HelioScreen

HelioNews

News about In Vitro Sun Protection Testing

HN 2015 N°22

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Congress & Events

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Porte de Versailles, Paris, 12-14 April 2016

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Editorial

Things are moving… slowly. The hope of an international accepted method for in vitro SPF within the ISO process has been stopped by some members of the group who finally stated there would be no enough advance to go on. Some of them never assisted or participated to the group since the beginning, other changed their mind despite of a prior unanimous acceptation from the group reported to the ISO committee. It is a fact, political was stronger than technical but it is clear we need an in vitro method and we also need a method which is reliable and give the same value in all worldwide laboratories (in vivo and in vitro methods). Indeed, as we had the knowledge of several in vivo SPF values from different laboratories, it was easy to compare the correlation in vivo/in vivo vs. in vivo/in vitro. The conclusion, it was at the same level!

Thus, today, everybody knows about the dispersion of the in vivo method and the fact than anyone can choose the institute to get the most convenient claimed SPF. So, I really wonder how to improve whatever the method in the future?

Our laboratory has been a great contributor and I really think it is better not to lose our time and money again following the proposal of starting everything again… at least in the ISO committee. I spend a lot of time in Asia and see how the European methods are credible, some times more than international ISO, so I hope something could come from the west again…

Dominique Lutz, CEO Scientist Manager

New claim for photostability performance

INTRODUCTION

One paradox of organic sunscreens is that in order for them to protect against UV radiation, they must be exposed to it, which induces their photochemical degradation and, in turn, a progressive loss in efficacy. Even if most experts would agree that indicating the photostability of a product would be of compelling interest for consumer safety, different methods have been proposed to determine this characteristic without validation mainly due to their lack of inter-laboratory reproducibility.
The aim of the present work (already published **[1]**)was to develop a reproducible in vitro method, based on UV transmission measurements at two irradiation doses, to test and rank sunscreens based on their photostabilities. This approach was used to assess some 107 sunscreens and shows how, by strictly controlling key parameters, comparisons between the photostabilities of products can be made, with potential for new label claims.

METHODS

Following are the main steps of the test procedure. Specific details of the test method and the criteria selected are given in the published article.

1. Conduct calibrations and validations of:
i) properties of the test plates[a]; ii) environmental conditions such as temperature at the substrate surface[b] (25°C); iii) the automatic syringe and balance for product application (1.3 mg/cm²); iv) the automatic/mechanical robot or device used for product spreading[c]; v) the UV spectrophotometer used for transmission/absorbance measurements[d]; and vi) the UV exposure source for irradiation, including the UV radiometer or spectroradiometer used[e].
2. Conduct blank measurements of a glycerin or white petroleum-treated plate for the reference "blank" to compare with subsequent absorbance measurements.
3. Spread the sunscreens at 1.3 mg/cm² in an automated manner onto molded polymethyl methacrylate (PMMA) plates (Helioplate HD6), and take at least three replicate in vitro absorbance measurements after a 30-min drying step, and prior to any UV irradiation; this establishes the initial UV spectrum or T0 (λ) data.
4. Expose the product-treated substrate to a first UV irradiation dose of 4 MEDs.
5. Measure the in vitro absorbance of the sunscreen after the first UV exposure to establish a second UV spectrum, or Tt1 (λ) data.
6. Expose the product treated-substrate to a second UV irradiation dose at 4 MEDs (i.e., for a total of 8 MEDs).
7. Again measure the in vitro absorbance of the sunscreen after the second UV exposure to determine a third UV spectrum, or Tt2 (λ) data.
8.Calculate two UV A+B residual efficiency percentages for the first (Tt1) and second (Tt2) UV exposure doses according to **Equation 1** for the result on a one plate and **Equation 2** for the arithmetic mean.

$$\text{UVA+B \%REn} = \frac{\sum_{290\ nm}^{400\ nm} 1 / T(\lambda)t - 1}{\sum_{290\ nm}^{400\ nm} 1 / T(\lambda)0 - 1} \times 100 \qquad \textbf{Eq. 1}$$

$$\text{UVA+B \%RE} = (\Sigma\ \text{UVA+B \%REn}) / n \qquad \textbf{Eq. 2}$$

Here, T(λ)0 is the transmission before irradiation, T(λ)t is the transmission after the irradiation dose and n representing the number of plates. The same equation is used for any UV irradiation dose.

