

Contents

Editorial

I. Fundamentals of In Vitro sunscreen testing methods.

- Introduction
- Parameters
- Conclusion

Guideline : Issues and methods of Sun Protection Evaluation.

Partnership between HelioScreen and Shiseido

II. Solar protection in Europe vs. USA ?

- Introduction
- Materials & Methods
- Results
- Conclusion

Scientifics articles

«375 CW» label

Congress & Events



in-cosmetics Hamburg

2014 april 1-3th

Visit us at **Booth 3E62**



Sun Protection & Anti Ageing Skin Care Conference Asia.

SINGAPORE - 2014 july 9-10th

Conference by **Dominique Lutz** about «**Sun protection evaluation Facts and Perspectives**»

HelioScreen Labs

44, rue Léon Blum

60100 Creil

Phone: +33 3 44 24 33 29

administration@helioscreen.fr

«HelioNews» published by HelioScreen Labs

Editor & Design:

S. MIKSA

smiksa@helioscreen.fr

Publisher:

D. LUTZ

dlutz@helioscreen.fr

Editorial

The long process that will lead us towards reliable In Vitro tests methods, reproducible and identical for all laboratories is far from being completed. I have been advocating for years for the reliability of the in vitro tests contributing as well to the common efforts to improve these methods. Significant progresses have been made and from a technical point of view we can hope solution very soon. I am still persuaded that the reliability of the in vitro tests will be far superior to that of current in vivo testing unless we will never know for this later. Unfortunately we are still far from awareness of the indispensable respect of rules and conditions by testers, industries, institutes not to mention customers who care little about the conditions in which their tests are carried out.

If since the beginning, those tests were only considered as a screening tool, it was mostly due to the way those who made them often thought they mastered everything. Every individual has his proper way, his proper contention, his so-called expertise. Almost nothing has changed since that starting period and these certainties and claims that could have condemn the In Vitro method, will go on again for a long time. Consequently the finalization of a universal method will be challenged but this time not for technical reasons.

As we have been done for years in the technique field, we will continue to fight but now in the cultural field because we know that we must contribute to change mentalities.

Dominique Lutz, CEO Scientist Manager

I. Fundamentals of In Vitro sunscreen testing methods.

I.a. Introduction

Since many years now, we have not been able to master (or at least reproduce) absorption measurement of a product spread over a substrate. However, this is the basic principle of sun protection evaluation by means of In Vitro methods (SPF, UVA-PF, CW...).

Certainly, it is not so easy to achieve but at least ... it's quite easy to find the reasons. Everyone would agree that the principle of this test is «only» to successfully reproduce a thin uniform layer of product on a non-smooth substrate for different products or several times for the same product.

Indeed, the physical law which governs absorption spectrum measurement is the Beer-Lambert law even though we know that due to the distribution of the product, modifications are required to take into consideration the whole surface (as proposed by O'Neils). But it is not uninteresting for the understanding to focus on suitably low surfaces in order to reason with this basic law which demonstrates a direct relationship between the path in a homogeneous body and the value of the absorption as function of the length wavelength (see Figure 1).

You would have understood, the principle of the method is based on the absorbance across the traversed path which is based on the distribution of the product which itself depends on the quality of the interfacial properties between the product and the substrate on which it is spread. This interface problem has long been mastered by other industries which can understand, edit and manage to such results that there astonish us every day. For example, definitely paste incompatible materials or apply surface treatments to radically change the properties of materials, textiles and polymers.

«We have often failed because we wrongly stated problem.»

The majority of cosmeticians or clinicians we are, always focused problem solving mimicking at best the In Vivo test that we want unconditionally correlate and unfortunately forgot to look at the problem as physicists and thermodynamicists. At the very best they understood its importance but believe that a thorough study of the physico-chemical mechanisms involved is disproportionate for cosmetic application. By contrast, do not they prepare carefully their walls before painting their kitchen with a beautiful lacquer? It is only their kitchen but they do not hesitate in studying the best conditions to get good result (choice of solvent, choice of applicator, surface preparation, etc).

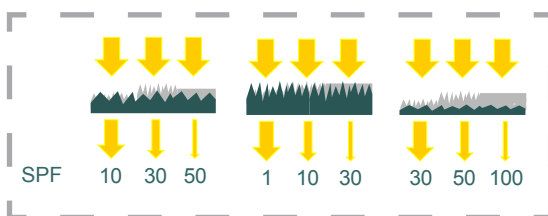


Figure 1. Representation of impact of the thickness distribution on the sun protection product.

