Skin Safety of the UV Absorbers by Measurement Cytotoxicity High Functional Product with Water-in-Silicone System

In-Young Kim, Min-Ho Kim, Sang-In Nam and Kil-Whan Chun
Cheiljedand R & D Center Cosmetics and Household products Div.
51-1, 3KA, Shinhung-Dong, Chung-Ku, Inchon, 400-103, KOREA

Key Words

Measurement Cytotoxicity, UV Absorbers, Water-in-Silicone System, SPF, Waterproof Sunscreen Product, Nylonpoly UVA/UVB, ZnO and TiO_2, In-vitro and In-vivo

Abstract

Consumers have recently preferred to purchase extensive UV intercepting products, which are waterproof and free from side effects on skin. Testing Cytotoxicity (in-vitro) in SR method, cell survival ratio of UV-B interceptors decreased above 0.08W/V%, and so did that of UV-A interceptors above 0.06W/V%. Also, Patch-test of inorganic UV interceptors resulted in no skin irritation even below 10.0 and 11.25. UV interceptors in the sunlight showed yellowish discoloration in 5 to 14 days. In absorption curves, UV-B was most suitable for Octyl methoxycinnamate (OMC) and UV-A for Butyl methoxy dibenzoylmethane (BMDM). For this reason, Nylonpoly UVA/UVB the material of OMC and BMDM coated with Nylon & polyethylene, was used as the organic UV interceptor. And zinc oxide (ZnO) and titanium dioxide (TiO_2) was used as inorganic UV interceptors. The appropriate mixture ratio of ZnO and TiO_2 was 6 to 4. 6% of ZnO, 4% of TiO_2 and 5% of Nylonpoly UVA/UVB were all combined with our sunscreen cream. The SPF value of in-vivo applied to a guinea pig was 34.9 and that of in-vivo was 38.5.

Cyclomethicone and dimethicone were used in water-in-Silicone system. Cetyl dimethicone and sorbitan sesquioleate were used as emulsifiers and $MgSO_4 \cdot 7H_2O$, Mg-stearate/Mg-Al-stearate copolymer as emulsification stabilizers.

In practical application, each SPF duration of O/W type and W/S type containing sunscreen cream of the same content showed that W/S type of sunscreen cream was 5 times as durable as the other. This product is fit for using in swimming, climbing or skiing. This research is to minimize skin trouble caused by UV interceptors and to make one with proper softness, skin safety and UV intercepting efficiency.

1. Introduction

A method of SPF measurement on UV intercepting products has been different from nation to nation and company to company. But in 1978, FDA of the USA established a standard of SPF measurement and in 1983, 1984, Australia and Germany began to set up their own standards. Materials of basic cosmetic products have recently been extracted from natural objects such as animals and plants. Ultraviolet ray is the wave length of 200 nm–400 nm and divided into UV-B of 280–320 nm and UV-A of 320–400 nm. It is said that the formation of skin cancer begins with the damaged DNA of skin cells and mutation of cells by deformed DNA is the primary cause of skin aging. Especially UV-B of ultraviolet ray should be prevented because it causes skin irritation and related with skin cancer. Although UV-A has been considered safe except producing melanin on skin and facilitating melanin, it is known as a primary cause of skin irritation and skin cancer with the recent break-down of the Ozone layer and skin trouble and immunity restraint caused by active oxygen. UV-A has more skin permeability and more troubles with skin oxidation than UV-B does. To solve this, ultraviolet ray should be completely shut off from skin. Though today, UV-B and UV-A interceptors have been developed, most of the those products are only allowed to use within a limited amount due to their toxicity and safety of ray.

In addition, UV interceptors are mainly composed of organic synthetic materials and its cytotoxicity is believed to be a primary assignment that cosmetic researchers have to deal with. For this reason, there have been many theories on high values of SPF by applying an inorganic UV protector. And there also natural synthetic UV interceptors using a group of Amino acids have been produced to stabilize emulsification. In order to capsulated Suncaps (Sunsmart Co. USA) with Octyl methoxycinnamate (OMC) in matrix and Zinc oxide (ZnO), many capsulated goods with PMMA (polymethylmathacrylate) are produced now.

