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PRÜFBERICHT

In Vivo Bestimmung des Sonnenschutzes

International Sun Protection Factor (SPF) Test Method - 2006

nach COLIPA - CTFA SA - JCIA - CTFA US

In Vivo Bestimmung der Wasserresistenz

Sun Product Water Resistance - 2005 (Spa Pool)

nach COLIPA

Ort der Durchführung:	**Institut Dr. Schrader Hautphysiologie** **Max-Planck-Straße 6** **37603 Holzminden - Deutschland**
Auftrags-Nr.:	**295-HP-09-000-09-092**
Projekt-Nr.:	**HP-09-040b**
Berichtsdatum:	**12.03.2013**

Kunde:

Testprodukt: **Sonnenschutzcreme UV 30**

Entwicklungsnr. 468 (80427-01)

im Auftrag der
Adolf Würth GmbH & Co. KG
Reinhold-Würth-Straße 12-17
74653 Künzelsau-Gaisbach
Deutschland

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SPF-Test / Wasserresistenz

Zusammenfassung

Testprodukt: **Sonnenschutzcreme UV 30**
Entwicklungsnr. 468 (80427-01)

im Auftrag der
Adolf Würth GmbH & Co. KG, Reinhold-Würth-Straße 12-17,
74653 Künzelsau-Gaisbach, Deutschland

Zielsetzung: Sonnenschutzfaktor- (SPF) und Wasserresistenz-Bestimmung auf Grundlage der Internationalen-SPF-Testmethode 2006 und der Richtlinie zur Auswertung der Wasserresistenzbestimmung von Sonnenschutzprodukten (COLIPA 2005)

Projektdurchführung: K. Schünemann, K. Kucher, B. Melching, D. Kordts, I. Mosebach, K. Kohlmetz

Testzeitraum: 02.03.2009 - 07.03.2009 / 26.03.2009 - 03.04.2009
Referenz: Standard P3
Probanden: 12 weiblich, 0 männlich / gesamt: n = 12
Alter: 21 - 65 Jahre
Dosisprogression: 12 %
Auftragsmenge: 2.00 ± 0.05 mg/cm²
Zertifikat Sonnensimulatoren: März 2008
Abweichung vom Protokoll: keine

Ergebnisse: Die in diesem Bericht dokumentierten Ergebnisse basieren auf dem in Anhang 2 beigefügten Protokoll.
Die statistischen Anforderungen der Prüfrichtlinie werden für Testprodukt und Referenzprodukt erfüllt.

Produkt	Mittelwert				Empfohlene Auslobung		
	SPF		Wasser-resistenz	WRR	SPF	Produkt-kategorie	Wasser-resistenz
	static	wet	[%]	[%]			
Testprodukt	38.2	26.2	67.7	65	**30**	**Hoher Schutz**	**Ja**
Standard P3	15.1			--	innerhalb der vorgegebenen Grenzen		

In dieser Untersuchung erreicht das Testprodukt einen mittleren SPF von 38 vor Wässerung und einen mittleren SPF von 26 nach Wässerung. Daraus kann ein Label SPF von 30 (Hoher Schutz) abgeleitet werden.

Das Testprodukt erreicht das Kriterium 'Wasserresistent'.

Dr. Andreas Schrader

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Dr. Mathias Rohr (Direktor)

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ANHANG 1

Daten - Statistik

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International Sun Protection Factor (SPF) Test Method - 2006

COLIPA - CTFA SA - JCIA - CTFA US

Institut Dr. Schrader Hautphysiologie

12.03.2013

Sun Product Water Resistance - 2005 (Spa Pool)