All elements presented in this article are only a focus of the current knowledge, sometimes very recent. This is rarely followed but what is described is essential, inescapable to master reproducibility and so ensure a reliable method. This is not an opinion on the matter but only the reminder of physical and chemical laws that govern the properties of interface, no one can ignore. However, some will still say that their expertise and experience is enough. Moreover, they will justify by claiming that they did not check the validity of these essential rules. And this is true in their case for two reasons.

The first reason is that when tests are carried out «roughly» on an unsuitable substrate under basic conditions, the variability is such - when it can be checked - that the improvement produced by the control of a particular parameter is invisible. For example, the tire pressure of a car is an important performance factor. If you have an old 60s car, probably monitoring this parameter will not bring any significant improvement.

The second reason is that ALL parameters can be important... or not because the result is always product dependent. The physicist will immediately understand! But sometimes it will be sufficient to choose «bad» product(s) to demonstrate that a parameter is not important. For example, about 1 in 2 has a smartphone. If you have a panel of people 0-3 years old, no doubt that the number will be reduced to 0.

So, before understanding and master any phenomena, it is important to analyze all its aspects.

I.b. Parameters

Substrate

It is understandable that with a roughened substrate, the prerequisite is to ensure that the topography is always the same in order to have the same thickness of products anywhere. This has been demonstrated many times in many publications or choice in the most important international methods including ISO 24443:2012 concerning the determination of the UVA-PF In Vitro.

The substrate's injection (molded plate) allows a total reproducibility of all parameters incommensurate with sandblasting which is difficult to reproduce. Regarding sandblasting, the most common technique is to manually sandblasted a large plate and then cut it out. Of course, it is totally impossible to have a perfect reproducibility. However, a recent proposal (already presented in the previous HelioNews HN16) offers individually molded sandblasted plates by means of a mechanical system that ensures complete reproducibility (see Figure 2).

On the other hand, whatever the process, it is essential to have the guarantee of topography measurements. These parameters should not be restricted to an average roughness Ra or Sa. An average is only valid with the knowledge of the dispersion. An average plate 6 microns can vary between 5 and 7 or between 2 and 10 microns, the developed surface will not be the same at all. Of course, this distribution must be the same from one plate to another (see Figure 3).

Everyone knows that the results will greatly depend on the roughness which is generally accepted that 4-5 microns is an acceptable average (see Figure 4). This does not prevent some methods such as the FDA rules in 2011 to allow for sun protection evaluation on a so large range that the choice of the roughness depend the result (between 2-7 microns, ie the old sandblasted plates). It is a scientific nonsense as if a HPLC assay of an raw material would be possible in a method with all kind of solvent!

Obviously, to be sure to have reproducible plates, it must be able to control. Most of the plates on the market are not controlled

