Cosmetics Europe
the personal care association

COSMETICS EUROPE:

GUIDELINES FOR EVALUATING SUN PRODUCT WATER RESISTANCE

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Contents

1 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, very water resistant or waterproof.

In order to substantiate these product efficacy claims, a number of methods have 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 (SABS1557).

The method for determining water resistance requires a sun protection factor to be measured following a defined water immersion procedure. This procedure utilises a spa-pool, a Jacuzzi® or a bathtub, each of which may vary in design but must comply with the guidelines. These guidelines describe the procedure for water immersion. The SPF measurement procedure is that described by the International Sun Protection Factor (SPF) Test Method (COLIPA document number 001-2003; February 2003). All references to “The International Sun Protection Factor (SPF) Test Method” herein, relate to that document or to later versions as may be published from time to time. The reader should ensure that the current version of the International Sun Protection Factor (SPF) Test Method is followed.

2 AIM

The aim is to provide guidelines for determining the water resistance of a sunscreen product by assessing and 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).

3 TEST METHOD

3.1 Outline of the Method

The principle of the water resistance test is to compare the Sun Protection Factor for a sunscreen product after a period of immersion in water with the original, static SPF of the product determined according to the International Sun Protection Factor (SPF) Test Method. The Sun Protection Factor value on an individual subject (SPF_i), for any product, either before or after water immersion, is defined as the ratio of the minimum erythemal dose on protected skin (MED_p) to the minimum erythemal dose on unprotected skin (MED_u) of the same subject.

i.e.

$$SPF_i = \frac{MED_p}{MED_u}$$

The static SPF (SPF_s) is determined according to the current published International Sun Protection Factor (SPF) Test Method. To determine the wet SPF (SPF_w), the International Sun Protection Factor (SPF) Test Method is followed to the point where the product under test has been applied to the volunteer's skin. Product treated skin is then immersed in water according to the following schedule:

- Allow 15 to 30 minutes drying time after product application
- First 20 minute immersion in water
- Allow 15 minutes drying time (no towelling)
- Second 20 minute immersion in water
- Allow to dry for 15 minutes or until completely dry (no towelling)

The full laboratory water immersion procedure employing a spa-pool or equivalent is described in the Appendices. An alternative procedure, using a swimming pool, is also acceptable but this method is difficult to standardise and is therefore not included in the scope of these guidelines. Laboratories employing the use of a swimming pool for water resistance measurements should ensure that all immersion conditions described within this method are accurately reproduced and that the spirit of these guidelines for spa-pools is observed.

On completion of the water immersion procedure, the International Sun Protection Factor (SPF) Test Method is resumed at the point immediately after the product was applied and allowed to dry on the volunteer's skin. This then completes the wet SPF determination. This wet SPF can then be compared with the SPF obtained for product treated skin, not immersed in water (ie the static SPF).

3.2 Test Subject Selection

Members of the volunteer panel should fulfil the selection criteria with regard to skin type, skin condition, medical condition and time since last test, as laid out in the International Sun Protection Factor (SPF) Test Method.

There should be a minimum rest period of two months before participation in a new water resistance retention test or any other test involving exposure of the skin to ultraviolet radiation (eg SPF test).

3.3 Test Area

The anatomical test area to be used is restricted to the back, between the scapula line and the waist, with the exact locations chosen to ensure complete submersion in the immersion equipment. Test product application to test sites should be randomised on each individual subject and over the whole test panel.

3.4 Source of Ultraviolet (UV) Radiation

An artificial source of UV radiation (solar simulator) should be used. The specification for an appropriate artificial UV source is that defined in the International Sun Protection Factor (SPF) Test Method. Control of the UV source quality should be according to the COLIPA Guidelines for Monitoring UV-Light Sources.

3.5 Reference Water Resistant Sun Product

Any laboratory following the Guidelines for evaluating Sun Product Water Resistance should validate their procedures at least every four months, by testing the Standard Water Resistant Sun Product coded P2, with nominal SPF of 12/15. A fresh sample of P2, not older than 12 months of age, shall be used. The expected level of water resistance should be > 50%.