Main factors causing skin irritation are mixture of cosmetic components, pH, emulsificated form, an antiseptic and an aromatic and all these raw materials should be properly combined. All companies in this field are devoting their efforts to making safe products from cytotoxicity. Especially, more efficient UV interceptors are need in sporting fields such as swimming, skiing or mountain climbing. So, durability and waterproofing of the UV interceptors become very important.

In this study, we have tested cytotoxicity of the UV interceptors and tired to find some materials which are least irritating with skin. With the resent crucial cytotoxicity of UV interceptors, we tired to minimize cytotoxicity by using some raw materials that contact skin indirectly by coating with a measuring the range of UV extinction of organic and inorganic UV interceptors. SPF effect was evaluated by the method of O/W and W/S emulsification, and the intercepting effect was measured by comparing SPF in in-vitro and in-vivo.

2. Materials And The Method Of Measurement.

2.1 Materials

To measure skin irritation of UV interceptors, we used organic and inorganic UV interceptors in UV-B and UV-A ranges. For emulsifiers used in UV intercepting cream, W/O emulsifier of Cetyl dimethicone copolyol (Goldshmidt, Germany) and Sorbitan sesquioleate (ICI, America) were used. The silicone oil was used to improve a using sense, and Magnesium-aluminum silicate copolyol (ECC, America) and Magnesium stearate (Witco Organic) were used as a steady increaser. Also, to remove the irritation of an organic UV interceptor, Nylonpoly UVA/UVB (Creations Couleurs, USA) coated with Nylon and polyethylene were used for OMC and BMDM. Lastly, we used ZnO and TiO_2 to obtain a high SPF value.

In this experiment, reagents were used without refining exclusive cosmetic materials. Cosmetic ingredients were Vitamin-E and Sodium hyaluronate . Positive/negative de-ion water passage ion exchange resin top was used as distilled water.

2.2 The method measurement skin irritation of UV interceptors.

For a cell, normal human fibroblast was cultured by combining 5% of CO_2 at 37°C with DEME containing 5% of fetal bovine serum. The cell collected with Neutral Red Assay was diluted by a culture medium containing 2% of serum and inoculated 90 µl of cell suspension (2500–3000 cells/well) in each well of 96 well tissue culture plate and then, cultured at 37°C with 5% of CO_2 for 2 days. After culture, it was exchanged with a new culture medium of 90 µl and treated with 10 lml of a testing material, and then cultured again for 2 days. Into each well padding culture, 100 µl of neutral red solution (50 µg/µl) was added and kept for 3 hours. We extracted Neutral Red in a cell y using 1.0% of acetic acid/50% of ethanol solution after treating with 10% of formalin/1.0% of $CaCl_2$, 100 µl, condensed in lysosome of a cell which was not damaged or alive when Neutral Red Passed Complete Plasma membrane. Extracted Neutral Red was directly measured for its cytotoxicity in 540 nm with ELISA reader.

2.3 Measurement of SPF

2.3.1 In-vitro measurement

To measure UV intercepting value of UV-B and UV-A, we made the most stable W/O emulsion cream containing fixed amount of UV intercepting material. And then we measured the SPF with multiport Solar UV Simulator (USA, Solar Light Co., (Fig.1) and SPF-290 Analyzer (Fig.2). The sample for the measurement was 2.0 µl/cm².

2.3.2 In-vivo measurement

For the measurement, we used a guinea pig and Ultra Solar Simulator (USA) as a measuring instrument. We tried to obtain reliable SPF value by comparing with the result of established in-vivo.

