



# STEPS



1. PRODUCT APPLICATION



2. WATER RESISTANCE CHALLENGE



3. SOLAR SIMULATOR



4. READING OF RESULTS

## SPF ISO 24444 Protocol Testing

The ISO 24444 SPF test is now adopted in nearly 60 countries

### Supportable Claims

- Sunscreen Efficacy
- SPF Number

### Applicable Countries

Harmonisation of this test allows us to certify for the countries below...

Australia
New Zealand
European Union 28 countries
UK
India
Japan
Taiwan
Korea
MERCOSUR 6 countries
Canada
ASEAN 10 countries
South Africa
Mexico
Chile
Russia
Israel
India
Russia
Japan

Note that in some countries, alternative methods are accepted.

### Product Application

A very accurately measured and controlled amount (2 mg/sq cm) of product is applied to a marked out area of skin. The product is evenly spread, using a standardised technique. The product is then allowed to dry for 15 to 30 minutes.

### Water Resistance

For most countries, 40 minutes or 80 minutes. Up to 4 hrs is permitted in Australia.

### Solar Simulation

A Solar simulator, which has been designed and calibrated to imitate the spectrum of sunlight, is used to apply small incremental doses of light to the protected area. An unprotected area and an area with a Standard Sunscreen applied, are also exposed. Overnight, a mild erythema (slight sunburn) develops at test sites where the SPF has been exceeded.

### References

#### [Monographed Methods]

ISO 24444 : 2010

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## SPF TEST ACCORDING TO THE ISO 24444 PROTOCOL

### 1. Objective

The test panel will be convened to evaluate the effectiveness of a test material as a sunscreen product by determining the Sun Protection Factor (SPF) on human skin as described in the document: International Standard ISO 24444 - Cosmetics - Sun Protection Test methods -in-vivo determination of the sun protection factor (SPF).

### 2. Test Material Handling

The record of the sample is entered into a log identifying the lot number, sample description, batch number, sponsor, date received and tests requested. Samples are retained for a period of two years beyond final report generation.

### 3. Ethical Principles for Conduct of Study

3.1 The study will be conducted in accordance with the principles as described in the document WMA Declaration of Helsinki – for Medical Research Involving Human Subjects.

### 4. Standard for Inclusion of a Panelist in a Study

- 4.1 Individuals over the age of consent.
- 4.2 Individuals free of any dermatological or systemic disorder which would interfere with the results, at the discretion of the investigator.
- 4.3 Individuals who have completed a preliminary medical history evaluation.
- 4.4 Individuals who have read, understood and signed an informed consent document relating to the specific study to which they are subscribing.
- 4.5 Individuals with no known abnormal response to sunlight.

### 5. Standard for Exclusion of a Panelist from a Study

- 5.1. Individuals taking medication which in the opinion of the investigator would mask or interfere with the results.
- 5.2. Individuals with chronic skin allergies.
- 5.3. Individuals with suntan or sunburn.
- 5.4. Individuals with abnormal reaction to the sun.
- 5.5. Pregnant or lactating females.
- 5.6 Subjects accustomed to using sun beds.
- 5.7 Subjects who have participated in an SPF study within the last two months.



## 6. Informed Consent and Medical History Forms

An informed consent will be obtained from each volunteer prior to initiating the study describing reasons for the study, possible adverse effects, associated risks and potential benefits of the treatment and their limits of liability. Panelists sign and date the informed consent document to indicate their authorisation to proceed and acknowledge their understanding of the contents. Each subject is assigned a permanent identification number and completed an extensive medical history form. These forms along with the signed consent forms, are available for inspection only on the premises of Dermatest Pty Ltd and during normal office hours.

## 7. Panel Composition

a minimum of ten healthy volunteers, between the age of consent and 70 years will be recruited for this study. The panel consisted of fair skin individuals with Fitzpatrick skin types I, II or III.