a. Helioplates HD6, HelioScreen; b. HD-THERMASTER, HelioScreen; c. HD-SPREADMASTER, HelioScreen; d. UV-2000S, Labsphere Inc.; e. Pre-Irradiation Solar Simulator Model 16S-300-009 and UVB sensor PMA 2101 biologically-weighted erythema, Solar Light Company Inc.

RESULTS & DISCUSSION

a) Photodegradation Results

In the first step of this process, the photostability behaviors of the all products were assessed and three common evolutions of residual efficacy, depending on the photo-behavior of the product, were observed: no photodegradation (a), linear photodegradation (b) or polynomial photodegradation (c). This fact lead to considerIn addition, the degradation differently impacted UVB alone, UVA alone, or both UVA+B.

Using theses results, photostability levels were defined and ranked in order to represent the current sunscreen market. For this purpose, four categories according to Superior, Moderate, Minimum and None have been proposed with respectively a representation of 40%, 30%, 20% and 10% of all tested products (**Figure 1** represents the photostability behavior ranking used as an "abacus").

However, determining a product's photodegradation behavior requires more than one UV exposure to reliably rank its photostability. This is because any sun-induced photodegradation could be highly variable and dependent upon in situ circumstances, even with strict protocols. Consequently, the present method does not attempt to precisely define a product's photostability level after one unique in situ UV exposure, but instead sets a maximum limit of photodegradation that might be considered reasonable for a product after two standardized exposures. Even if, in theory, any two different irradiation doses could be selected, an interesting compromise between the testing time possible in a laboratory (for practical reason) and the minimum requirement to ensure reproducibility (e.g., drying influence without UV exposure) lead to select a first exposure dose at 4 MEDs and a second at 8 MEDs (equal to twice the first).

b) Labeling Proposal

Table 1 therefore summarizes the minimum requirement of UVA+B %REf for the two irradiation steps using the test method proposed and a possible photostability ranking based on a number of plus signs (+). This **Table 1** have to be used to allocate plus ratings on the basis of both the first and second exposure UVA+B %RE.
Finally, **Table 2** offers a logo depiction for product labels based on the results from **Table 1**. Through this proposal, manufacturers could display photostability information for consumers in a simple manner to indicate the reliability of the SPF sun protection provided during use.

CONCLUSION

Proposed here is a test method to determine the photostability behavior of sunscreens based on 107 commercial sunscreen products tested and according to a minimum of two UV irradiation doses; i.e., 4 MEDs and 8 MEDs.

First, by tightly controlling different key parameters, the method provided consistent and reproducible results (Coefficient of Variation equal to 0.2% and 0.3% for 4 MEDs and 8 MEDs, respectively). Obviously, if the different key parameters are not strictly followed, the reliability of the method could be challenged.

From the all tested products, three different photodegradation behaviors were observed (linear and polynomial photodegradation, as well as no degradation) and the results were used to rank the products based on the residual efficiency of their UV A+B parts, or UVA+B %RE.

These results were then used to propose a pragmatic way, on finished product labels, to communicate photostability to consumers: using a photostability rating as a new product claim. Finally, this study confirmed the need to control the photostability behavior of sunscreen products for the safety of consumers. This, in turn, should save on costs and improve the efficacy of ad hoc UV filter combinations used during sunscreen product development.