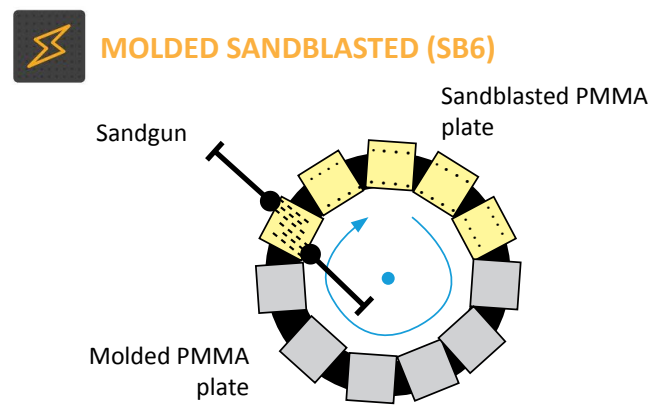


Figure 2. Innovative mechanical sandblasting process

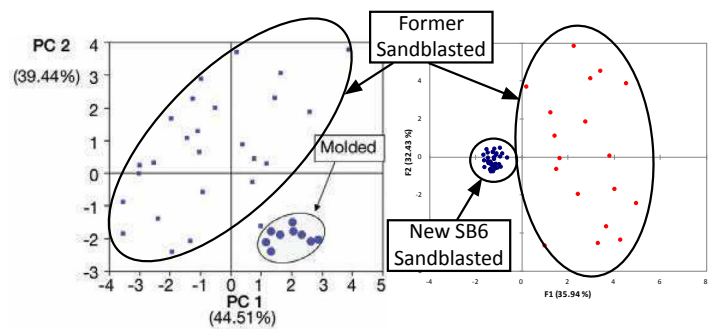


Figure 3. Topographic parameters reproducibility according to substrate

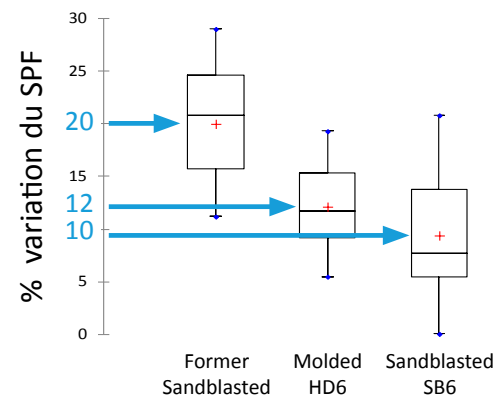


Figure 4. In Vitro SPF variation according to substrate

or sometimes assert that gives no warranty average roughness. Imagine measuring the size of a needle with a meter? However, devices have been described in ISO 24443:2012 as the profiler without contact optical measurement based on the principle of the aberration of monochromatic light.

Product and temperature control

We must be able to control all products and we know that the method is product dependent. However, for thermodynamic reasons, it is essential that the substrate and product are packaged at the same temperature at least 24 hours before test. A thermodynamicist will have quickly understood.

However, he shall also have understood as demonstrated by a recent article, it is essential to control and maintain the temperature at the interface during the application, the spreading, the drying steps of the product and even UV exposure. Furthermore, it has been demonstrated that the very precise control of room temperature is not sufficient and – on a large panel of products - more than half of the products are more or less sensitive with this never checked until now.

Product quantity

If historically it was spread a quantity of $2\text{mg}/\text{cm}^2$, again, this is due to the popular belief that In Vivo test should be mimicked. Fortunately, we realized pretty quickly that the test was in fact a test of physical measurement and the amount was intended to always measure the same film thickness. Furthermore, it is logical that this amount is related to the roughness of the plate. This also reiterates the importance of the topography of the plate and its control in order to have always the same thickness of product. It is a rule of common sense, but there are still UV analyzer manufacturers that do not give good advice or ignore it. The manufacturer of diesel vehicle will always recommend the right fuel.

UV exposure

UV irradiation conditions, is an essential parameter. It has been little taken into consideration at the beginning of sun protection assessment but were quickly governed to improve the reliability of tests. It is clear that over the goal of having an inter-laboratory result reproducible and therefore a very same thickness of spread product, this result must be also identical after irradiation step. However, many devices of UV exposure on the market have for example a cooling system based on air flow which can disrupt the distribution of the product to the substrate surface and thus ultimately lead to different results.

Spreading

Quality of the spreading is linked to the properties of the product itself but also to the thermodynamic conditions. Indeed, while spreading the product, it will be affected by the spreading stress and its conditions. Depending on these conditions, results in spreading can be very variable.

«A recent proposal demonstrated a robotic spreading which almost completely ensures reproducibility.»

To rough a substrate is not as some still believe to mimic the surface of the skin. But this is why it has long been spread $2\text{mg}/\text{cm}^2$ and that many laboratories worldwide continue to do that. Moreover, as the skin surface is irregular, it will create a lot of problems for zero profit. The roughness of the substrate is necessary for providing the necessary energy for spreading the product (solid or liquid to solid) as when you need to shake two immiscible liquid / liquid products. Its value is linked to the ad hoc thickness to perform the measurement ... if the product does not have too much

Partnership between HelioScreen and Shiseido

The Japanese cosmetic company SHISEIDO (Tokyo, Japan) and our laboratories HELIOSCREEN (Creil, France) have decided to start collaboration in the field of In Vitro sunscreen testing. The two companies involved in the international work on the reliability of these tests advocate for the possible expectation of an in vitro method for SPF determination in a very short delay. Nevertheless, current and recent conditions for performing such testing must be now strictly, known, accepted and followed by all laboratories worldwide.

New compulsory requirements such as control of the temperature and pressure can be insured by the means of appliances respectively developed by HelioScreen and Shiseido, the HD-THERMASTER and Finger Pressure Sensor (see Figure 5). Both appliances have been described and validated in scientific publications and international workshops recently. As a first decision for this agreement, these innovative appliances are now worldwide distributed by HelioScreen, HelioScreen Asia Co., Ltd. and their representatives on the 5 continents.



