

# Testing of Sunscreen Products

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**HBA 2010 Session on September 29<sup>th</sup>, 9.00 -10.30 AM: Sunscreens - A Mini-Symposium**

**Moderator: Dr. Nadim Shaath**

# Abstract

Three years ago the FDA published proposed rules on UVA protection, which include an evaluation of sunscreen efficacy *in vivo* (UVA-PF) and *in vitro* (UVAI/UV ratio after pre-irradiation), and require using both methods to determine the magnitude and breadth of UVA protection. The FDA has expressed its vision that balanced and photostable UVA/UVB protection provided by sunscreen products needs to be ensured. New regulations have not been finalized yet; however, they have already triggered certain sunscreen trends in the US, which became most apparent in 2009-2010 due to the duration of a new product development cycle in the US sunscreen industry.

Numerous sunscreen products were tested to determine their UVAI/UV ratios, UVA and UVB photostability. Methodological, technical and instrumental aspects of the testing methodologies utilized in this study will be presented. It was found that sunscreen products launched in the US in 2009 and especially in 2010 have sufficient photostability and progressively higher SPF values (50, 55, 70+, 85, 90+, 100+). In addition, leading manufacturers more frequently claim various skin benefits, for example product's ability to counter the skin damage on cellular level, etc.

Sunscreen trends that emerged in the US in 2009-2010 will be discussed in detail. In general, some of these trends can be considered beneficial to the consumer. At the same time, majority of sunscreen products tested belonged to the "High UVA Protection" category and failed to fulfill the "Highest UVA Protection" criterion. Reaching the highest UVAI/UV ratio, especially for sunscreens with SPF 30+ is a technical challenge that cannot be effectively addressed with the use of sunscreen actives currently approved in the US.

# Introduction

- **More than three years** ago the FDA introduced new proposed rule on UVA protection [1] that includes testing of sunscreen efficacy *in vivo* (UVA-PF) and *in vitro* (UVAI/UV ratio after pre-irradiation) and requires using both methods to determine the magnitude and breadth of UVA protection

- The FDA's vision is to ensure balanced and photostable UVA/UVB protection; agency believes that both UVB and UVA radiation protection are equally important and more protection against UVA radiation damage is better for consumers' health

**However, as of today the FDA is still finalizing new regulations...**



# Introduction - Continued

## In the meantime:

- The delay in the final ruling by the FDA has led to the regulation "vacuum" in the US sunscreen industry
- US consumers became concerned, petitions are being filed...
- New sunscreen trends in the US have emerged
- SPF "race" is on-going ...
- US sunscreen manufacturers do not have a unified position regarding the FDA proposed ruling
- EWG has created the 2010 list of top-rated sunscreen finished products available in the US - based on the simulated calculations and literature references - without conducting actual *in vitro* testing of these products

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"Well, don't just stand there looking precancerous."

# Comparison of the *in vitro* test methods used for sunscreens

<i>In Vitro</i> Method Parameters	FDA Proposed Rule (August 27, 2007)	COLIPA (2009)	Boots star rating system (2008 Revision)
Pre-Irradiation Dose	Equal to SPF of sunscreen product multiplied by 200 J/m <sup>2</sup> -eff multiplied by 2/3. UV radiation dose of 200 J/m <sup>2</sup> -eff is equivalent to one minimal erythema dose (MED).	D (dose) = UVAPF0 × D0 J/cm <sup>2</sup> ; D0 is unit UVA dose per unit UVAPF0, to be applied with the UV source spectrum, experimentally determined to achieve a fair correlation between <i>in vitro</i> UVAPF and <i>in vivo</i> PPD values. D0 value is fixed at 1.2 J/cm <sup>2</sup> UVA.	Fixed pre-irradiation dose: total UVA dose delivered during the product exposure to UV-radiation should be 17.5 J/cm <sup>2</sup> (representing 60 minutes of 'standard' sun equivalent UVA). The time taken to deliver this dose will vary according to the chosen flux
Irradiation Source	Not Specified. FDA proposed to utilize pre-irradiation dose in terms of "erythema effective dose" in order to allow various solar simulators to be used.	The spectral irradiance at the exposure plane of the artificial UV source should be as similar as possible to the irradiance at ground level under a standard zenith sun as defined by COLIPA (1994) or in DIN 67501 (1999).	The exposure source should be as similar as possible to COLIPA (1994) reference spectrum
Irradiation Flux	Not Specified	The UV irradiance must be within the following acceptance limits (measured at sample distance): Total UV irradiance (290 to 400 nm) 50 – 140 W/m <sup>2</sup> ; Irradiance ratio of UVA (320 to 400 nm) to UVB (290 to 320 nm) 8 – 22.	Total UV irradiance (290nm to 400nm) not less than 45W/m <sup>2</sup> and should not exceed 75W/m <sup>2</sup> . The UVA (320nm to 400nm) irradiance no less than 90% of total UV irradiance and no more than 97% of total UV irradiance.
Application Dose	2 mg/sq.cm	0.75 mg/sq.cm	1 mg/sq.cm
Efficacy parameter	UVA/UV Ratio	(a) Ratio of UVA PF to SPF (b) Critical Wavelength	UVA/UVB Ratio
Criteria	>0.2 to >0.95	(a) at least 1/3; (b) >370 nm	0.61 to >0.91
Label claim	1 to 4 stars	UVA logo	3 to 5 stars
Substrate	Quartz (The FDA requested comment regarding suitability of other possible substrates)	PMMA	Quartz or PMMA. Alternatives may be used as substrate if their suitability was demonstrated.
Substrate temp.	Not specified	Less than 40 deg. C	From 20 deg. C to 40 deg. C