3. Results And The Discussion

3.1 UV Protector

Nowadays, the harmfulness of ultraviolet ray on skin is percepted as serious and the results has become not a problem with only cosmetic but the problem with environmental pollution of the earth. Therefore, products with high SPF also need safety of skin and stability of components. To enhance the effect of intercepting ultraviolet ray, combining the materials having on effect of intercepting ultraviolet ray is necessary. UV interceptors are classified into an organic UV absorber and an inorganic UV interceptor (Table 1). Inorganic UV disperser is composed of metal oxide. UV intercepting mechanism of the ultrafine particles of metal oxide acts in complex condition of absorption and dispersion, and this is called a general interceptor. An absorber and an interceptor are extensively called a UV interceptor. This UV interceptor ha a wide intercepting range from ultraviolet ray and is widely applied to various products. Today, the main characteristics of UV interceptors of UV-B and UV-A are as follows:

(A) UV-B Intercepting effect

UV-B (Fig.3) cause acute inflammation or irritation with skin, increases melanin pigment, and becomes a cause of spots and freckles. A lot of products with high SPF value appear on the market.

UV intercepting mechanism of ultrafine TiO_2 is according to combining action of absorption and dispersion. There is a report that the smaller diameter of the particle, the higher absorption and the bigger diameter, the higher dispersion. Ultraviolet TiO_2 has different reflective index according to its crystallization. TiO_2 with small reflective index is in the course of developing now. It also has a high transparency to a visible ray and the case of getting loose in white on the skin is rare. The reflective index of TiO_2 is about 1.9. This value is a little higher than the reflective index of oil (1.5) and when it is applied onto the skin, it shows a transparent color. In it may cause raging fire, discoloration, condensation and division of combining ingredients. For these reasons, Silica, Alumina and Silicone are used to treat with the surface and it contributes to dispersion and waterproof.

(B) UV-A Intercepting effect

UV-a indicates the range of 320 ~ 400 nmand contains about 90% of ultraviolet ray which reaches the surface of the earth. Though the effect of UV-A is smaller than that of UV-B, UV-A reaches deep skin (Fig.3) to the true inner skin and may cause a chronic skin trouble to lead skin aging. On the other hand, UV-B is absorbed or dispered only on the outer skin. Main effects of UV-A on skin is as follows:

(a) Immediate melanin by the temporal oxide of Melanin.
(b) Though weak to cause inflammation or irritation, it cause delay-type melanin.
(c) Increases a bad effect of UV-B.
(d) Increases pigment formation.
(e) Reaches the true inner skin and changes the fiber of the true inner skin.
(f) A cause of aging by promoting wrinkle formation.

A lot of studies on cytotoxicity of UV-A are now in the course of research. Established UV interceptors are mainly Benzophenone or BMDM but, discoloration of them is a problem. Therefore, ZnO is widely used as an up to date UV-A interceptor and an inorganic system UV interceptor.

Recently, ZnO is one of noticed materials because of its high safety and excellent ray stability. UV absorber has the largest absorption wave length around 320 nmand can cover relatively large range from UV-B and UV-A. Comparing with ultrafining oxidized Titan, ZnO has no Cloudiness in high concentration and sensuous property.

3.2 Absorption spectrum of UV interceptor

We measured UV absorption spectrum of organic UV interceptors which were widely used nowadays (Table 1). UV was measured within the range of 200 – 400 nm (Fig.4a). UV-B interceptors showed the largest absorption curve in the range of 280 – 320 nm and UV-A interceptors showed its largest curve in the range of 280 – 360 nm (Fig.4b). For absorption wave length of interceptors ZnO (0.03 μm) had a high extinction from 280 to 400 nm and TiO_2(0.03 μm) showed the largest absorption curves at 318 nm and 382 nm(Fig.4c).

Ferrous oxide and Talc are regarded as having UV intercepting effect because they showed a tiny absorption curve in the range of UV.

