HelioScreen

HelioNews

News about In Vitro Sun Protection Testing

HN 2014 N°19

Contents

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Editorial

We can take more and more for sure, an In Vitro SPF method will be available in the next years. Clearly the way is still long because the level of requirements for repeatability, reproducibility between laboratories and finally correlation have been fixed higher than the existing In Vivo methods. Moreover, we have, on these In Vivo values, more or less no information about reproducibility. It is quite sure correlation step will be challenging as long we have also some suspicions about In Vivo results for same product performed in different laboratories or countries. It is already huge problems to solve but there is another reason to be concerned by the last steps to get at least a reliable method.

Beyond people who are expecting for a method very soon on the base on the recent results about reproducibility due to the opportune proposal of the robot and demonstration it is the only way to get it, most are thinking we have just now to fix the conditions of quantity, substrate etc… to reach the method. But the solution will not be unique. The solution will come from an adaptation depending of the kind of product and surely the application on several substrates. Most of them will surely fight this approach as too complicated and just waiting for the best mean correlation between the former In Vivo method and the new In Vitro one. They surely forget it is the case for plenty of methods such as viscosity, HPLC, etc…

Already some people have challenged price of new equipment required to ensure reliability arguing it is too expansive for developing countries. Let's be serious, How do they do with in vivo testing now? And price of in vitro will be still cheaper than In Vivo. Anyway, there are already at least 2 laboratories which propose testing services with these technologies such as robotic spreading and so accuracy for the same price than before: One in Europe the other one in Asia.

Dominique Lutz, CEO Scientist Manager

I. The ISO 24445*: What could be the changes brought by the future In Vitro SPF standard?

I.a. Introduction

While obligations and requirements for the In Vivo SPF assessment have been strictly defined since many years, nothing has been yet decided for In Vitro SPF testing. **Strongly recommended for the gradual replacement of In Vivo methods** on ethical grounds, they are also essential for certain assessments which cannot be carried out by means of In Vivo tests (i.e. establishing a ratio such as Critical Wavelength/Broadspectrum/ Boots Star Rating system).

If used properly, they are perfectly suited for the overall assessment of the prerequisites allowing the relevant scaling of effective sun protection. Although, the use of In Vitro method has evolved considerably, it is often **performed without enough specifications** and latest discoveries about the SPF assessment. Nevertheless, the future ISO 24445 international standards for In Vitro SPF assessment is therefore expected to change the current situation. Even if, nothing has been decided yet, the present paper imagines what could be the changes brought by this **new SPF standard based on latest innovations**.

I.b. Unavoidable evolution of the conditions of implementation

These methods, which are seemingly more affordable than the In Vivo ones, are, nevertheless, **not easy to master**. It is in fact one of the major reasons which has often suggested that they were unreliable because they were used in an amateur manner. They were seen as backup methods, usable at the best for screening purposes. There are new players on the market which provide tests without taking into consideration the need to justify either their experience or the minimum requirements to carry them out. Several organizations composed of experts such as Cosmetics Europe (ex-Colipa), publications of independent experts and, more recently, a coordinated development action have clearly demonstrated, within the ISO framework, the importance of strictness, and have also set some basic rules which should be observed in order to get the best of these methods. The implementation of these methods and the publication of the ISO 24443:2012 standard, with all the related enforcement measures, have already changed the situation and forced the testers to fulfill the requirements but this was only the first step… **A new step is necessary for the future ISO 24445 standard**…

*The official ISO 24445 standard has not been developed and published yet. Thus, the information contained in the present paper about the future ISO 24445 provided herein by HelioScreen are presented in good faith and believed to be accurate.

I.c. The requirements of the future ISO 24445 standard

This future In Vitro SPF standard, which incorporates many of the recommendations issued by the ISO 24443 for the In Vitro determination of UVA protection, also **includes new requirements and appliances**. The **Table 1** summarizes the different specifications which should be respected in order to ensure reliability of the In Vitro SPF method.