«Read the full article on the website : http://www.cosmeticsandtoiletries.com/testing/efficacyclaims/Dynamic-Photostability-Test-Method-for-Additional-Sunscreen-Claims-337631501.html»

Figure 1. Photostability Ranking «Abacus»

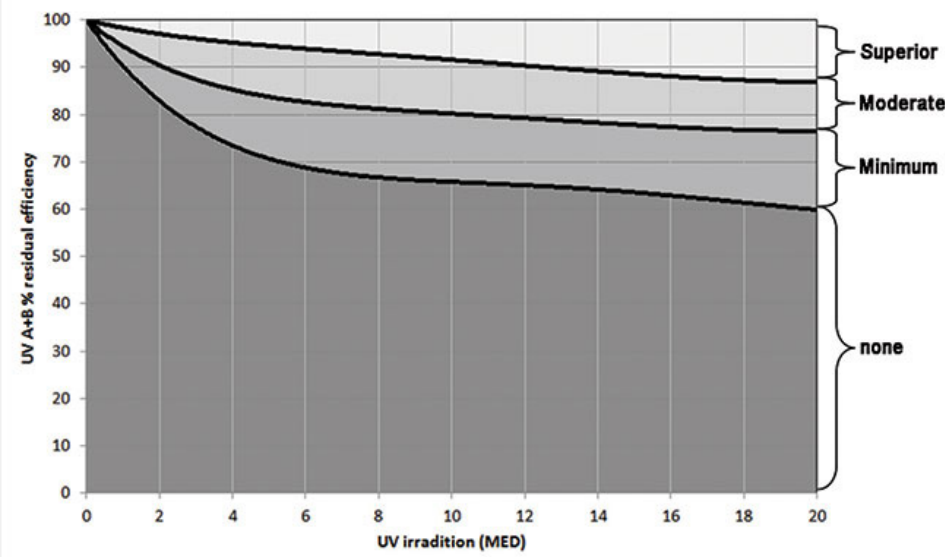


Table 1. Plus' Category According to UV irradiation Steps and UV A+B Residual Efficiency

Photo-stabitility categories for UV_{A+B} %RE_f		1st irradiation dose at 4 MEDs			
		UV_{A+B} %RE_f 100 – 95.2%	UV_{A+B} %RE_f 95.2 – 85.3%	UV_{A+B} %RE_f 85.3 – 73.5%	UV_{A+B} %RE_f Less han 73.5%
2nd irradiation dose at 8 MEDs	UV_{A+B} %RE_f 100 – 92.8%	+++	++	+	None
	UV_{A+B} %RE_f 92.8 - 81.2%	++	++	+	None
	UV_{A+B} %RE_f 81.2 - 66.7%	+	+	+	None
	UV_{A+B} %RE_f Less than 66.7%	None	None	None	None

Table 2. Proposed Label Logo for Photostability

Classification	Superior	Moderate	Minimum	none
Plus category	+++	++	+	n/a
Label proposal	PHOTO-STABILITY +++	PHOTO-STABILITY ++	PHOTO-STABILITY +	n/a

Reproducibility of *in vivo* Water Resistance value

INTRODUCTION

Nowadays, in addition to the well know Sun Protection Factor (SPF) or UVA Protection Factor (UVA-PF), the Water Resistance (WR) for assessing sun product water resistance was often measured. Even if, several In Vitro methods have been published **[footnotes]**, only In Vivo method can be used for consumer guidance. For this purpose, different current standardized methods can be already used through the FDA monograph 2011, the AS/NZS2604:2012 or the COLIPA - Guidelines for Evaluating Sun Product Water Resistance 2005. Moreover, to access a worldwide reproducible method, the harmonization of WR determination have been started and still in progress through the ISO 16217 ("Cosmetics — Sun protection test methods - Water resistance — Water immersion procedure") and ISO 18883 ("Cosmetics - Sun protection test methods – Water resistance - Determination of percentage of water resistance").

"...a same product can be claimed Water Resistance or No Water Resistance due to In Vivo variability."

From a regulatory point of view, two principal ways could be used to support either water resistant or very water resistant claim after different immersion times with (i) an absolute value by labeling the SPF after immersion test (SPFw) or (ii) a relative value by comparing the SPF before immersion (SPFs) with the SPFw. For remind, the In Vivo SPF assessment is carried out by measuring the Minimal Erythemal Dose (MED) which consist of comparing the ultra-violet (UV) radiation dose required for the appearance of a first unambiguous biological endpoint, in this case skin redness, with and without sunscreen product protection.