# Determination of UVAI/UV Ratio and UVA Category

The FDA proposed rule contains the following steps:

- (1) Pre-irradiation of the substrate\* with applied sunscreen (2 mg/sq.cm)

The FDA specified the pre-irradiation dose (PID) in terms of “erythema effective dose “calculated by weighting the output spectrum of the solar simulator with the reference action spectrum for erythema as defined by CIE:

$$\text{PID (J/m}^2\text{-eff)} = \text{SPF} * 1 \text{ MED} * 2/3 \text{ where } 1 \text{ MED} = 200 \text{ J/m}^2\text{-eff}$$

- (2) Calculating the value of mean transmittance, its standard deviation, the coefficient of variation which gives an indication of the uniformity of the sunscreen layer and must be within the recommended range (< 10%)

- (3) The determination of the index of UVA protection (or UVAI/UV Ratio) by using the absorbance average values for each wavelength converted from Transmittance values:

$$A(\lambda) = -\log T(\lambda) \text{ between } 290 \text{ to } 400 \text{ nm at } 5 \text{ nm intervals for each of required } 12 \text{ locations}$$

\*The FDA proposed roughened quartz plates as substrate in the *in vitro* portion of its UVA test method **while requesting comments regarding the suitability and availability of quartz and other possible substrates**. The suitability of Vitro Skin® N-19 [2] as alternative substrate was demonstrated by O. Dueva-Koganov *et. al.* [3]

## Determination of UVAI/UV Ratio and UVA Category - Continued

The index of UVA protection is calculated as the area per unit wavelength under UVAI portion of a plot of Absorbance,  $A(\lambda)$  versus wavelength, divided by the area per unit wavelength under the entire UV portion of the curve.

At least five repetitions are required, and the mean is used to determine the rating for the sunscreen:

$$\frac{UVAI}{UV} = \frac{AreaUVAI / \lambda}{AreaUV / \lambda}$$

*The integrals in this formula are calculated using Simpson's rule for irregular areas, and action spectrum factor is equal to 1 for all wavelengths*