Indeed, first of all, as for the ISO 24443, **topographic parameters of substrate must be controled and respected a control chart for both molded and sandblasted plates**. The characteristics and the performance of the spectrophotometers and their regular checking with specific calibration, will be documented. The product irradiation level is defined by a strict quantity of UV (J/m²). It will be mandatory to control the emission of the source in order to calculate and justify the dose. **The temperature of the interface substrate/ sample must also be controlled during whole process.** The control of the source on each irradiation requires adequate equipment: a spectroradiometer in the best case or, in the worst case, at least a radiometer. The specific terms of the statistical calculation of acceptance or rejection on a minimum number of 4 plates must also be documented.

Furthermore, the method consists in assessing the residual transmission of a product spread in a thin layer on a substrate, a most important fact! HelioScreen has been the first to demonstrate*, and then ISO, that **automated spreading influences the result to improve reproducibility**. This is why ISO could recommend a single way of spreading (e.g. by means of a robot) that guarantees the state of the spreading. Having reached the same conclusions, all different worldwide standards and methods should suggested a change of the recommendation already published.

Finally, about the process for performing the test, a protocol's draft has been also presented (see **Table 2**) in the present HelioNews. It has been based on latest results for a reliable In Vitro SPF method in term of reproduciblity and correlation with In Vivo SPF value. For this last part, we are convinced that an **multiple-method could be a convenient solution with different product's groups**. Futhermore, the compliance with these requirements when performing a test must be completely documented in the repport. Thus, the test report on the determination of the In Vitro SPF of sun protection product should contain at least several compulsory information such as described in **Table 3**.

I.d. Conclusion

The future ISO 24445 standard will become applicable in many countries either through its direct application or because it will define the application conditions of the method for determining the basis of other In Vitro tests. The strict conditions laid down appear therefore as a prerequisite for the accomplishment of relevant In Vitro SPF tests. **The user performing the tests by itself or having them conducted by other parties will have to make sure that these rules and obligations have been respected.** We are confident that this future In Vitro SPF standard will change the way of sunscreen testing and improve consumer health sun protection.

* In Vitro UV Testing—Robot vs. Human Spreading for Repeatable, Reproducible Results, S. Miksa, D. Lutz and C. Guy, Cosmetics & Toiletries, October 2013

Table 1. Key parameters of In Vitro SPF sunscreen testing method

Factor	Comments	Key figures		
		Parameter	Molded	Sandblasted
Substrate	- Manufactured in PMMA - Checked with a profilometer - Delivered with a quality certificate	Ra (µm) Rv (µm) Rdq (°) A1 (µm²/mm) Ssc (L/µm) Vvv (mL/m²)	4.85 ± 0.32 13.04 ± 0.63 11.12 ± 1.29 239.75 ± 44.51 0.03 ± 0.01 1.04E-6 ± 6.19E-7	4.19 ± 0.51 11.40 ± 2.50 11.00 ± 1.94 238.25 ± 72.66 0.03 ± 0.01 8.70E-7 ± 2.37E-7
Environment	- Control of temperature at substrate surface - No direct sunlight - No direct air flow	25°C ± 2°C Steps: application, spreading, drying, UV exposure		
Product	- Application by means of automatic syringe - Precision balance	1.3 mg/cm² for PMMA molded plate 1.2 mg/cm² for PMMA sandblasted plate		
Spreading	- Use an automated robot (e.g. no manual spreading)	Robot with 6 motors 3 circular strokes and 3 linear strokes in 1 min Tool's hardness between 20-25 shores		
Solar simulator	- Checked with a spectroradiometer - As similar as possible to institute irradiance used during In Vivo SPF test (UV SSR source)	Irradiance level (40 – 200 W/m²) UVA/UVB ratio (8 – 22) No heating and air flow Beam uniformity (< 10%)		
UV analyzer	- Include all absorbance and scattering effects	Dynamic Range Limit at 2.2 abs (DO) Linearity Limit at 85% Wavelength accuracy test Integrating sphere Measurement each 1 nm		
Calculation	- Use the UV SSR / Natural Sunlight source and Erythema action spectrum for calculation - Arithmetic mean	C_{MOLD} and C_{SAND} Dcoeff CI[95%] < 17%		