COLIPA

Kunde: / Projekt-Nr.: HP-09-040b

VOLLSTUDIE — **Sonnenschutzcreme UV 30 Entwicklungsnr. 468 (80427-01)**

Probandencharakterisierung						Testprodukt							Standard P3		Datenausschluss-kriterium	Auftragsmenge		
						Static			Wet (Spa Pool)							Testprodukt		P3
Nr	Initialen	Alter	Geschl.	Hauttyp	ITA°	MEDu [mJ/cm²]	MEDp [mJ/cm²]	SPF_{static}	MEDu [mJ/cm²]	MEDp [mJ/cm²]	SPF_{wet}	Wasser-resistenz [%]	MEDp [mJ/cm²]	SPF		Static [mg]	Wet [mg]	[mg]
1	N.R.	23	w	I	62	19.3	648.6	33.6	19.3	432.4	22.4	65.6	328.2	17.0		74	73	71
2	M.H.	21	w	I	56	18.7	627.5	33.6	18.7	418.3	22.4	65.6	226.0	12.1		72	73	74
3	V.F.	22	w	II	52	24.9	870.5	35.0	24.9	621.8	25.0	70.6	422.8	17.0		72	74	73
4	S.A.	49	w	II	53	19.6	686.8	35.0	19.6	438.0	22.3	62.7	265.9	13.6		73	73	71
5	M.J.	50	w	III	34	27.8	887.3	31.9	27.8	554.6	19.9	61.3	377.1	13.6		72	72	74
6	A.R.	40	w	II	54	21.6	967.4	44.8	21.6	677.2	31.4	69.3	327.8	15.2		74	73	72
7	L.R.	47	w	II	47	21.1	601.2	28.5	18.9	420.8	22.3	77.6	320.5	15.2		72	72	72
8	S.W.	33	w	II	43	25.0	1122.1	44.8	25.0	701.3	28.0	61.6	425.8	17.0		74	74	73
9	A.G.	43	w	I	63	18.9	848.6	44.8	16.9	594.0	35.1	77.9	287.5	15.2		74	74	75
10	B.Z.	43	w	II	53	19.6	784.1	40.0	19.6	490.1	25.0	61.5	297.5	15.2		74	74	72
11	B.H.	61	w	II	48	21.0	1051.5	50.2	18.7	657.2	35.1	69.4	318.1	15.2		74	70	74
12	D.M.	65	w	I	58	16.3	583.5	35.7	14.6	364.7	25.0	69.1	248.0	15.2		72	72	72
Anzahl						12	12	12	12	12	12	12	12	12		12	12	12
Mittelwert						21.2	806.6	**38.2**	20.5	530.9	**26.2**	**67.7**	320.4	**15.1**		73.1	72.8	72.8
Standardabweichung						3.3	181.3	6.6	3.8	117.4	5.2	5.8	63.0	1.5		1.0	1.2	1.3
Konfidenzintervall (CI)								** 4.2			** 3.3	* 2.3		0.9	Auftragsfläche [cm²]	36	36	36
Konfidenzintervall [%]								11.0			12.5	3.4		6.3	mittl. Auftragsm. [mg/cm²]	2.03	2.02	2.02
untere Grenze des CI								33.9			22.8	65.4		14.1	Standardabweichung	0.028	0.033	0.036
obere Grenze des CI								42.4			29.5	70.0		16.1				
Label SPF / WRR								**30**				**65**						
Wasserresistenz												Ja						

WRR - Water Resistance Retention; * 90 % einseitig; ** 95 % zweiseitig

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ANHANG 2

Protokoll

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SPF – Water Resistance (Spa Pool)

1 International Sun Protection Factor (SPF) Test Method - 2006

The test is based on the International-Sun Protection Factor-Test Method (I-SPF-TM) as defined in 20061 by COLIPA – CTFA SA – JCIA – CTFA US.

Test areas (6 x 6 cm2) on subjects' backs are coated with 2.00 ± 0.05 mg/cm² of test product. The quantity applied is controlled by reweighing. Between product application and irradiation a waiting time of 15 minutes is realized. Irradiation is carried out by 6 different doses. 20 ± 4 hours after the end of irradiation the resultant erythema is used to ascertain the minimum erythema dose (MED). The individual Sun Protection Factor (SPFi) is determined from the ratio of MEDu (MED of untreated skin) and MEDp (MED of protected skin) of the area treated with the product in question.

An SPF result obtained using the I-SPF-TM 2006 is expressed as the arithmetical mean of the individual SPF values obtained from the total number of subjects used, including the calculation of the 95%-Confidence Interval (95%-CI).

