HelioNews 2011 N° 12

www.HelioScreen.fr

HelioNews

The current events of the sun protection proposed

CELEBRATING 15 years 1999-2014 SPECIAL REPUBLICATION

HelioScreen

We will be at Sunscreen symposium Florida Sept 2011.

SOCIETY OF COSMETIC CHEMISTS SCC Dedicated to the Advancement of Cosmetic Science

Summary

● ● ● ●

HelioScreen Labs
44, rue Léon Blum
60100 Creil
Phone: +33 3 44 24 33 29
administration@helioscreen.fr

Updated version

Few modifications have been performed (highlighted with *)in order to avoid misleading in comparison with original version in French.

Editorial …

At last, the FDA has proposed in the June 2011, its final rule for labelling of sunscreen products. Let's remind the 2007 proposals calling for comments had been supposed to be more or less in line with the global final requirements especially for UVA claiming .

It was really a surprise than the requirement for UVA claim has been revised to keep with conditions closer to what is proposed in EU: the critical wavelength . But, forgotten the requirement of the ppd UVApf either *In Vivo* or *In Vitro*! Another "good surprise" which became unexpected in the last month before publication, was that the conditions of testing were proposed so as to feet as much as possible with existing or expecting ISO rule (*In Vitro* or *In Vivo*).

We have been quite happy to learn at least FDA adopt the PMMA substrate instead of "undeterminable" roughened Quartz and spread quantity of 2mg/cm². However quite a surprise to learn the roughness can be choose in the huge range (2 to 7 microns) with a quantity of 0.75mg/cm²!

We have been always advocating for 6 microns roughness instead of 2, and it was not the better surprise of the spring! Additionally, the required quantity has been fixed to 0.75mg/cm². It is in fact the one of the …'former" version of the Colipa recommendation. Clearly we could have expected for a quantity of 1.3 mg/cm², if the FDA, had referred to the "up date" revision of this method. But we can suppose it was a question of calendar.

So we decided to start a huge work to check these conditions (6 microns and 0.75mg/cm²) in the lab. So many products have been controlled… But now we are sure! Better to use the HD6 than HD2 ! A paper, to be published, has been prepared, rewieved and scheduled so quickly that it will be published in cosmetic and toiletries in October. I would like to thanks all people from my lab, the publisher and specially people who help me with so good advise to finalize. I do hope the final advice to use 6 instead of 2 will help some products in "border line" to be accepted with this new rule. D.L

In Vitro & In Vivo compliance packs offered for FDA

2006: COLIPA announced an in vitro method for measuring the UVA effectiveness of sunscreens which could supplement the reference in vivo method of persistent pigmentation A recommendation from EU commission recommended to go towards in vitro test when validated. It was based on the fact that the combination of in vitro evaluation together with in vivo experimentation would allow volunteer safety to be increased and thus the practical conditions of in vivo testing to be reduced. In Oct 2007,. Laboratoire Dermscan and HelioScreen Labs were combining their skills to offer a complete new in vitro / in vivo service in the field of sunscreen testing. With a **compliant SPF/ UVA Colipa compliant pack meeting all EU requirements**.

2011: The new FDA standard already modifies the way in vivo tests (SPF) as well as in vitro "Broad spectrum" enabling UVA protection factor evaluation to be performed. The two companies, experts in Solar products evaluation, decide to reinforce their partnership putting on top a **new FDA compliant pack meeting all the US market requirements**.Not only this partnership brings the most relevant technical answer to your need, but also allows the customers to have only one interlocutor for two expertises. (Sales: Emmanuelle PRUNEAU +33 (0)472 823 650)

FDA New Rule: Border line products in broadspectrum claiming: How to improve ??

See next pages...

A new representation of HelioScreen in Australia and New Zealand

After the last representations in USA (2009) and Japan (2010) China and Taiwan (2011), Helioplates are now directly available in Australia and New Zealand since last month.Customers from this part of the world can contact directly our representative Warsash Scientific Pty Ltd
Unit 7, 1 Marian Street Redfern NSW 2016
PO Box 1685 Strawberry Hills NSW 2012 AUSTRALIA Tel: +61 (0)2 9319 0122

«HelioNews» published by HelioScreen Labs
Updated version : S Miksa smiksa@helioscreen.fr
Editor : G holtzinger Design : H Pierrefiche
Publisher : D Lutz dlutz@helioscreen.fr

Patents ...