## 8. Solar Simulation

The light source employed is a small beam 150 watt Xenon Arc Solar Simulator (Solar Light Co., Philadelphia, Pennsylvania, Model 16S or Model 601) having a continuous emission spectrum in the UV range from 290 to 400 nm and compliant with the spectral performance requirements of the Annex B of the ISO protocol. Xenon arc is selected on the basis of its black body radiation temperature of 6000°K which produces continuous UV spectra (all wavelengths) substantially equivalent to that of natural sunlight. This device is equipped with a dichroic mirror (which reflects all radiation below 400nm) and works in conjunction with a 1 mm thick Schott WG320 filter (which absorbs >99% of radiation below 290nm) to produce simulation of the solar UVA@UVB spectrum. A 1 mm thick UG 11 filter (black lens) is added to remove reflected (infra-red, greater than 700 nm) heat and remaining visible radiation. UV radiation is monitored continuously during exposure using a Sunburn UV Meter/Dose Controller System (Solar Light Co) . Measurements are taken at a position within 8 mm from the surface of the skin. The field of irradiation is >0.5 cm in diameter, with at least 1 cm between each adjacent site. Realignment of the Light Sources and calibration of the sunburn meters are conducted by independent certification facilities and adjustment to light source power supply only by the Director. The calibration data for each solar simulator will appear on the test report in the following tabular format.



Spectral Range nm	Measured % RCEE Limits		Solar Sim			
	Lower	Upper	No:			
<290		<0.1				
290 to 300	1.0	8.0				
390 to 310	49.0	65.0				
290 to 320	85.0	90.0				
290 to 330	91.5	95.5				
290 to 340	94.0	97.0				
290 to 400	99.9	100.0				

### 9. Determination of Water Resistance (Where Conducted)

This does not form part of ISO 24444, but is a requirement of AS/NZS 2604 (2012) for those products making a water resistance claim. This test is employed to determine the substantivity of a test product and its ability to resist water immersion. The procedure described in Appendix B of AS/NZS 2604 is followed. The immersion schedule is listed as follows:

9.1 Subject immersed in the Spa pool for 20 min with 4 min of air agitation.

9.2 5 minutes rest period out of the water. This sequence is repeated for the required time period. Immersion is achieved indoors in a circulating whirlpool tub through 1.5 cm diameter ports. The water is maintained at an average temperature of 33°C +/- 2°C. The pH of the water is maintained between 6.8 and 7.2.

The test area is air dried prior to exposure from the solar simulator using 25% increments. The exact series of exposures given is determined by the control MED and the expected SPF of the product.

Water Resistance Category Description for AS/NZS 2604 (2012)

Tested SPF after Immersion	Maximum Water Resistance Claimable
At least 4 but less than 8	No Claim
At least 8 but less than 15	40 min
At least 15 but less than 30	2 hrs
At least 30 or above	4 hrs

### 10. Determination of the Sun Protection Factor

One test site area of 40 sq.cm serves to determine each subject's Minimal Erythema Dose (MEDu). This is executed by exposing the back to a series of 5 timed



incremental UV exposures at 112% or 125% intervals. The individual subject's MED<sub>u</sub> is the shortest time of exposure that produces perceptible unambiguous redness at 16 to 24 hours post irradiation. The test area is described as the infrascapular area of the back to the right and left of the midline. The application area is 40 sq.cm. The product is delivered to the test site through plastic volumetric syringes. The material is evenly applied to a rectangular area for a final covering of 2.0 mg/sqcm +/- 2.5%.

The product is deposited in a series of evenly distributed spots and then spread evenly with a fingertip. Product application, UV exposures and measurements of responses will be conducted in stable environmental conditions with the room temperature maintained between 18°C and 26°C.

15 to 30 minutes after application, a series of 5 UV light exposures in 112.5% increments calculated from previously determined MED's bracketing the expected SPF will be administered from the solar simulator to subsites, each with an area of not less than 1 cm sq and spaced at 1 cm separation within the treated area. On the actual day of testing another series of exposures is administered to an adjacent untreated site of unprotected skin to re-determine the MED. An adjacent test site is then selected to perform a static determination on the test substance.