3.3 The stability of organic UV interceptor

We obsorved UV interceptors in table 1 after making the concentration of each sample as 3% in mineral oil as a solvent for 2 weeks in sunlight. As a result of the observation, all materials of UV-B and became discolored but, among them, OMC and BMDM was least discolored (Fig.5)

3.4 Skin irritation test of UV interceptor

The experiment on skin irritation (In-vitro) was performed in the method of section 2.2. Survival ratio (%) of a cell was measured by increasing the concentration as 0.001 – 0.8W/V%. As a result of the experiment an UV-B interceptor starts reducing its survival ratio above 0.08W/V% (Fig.6). As a result of the experiment an UV-A interceptor starts reducing its survival ratio above 0.06W/V% (Fig.7) But, Nylonpoly (UVA/UVB showed 88% of survival ratio even above up to 0.8W/V%. With the result, we could find that in the respect of skin irritation Nylonpoly (UVA/UVB (Fig.8) is safer than organic UV interceptors. The skin safety of ZnO and TiO_2 can be proved with a general material but, in this experiment, used without experiment because of the difficulty combining a culture medium. Also, Patch-test of inorganic UV interceptors resulted in no skin irritation even below 10 and 11.25 (Table II).

3.5 Mixing condition of ZnO and TiO_2

We measured SPF by changing mixing rate to Blank, 10:0, 0:10, 5:5, and 6:4 based on total 10% of ZnO and TiO_2. The SPF value when ZnO:TiO_2 was mixed in 5:5 was 16.5 and when it was changed to 6:4, SPF value was 19.8. the rate of ZnO and TiO_2,

Therefore, was decided as 6:4 in this experiment (Table III, Fig.9). It is considered that the reason may be a rising effect of two raw materials because ZnO shows extensive UV absorption in the range of 200 – 400. W/S type sunscreen cream was made by mixing 6% of ZnO and 4% of TiO_2 and 5% of Nylonpoly UVA/UVB. And its SPF was 38.5 (Fig.9).

3.6 SPF Measurement (In-vitro and In-vivo)

Up to now, we measured SPF, comparing in in-vivo and in-vitro by mixing ZnO, TiO_2 and Nylonpoly UVA/UVB properly in consideration of the stability and safety of UV interceptors.

For in-vitro SPF measurement, the same method as FDA regulation was used and in-vivo SPF measurement was performed after removing hairs of a guinea pig, with ultra solar simulator (USA). The result showed that the value of in-vivo SPF was 34.9 (Fig.12) and that of in-vitro SPF measured with SPF-290 analyzer was 38.5 (Fig.10). The SPF difference like this is regarded as an error of the experiment method.

3.7 Water-in Silicone (W/S) emulsion system

Water-in oil system has various defects such as heavy using sense and oil sense. To improve the problems, Silicone system with a square or pentagonal ring is widely used and Cyclomethicone and dimethicone are also generally used. Cyclomethicone is light oil with low volatilization and has a good common use with other solvents. For its emulsion, a Surface active agent with a silicone ring is usually used. V) sunscreen cream.

The emulsifier used in W/S was Cetyldimethicone cocpolyol and Sorbitan sesquioleate and as a stabilizer, $MgSO_4 \cdot 7H_2O$, Mg-stearate, Mg-Al-stearate copolymer were used. The durability of SPF was measured with W/S (Table IV) type products and O/W-type (Table V) In a general O/W type sunscreen cream, its durability decreased to 6.5 in 20 minutes after 1 application and in W/S type Sunscreen cream, its SPF value kept the same 25.7 after 100minutes (Fig.11).

Waterproof testing, 0.3g of the sample cream was applied to the skin in an area of 30 cm^2. After that, the skin was immersed in water for 20 min. then left out of the water for 20 min. This procedure was repeated three times and then the skin was irradiated.

The sunscreen cream prepared with water-in silicone system was recognized higher SPF value than that of O/W system under both static and waterproof testing.

4. Conclusion

This study was performed to find the raw material least irritating with skin by testing the skin toxicity and safety and the stability of a synthetic UV interceptor and pigments. Nowadays, because skin irritation of an organic UV interceptor is on the rise, this study have tired to indirectly contact skin by coating the synthetic raw materials.