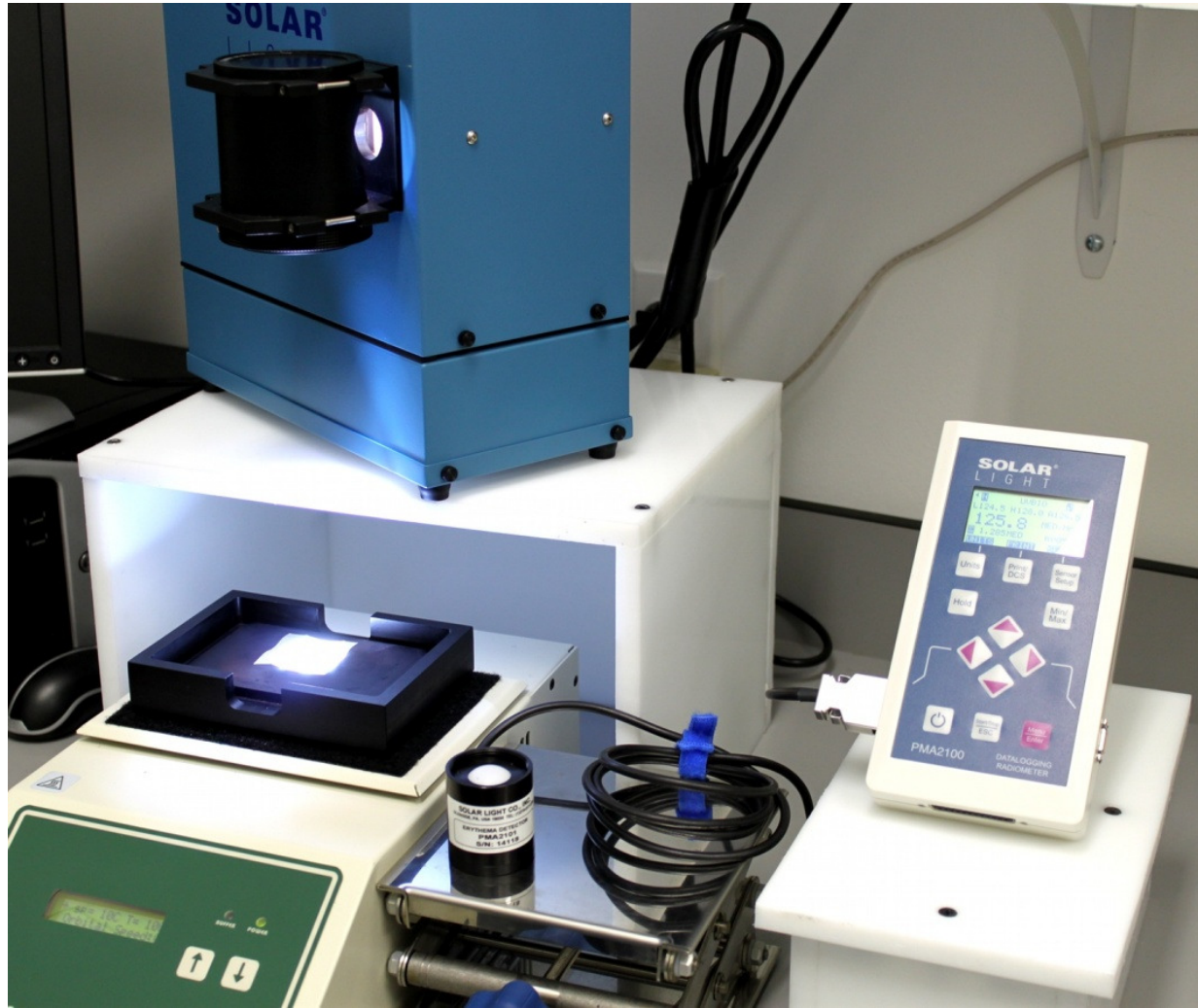
**The UVA ratings are categorized as:**



<i>In Vitro</i>	0.2-0.39	0.40-0.69	0.70-0.95	> 0.95
<i>In Vivo</i>	2 to under 4	4 to under 8	8 to under 12	12 or more

*Note. The FDA requires using both in vivo and in vitro UVA radiation testing methods to ensure that the magnitude and breadth of UVA protection is determined. The FDA asks US sunscreen manufacturers to declare, in addition to the SPF, the level of UVA protection, expressed in five categories: "No UVA protection," "Low," "Medium," "High," and "Highest."*