- **Patent** WO/2011/063329 Publication Date: May 26, 2011 Assignee: GL Grune ***Broad-spectrum uva stable, non-toxic, zinc oxide based complex :*** Revealed in this patent is a new compound that also is presented as a dispersion with broad UVA and UVB spectral absorption stability, heat absorption, and insulation properties, as well as antimicrobial water resistance and high SPF properties. The zinc oxide complex described comprises distilled water buffered to a pH of > 10 and zinc oxide particles. According to the invention, the complex is made from natural-based, earth-derived, Ecocert-certified ingredients, is nontoxic and may be used or combined with any compound wherein UVA/UVB stability is desired. The applications for the complex are numerous including sunscreens and cosmetics.

- **Patent** WO/2011/061133 Publication date: May 26, 2011 Assignee: DSM IP Assets B.V., T Satzinger and H Westenfelder ***:Topical compositions having TiO_2 particles with improved water resistance.*** The present invention relates to a method of improving the water resistance of micronized double coated titanium dioxide (TiO_2) particles having an inner inorganic silica coating and an outer silicone coating. Said particles are incorporated into a topical composition in the form of a dispersion in C_{12-15} alkyl benzoate and polyglyceryl-2 dipolyhydroxystearate. Furthermore, the invention relates to the topical compositions comprising the dispersions, including sunscreens.

- **Patent** US 20110052516 Publication date: March 3, 2011 Inventor: Christine Mendrok-Edinger : ***Phosphate ester surfactants to increase SPF.*** This technology relates to the use of phosphate ester surfactants for increasing the sun protection factors of topical compositions that comprise at least on UV filter. Furthermore, the invention relates to topical compositions comprising a phosphate ester surfactant and at least 4.0% w/w of butyl methoxydibenzoylmethane in combination with additional UV filter substances.

- **Patent** US 7906108 Publication date: March 15, 2011 :***Polysiloxane sunscreens.*** As The present invention relates to novel sunscreens based on polysiloxanes and to their preparation and use, especially in formulations for protection against harmful effects of sunlight.

- **Patent** US 7998509 Publication Date: Aug. 16, 2011 ***Use of stinging cells/capsules for the delivery of active agents to keratinous substances.*** According to one aspect of the present invention there is provided a method of delivering an active agent into a keratinous substance, the method comprising applying a composition comprising the active agent disposed in or around at least one stinging capsule to an outer surface of a non-skin keratinous substance and triggering a discharge of the at least one stinging capsule to thereby deliver the active agent into the non-skin keratinous substance.

.. It happened under the sun

- **Comparatively Speaking: SPF and Calculated SPF**

By: Anthony J. O'Lenick Jr., Siltech LLC; and Dennis Lott, Tanning Research Laboratories Inc. August 16, 2011*

The principle to determine In Silico SPF from the knowledge of ingredients and formulas has already be proposed by BASF and Coty On the other hand testing requires some specifics rules which could affect the SPF. This publication point out the difficulty to adequately calculate SPF from a combination of sunscreen actives and Additional factors included: ingredients in the formulation such as solvents, film formers, emulsifiers and water resistant agents; interactions between ingredients, ability of the formulation to be uniformly spread on the skin; the photostability of the formulation, and product migration.

The very important conclusion is that:

The formulation of effective sun protection products requires cooperation between the formulator, the raw material supplier and the product testing laboratory to assure that the product that is effective at using the least amount of sunscreen agent for the highest SPF.

- Parabens: there is still a debate in France

In France, There is always a great concerned about the possible consequences resulting from the proposed bill aiming to ban some families of substances classified as endocrine disruptors in consumer products. While the controversy is now extending to medicines, the cosmetics industry, directly concerned by the ban on parabens, appears to be torn between scientific reality and the need to meet consumer demand. While it is still not included in the agenda of the Senate, the bill proposed by Yvan Lachaud (an MP from the 'Nouveau Centre' party, centre right), adopted on first reading by the National Assembly with the aim to ban the use of phthalates, parabens and alkylphenols in consumer goods, continues to cause some turmoil in France and Europe.