In the present study, we only focused on the relative value of WR. Indeed, the possible lack of In Vivo SPF reproducibility (i.e. equal to absolute value of WR) has been already demonstrated on few products in a previous **HelioNews 20** and expanded for plenty of products in an article to be published in a future scientific magazine soon. For this purpose, we attempt to estimate with few data an example of the variability of the percentage of Water Resistance (%WRR) afforded by sunscreen products and there consequences. For that, we gather In Vivo SPF before and after water immersion values from the same testing laboratory (Intra - repeatability) and from different testing laboratories (Inter - reproducibility).

METHOD

The individual percentage of Water Resistance (%WRRi) based on the individual SPFw (SPFiw) and individual SPFs (SPFis) has been calculate according to **Equation 1**.

$$\%WRRi = \frac{(SPFiw - 1)}{(SPFis - 1)} \qquad \textbf{Eq. 1}$$

The mean percentage water resistance retention (%WRR) is expressed as the arithmetic mean of the 'n' %WRRi values. Furthermore, a product will be considered water resistant if the 95% confidence interval on the SPFs was within ± 17% of the SPFs and the value for the 90% lower unilateral confidence limit [mean %WRR – d] is greater than or equal to 50% with d calculated according to **Equation 2**.

$$d = \frac{tu \bullet s}{(n)^{1/2}} \qquad \textbf{Eq. 2}$$

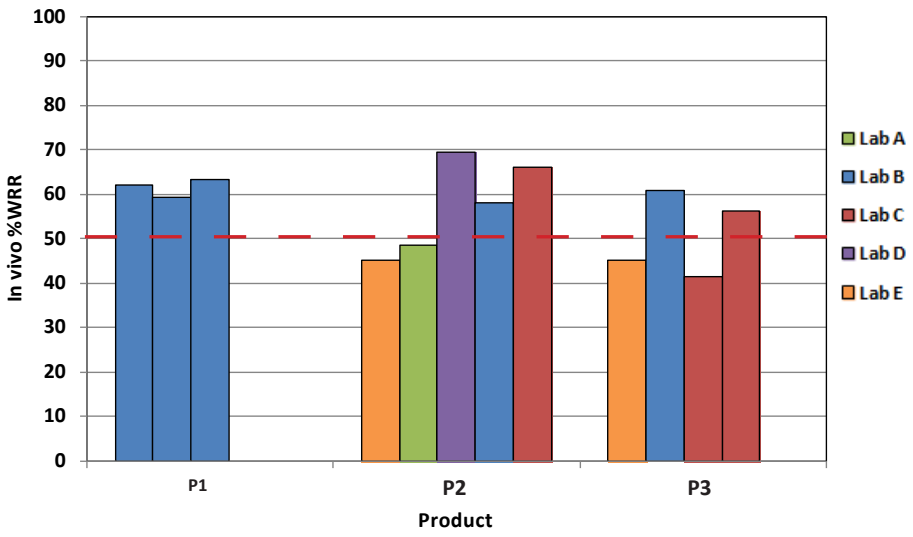
where tu is the t value from the "one-sided" Student-t distribution table at a probability level p=0.10 and with n-1 degrees of freedom; s is the standard deviation; and n is the total number of volunteers in test.

RESULTS AND DISCUSSION

The **Figure 2** shown different %WRR results for the products tested in different laboratories according to the COLIPA - Guidelines for Evaluating Sun Product Water Resistance 2005.

First of all, as it is well known, the variability is clearly always product dependent and we could have a great reproducibility for Intra and Inter laboratories as for the product P1 within the Lab B, the P2 between the Lab E & A and the P3 between the Lab E and C.

Figure 2. %WRR according to in vivo testing laboratories



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Agin, P. Water resistance and extended wear sunscreens. Dermatol. Clin. 24, 75–79 (2006).

Ahn S, Yang H, Lee H, Moon S, Chang I. Alternative evaluation method in vitro for the water-resistant effect of sunscreen products. Skin Res Technol. 2008 May 14:187-91.

Bielfeldt S, Röck C, Wilhelm KP. Chances and limits of an improved method to assess water resistance of cosmetic sunscreen products in vitro on polymethylmethacrylate plates. Int J Cosmet Sci. 2013 Feb 35:89-93.