A reference sunscreen, as described in Annex c of the ISO 24444 Standard, is also applied to each of the test subjects, utilising an application exposure procedure which is the same as that utilised for the test product.

Following UV exposure to all test sites, the product is gently removed using moist soft tissue together with ethanol if needed.

### 11. Evaluation of Response

The volunteers are instructed to return to the testing facility sixteen to twenty four hours post exposure, for evaluation of delayed erythemic response. The smallest exposure or the least amount of energy required to produce unambiguous redness (MED<sub>p</sub>) in the treated site is recorded. The SPF is then calculated by the equation: MED<sub>p</sub> ( Protected Skin ) / MED<sub>u</sub> (Unprotected Skin) = SPF<sub>i</sub> (calculated to one decimal place).

### 12. Calculations and Statistics

The SPF<sub>i</sub> values from an initial panel of the first 10 test subjects are sequentially evaluated in order to determine a provisional mean Sun Protection Factor (SPFn1). The statistical criteria described in the test method were then applied to determine a confidence interval and statistical variance. Where necessary, additional subjects will be tested according to the protocol if the first 10 results are not found to be within the specified range.

### 13. Rejection Criteria

Panelist's results will be rejected and the panelist replaced if:

13.1. The responses on the treated test site are randomly absent or out of sequence. This is an indication that the products are not spread uniformly.

13.2. An MED could not be obtained due to elicited response at all exposure sites.



13.3. The exposure series failed to elicit an MED response on either the untreated or the applied skin areas.

The test is then considered a technical failure and the subject's data is discounted.

**14. Individual Panelist Results**

These will be set out in the attached report in ISO 24444 spreadsheet format.

**15. Observations**

No adverse effects or unexpected reactions of any kind will be recorded.

**16. Archiving:** All original samples, raw data sheets, technicians notebooks, correspondence files and copies of final reports and remaining specimens are maintained on premises of Dermatest Pty Ltd in limited access storage files. A duplicate disk copy of final reports is archived separately off site.

**17. Colour Discrimination Test**

All technical employees of Dermatest Pty Ltd who are involved in scoring of exposed skin spots are required to take and pass a visual colour discrimination examination using the Farnsworth-Munsell 100 Hue Test.

Labelled category	Labelled sun protection factor	Measured sun protection factor (measured in accordance with the principles recommended in point 10 (a))
'Low protection'	'6'	6-9,9
	'10'	10-14,9
'Medium protection'	'15'	15-19,9
	'20'	20-24,9
	'25'	25-29,9
'High protection'	'30'	30-49,9
	'50'	50-59,9
'Very high protection'	'50 +'	60 ≤

SPF Found		Category Description
Tested SPF	Label SPF	
4-14	4	Low
	6	Low
	8	Low
	10	Low
15-29	15	Medium or Moderate
	20	
	25	
30-59	30	High
	40	High
	50	High
60 or higher	50+	Very High

**18. Certification for E.U. Compliance**

Where requested, an additional certification, indicating compliance with the E.U. requirements for sunscreens, can be provided.



### 19. Certification for AS/NZS 2604 (2012)

Where requested, an additional certification, indicating compliance with AS/NZS 2604 (2102) requirements for SPF and Water Resistance requirements for sunscreens, can be provided.

### References

1. Dermatest SOP – 010 Procedure for Conducting an SPF Study.
2. International Standard ISO 24444 (2010) - Cosmetics - Sun Protection Test methods *-in-vivo* determination of the sun protection factor (SPF) Factor (SPF).
3. Australian New Zealand Standard AS/NZS 2604 (2012) – Sunscreen Products – Evaluation and classification.
4. Commission Recommendation of 22 September 2006 on the efficacy of sunscreen products and the claims made relating thereto. *Official Journal of the European Union L 265/39*
5. WMA Declaration of Helsinki – for Medical Research Involving Human Subjects.