Table 3. Information contained in final report

Description of parts
Identification of the testing laboratory
Sample identification;
Plate manufacturer and batch code;
Description of the instruments used with manufacturer and instrument model, e.g. automatic serynge, balance, automated spreading, spectrophotometer and solar simulator;
Application rate of the sunscreen (mg/cm²);
Temperature control at substrate surface (°C);
UVB and UVA irradiance ($W.m^{-2}$);
Mean UV exposure dose used to irradiate the test sample and exposure time (hh/mm/ss);
The calibration factor "Y" used to adjust the UVA radiometer measurement with the reference spectroradiometer measurement of the UV exposure source;
Results of calculated In Vitro SPF value with Standard Deviation and Confidence Interval;
Mean UV absorbance values at each 1nm wavelength increment for the test sample;
Reference data for the samples reference material with date of testing;
Identification of individual conducting the test;
Date of measurement;
Reference to this test method, e.g. ISO 24445

Table 2. Protocol for test performing

Step 1

Conduct the calibration and validation of the test equipment, including (i) the properties of the test plates, (ii) the environment condition including temperature at substrate surface, (iii) the automatic syringe and balance for product application, (iv) the robot used for product spreading, (v) the UV spectrophotometer used for transmission/absorbance measurements and (vi) the UV exposure source used for UV irradiation including the UV radiometer (or spectroradiometer) used to measure.

↓

Step 2

Conduct blank measurements of a glycerine (or white petroleum) treated plate for the reference "blank" for the subsequent absorbance measurements.

↓

Step 3

Selection of products groups.

↓

Step 4

In Vitro absorbance measurements of the sunscreen product spread on molded and/or sandblasted PMMA plates (at least 4 repeatability) by means of automated spreading, after drying step and prior to any UV irradiation. Acquisition of initial UV absorbance spectrum with $Ai_{0\text{-}MOLD}(\lambda)$ and/or $Ai_{0\text{-}SAND}(\lambda)$.

↓

Step 5

Mathematical adjustment of the initial UV absorbance spectrum using coefficients C_{MOLD} and/or C_{SAND} to achieve $Ai(\lambda)$ and finally an In Vitro $SPFi_0$ (no UV dose).

↓

Step 6

A single UV exposure dose D (J/cm^2) is calculated according to $SPFi_0$ and D_{coeff}.

↓

Step 7

In Vitro absorbance measurement of the sunscreen product after UV exposure. Acquisition of second UV spectrum with $Aii_{MOLD}(\lambda)$ and/or $Aii_{SAND}(\lambda)$ data.

↓

Step 8

Mathematical adjustment of the second absorbance spectrum (following UV exposure) and by multiplying with the same C_{MOLD} and/or C_{SAND} to achieve $Af(\lambda)$ and finally the In Vitro SPFi.

↓

Step 9

Calculation of the arithmetic mean for the In Vitro SPF and confidence interval CI [%].

PA Rating System: Worldwide misleading

Beyond UVB protection, recent studies also have shown the importance of protection against UVA rays (320 nm–400 nm). Different classification system for UVA protection level have been proposed such as UVA Logo, Broadspectrum, Boots Star Rating system and PA Rating System...

Since first PA Rating System proposed by the JCIA (Japan Cosmetic Industry Association) in 90's, a huge evolution has been done about In Vivo PPD method but also In Vitro UVA-PF method. Sometimes people makes a confusion and thinks the the PA Rating System has been proposed by the JCIA, is based on in vivo method ans is the unique PA rating method. **This is not the reality.** Method is one thing classification another one and the latest is related to local labeling regulatory. The **Table 4** shown the last updated PA Rating system.