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

3.6 Product Quantity and Application

Product should be applied at a precise application rate of 2.0 mg/cm² (± 2.5%). The amount of product applied and the uniformity of spreading on the test sites affect the magnitude and variability of the test results. It is therefore important to follow the recommendations set out in the International Sun Protection Factor (SPF) Test Method for product application quantity, product application technique and minimal area of the product application site.

3.7 Determination of the Minimum Erythemal Dose (MED)

Minimum erythemal doses (MEDs) are determined according to the appropriate section of the current International Sun Protection Factor (SPF) Test Method. The dose increment used to determine the unprotected and protected MEDs for the wet SPF must be the same as used to determine the MEDs for the static SPF.

SPF measurements before and after water immersion ("static" and "wet" SPF) must be determined in the same laboratory on the same panel of volunteers as part of the same test sequence.

3.8 Number of Test Subjects

Up to 25 volunteers 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 Section 4 below and to the International Sun Protection Factor (SPF) Test Method.

3.9 Water Immersion Procedure

The procedure for water immersion requires that the entire product treated site be fully submersed in water for various defined periods of time. A spa-pool, Jacuzzi or bathtub with water circulation is the only recommended equipment and a full description of the immersion procedure is given in Appendix A.

3.10 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 (Appendix B).

4 CALCULATIONS AND DATA HANDLING

The following calculations should be performed:

4.1 Mean static SPF (SPF_s)

The static SPF (SPF_s) 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_s and the 95%CI should be calculated according to the guidelines described in the International Sun Protection Factor (SPF) Test Method. A test will be considered acceptable if the 95% confidence interval on the mean static SPF (SPF_s) is within ± 17% of the mean static SPF (SPF_s).

4.2 Individual Percentage Water Resistance Retention

An individual Percentage Water Resistance Retention ($\%WRR_i$) value should be calculated for each individual subject according to the formula below:

$$\%WRR_i = \frac{(SPF_{iw} - 1)}{(SPF_{is} - 1)} * 100$$

Where: SPF_{iw} = individual wet SPF after water immersion
SPF_{is} = individual static SPF

4.3 Mean Percentage Water Resistance Retention

The mean percentage water resistance retention (%WRR) is expressed as the arithmetic mean of the 'n' individual %WRR values ($\%WRR_i$).

4.4 Calculation of Lower Confidence Limit on the Mean Percentage Water Resistance Retention

The confidence in the value for the mean percentage water resistance retention (%WRR) is expressed by way of a unilateral 90% confidence interval. The unilateral confidence interval is used because it is only necessary to predict the lower confidence level for the mean %WRR, since this is all that is needed to be confident that the product performs to a minimum level of water resistance. An upper confidence limit is not required, because it is not important to know the upper level of water resistance performance in this test. The level of confidence is set as 90% rather than the conventional 95% to allow for the increased biological variability that arises from the two major steps in the measurement procedure. In addition to the variability arising from the SPF determination (which is usually assigned a 95% confidence) there is the additional variability that arises from the water immersion procedure. In recognition of this increased, expected variability, a 90% confidence level is chosen rather than 95%.

The 90% unilateral confidence interval for the mean %WRR is calculated as:

$$[mean\%WRR - d]$$

with d calculated as:

$$d = \frac{t_u * s}{\sqrt{n}}$$

where s = standard deviation
n = total number of volunteers in test
t_u = t value from the "one-sided" Student-t distribution table at a probability level p=0.10 and with n-1 degrees of freedom

n	10	11	12	13	14	15	16	17	18	19	20
t_u values	1.383	1.372	1.363	1.356	1.350	1.345	1.341	1.337	1.333	1.330	1.328

4.5 Acceptance Criteria for Water Resistance Testing

A product will be considered water resistant if the value for the 90% lower unilateral confidence limit [mean %WRR – d] is greater than or equal to 50% AND the 95% confidence interval on the mean static SPF was within ± 17% of the mean static SPF.

