO 24444.

4.5 Water immersion procedure

The static SPF (SPF_{is}) is determined in accordance with the ISO 24444.

To determine the SPF post water immersion (SPF_{iwr}), ISO 24444 shall be followed to the point where the product under test has been applied to the subject's skin.

Product treated skin is then immersed in water according to the process described in ISO 16217.

The following sequence of immersion and 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 (no towelling- off of test sites) in the air for at least 15 min.

No water droplets shall be visible and additional drying time may be required (at most 60 min) prior to initiating any exposure as described in ISO 24444.

This SPF post water immersion (SPF_{iwr}), shall then be compared with the static SPF (SPF_{is}), and the percentage of water resistance shall be calculated.

4.6 Reference water resistant sun product

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

Inclusion of the reference sunscreen in respect of water resistance performance is not required for each test subject and may be validated separately at least every 8 weeks (or 10 tests when more than 10 tests are conducted within 8 weeks).

The static SPF with no immersion shall be validated as described in ISO 24444.

The mean SPF of P2 after water exposure shall fall between the values specified in [Annex C](#).

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

4.7 Determination of the minimum erythematous dose (MED)

Minimum erythematous doses (MEDs) are determined according to the appropriate clause of the ISO 24444. The dose increment used to determine the unprotected and protected MEDs for the SPF after immersion shall be the same as used to determine the MEDs for the SPF before immersion.

SPF measurements before and after water immersion shall be determined in the same laboratory on the same panel of subjects as part of the same test sequence.

4.8 Number of test subjects

According to ISO 24444, up to 25 subjects should be selected for the study. A minimum of 10 subjects and up to 20 valid subjects should complete the test, with the exact number being defined by the need to satisfy the prescribed statistical acceptance criteria.

For details of statistical definitions, procedure and calculations refer to [Clause 5](#) and to ISO 24444.

4.9 Test chronology

The sequence in which the static and wet SPFs are determined may be critical and so it is strongly recommended that they are determined in the sequence described in the test procedure chronology (see [Annex B](#)).

5 Calculations and data handling

5.1 General

The following calculations shall be performed.

5.2 SPF before immersion

The SPF before immersion is calculated as the mean of the total individual static SPF values (SPF_{is}), determined on all subjects completing the procedure. A corresponding 95 % bilateral confidence

interval (95 %CI) should also be calculated. Both the SPF before immersion and the 95 %CI shall be calculated according to the guidelines described in ISO 24444.

A test will be considered acceptable if the 95 % confidence interval on the mean SPF before immersion is within ± 17 % of the mean static SPF (SPFs).

5.3 SPF after immersion

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.

5.4 Individual percentage of water resistance

An individual percentage of water resistance (%WR_i) value should be calculated for each individual subject according to [Formula \(1\)](#):

$$\%WR_i = (SPF_{iwr} - 1) / (SPF_{is} - 1) \times 100 \tag{1}$$

where

SPF_{is} is the individual SPF before water immersion; MED_{isp}/MED_{isu};

SPF_{iwr} is the individual SPF post water immersion; MED_{iwrp}/MED_{iwru}.

Both measured on the same subject.

5.5 Mean percentage of water resistance

The mean percentage of water resistance (%WR) is expressed as the arithmetic mean of the “n” individual %WR values (%WR_i).

5.6 Calculation of lower confidence limit on the mean percentage of water resistance

The confidence in the value for the mean percentage of water resistance (%WR) is expressed by way of a unilateral 90 % confidence interval.

The 90 % unilateral confidence interval for the mean %WR is calculated as [Formula \(2\)](#):

$$\text{mean \%WR} = \text{mean \%WR}_i - d \tag{2}$$

where *d* is calculated as

$$d = t_u \times s / \sqrt{n}$$

where

s is standard deviation;

n is the total number of volunteers in test;

t_u is the t value from the “one-sided” Student-t distribution (see [Table 1](#)) at a probability level *p* = 0,10 and with *n*-1 degrees of freedom.