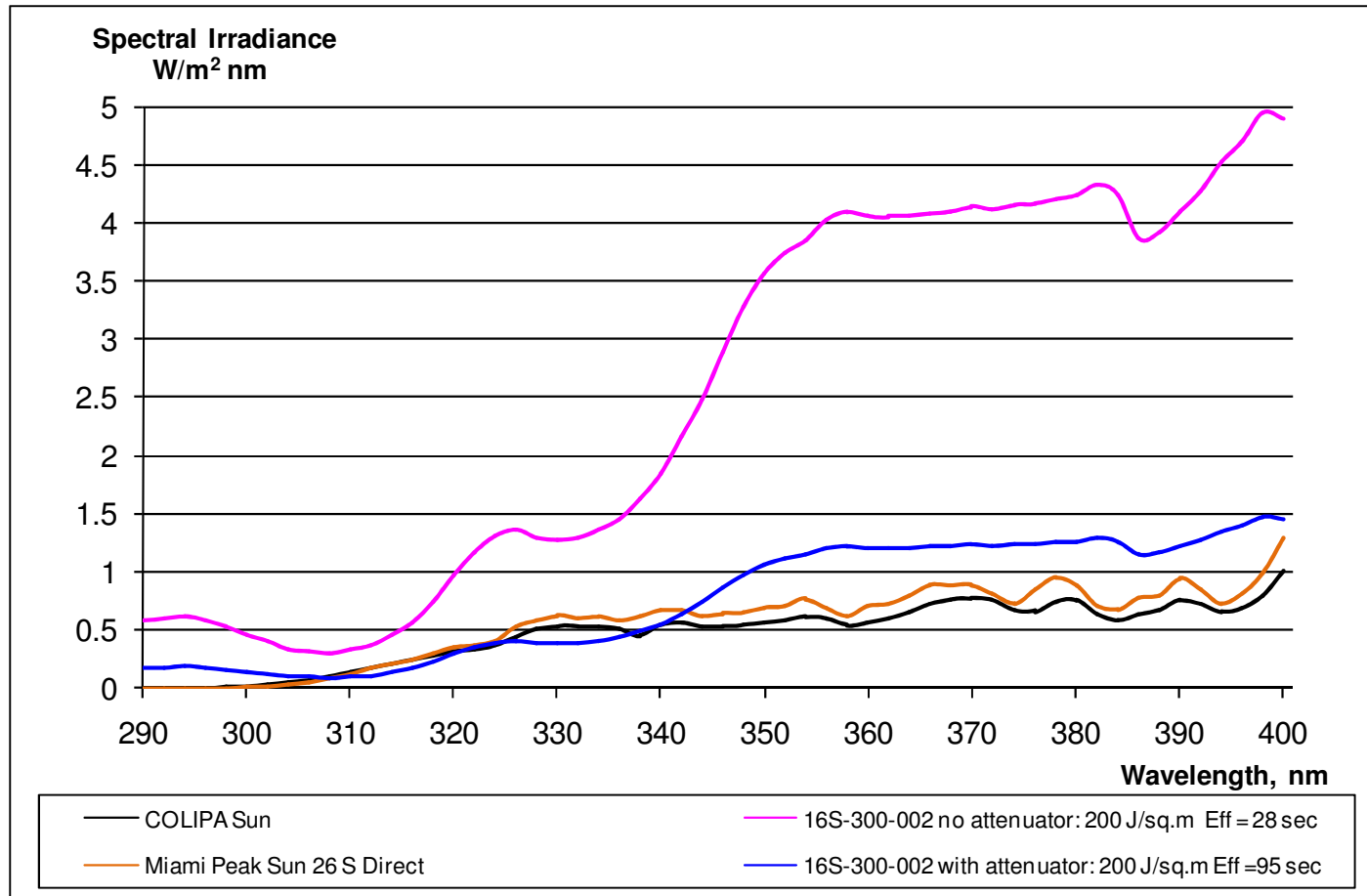
# Pre-Irradiation Equipment



**16S-300-002 Solar Simulator - full spectrum sunlight (Air Mass 1.5); variable 1 to 4 sun output intensity; vertical beam adapter redirecting the light beam to point downward (spot diameter 33 mm); XPS 400 - a precision current source for 16S-300-002; PMA2100 Radiometer with PMA2101 Detector (all from SolarLight Company, PA) [4]; Peltier-cooled surface (SC25 from Torrey Pines Scientific)**

# Pre-Irradiation Equipment/Criteria - Continued

## Spectral Irradiance of 16S-300-002 Solar Simulator (SolarLight Company, PA)



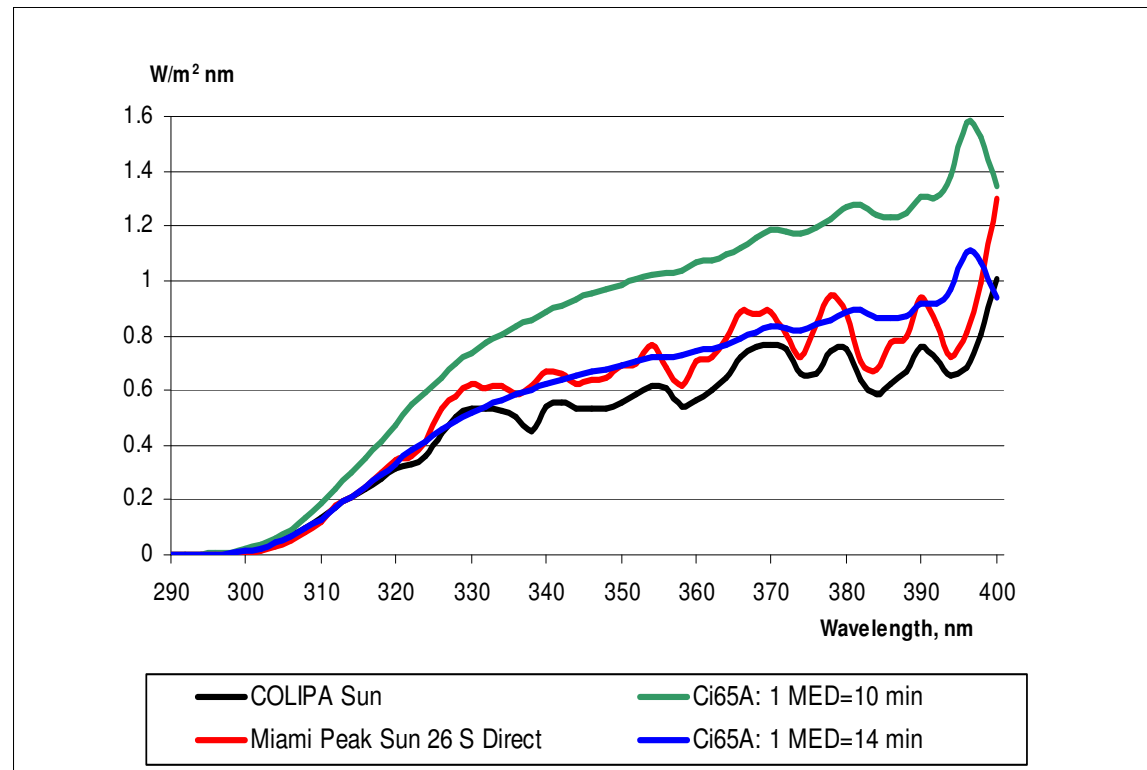
**Pre-irradiation criteria can be fulfilled with 16S-300-002 Solar Simulator (SolarLight Company, PA) that produces full spectrum sunlight (Air Mass, AM 1.5) with variable 1 to 4 sun output intensity [4]**