Figure 5. Finger Pressure Sensor

This just a starting point for a collaboration of the two companies which are also working on different subjects in order to be able to make very soon some concrete proposals. The beginning of an exciting collaboration for development of a reliable In Vitro sun protection assessment method for ethical, practical and economical reasons.

repulsion for the substrate.

On the other hand, the pressure must be checked for applying the product in order to improve reliability in the same manner we solve and control a mixing speed if you want to master the intimate mixture of a liquid product with another incompatible. This is a factor which is also known recently and has been the subject of recent publications but again our physicist will not be surprised.

Recent proposals of appliances allow mastering these parameters but some people still believe be able to control pressure by means of their own expertise. It is important to remember that the obvious goal is to have the same result in all laboratories so it will be necessary to accept an universal application pressure or spreading method.

Strict control of these parameters will be sufficient to ensure the result?

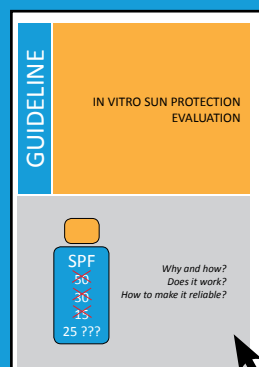
If all laboratories would already applied rigorously the rules above, we could reach a certain reproducibility even if some products would still have variability but probably much better than In Vivo tests. It is also surprising to have few data on the reproducibility of In Vivo tests and when they exist, they involve few laboratories and few products. Interesting no?

Unfortunately, even being very well trained, we still have major impossibility for humans to always reproduce the same spreading and / or the same pressure. A recent proposal demonstrated a robotic spreading which almost completely ensures reproducibility. This has just been published and again demonstrated in the context of an international Workshop performed in our laboratory on different machines versus different operators.

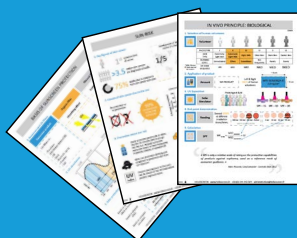
The laboratories or institutes which offer tests to customers cannot ignore it, as it has been the case for laboratory equipment and control for In Vivo tests few years ago.

Guideline: Issues and methods of Sun Protection Evaluation

Discover the fundamentals of sun protection and key parameters of In Vitro testing.



- Sun risk
- Basis of sun protection
- In Vivo / In Vitro principle
- Key parameters of In Vitro method



II. Solar protection in Europe vs. USA ?

II.a. Introduction

Nowadays, it is well known that beyond UVB protection, the UVA protection is also important and sunscreens must provide a total protection. The regulation in USA about sunscreen market requires the SPF (Sun Protection Factor) assessment by In Vivo method and the CW (Critical Wavelength) by means of In Vitro method ≥ 370 nm. Both values act as an indication about UVB and UVA protection.

In Europe, the regulation requires besides two values previously mentioned to determine the UVA-PF by In Vitro or In Vivo method and at least equal to $1/3$ of the SPF.

Thus, the aim of this study is to determine, through a lot of products used in the USA and Europe, if a regulation ensures to consumers better protection against UVA.

II.b. Materials & Methods

In Vitro methods:

- FDA rules 2011
- ISO 24443:2012

Equipments:

- Product application: Automatic serynge
- Substrate: Molded PMMA HD6
- Robot spreading: HD-SPREADMASTER
- Temperature control: HD-THERMASTER
- UV exposure: Suntest CPS+ and Solar Light 16S
- UV analyzer : Labsphere UV-2000S

II.c. Results

The results of the percentage of products which have a CW ≥ 370 nm **AND** a UVA PF $\geq 1/3$ SPF are available in **Figure 6**. Thus, only about 20% of products sold in the U.S. are in compliance with European regulations (against 94% of products sold in Europe). In other words, products marketed in the USA offer lower protection against UVA.

On the other hand, the graph shown in **Figure 7** allows visualization of the ratio UVA-PF/SPF assessment (mean and standard deviation) based on the CW for all products. So it seems that the majority of products which have a CW ≥ 375 nm also have a UVA-PF $\geq 1/3$ SPF ratio.