FDA new rule : Border-

preamble

This article is partly extracted from presentation [1] which will be presented during Sunscreen symposium in Florida (Sept 11) by D Lutz (HelioScreen labs).

Introduction

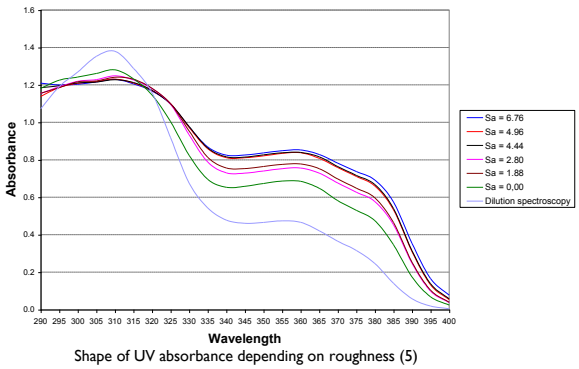
Considering the work done about normalization at a world while level within the ISO TC217 group for *In Vivo* and *In Vitro* SPF and UVA protection, it appears there is a global consensus emerging about some methods, unless for *In Vitro* SPF which is still is discussion. We could expect an harmonization of the "local" regulations and rules.

EU had the advantage to have already precise in 2006 the requirement for global testing procedure (SPF+UVA). The fact is the methods proposed by ISO are based on what is already more or less recommended in EU. Australia is ready to change the AS/NZS 2604 norm before the end of the year with an expected and announced harmonization in coherence with ISO process. FDA seemed to go towards some thing different with a quite 4 years old proposed rule but has at last publish in June the final rule regarding labelling for OTC sunscreen products [2] including the testing procedures- for the SPF, water resistance and broad-spectrum tests. The compliance date for application is June18.2012.

Concerning UVA, the rule implements a pass/fail test based on the *In Vitro* critical wavelength method (CW). We already knew this method as it is part of the EU recommendations [3] with condition for passing the test to claim a CW not below 370 nm. As a matter of fact FDA also took into account the recent developments in the rest of the word and specifically EU and ISO. This is why they also finally recommend PMMA as substrate.

FDA refers to Colipa UVA recommendations [4] to specify the degree of roughness. This has been done before the recent modifications of COLIPA (2011) which is now in agreement with ISO 24443 to recommend the 6µ roughness with a quantity of 1.3mg/cm².So with FDA rule, the "Sa" values have been proposed in the range of 2 to 7 nm for a quantity of 0.75mg/cm² to "ensure UV radiation transmitted through sunscreen is within the dynamic range of UV detectors".

Unless the same endpoint of the CW tests, we will have to provide two determinations in case a product is marketed in EU and US.



Shape of UV absorbance depending on roughness (5)

In our laboratory, before the up dating of the method, we had been already using the HD6 for COLIPA UVA pf determination It was not out of the

Line Products in Broadspectrum claiming ! How to manage ??

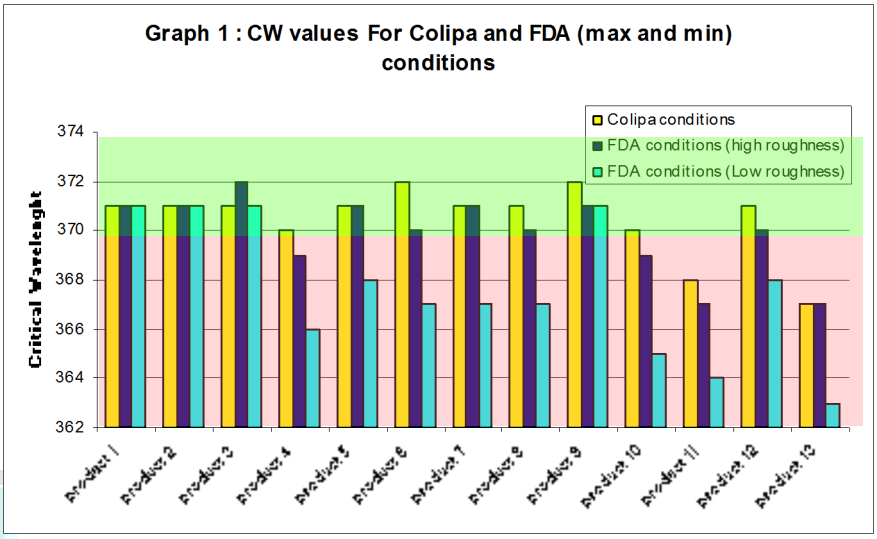
recommendation and as we trusted this modification for the reliability of correlation. Slight higher results could be obtained with these plates for certain products.