Choquenet B, Couteau C, Paparis E, Coiffard LJ. Development of an in vitro test to determine the water-resistance of sunscreens. Pharmazie. 2008 Jul 63(7) :525-7.

M. Pissavini, V. Alard, V. Perier, D. Lutz et al. In vitro assessment of water resistance of sun care products: a reproducible and optimized in vitro test method. International Journal of Cosmetic Science, 2007, 29, 451–460

Nevertheless, for some borderline products such as the samples P2 and P3 , we can observe a poor reproducibility and repeatability for In Vivo %WRR assessment for Inter and Intra labs. Indeed, the product P2 can claim Water Resistant from the 3 different laboratories D, B and C (respectively %WRR equals to about 70%, 58% and 66%) but the same product cannot claim Water Resistance from the 2 other laboratories E and B (respectively %WRR equals to 45% and 48%). With the product P3, from the same laboratory, the same product can also be claimed or no Water Resistance according to the test replicate (i.e. the Lab C measured a %WRR equals to 40% and another one equals to 56%).
In other words, according to the test replicate (inter or intra labs), a same product can be claimed Water Resistance or No Water Resistance due to In Vivo variability.

CONCLUSION

Currently, the In Vivo Water Resistance test according to COLIPA - Guidelines for Evaluating Sun Product Water Resistance 2005 is one of the most worldwide used for Water Resistance claiming based on relative value. However, as it has previously been demonstrated, even if the reproducibility could be sufficient for some products, for another one the variability can lead to confuse consomer guidance with wrong efficacy claim. Obviously, it is the case for this WR method but without any doubt, other current WR methods will show the same issues.

Fortunately, an ISO process is in progress with the goal to obtain international and reproducible WR method soon. For this purpose, the method will be improved in terms of specification but as an evidence, beyond economical reasons, to bring the proof of reliability of these future standards for ethical reasons, this future ISO method must be tested and used with plenty products (and not only 1 or 2 products...) and with several multi-center studies.

Finally, looking back, criteria for success have never been defined among laboratories by which we can consider the In Vivo WR method to be sufficiently accurate and reproducible for consumer health, for predicting what will happen in the marketplace and with the consumer. In absence of any definition, for practical and for health protection reasons, we need to agree how much overestimation or underestimation of the WR protection level between different In Vivo values for the same sunscreen product is tolerable. As it is a relative value, it seems reasonable that above a maximum of 10%, the method could be considered a failure in a larger correlation field of randomly picked and tested samples anywhere in the world.

Scientifics articles and conferences

[1] Cosmetics & Toiletries - November 2015
S. Miksa, D. Lutz and C. Guy. Photostability Test for Additional Sunscreen Claims - Part I: Protocol Setup - Part II: Calculations and Results - Part III: New claim.

Florida Sunscreen Symposium, Orlando - September 2015
T. Harding, S. Miksa and D. Lutz. Aspects of in vitro material handling and new developments in in vivo and in vitro automation.

Sun Protection Conference, London - June 2015
D. Lutz. In vitro SPF for label claim: fact or fiction?

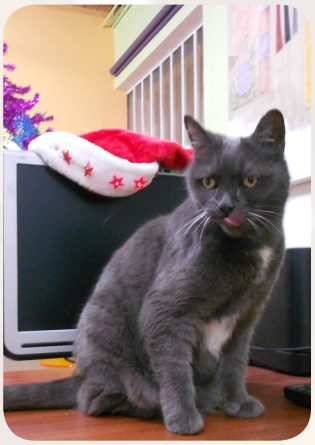
Cosmetics Business Regulatory Summit, Brussels - May 2015
S. Miksa. How to ensure your innovative ideas comply?

in-cosmetics Workshop, Barcelona - April 2015
D. Lutz. UVA protection factor in vitro method.

All the team of HelioScreen & HelioScreen Asia Co., Ltd. wish you a Happy Holiday and a joyful New Year

Sébastien MIKSA | Lydia MINIC | Céline GUY | Dominique LUTZ | Nicolae POPA

Elise DELAMOUR | Budsarin TIPLAMAI



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