II.d. Conclusion

The majority of sunscreens sold in the U.S. have relatively low effective protection against UVA even if the CW (broad spectrum) is ≥ 370 nm. Indeed, compared to marketed products in Europe which have a UVA-PF $\geq 1/3$ of the SPF (and obviously a CW ≥ 370 nm), few products in USA reach such level of UVA protection.

To provide more effective protection for consumers, it is recommended that the USA regulatory evolves according to the European guidelines for the determination of the UVA-PF $\geq 1/3$ SPF or at least have a CW ≥ 375 nm.

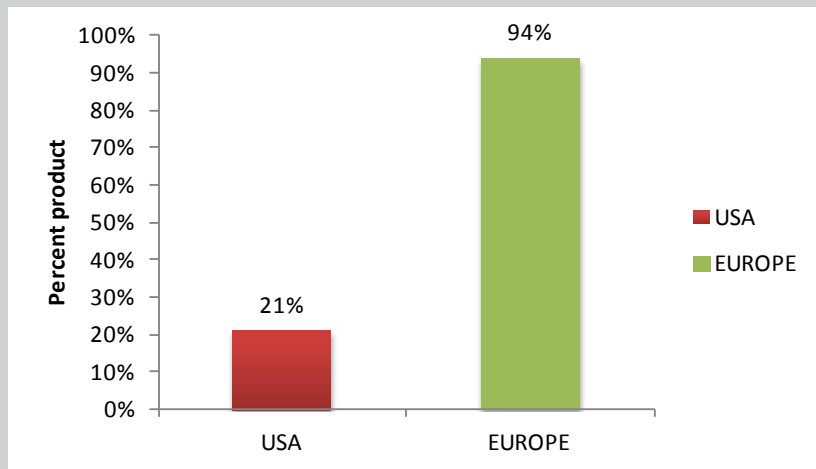


Figure 6. Percentage of commercial product in Europe or USA with a CW ≥ 370 nm **AND** a UVA-PF $\geq 1/3$ SPF

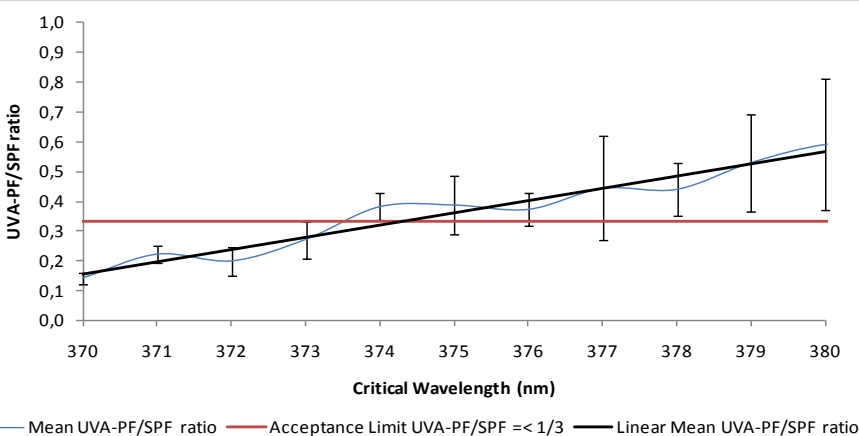


Figure 7. Evolution of UVA-PF/SPF ratio according to CW

Scientifics articles

Cosmetics & Toiletries, Avril 2014:

- S. Miksa, D. Lutz et C. Guy. *Sand Blasting to Improve Reproducibility of In vitro Sunscreen Evaluation.*

PHOTODERMATOLOGY, PHOTOIMMUNOLOGY & PHOTOMEDICINE, April-June 2014:

- U. Osterwalder, M. Sohn and B. Herzog. *Global state of sunscreens.*

- B. Petersen and H. Christian Wulf. *Application of sunscreen – theory and reality.*

- J. Krutmann et al. *Towards standardization of UV eye protection: what can be learned from photodermatology?*

- S. Grether-Beck, A. Marini, T. Jaenicke and J. Krutmann. *Photoprotection of human skin beyond ultraviolet radiation.*

H&PC Today Avril 2014:

- S. Miksa, D. Lutz et C. Guy. *Improvement of In Vitro sunscreen testing inter-laboratories reproducibility by means of reference absorbance curve.*

«375 CW» label

HelioScreen, pioneer in In vitro sunscreen testing proposes you the label «375 CW» for greater effective UVA protection. This label guarantees a critical wavelength ≥ 375 nm in total compliance with FDA rules 2011 or ISO24443:2012 regulations.