1. Consumers have recently preferred to purchase extensive UV intercepting products, which are waterproof and free from side effects on skin. Testing Cytotoxicity (in-vitro) in NR method, cell survival ratio of UV-B interceptors decreased above 0.08W/V%, and so did that of UV-A interceptors above 0.06W/V%. Also, Patch-test of inorganic UV interceptors resulted in no cytotoxicity even below slight irritation.

2. UV interceptors in the sunlight showed yellowish discoloration in 5 to 14 days. In absorption curves, UV-B was most suitable for Octyl methoxycinnamate (OMC) and UV-A for Butyl methoxy dibenzoylmethane (BMDM). For this reason, Nylonpoly UVA/UVB the material of OMC and BMDM coated with Nylon & polyethylene, was used as the organic UV interceptor.

3. Zinc oxide (ZnO) and titanium dioxide (TiO_2) were used as inorganic UV interceptors. The appropriate mixture ratio of ZnO and TiO_2 was 6 to 4. 6% of ZnO, 4% of TiO_2 and 5% of Nylonpoly UVA/UVB were all combined with our Sunscreen Cream. The SPF value of in-vivo applied to a guinea pig was 34.9 and that of in-vivo was 38.5. Cyclomethicone and dimethicone were used in water-in-Silicone system. Cetyl dimethicone and sorbitan sesquioleate were used as emulsifiers and $MgSO_4 \cdot 7H_2O$, Mg-stearate/Mg-Al-stearate copolymer as emulsification stabilizers.

4. In practical application, each SPF duration of O/W type and W/S type containing sunscreen cream of the same content showed that W/S type of sunscreen cream was 5 times as durable as the other. This product is fit for using in swimming, climbing or skiing.

This research is to minimize skin trouble caused by UV interceptors and to make one with proper softness, skin safety and UV intercepting efficiency.

References

1. T. Hikima and K. Ogino, *Fragrance Journal*, 7, 37-44 (1995)
2. Martin M. Rieger, *Cosmetics & Toiletries*, 102, 91-96 (1987)
3. Alfred J. Rieger, *Cosmetics & Toiletries*, 102, 102-106 (1987)
4. Ward L. Billhimer, *Cosmetics & Toiletries*, 102, 83-89 (1987)
5. Nadim A. Shaath, *Cosmetics & Toiletries*, 102, 21-36 (1987)
6. Rosario Oteri, Steve Johnson and Susan Dastis, *Cosmetics & Toiletries*, 102, 107-109 (1987)
7. Charles Fox, *Cosmetics & Toiletries*, 102, 69-81 (1987)
8. Martin Stockdale, *Cosmetics & Toiletries*, 102, 111-115 (1987)
9. Nadim A. Shaath, *Cosmetics & Toiletries*, 102, 69-81 (1987)
10. Mark A. Mitchnick, *Cosmetics & Toiletries*, 107, 111 (1992)
11. M.W. Brown and E. Galley, *Cosmetics & Toiletries*, 105, 69 (1990)
12. Robert P. Brady and Karen L. Kelley, *Cosmetics & Toiletries*, 105, 25 (1990)
13. Anthony J. Petro, *Int. J. of Cosmet. Sci.*, 3, 185-196 (1981)
14. Bonnie Agnes Loudin, *J. Toxicol-Cut. & Ocular Toxicol*, 4 (2), 135-140 (1985)
15. T. Ogawa, H. Kaminaga, H. Kakishima and T. Yoneya, 香粧會誌 116 (2), 86-89 (1992)
16. Emi Tada, Michiyo Maruyama and Yoko Todoroki, 香粧會誌 16 (3) 149-152 (1992)
17. Ogawa K., Odagiri H. and Kumagai S., *IFSCC*, 623-640 (1994)
18. Pearse A. D., Int. *J. Cosmet. Sci.*, 15, 234-244 (1993)
19. Sayre Robert M., *J. Soc. Cosmet. Chem.*, 31, 133-143 (1980)
20. Luciann L.H. and Pentland Alice P., *J. Invest. Dermatol.*, 1. 35-41 (1993)

Table 1. Major sunscreen agents have been used in Korea.