1.1 Reference Product

A reference formulation is included in the test to be used as a methodological control to verify the test procedure. Three reference products are available named P2, P3 and P7 (only if expected SPF < 20). The following acceptance limits are given:

Tab. 1: Reference Products

Reference Product	Mean SPF	Expected Range (± 2 SE)	
		Lower Limit	Upper Limit
P2	16.6	14.2	19.0
P3	16.2	13.8	18.7
P7	5.1	4.4	5.9

1.2 Selection of Subjects

Subjects taking part in this test are selected on the basis of Fitzpatrick's skin type table or on the basis of skin colour typing by colorimetric measurements, respectively. They correspond to the majority of users as far as their skin sensitivity classification is concerned. The following types result:

Tab. 2: Skin colour typing

Phototype	Skin colour typing	expected ITA° value*		Phototype	Skin colour typing	expected ITA° value*
Type I	very light	> 55°		Type IV	tan (or matt)	from 41 to 28°
Type II	light	from 42 to 55°		Type V	brown	- 30 to 10°
Type III	intermediate	from 29 to 41°		Type VI	black	< - 30°

* ITA° = [Arc Tangent {(L*-50) / b*)}] 180 / 3.14159

The following persons are automatically excluded from the test group: pregnant or lactating women - subjects under medication (e.g. cortico therapy) - subjects with dermatological problems - subjects who have a history of abnormal response to sun - subjects accustomed to using UVA sun beds.

According to the Declaration of Helsinki[2] subjects consent to the study in writing. Beforehand they are informed about the study, its objectives, probable benefits, potential risks and troublesome aspects, as well as about rights and responsibilities.

1.3 Excitation Spectrum

A 300 W Xenon light source is used by the Multiport-601 system for excitation. By filtering with a WG 320 and a UG 11 filter the accuracy of the International-SPF-Test Method for the excitation source is guaranteed. These criteria are checked frequently. The excitation spectrum is recorded with a PMT detection system (photo-multiplier-tube and double monochromator). From this the erythemal effectiveness of each wavelength band is calculated as a percentage of the total erythemal effectiveness from < 290 to 400 nm (Relative Cumulative Erythemal Effectiveness) and compared to the guidelines. The wavelength accuracy of the detection system is checked frequently with a Holmiumoxid filter. This guarantees an accurate International-SPF-Test Method spectrum at any time.[3,4] In addition an external certificate of the spectral conformity with the guideline is available.

Irradiation spots of a Multiport-601 have an area of 0.64 cm², each. Exposed sub-sites have a distance of 0.8 cm.

1.4 Number of Subjects

The number of subjects is restricted to a minimum of 10 and a maximum of 20 valid results in the International-SPF-Test Method. A minimum of 10 volunteers is sufficient if the 95 % confidence interval of the mean SPF falls within a range of ± 17 % of the mean SPF. Otherwise, the number of subjects is increased from 10 until the statistical criterion is met. A maximum of five individual results may be excluded from the calculation of mean SPF. Each exclusion has to be justified. All individual results are included in the report. Test data are rejected under the following circumstances: 1) The exposure series on a subject fails to elicit an erythemal response on any sub-site, 20 ± 4 hours after exposure. 2) Erythemal responses within an exposure series are randomly absent 20 ± 4 hours after exposure. 3) All sub-sites in the exposure series show an erythemal response 20 ± 4 hours after exposure.

1.5 Product Quantity

The I-SPF-TM recommends an amount of application of 2.00 ± 0.05 mg/cm². Annex 1 gives a table of the individual amounts of application with statistics. The amounts of application are determined with the aid of a precision balance before and after product application.

1.6 Ambient Conditions

Product application, UV-exposure and MED assessment are carried out in stable ambient conditions. Testing is performed in an air-conditioned laboratory with a room temperature of 25 ± 1°C.

[1] International Sun Protection Factor (SPF) Test Method, COLIPA, CTFA-SA, JCIA, CTFA, 2006
[2] Declaration of Helsinki, 52nd World Medical Assembly, Edinburgh, Scotland, October 2000
[3] Rohr M., Schrader A., Schrader K.; Die Bestimmung des Sonnenschutzfaktors nach COLIPA; Parfümerie und Kosmetik, Hüthig Verlag Heidelberg, 12 – 19, 5/1998
[4] Rohr M., Schrader A.; Quality of irradiation – An essential criterion of SPF determination. SÖFW Journal, 3, 2003

1.7 Incremental Progression of UV-Dose

A geometric progression of 1.12 or 1.25 can be used. If the expected SPF is > 25 a progression of 1.12 has to be used. Irradiation is carried out by 6 different doses.