It has been demonstrated that it is the same consequences for the CW values, and it was also our experience in our institute.

Unless we propose both HD2 and HD6 plates we have been always advocating for the 6 µ roughness. When FDA proposed this range of roughness, it seemed to us logical to check influence of roughness and reduced quantity for 6µ plates. We checked it on a great number of products as we did for the Colipa UVA method in 2007. We ran this study in the last weeks and results will be published very soon in the October issue of cosmetic and toiletries under the title ***"New FDA Rule For Broadspectrum Labelling. Some Keys About Substrate to Ensure Reliablity". By D Lutz and al.***

Additionally, and later on this study, it appeared also important to compare results following COLIPA and FDA guide lines for the same products. We decided to investigate in the case of 13 specifics border line", failed or limit-accepted products for the CW test on "Colipa conditions" (HD6 1.3mg/cm²). How about their CW in case, as for COLIPA UVA, we would have applied on the lower allowed level of roughness?

This is the goal of this study based on marketed products which have been sorted from our customer data base (products tested in the past for determination of UVA pf Colipa and CW in the former version but realized on 6µ plates). We also decided to test again the Colipa UVApf , this time on the HD2 plates. We already know about the behaviour as long it has been demonstrate by Colipa and in home checking. We limited this duplication at 4 products within the 13 along we did not know the *UVA pf in Vivo* value for the other products.



Materials /

FDA and COLIPA conditions have been followed:

Appliance: We used a Labsphere UV 2000 which is relevant either with the COLIPA and FDA rule

Irradiation: We used a calibrated SUNTEST CPS + delivering 71.3 W/m² in UVA for a total irradiance of 500W:m². This appliance is equipped and disposed in a specific "conditioned room" in our lab to ensure the control of temperature under 40°c.

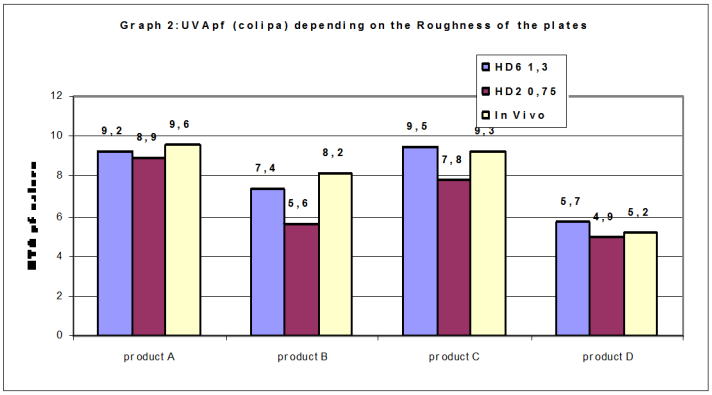
Substrate: We used HD2 and HD6 plates from our companies. These plates are controlled and guaranteed for HD6 within the Colipa requirements.

Pre conditioning of Plates and Products: Following our internal procedure, both plates and product have been conditioned in an oven at a controlled temperature between 27 and 29°c for 24H before testing.

Blank and Controls: to control the appliance Colipa procedure have been followed with a Holmium sampler and HD0 plates for linearity control.

Procedure: We spread the required quantity on the plate and spread out. 9 points were measured on each plate and 3 plates per products. All the measurements have been duplicated with 2 operators.

Irradiation/



For complying with FDA, we applied a pre irradiation dose of 4 times 200J/m²-eff, which represents 8 j/cm² in UVA-UVB . With our conditions (7.62mw/cm² in total flux) the exposure time was 17.65 mn.

For complying with COLIPA, we applied the dose of 1.2x c "C" adjusted UVA pf value.