4.6 Very Water Resistant Claims

Products which are designed to provide extra water resistance should be tested by adding a further two twenty-minute water immersion periods to the water immersion procedure described in sections 3.1, 3.9, Appendix A.3 and Appendix B.2. The revised water immersion procedure for very water resistance testing is then:

- Allow 15 to 30 minutes drying time after product application.
- First 20 minute immersion in water
- Allow 15 minutes drying time (no towelling)
- Second 20 minute immersion in water
- Allow 15 minutes drying time (no towelling)
- Third 20 minute immersion in water
- Allow 15 minutes drying time (no towelling)
- Fourth 20 minute immersion in water
- Allow to dry for 15 minutes or until completely dry (no towelling)

After completion of the water immersion procedure, the SPF test is resumed as for the standard water resistance test and the calculations described in section 4 are performed. A product will be considered very water resistant if the value for the 90% lower unilateral confidence limit [mean %WRR – d] is greater than or equal to 50% AND the 95% confidence interval on the mean static SPF was within ± 17% of the mean static SPF.

APPENDIX A: WATER IMMERSION PROCEDURE

A.1 Equipment Specific to procedure (immersion equipment)

- Spa pool, Jacuzzi® or bathtub with water jets to circulate water. Air jets (bubbles) should not be used.
- Sufficient capacity to enable complete immersion of all test areas with no contact between subject and any surface.
- Means of controlling water temperature.
- Ease of cleaning, sanitising, filling and emptying should be ensured.
- Security: Non-slip coatings should be used and volunteers supervised at all times.

A.2 Water Quality

- The immersion equipment should be filled with water complying with EC Council Directive 98/83/EC (3rd November 1998) governing the quality of water intended for human consumption. In addition, the maximum permitted level for magnesium and calcium (combined) is 500mg/l and the minimum is 50 mg/l.
- The temperature of the water should be 29ºC ± 2ºC.
- The water in the immersion equipment should be sanitised with bromide or chlorine according to the manufacturer's recommendations. Where no chemical sanitation procedure is adopted, the equipment should be emptied, cleaned and refilled between each volunteer that uses the pool.
- It is recommended that the pool or spa should be emptied and refilled every day during testing activity.