Table 4. PA Rating System

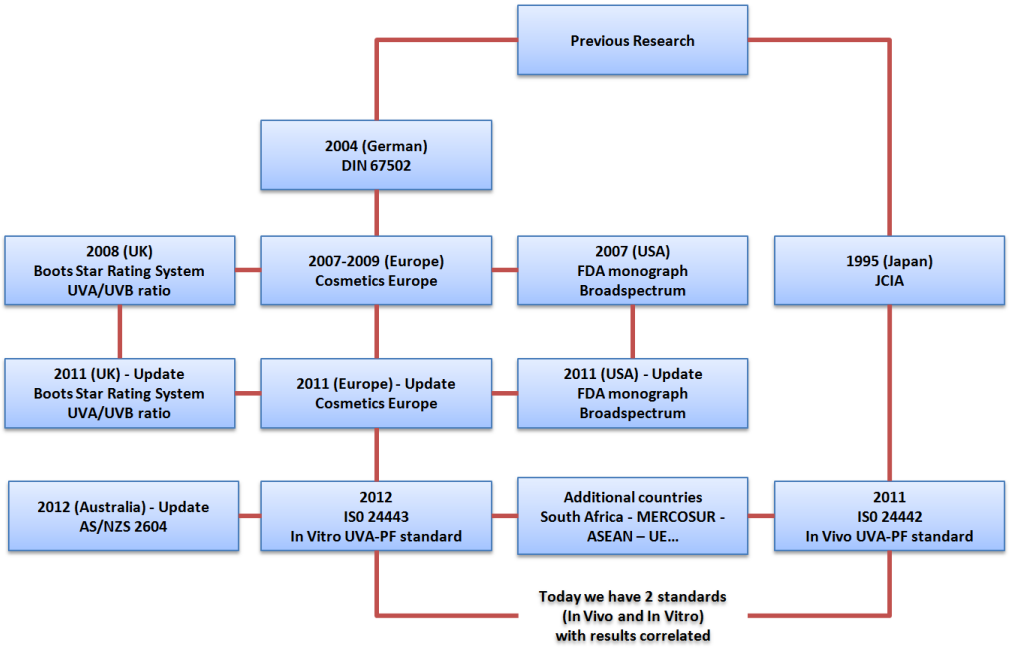
Level of performance	PFA value	Protection Grade
Low	2 to < 4	PA+
Medium	4 to < 8	PA++
High	8 to < 16	PA+++
Very High	≥ 16	PA++++

Furthermore, nowadays, whatever the UVA methods used, they are all linked and correlated. The history of UVA protocols is summarized in **Graph 1**. For example, different information can be extracted from official standards:

1) From the scope of the ISO 24442:2011 (In Vivo UVA-PF) standard: « This International Standard specifies the method for determining ultraviolet protection factor A (UVA-PF) of a sunscreen by the method of evaluation of persistent pigmentation according to the lines recommended by the Japan Cosmetic Industry Association (JCIA) in 1995.»

2) From the scope of ISO 24443:2012 (In Vitro UVA-PF) standard: «This International Standard specifies an in vitro procedure to characterize the UVA protection of sunscreen…[…]… These include calculation of Ultraviolet-A Protection Factor (UVA-PF) (correlating with in vivo UVA-PF from Persistent Pigment Darkening (PPD) testing procedure), critical wavelength and UVA absorbance proportionality.»

Graph 1. Historical of UVA-PF method



To conclude, what is important is the value we use is guaranteed by the method described whatever the system of classification. The classification of UVA protection level according to PA Rating System can be assessed by any international recognized methods (in compliance with local classification regulations).

New product from HelioScreen:
The software HelioSoft for sun protection calculation

HelioSoft

by HelioScreen

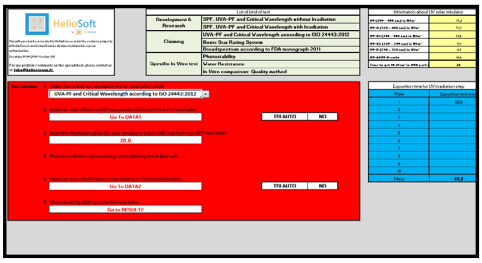
Since 1999, HelioScreen is involved in In Vitro sun protection assessment of cosmetics products. As an recognized expert in the field of In Vitro sunscreen testing, HelioScreen develops his own software for the sun protection calculation in order to ensure to customers a high reliability. It has been improved and updated in order to propose the software HelioSoft for the laboratories which perform test.