# Pre-Irradiation Equipment/Criteria- Continued



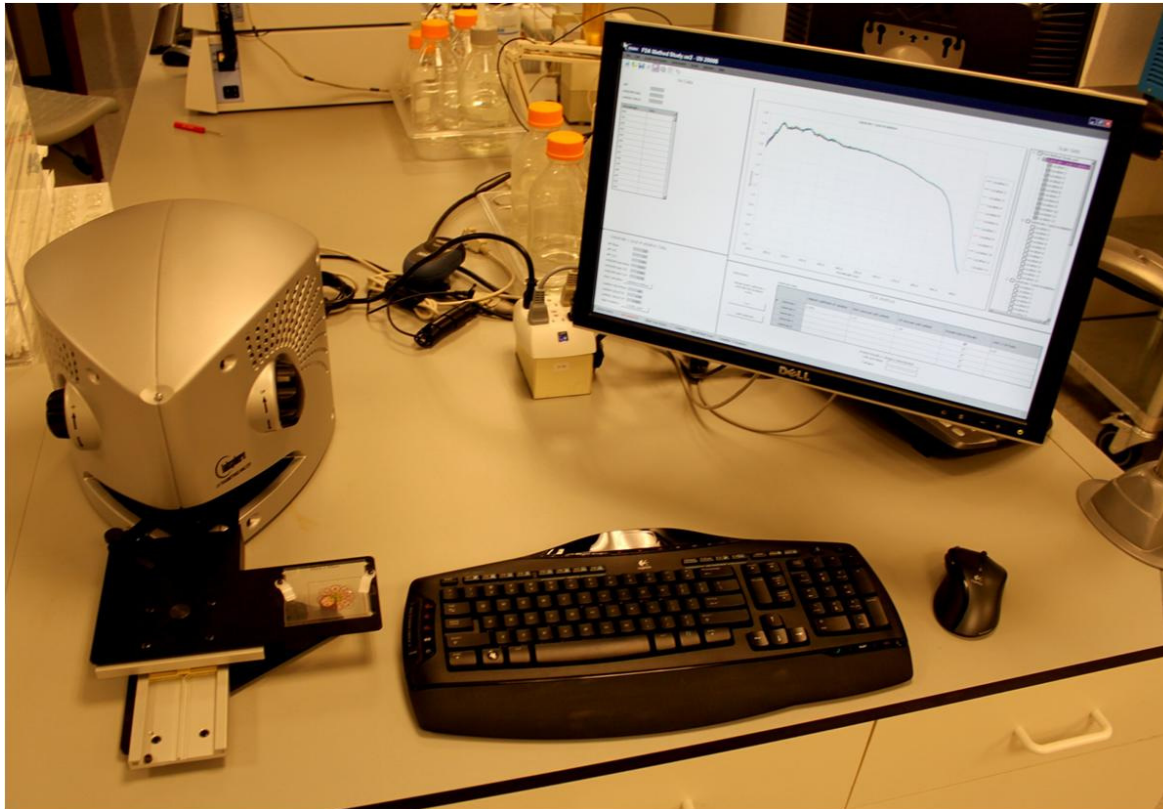
**Weather-O-Meter Ci65A (Atlas)**

**Spectral Irradiance of Ci65A with Right Light™ inner/Quartz outer**



**Pre-irradiation criteria can be fulfilled with Weather-O-Meter Ci65A with Right Light inner/Quartz outer filter combination from Atlas [3, 5]**

# Transmittance Analyzers/Software



LabSphere UV 2000S

## LabSphere UV 2000S - Technical Characteristics/Software [6]

- Xenon flash 10W/ Integrating sphere, photodetector/continuous emission spectrum from 290-400nm; sufficient illumination at each wavelength across the spectrum from 290-400nm, but not in excess of 0.2 J/cm<sup>2</sup>; sufficient dynamic range being  $\geq 2.7$  A at each wavelength across the spectrum from 290-400nm

-on April 30, 2009 LabSphere has released version 1.2 software to be used with UV 2000S instrument and that was the first system on the market capable of performing *in vitro*, FDA proposed, Boot Star, COLIPA, and user-defined SPF measurement methods; software will be further updated to support evolving regional methods (e.g., revised Boot Star, PA+ Method, UVAI/UV Ratio)

# Transmittance Analyzers/Software – Continued



Optometrics SPF 290S

## Optometrics SPF 290S - Technical Characteristics/Software [7]

- Xenon 125 W/ Integrating sphere, monochromator, photomultiplier; Dynamic range 2.5 A
- in 2009 Optometrics has released WinSPF Software V4.0 that provides compliance with the US FDA UVA *in vitro* testing procedures (as proposed) and performs the calculations and ratings prescribed in the Federal Register thus allowing SPF 290S user to evaluate their products to ensure they conform to US Federal labeling guidelines

**Both transmittance analyzers, Labsphere UV 2000S and Optometrics SPF 290S can be successfully utilized; it takes somewhat less time to conduct the measurements on Labsphere UV 2000S VS. Optometrics SPF 290S**