Ingredients	λmax(nm)	Max (%conc.)	Trade Name
UV-B Absorbers			
Octylmethoxy cinnamate	311	7.5	Parsol MCX
(DEA)-methoxy cinnamate	290	8.0	Bernel Hydro
Octyldimethyl-PABA	311	9.0	Eusolex 6007
2-phenylbenzimidazole-5-sulphonic acid	310	4.0	Eusolex 232
Homosalate	306	10.0	Kemester HMS
Octocryene	303	10.0	Uvinul N-539
UV-A Absorbers			
Butyl-methoxydibenzoylmethane	343 ~ 365	5.0	Parsol 1789
Benzophenone-1	292 ~ 332	-	Uvinul 400
Benzophenone-2	345 ~ 354	-	Uvinul D-50
Benzophenone-3	295 ~ 330	2 ~ 6	Uvinul M-40
Benzophenone-4	289 ~ 332	5 ~ 10	Uvinul MS-40
Benzophenone-12	280 ~ 342	-	Uvinul 408
Physical Pigments			
Titanium dioxide	320	2 ~ 25	Ultrafine TiO2
Zinc Oxide	380	2 ~ 25	Z-COTE HP-1
Ferrous Oxide	280 ~	-	-
Talc 1623	280 ~	-	-

Table II. Cytotoxicity measurements by Patch-test.

Samples	Result of evaluation (No. of Subjects)					Average skin irritation range(%)	Remarks
	+++	++	+	±	-		
UV-B sunscreen agents							
Octylmethoxycinnamate	-	1	2	7	9	18.75	Parsol MCX
DEA-methoxycinnamate	-	1	3	8	8	21.25	Bernel Hydro
Octyldimethyl PABA	-	1	5	7	7	25.00	Eusolex 6007
2-phenylbenzimidazol-5-sulphonic acid	1	1	4	6	8	26.25	Eusolex 232
Homosalate	-	2	3	10	5	27.50	Kemester HMS
Octocrylene	-	1	6	4	9	23.75	Uvinul N539
UV-A sunscreen agents							
Butyl methoxydibenzoylmethane	-	1	3	8	8	21.25	Parsol 1789
Benzophenone-1	2	3	4	5	6	37.50	Uvinul-400
Benzophenone-2	-	2	6	8	4	32.50	Uvinul-D50
Benzophenone-3	-	1	5	9	5	27.50	Uvinul-M40
Benzophenone-4	1	1	3	8	7	26.25	Uvinul-MS40
Benzophenone-12	2	1	4	9	4	35.00	Uvinul-408
Pigments							
Titanium dioxide(TiO_2)	-	-	2	4	14	10.00	UV-Titane M160
Zincoxide(ZnO)	-		2	5	13	11.25	

$$\text{Average Skin Irritation Range} = \frac{\Sigma(\text{Results of evaluation X Standard numbers of person})}{\Sigma(\text{total persons X numbers of sincere marked irritation})} \text{ X } 100$$

#. Standard of Evaluation

Irritation Range		Values of standard valuation	Decision of Skin Irritation	Range
Sincere marked irritation	+++	2.0~	Sincere marked irritation	above 40
Marked irritation	++	1.5	Moderate marked irritation	20 ~ 30
Moderate marked irritation	+	1.0	Slight marked irritation	10 ~ 20
Slight marked irritation	±	0.5	No irritation	0 ~ 10
No irritation	-	0		

Table III. SPF in-vitro Measurement of a Water-in-Silicone type Cream with SPF-290Analyzer. (Average values:n=10)

Ratio (Total : 10%)	SPF	Remarks
ZnO : TiO_2 (10 : 0)	14.9	
ZnO : TiO_2 (0 : 10)	10.6	
ZnO : TiO_2 (6 : 4)	19.8	
ZnO : TiO_2(5 : 5)	16.5	
Nylonpoly™UVA/UVB(5%)	11.4	
ZnO:TiO_2:Nylonpoly(6%:4%:5%)	38.9	

Table IV. Formula of Water-in-Silicone type Sunscreen cream.