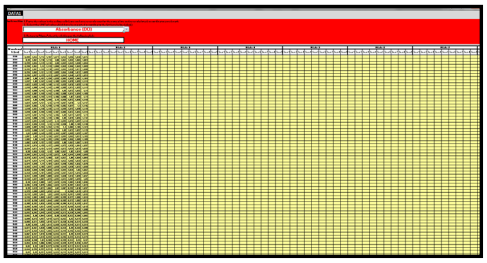
By means of this software, you just need to introduce your raw data (with selection of unit) between 290-400 nm, and you obtain all information required for screening such as **SPF, UVA-PF, Critical Wavelength, Dispersion, UVB/UVA ratio**...

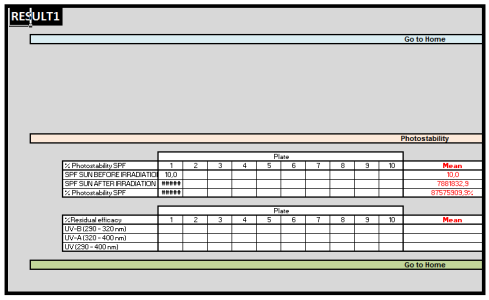
The different worldwide methods and standards (**ISO 24443:2012, Boots Star Rating system, FDA rules**...) with all information required about the process such as irradiation time (with information about irradiance in the software), calculation of confidence interval... are also available.

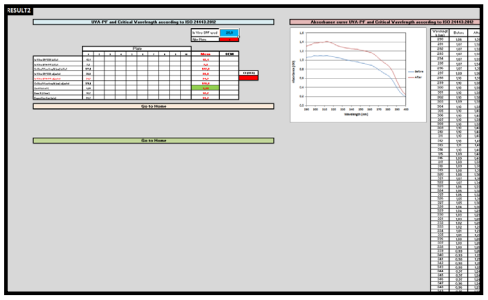
Finally, you will also have access to further information from HelioScreen methods such as the **In Vitro comparison method for quality control**, the photostability performance...

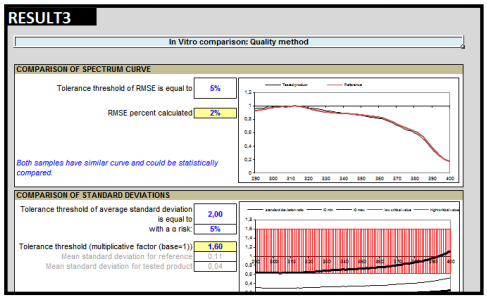
This software very easy to use and in Excel form, is clearly well adapted for In Vitro sunscreen testing and it will help you in the suncare product developement and claiming. Please feel free to contact us if you need further information about this software, we will be pleased to help you.











Audit our Quality System
ISO 9001 certified

HelioScreen's quality system is an integral part of all operations related to the realization of tests and the sale of substrates. Our laboratory is yearly checked and **Bureau Veritas Certification ISO-9001 certified since 2010** as «Research, development and evaluation of the effectiveness of the solar protection in cosmetics or pharmaceuticals.[...]».

We have established several processes in oder to control and improve activities. Furthermore, beyond our quality policy, our system is based on the «One Writing System» which ensures total traceability. The satisfaction of our customers is our goal.

ISO 9001
BUREAU VERITAS Certification

Scientifics articles

Photodermatol. Photoimmunol. Photomed. • April/June 2014:
- J. Frank Nash and Paul R. Tanner. *Relevance of UV filter/sunscreen product photostability to human safety*

INNOLAB Magazine • July/August 2014:
- B. Tiplamai, S. Miksa, D. Lutz and C. Guy. *Key parameters for reliable In Vitro sunscreen testing method*

EuroCosmetics • August 2014:
- S. Miksa, D. Lutz and C. Guy. *Development of sunscreens: Basis for efficacy formulation*

C&T • September 2014:
- S. Miksa, D. Lutz and C. Guy. *Improving the UV Exposure of Sunscreen During In vitro Testing*

SPC Supplement • September 2014:
- D. Lutz, S. Miksa and C. Guy. *The 10 reasons and hopes about In Vitro SPF method*

SPC Asia• November 2014:
- S. Miksa, D. Lutz, C. Guy and B. Tiplamai. *Advantages of In Vitro sun protection assessment methods*