## **EWG – 2010 Top-rated sunscreens**

**EWG top-rated sunscreens all contain the minerals zinc or titanium. According to EWG they are the right choice for people who are looking for the best UVA protection without any sunscreen chemical considered to be a potential hormone disruptor. None of the top-rated products contain oxybenzone or vitamin A and none are sprayed or powdered [8]**

**Seven products from EWG Top sunscreens list and one from Non-mineral options list [8] were tested *in vitro* according to the FDA proposed rule [1, 3]; these products were also evaluated in BASF Sunscreen Simulator [9] using label information regarding concentrations of sunscreen actives**

# Test articles selected from EWG Top sunscreens and Non-mineral options lists [8]

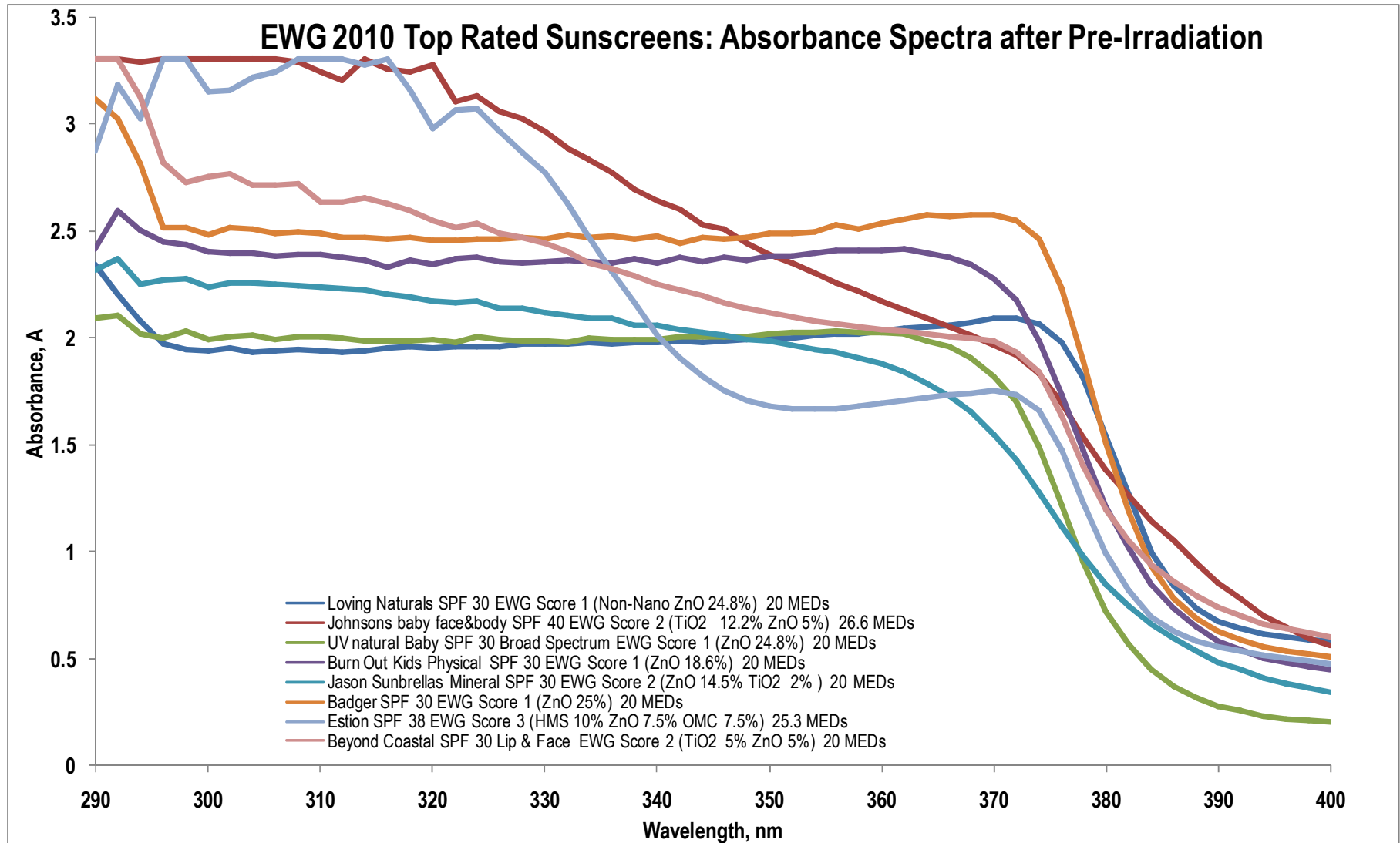


## Testing of products from EWG Top sunscreens and Non-mineral options lists

Sunscreen Products from EWG 2010 Top-Rated List [8]	EWG Overall Score* [8]	UVAI/UV Ratio (Star Rating) After Irradiation TESTED	UVAI/UV Ratio After Irradiation CALCULATED on BASF Sunscreen Simulator [9]	UVA/UVB Ratio after Irradiation TESTED	UVA/UVB Ratio after Irradiation CALCULATED on BASF Sunscreen Simulator [9]
Loving Naturals SPF 30 (Non-Nano ZnO 24.8%)	1	0.89 (Three Stars)	0.9-0.96	0.85	0.85-0.94
Johnsons baby face & body SPF 40 (TiO2 12.2% ZnO 5%)	2	0.69 (Two Stars)	0.78	0.62	0.65
UV natural Baby SPF 30 Broad Spectrum (ZnO 24.8%)	1	0.8 (Three Stars)	0.9-0.96	0.74	0.85-0.94
Burn Out Kids Physical SPF 30 (ZnO 18.6%)	1	0.83 (Three Stars)	0.9-0.96	0.78	0.84-0.94
Jason Sunbrellas Mineral SPF 30 (ZnO 14.5% TiO2 2%)	2	0.75 (Three Stars)	0.86-0.89	0.68	0.78-0.79
Badger SPF 30 (ZnO 25%)	1	0.85 (Three Stars)	0.9-0.96	0.791	0.85-0.94
Estion SPF 38 (Homosalate 10% ZnO 7.5% Octinoxate 7.5%)	3	0.61 (Two Stars)	0.63	0.521	0.49
Beyond Coastal w/ Z-cote SPF 30 Lip & Face (TiO2 5%, ZnO 5%)	2	0.74 (Three Stars)	0.78	0.645	0.65

\* SCORE KEY:  0-2 recommended  3-6 caution  7-10 avoid

# Testing of products from EWG Top sunscreens and Non-mineral options lists - Continued



# Testing of selected sunscreen products launched in the US in 2009-2010

Commercial Product	Sunscreen Actives	MEDs	UVAI/UV Ratio (Star Rating)
Banana Boat Sport Performance Broad spectrum sunblock AvoTriplex™ photostable UVA technology Long lasting UVA/UVB protection Ultra sweatproof, Non-greasy, Waterproof <b>SPF-15</b>	Avobenzone 1% Homosalate 6% Octocrylene 0.8% Oxybenzone 2%	10.0	0.741 (Three Stars)