Table 1 — “one-sided” Student-t distribution

<i>n</i>	10	11	12	13	14	15	16	17	18	19	20
<i>t_u</i>	1,383	1,372	1,363	1,356	1,350	1,345	1,341	1,337	1,333	1,330	1,328

A product is considered water resistant if the value for 90 % lower unilateral confidence interval satisfies the local requirements and the mean SPF before immersion is within ± 17 % of the mean.

5.7 Acceptance criteria for reference product P2

Acceptance range was determined after collection of worldwide values from various laboratories and set according to ISO 13528.

Wet SPF: Mean value: 11,5 acceptance range [9,0 to 15,0]

WR%: Mean value: 68,1 % acceptance range [50 to 85], calculated as described in [5.6](#).

6 Test report

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

- a) the period of time of the SPF water immersion test (40 min, 80 min, etc.);
- b) the temperature of the simulated swim test device and the environment;
- c) the water immersion test conditions as designated in [Annex A](#): Conductivity, water flow;
- d) the static SPF_{is} values obtained for each test subject, with expression of the mean static SPF, standard deviation and 95 %IC interval, 17 % of the mean;
- e) the mean water resistance SPF_{iwr} values obtained for each test subject, with expression of the mean water resistance SPF and standard deviation;
- f) the individual percentage of water resistance ($\%WR_i$) obtained for each test subject, with expression of the mean percentage of water resistance ($\%WR$), standard deviation and 90 % unilateral confidence interval (D);
- g) the value of [$\%WR-D$];
- h) the value obtained for the water resistant reference sunscreen P2 and date of obtention.

Annex A (normative)

Simulated swim test device design

The device shall meet the requirements described in ISO 16217:2020, Annexes A and B.

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.

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 subjects 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 test device shall be 0,02 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 water proof.

Generally, the water to be used starts as potable water. The water used for each immersion test shall comply with the following. All measurements should be conducted before the test volunteer(s) enter 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 or sodium hydrogen sulfate shall be used if necessary to adjust pH downward and Sodium bicarbonate to adjust pH upward.

Water quality shall be compatible with local health and safety requirements. Water hardness may be determined using data supplied by water authorities or by analytical determination.

Annex B (normative)

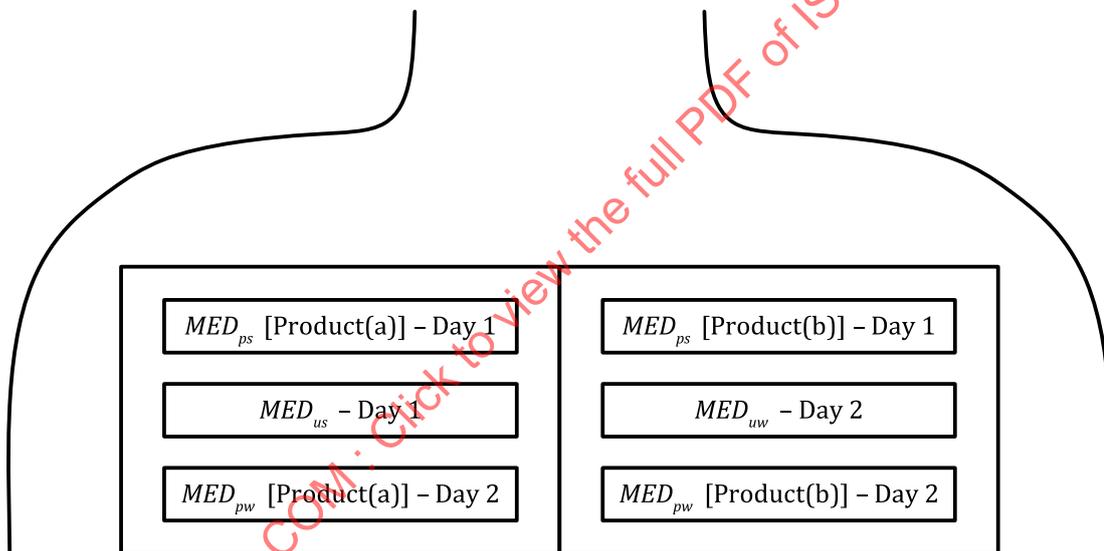
Test procedure and chronology

B.1 Randomization of test sites

The test procedure allows for two or more products to be tested at the same time.