A.3 Immersion Conditions

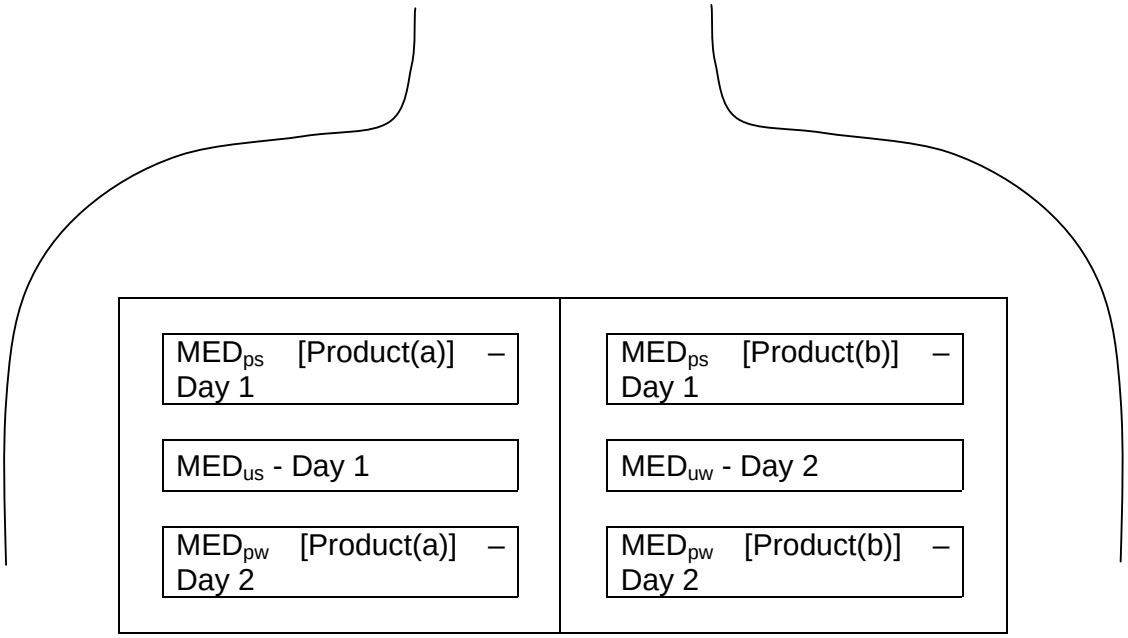
- Test product is applied to the test area (back) designated for water immersion according to the current International Sun Protection Factor (SPF) Test Method.
- 15 to 30 minutes drying time must elapse after application of the test product to the skin and before immersion in water.
- The immersion equipment should be furnished with a means of continuous water circulation which does not direct water onto the test areas and produces a rate of flow which adequately simulates moderate activity.
- The volunteer is then immersed in the immersion equipment for 20 minutes ensuring complete immersion of the test sites under the water and avoiding contact between any test site and any part of the immersion equipment.
- The volunteer exits the immersion equipment after the first 20 minute immersion and the product treated site is allowed to air-dry (with no towelling) for a further period of 15 minutes.
- The volunteer is then immersed in the immersion equipment for a second period of 20 minutes, again ensuring complete immersion of the test sites under the water and that there is no contact between any test site and any part of the immersion equipment.
- The volunteer exits the immersion equipment after the second 20 minute immersion and the product treated site is allowed to air-dry (with no towelling) for a further period of 15 minutes or until completely dry. There should be no visible water drops on the test site at the end of this drying period. If water droplets remain on the test site, then drying time should be extended.

- After the skin has completely dried, the SPF of the product applied to the test sites and immersed in water is then determined according to the International Sun Protection Factor (SPF) Test Method.
- Further volunteers are recruited and the same procedure followed until sufficient volunteers have generated sufficient data to comply with the statistical criteria.

APPENDIX B: TEST PROCEDURE and CHRONOLOGY

B.1 Randomisation of Test Sites

The test procedure allows for two or more products to be tested at the same time. The following illustrative example of product positioning indicates how multiple products may be positioned on a single subject. Test site positioning should always be randomised on each individual subject and across the whole panel of volunteers.



MED_{us} = unprotected, static MED (non immersed in water)
MED_{uw} = unprotected, "wet" MED (post-immersion in water)

MED_{ps} = protected static MED (non immersed in water)
MED_{pw} = protected "wet" MED (post-immersion in water)

B.2 Test Chronology and Procedure

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

- **DAY 1**

(i) Determine the unprotected, static MED (MED_{us}) on previously unexposed skin.
(ii) Apply product to designated static SPF sites (MED_{ps} [Product(a)]; MED_{ps} [Product(b)] …etc.).
(iii) Allow 15 to 30 minutes for the product to dry.
(iv) Expose the product treated static sites (MED_{ps} [Product(a)], MED_{ps} [Product(b)] …etc.) to progressive UV doses according to the International Sun Protection Factor (SPF) Test Method.

- **DAY 2**

(i) Assess the erythemal reactions from previous day's UV exposures on unprotected static MED_{us} sites and determine the static MED_{us} for unprotected, non-immersed skin.
(ii) Assess the erythemal 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.
(iii) Calculate the individual static SPF (SPF_{is})
(iv) Apply product to test sites designated for water immersion (MED_{pw} [Product(a)], MED_{pw} [Product(b)] …etc.).
(v) Allow to dry for 15 to 30 minutes.
(vi) Immerse volunteer in water for 20 minutes with continuous water circulation.
(vii) Remove volunteer from immersion equipment and allow test sites to air-dry with no towelling for 15 minutes.
(viii) Return volunteer to immersion equipment and immerse in water for a further 20 minutes with continuous water circulation.
(ix) Remove volunteer from immersion equipment and allow test sites to air-dry with no towelling for 15 minutes or 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.
(x) Expose product treated water immersion sites (MED_{pw} [Product(a)], MED_{pw} [Product(b)] …etc.) to progressive UV doses according to the International Sun Protection Factor (SPF) Test Method.
(xi) Repeat unprotected MED (MED_{uw}). Note that this unprotected MED is post water-immersion, so as to be compatible with the protected wet MED_{pw}.