Coppertone Nutra Shield Sunscreen Lotion Broad spectrum UVA/UVB with Dual Defense™ (sun protection + nourishing antioxidants to promote natural skin repair) Helps prevent premature skin aging <b>SPF-30</b>	Avobenzone 2% Homosalate 15% Octisalate 5% Octocrylene 2% Oxybenzone 5%	20.0	0.794 (Three Stars)
Banana Boat Sport Performance Broad spectrum sunblock AvoTriplex™ (photostable UVA technology) Long lasting UVA/UVB protection Ultra sweatproof, non greasy, waterproof <b>SPF-50</b>	Avobenzone 1.5% Homosalate 15% Octisalate 5% Octocrylene 1.25% Oxybenzone 6%	33.3	0.807 (Three Stars)
Neutrogena Ultimate Sport Sunblock Lotion with Helioplex® (broad spectrum UVA/UVB) ultra sweat proof, waterproof <b>SPF-55</b>	Avobenzone 3% Homosalate 10% Octisalate 5% Octocrylene 4% Oxybenzone 5%	36.7	0.845 (Three Stars)
Neutrogena Sensitive Skin Sunblock Lotion Broad spectrum UVA/UVB PureScreen™ Naturally-sourced sunscreen ingredients (fragrance free, waterproof, sweatproof) <b>SPF-60+</b>	Titanium Dioxide 4.9% Zinc Oxide 4.7%	40.0	0.682 (Two Stars)
Neutrogena Age Shield FACE sunblock Superior anti-aging protection with Helioplex (broad spectrum UVA/UVB) Advanced sunscreens technology shields skin 6 layers deep from skin aging UVA rays Anti-oxidants combat free radicals that accelerate the signs of aging Fragrance-free, oil-free, non-greasy feel <b>SPF-70+</b>	Avobenzone 3% Homosalate 15% Octisalate 5% Octocrylene 2.79% Oxybenzone 6%	46.7	0.893 (Three Stars)
Coppertone NutraShield Sunscreen Lotion Broad spectrum UVA/UVB with Dual Defense (sun protection + nourishing antioxidants to promote natural skin repair) Helps prevent premature skin aging <b>SPF-70</b>	Avobenzone 2% Homosalate 15% Octisalate 5% Octocrylene 2% Oxybenzone 6%	46.7	0.853 (Three Stars)
Neutrogena Ultra Sheer Dry-touch sunblock With Helioplex, broad spectrum UVA/UVB Light weight, clean feel (fast absorbing, waterproof) <b>SPF-85</b>	Avobenzone 3% Homosalate 15% Octisalate 5% Octocrylene 4.5% Oxybenzone 6%	56.7	0.852 (Three Stars)
Neutrogena Age Shield FACE sunblock Superior anti-aging protection with Helioplex (broad spectrum UVA/UVB) Advanced sunscreen technology shields skin 6 layers deep from skin aging UVA rays Combats free radicals that accelerate signs of aging <b>SPF-90</b>	Avobenzone 3% Homosalate 15% Octisalate 5% Octocrylene 4.5% Oxybenzone 6%	60.0	0.867 (Three Stars)