2 Determination of Water Resistance

For the determination of water resistance the SPF testing is carried out twice at each subject. On one side of the back an SPF measurement according to the I-SPF-TM-2006 is carried out, whereas on the contra lateral side SPF testing is combined with a standardized watering procedure.

Watering of the test areas takes place in accordance with the COLIPA Guidelines of Evaluating Sun Product Water-Resistance[5]. Subjects stay in a Spa Pool twice for 20 minutes. A short outline of the schedule as well as the technical conditions of this watering procedure is specified in table 3.

Tab. 3: Water-Resistance-Testing

1	Application of product	2.0 ± 0.05 mg/cm²
2	Waiting time / Drying time	15 min
3 / 7	First stay in Spa Pool	20 min
4 / 8	Drying time at air	15 min
5 / 9	Second stay in Spa Pool	20 min
6 / 10	Drying time at air	15 min / or up to complete drying of test areas
11	Irradiation of watered areas	

The following technical parameters are ensured:

1. Water in the Spa Pool is held with a water jet in motion. Air jets (bubbles) are not used.
2. Test areas are completely covered by water. During watering no contact of test area and any surface of the Spa Pool is guaranteed.
3. Water quality corresponds to the EC Council Directive 98/83 EC (November 3rd, 1998).The content of magnesium and calcium is appropriate between 50 mg/ml and 500 mg/ml.
4. Water temperature is 29 ± 2 °C.
5. Water is sanitised by chlorine.

The individual and mean percentage water resistance (%WR_i) are calculated by the following formulae:

$$\%WR_i = \left(\frac{SPF_w^i - 1}{SPF_s^i - 1}\right).100 \qquad \text{mean}\%WR = \frac{\sum_{i=1}^{n} \%WR_i}{n}$$

SPF_s^i = individual SPF without water immersion (static)
SPF_w^i = individual SPF after water immersion (wet)
%WR_i = individual percentage water resistance
mean %WR = mean percentage water resistance

Taking into account the COLIPA Recommendation N°16 (2005) dealing with water-resistance labelling the Water Resistance Retention (WRR) is calculated as the lower unilateral 90 % confidence limit of the arithmetic mean of the individual %WR_i.

$$WRR = \text{mean } \%WR - d \qquad d = \frac{t_u \cdot s}{\sqrt{n}}$$

s = standard deviation
n = total number of subjects 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

A product reaches the criterion of the Water Resistance examination if the WRR value is ≥ 50 % and will fulfil for the static SPF value the statistic criteria of the international SPF test method of 2006.

3 Labelling of SPF Numbers and Optional Product Categories

Based on the European Commission Recommendation from September 2006[6] and COLIPA statement (26.09.2006), basis of labelling should be the mean SPF obtained in application of the I-SPF-TM-2006. The efficacy of sunscreen products should be indicated on the label by reference to categories. The variety of numbers used on labels for indicating the SPF is restricted to those and only those shown in Table 4. The range of SPF's for each category and the respective labelling is defined as follows:

Tab. 4: Categories and possible SPF

Labelled Category	Labelled SPF	Measured SPF
Low Protection	6	6.0 – 9.9
	10	10.0 – 14.9
Medium Protection	15	15.0 – 19.9
	20	20.0 – 24.9
	25	25.0 – 29.9
High Protection	30	30.0 – 49.9
	50	50.0 – 59.9
Very High Protection	50+	≥ 60

The minimum degree of protection provided by a sunscreen product is an SPF of 6. The category of sunscreen products should be indicated on the label at least as prominently as the SPF.

[5] Guidelines for Evaluating Sun Product Water Resistance, COLIPA, 2005

[6] Commission Recommendation of 22 September 2006 on the efficacy of sunscreen products and the claims made relating thereto (notified under document number C(2006) 4089) (Text with EEA relevance) (2006/647/EC); Official Journal of the European Union, L 265/39