- **DAY 3**

(i) Assess the erythemal reactions from previous day's UV exposures on unprotected wet MED_{uw} sites and determine MED_{uw} for unprotected skin, after water immersion.
(ii) Assess the erythemal reactions from previous day's UV exposures on product protected, wet MED_{pw} sites and determine MED_{pw} for product protected, water-immersed skin.
(iii) Calculate individual wet SPF (SPF_{iw}) according to the International Sun Protection Factor (SPF) Test Method guidelines.

NOTE:

- If necessary, the test may be shortened to cover only 2 days by combining days 1 and 2. In this case, the static MED and wet MED determinations are then conducted on the same day with an assessment of all erythemal responses on the start of Day 2. If this shortened procedure is adopted, then the investigator should be aware that water immersion subsequent to exposing the skin to UV-light in order to determine the static SPF, might affect the static SPF measured.

- Conducting a static SPF on skin which has been previously hydrated or is at an altered temperature as a result of a previous water immersion may lead to variability in the SPF measured. Since this does not happen in a standard SPF test, the static SPF must not be determined after immersion in water.

- It is also possible to conduct the wet SPF determination several days after the static SPF. However if this amended procedure is adopted, it is essential that an appropriate repeat, unprotected MED is performed on every day that product-protected exposure occurs.

- Static and wet SPFs should always be determined on the same volunteer in the same laboratory using the same instrumentation and test conditions.

APPENDIX C: WATER RESISTANT REFERENCE SUN PRODUCT (P2)

C.1 Formula Details

Ingredients	INCI Name	% W/W
Phase 1	Lanolin	4.5
	Theobroma Cacao	2.0
	Glyceryl Stearate ("Glyceryl Monostearate SE")	3.0
	Stearic Acid	2.0
	Octyl Dimethyl PABA	7.0
	Benzophenone-3 ("Oxybenzone")	3.0
Phase 2	Water	71.6
	Sorbitol	5.0
	Triethanolamine	1.0
	Methylparaben	0.3
	Propylparaben	0.1
Phase 3	Benzyl Alcohol	0.5

C.2 Manufacturing Process

i. Melt the ingredients of the fatty Phase 1 and heat to 80-85 °C.
ii. Heat Phase 2 to 80-85 °C, until completely solubilised.
iii. Add Phase 1 into Phase 2, while stirring Phase 2 with a homogeniser (Moritz type).
iv. Cool to 50 °C while stirring, then add Benzyl Alcohol and complete cooling. Compensate for water loss and homogenise.

C.3 Physiochemical Specifications

Appearance: White yellowish fluid emulsion
pH: 8.6 ± 0.5
Viscosity: 2.5 poises (at 10mn, Contraves TVB rheometer, rotary body N°3)
Density: 0.95 $g.cm^{-3}$

C.4 Analytical Data

HPLC: Octyl Dimethyl PABA: 6.9 to 7.1 % w/w
Benzophenone-3: 2.8 to 3.2 % w/w

C.5 Photometric Data

Typical data for a 100 mg/l solution in Isopropanol:

λmax.:	309.4 nm	Abs. Max.:	0.909
λ:	290.0 nm	Abs.:	0.540
λ:	320.0 nm	Abs.:	0.671
λ:	340.0 nm	Abs.:	0.120
λ:	400.0 nm	Abs.:	0.000

C.6 Formulation Stability

At least 2 months at 45 °C and 12 months at 20 °C.

C.7 Expected SPF and Water Resistance Values

SPF 12 to 15
Water Resistance > 50%

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