# Conclusions

- Methodological, technical and instrumental aspects of the sunscreen's *in vitro* testing methodologies were discussed
- Selected sunscreen products were tested to determine their UVAI/UV ratios
- Sunscreen products launched in the US in 2009 and especially in 2010 have sufficient photostability and progressively higher SPF values (50, 55, 70+, 85, 90+ 100...)
- The deficiencies of sunscreen simulator modeling/calculation approach used by EWG to evaluate the efficacy of commercial sunscreen products without conducting actual *in vitro* testing were discussed
- Leading sunscreen manufacturers more frequently use non- OTC claims regarding various skin benefits, for example sunscreen product's ability to counter the skin damage on cellular level, etc.
- Some of these sunscreen trends can be considered beneficial to the consumer
- The majority of US sunscreen products tested belonged to the “High UVA Protection” category (Three Stars) and failed to fulfill the “Highest UVA Protection” criterion (Four Stars)
- Reaching the highest UVAI/UV ratio (Four Stars), especially for sunscreens with SPF 30+ is a technical challenge that cannot be effectively addressed with the use of sunscreen actives currently approved in the US

# References

1. Federal Register, 21 CFR Parts 347 and 352 Sunscreen Drug Products for Over-the-Counter Human Use; Proposed Amendment of Final Monograph; Proposed Rule, 72(165), 49070-49122, §352.71, (2007)
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# Acknowledgements

**I am very grateful for the support of all my  
colleagues at IBT, Ossining, NY, especially:  
Steven G. Micceri and Art Duev**

**Thank you!**

## Attachment. Very Water Resistance *In Vivo*: COLIPA, FDA current/proposed

Test Conditions and Very Water Resistant (VWR) Criteria	COLIPA Guidelines for Evaluating Sun Product Water Resistance; <a href="http://www.colipa.com">http://www.colipa.com</a> (December 2005)	FDA Food and Drug Administration 21 CFR Sec. 352.76: Determination if a product is very water resistant	Proposed Amendment of Final Monograph; Proposed Rule, 72(165), 49070-49122 (2007) Sec. 352.76: Determination if a product is very water resistant (amended )
Temperature of water	29 °C +/- 2 °C	23 °C - 32 °C	23 °C - 32 °C
Immersion	Four immersions (baths) of 20 minutes, each followed by a 15 min rest/air dry period without toweling	Four 20 min immersion intervals each followed by a 20 min rest/air dry period without toweling (80 min water exposure total)	Four 20 min immersion intervals each followed by a 20 min rest/air dry period without toweling (80 min water exposure total)
Criteria for VWR (Waterproof) Claim	The remaining SPF after four 20 min water exposures is <b>≥ 50%</b>	Label SPF VWR value that is determined after 80 minutes (total) of water immersions	Label SPF VWR and <b>UVA (UVA-PF)</b> VWR (if appropriate) values determined after 80 min of water immersion

Sunscreen with initial (static) **SPF 50** can be claimed as **SPF 50 VWR** according to COLIPA if the remaining SPF after total 40 min of water exposure is just **SPF 25 (≥ 50% of initial SPF)**  
 Same sunscreen can be claimed as **SPF 25 VWR only** according to FDA  
**It is more difficult to fulfill FDA WR criteria while maintaining high sunscreen's efficacy**

## Attachment. Very Water Resistance In Vivo: COLIPA, FDA current/proposed

Test Conditions and Water Resistant (WR) Criteria	COLIPA Guidelines for Evaluating Sun Product Water Resistance; <a href="http://www.colipa.com">http://www.colipa.com</a> (December 2005)	FDA Food and Drug Administration 21 CFR Sec. 352.76: Determination if a product is water resistant	Proposed Amendment of Final Monograph; Proposed Rule, 72(165), 49070-49122 (2007) Sec. 352.76: Determination if a product is very water resistant (amended )
Temperature of water	29 °C +/- 2 °C	23 °C - 32 °C	23 °C - 32 °C
Immersion	Two immersions (baths) of 20 minutes, each followed by a 15 min rest/air dry period without toweling	Two 20 min immersion intervals, each followed by a 20 min rest/air dry period without toweling (40 min of water exposure total)	Two 20 min immersion intervals, each followed by a 20 min rest/air dry period without toweling (40 min of water exposure total)
Criteria for WR Claim	The remaining SPF after two 20 min water exposures is <b>≥ 50%</b>	Label SPF value determined after 40 minutes (total) of water immersions	The Label SPF WR and <b>UVA (UVA-PF)</b> WR (if appropriate) values determined after 40 min of water immersion

Sunscreen with initial (static) **SPF 50** can be claimed as **SPF 50 WR** according to COLIPA if the remaining SPF after total 40 min of water exposure is just **SPF 25 (≥ 50% of initial SPF)**  
 Same sunscreen can be claimed as **SPF 25 WR only** according to FDA  
**It is more difficult to fulfill FDA WR criteria while maintaining high sunscreen's efficacy**