Figure B.1 is an example of product positioning that indicates how multiple products may be positioned on a single subject.

Test site positioning shall always be randomized on each individual subject and across the whole panel of subjects.



Key

- MED_{us} is unprotected static MED (non immersed in water)
- MED_{uw} is unprotected "wet" MED (post-immersion in water)
- MED_{ps} is protected static MED (non immersed in water)
- MED_{pw} is protected "wet" MED (post-immersion in water)

Figure B.1 — Illustrative example of product positioning

B.2 Test chronology and procedure

The following procedure is followed for each subject and is repeated on between 10 and up to 20 subjects, until statistical criteria are satisfied.

B.2.1 DAY 1

B.2.1.1 Determine the unprotected, static MED (MED_{us}) on previously unexposed skin.

B.2.1.2 Apply product to designated static SPF sites (MED_{ps} [Product(a)]; MED_{ps} [Product(b)], etc.).

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B.2.1.3 Allow 15 min to 30 min for the product to dry.

B.2.1.4 Expose the product treated static sites (MED_{ps} [Product(a)], MED_{ps} [Product(b)], etc.) to progressive UV doses according to ISO 24444.

B.2.2 DAY 2

B.2.2.1 Assess the erythematous reactions from previous day's UV exposures on unprotected static MED_{us} sites and determine the static MED_{us} for unprotected, non-immersed skin.

B.2.2.2 Assess the erythematous reactions from previous day's UV exposures on product protected static MED_{ps} sites and determine the static MED_{ps} for product protected, non-water immersed skin.

B.2.2.3 Calculate the individual static SPF (SPF_{is}).

B.2.2.4 Apply product to test sites designated for water immersion (MED_{pw} [Product(a)], MED_{pw} [Product(b)], etc.).

B.2.2.5 Allow to dry for 15 min to 30 min.

B.2.2.6 Immerse subject in water for 20 min with continuous water circulation.

B.2.2.7 Remove subject from immersion equipment and allow test sites to air-dry with no towelling for 5 min to 20 min.

B.2.2.8 Return subject to immersion equipment and immerse in water for a further 20 min with continuous water circulation.

B.2.2.9 Remove subject from immersion equipment and allow test sites to air-dry with no towelling for at least 15 min and until test site is completely dry. Ensure that all test sites are free from water droplets. If water droplets are present, extend air-drying time, no longer than 60 min.

B.2.2.10 Expose product treated water immersion sites (MED_{pw} [Product(a)], MED_{pw} [Product(b)], etc.) to progressive UV doses according to ISO 24444 sun protection factor (SPF) test method.

B.2.2.11 Repeat unprotected MED (MED_{uw}). This unprotected MED is post water immersion, so as to be compatible with the protected wet MED_{pw} .

B.2.3 DAY 3

B.2.3.1 Assess the erythematous reactions from previous day's UV exposures on unprotected wet MED_{uw} sites and determine MED_{uw} for unprotected skin, after water immersion.

B.2.3.2 Assess the erythematous reactions from previous day's UV exposures on product protected, wet MED_{pw} sites and determine MED_{pw} for product protected, water immersed skin.

B.2.3.3 Calculate individual SPF after immersion (SPF_{ivr}) according to ISO 24444.

Static and wet SPFs shall always be determined on the same subject in the same laboratory using the same instrumentation and test conditions.

If necessary, the test may be shortened to cover only 2 days by combining days 1 and 2.