Results

Complete results on the COLIPA conditions and FDA limits conditions (2 and 6 µ :0.75mg/cm²), has been fully reported in graph1 and table 1. Results obtained previously while testing for COLIPA UVA are in column "Colipa conditions". The two columns for FDA conditions have been reported beside. We calculated each time the difference for the minimal and maximum proposed roughness by FDA. and in both cases, the difference between FDA and COLIPA conditions. The CW values are expressed at the mean value obtained by the two operator, then rounded with the higher value.

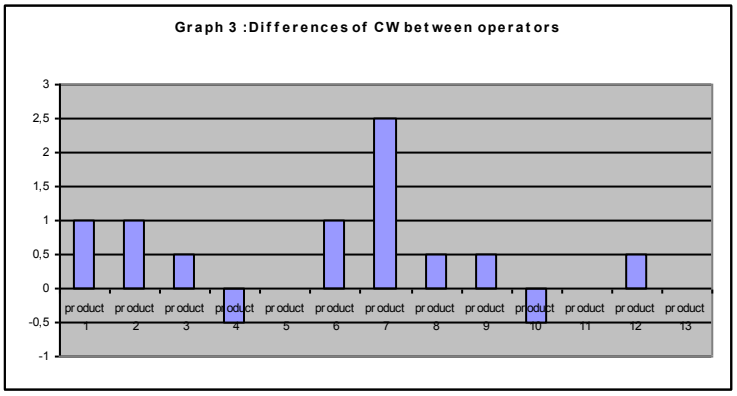
We also reported graph 3 the differences between our two operators.

All products have been previously measured with the conditions of COL-

Products		Colipa conditions	FDA conditions (high roughness)	FDA conditions (Low roughness)	Difference within FDA Conditions	Absolute Difference FDA (max)/ Colipa	Absolute Difference FDA (min)/ Colipa
Stick	product 1	371	371	371	0	0	0
Stick	product 2	371	371	371	0	0	0
Cream	product 3	371	372	371	1	1	0
Cream	product 4	370	369	366	3	1	4
Oil	product 5	371	371	368	3	0	3
Cream	product 6	372	370	367	3	2	5
Tinted cream	product 7	371	371	367	4	0	4
Milk	product 8	371	370	367	3	1	4
stick	product 9	372	371	371	0	1	1
Cream	product 10	370	369	365	4	1	5
Cream	product 11	368	367	364	3	1	4
stick	product 12	371	370	368	2	1	3
Milk	product 13	367	367	363	4	0	4
				Means	*2,31*	*0,69*	*2,85*

Table 1 CW values for Colipa and FDA* conditions (* at low and high level for roughness interval)

IPA (6µ /1.3mg/cm²) and we re evaluate 4 products within the 13 in the former conditions (2µ/0.75mg/cm²) in graph 2 reporting the values of the *In Vivo*. We did not relate the products as long they are products from customers.



Discussion

The 13 products had been previously evaluated either limit (11 products) or not complying (2 products) with the CW requirement (graph 1) in the conditions we used for the COLIPA UVA determination (HD6 plates 1.3mg/cm²).

It is clear results of CW on FDA conditions depend on the roughness between the two terminals of the intervals. For the low roughness most of the products (9) fail the test unless for the higher roughness, most of the product (9) passes the test. The difference is significant with an average close to 3 nm. That means within FDA conditions a product may pass or fail for the same test.

The proposal of such a wide range of roughness may appear astounding if we consider several papers from different authors [5] [6], who had demonstrated there is a great influence of the roughness on the shape of the curve.. Curiously, if FDA comments these choose to keep within COLIPA rules (now obsolete) they also mentioned the reference [5] (70 for FDA) and refers to the pressure of application instead of roughness!! Is there a misunderstanding?? Meantime, Colipa already up-dated its recommendation for *In Vitro* UVA ppd in 2011 by proposing the switch from 2µ (sand blasted) to6µ (better moulded or at least compliant with control card). It has been demonstrated to provide higher results unless both plates allow correlation. In other term the accuracy with *In Vivo* values is significantly better with 6µ plates.

Two products (4/10) passed with COLIPA condition but failed with FDA conditions (in the high value of roughness interval). But the difference is only 1 nanometer. Clearly it is sufficient the have the test failed. But it may be the variability of the method and it is advisable to confirm when there is such a result!

If we compare the FDA CW conditions to the COLIPA CW conditions, we note all products which pass the test for COLIPA also pass for FDA as long the higher roughness is chosen. The average difference between the two conditions is not significant as long the average is under 1 nm. This is not at all the case if the FDA CW test is done with the lower roughness for the test.

As there was a slight dispersion in the results between the two operators, we reported the value most of the time around or less 1nm with a maximum of 2.5 nm.(Graph 3) .The out lined product (7), a tinted cream, presented some difficulties to be spread. That demonstrate the difference for products 4 and 10 is not significant and all the products selected tested in Colipa conditions and FDA (higher value of the roughness scale) are comparable unless some difference in quantity apply and time of irradiation.

4 products have been re tested for UVA Colipa, on 2µ plates to compare the behavior with the former results. We can check results are in line with the conclusion of the COLIPA for product A and D. C is quite close but product B is underestimated both for 6 and 2µ plates.

FDA rule for CW determination allow some discrepancies as long the scale for roughness is quite wide. In case of border lined products, it may lead to reject product which would have been accepted in other conditions within the rule. The use of the high roughness for the plate allows getting very close result for FDA and COLIPA methods.

References:

(1) "Substrate: A key parameter for In Vitro determination". D Lutz Sunscreen symposium (Florida Sept 2011)

(2) Department of health and human services Foods and drug Administration 21 CFR Parts 201 and 310 (Dockets N°FDA-1978-N-0018) (formerly docket N°1978N-0038) RIN 0910-AF4

(3) European Commission Recommendation of the efficacy of sunscreen products and the claims made relating thereto. Official Journal of the EU L.265/39 2006/764/EC 39-43 Brussel 2006

(4) Colipa : method for the in-vitro determination of UVA protection provided by sunscreen products – guideline 2011

(5) Ferrero et al "Importance of Substrate Roughness for In Vitro Sun Protection Assessment". [IFSCC, Volume 9, No. 2 (2006)]

(6) D Moyal "UVA protection labelling and In Vitro testing methods Photochemical and Photobiological sciences 2010 .9. 516-523

Scientific articles

- Interpretation of SPF In vivo Results: Analysis and Statistical explanation By: Marc Pissavini and Olivier Doucet, Coty-Lancaster; and Olivier Brack, Statistique Industrielle KHI2 Consulting (KSIC) Posted: March 2011 C & T.

Pissavini and al explain how to interpret and use the In Vivo SPF results.

- Adapting SPF Testing Methods for Mineral Sunscreen Density

By: Paul G. McCormick, University of Western Australia

Posted: March 2, 2011, from the March 2011 issue of Cosmetics & Toiletries.

This article propose an adaptation of the applied quantity for measurement of SPF with such products.

Au Naturale Nanoparticle Sun Protection September 2010 Cosmetics & Toiletries. By Zhang and al

Zhang and al compared the optical extinction spectra of English ivy nanoparticles against that of TiO2. English ivy nanoparticles should have high transmittance in the visible UV region, which makes them "invisible,"

- "In Vitro and In Vivo new package Offered for Sunscreen Testing"

Laboratoire Dermscan and HelioScreen Labs were already combining their skills to offer a complete new in vitro / in vivo service in the field of sunscreen testing. The combination of these two areas of expertise specifically dedicated to sunscreen testing, is in line with the new recommendations of the European Commission and COLIPA , which favors in vitro test methods for the UVA evaluation of sunscreen products. As the new FDA rule propose to combine either In Vivo SPF/ Water resistance and In Vitro CW for broadspecrum claim, they have announced they decided to reinforce their collaboration with a new FDA pack including all required tests.

Companies information

- Intertek wants to partner with brands for quality and safety

the specialist in quality control and product safety, has been formally entrusted along with SGS, with the control for compliance of perfumes and cosmetic products exported to the Kingdom of Saudi Arabia,

Réglementation

- FDA Releases a Final Sunscreen Rule

After nearly a four-year wait for an action on sunscreens, the US Food and Drug Administration (FDA) has issued a Final Sunscreen Rule, which addresses the testing and labelling of OTC sunscreen products.

- FDA Releases Draft Guidance Toward Nanotech Regulation

According to the agency, nanotechnology is an emerging technology with a broad range of potential applications, applied in cosmetics, most notably in sun care. FDA has released a draft guidance to provide regulated industries with greater certainty over the use of nanotechnology.