Type	Ingredients	Contents(%)	Remarks
Oil phase	A) Dimethicone(6cs)	7.0	Emollient
	Cyclomethicone	8.0	Emollient
	Al-Mg silicate copolyol	3.0	Stabilizer
	Octyl dodecanol	5.0	Emollient
	Magnesium stearate	0.5	Stabilizer(W/O)
	Zinc stearate	0.3	Stabilizer(W/O)
	Cetyl dimethicone copolyol	1.5	W/O Emulsifier
	Sorbitan sesquioleate	0.5	W/O Emulsifier
	BHT	0.01	
	Propyl paraben	0.15	Preservatives
	Tocopheryl acetate	0.4	
Water phase	B) Xanthan gum(2% soln.)	5.0	Thickener
	Glycerin	3.0	Moisturizer
	1,3-Butylene glycol	2.0	Moisturizer
	Methyl paraben	0.2	Preservatives
	MgSO4.7H2O	0.5	Stabilizer
	Deionized Water	Q.S	
UV Base	C) Zinc oxide	6.0	UV screen agent
	Titanium dioxide	4.0	UV screen agent
	Nylonpoly™UVA/UVB	5.0	
Add	D) Perfume	0.2	

Preparation in the laboratory:

1. Melt components of water phase(A) at 80~82°C and mix intimately.

2. Heat components of oil phase (B) to 82~84°C and add to water phase(A) and UV base(C)while stirring.

Stir for 15minutes at this temperature(H/M speed:3,000rpm).

3). Start cooling and continue mixing with propeller stirrer speed 15 rpm.

4).When temperature drops below 45°C added additives(D) mix after each addition to get uniform

mixture.

5) At 30°C stop mixing, take sample for QC and when approved, pack the product.

Table V. Formula of Oil-in-Water type Sunscreen cream.

Type	Ingredients	Contents(%)	Remarks
Water Phase	A) Magnesium Silicate(10%soln.)	5.0	Thickener
	Xanthan gum (2%soln.)	5.0	Stabilizer
	TEA-stearate(10%soln.)	8.0	Consistency agent
	EDTA-2Na	0.02	Chelating Agent
	Glycerin	3.0	Moisturizer
	1,3-butylene glycol	2.0	Moisturizer
	Methyl paraben	0.2	Preservatives
	Deionized Water	Q.S	
Oil Phase	B) Dimethicone(6cs)	7.0	Emollient
	Cyclomethicone	8.0	Emollient
	Al-Mg silicate copolyol	3.0	Stabilizer
	Octyl dodecanol	5.0	Emollient
	Magnesium stearate	0.5	Stabilizer
	Zinc stearate	0.3	Stabilizer
	Glyceryl stearate PEG-100 stearate	1.5	O/W Emulsifier
	Polysorbate-60	1.0	O/W Emulsifier
	BHT	0.01	
	Propyl paraben	0.15	Preservatives
	Tocopheryl acetate	0.4	
UV base	C) Zinc oxide	6.0	UV screen agent
	Titanium dioxide	4.0	"
	Nylonpoly ™ UVA/UVB	5.0	"
Additives	D) Polyacrylamide/C13-14isoparaffin/ laureth-7(SEFIGEL #305)	0.3	Thickening agent
	Perfume	0.2	

Preparation in the laboratory:

1). Mix ingredients of oil phase (A) and start heating up (70~72°C) to melt Oil phase.

2). Mix ingredients of water phase (B) and start heating up (72~74°C)

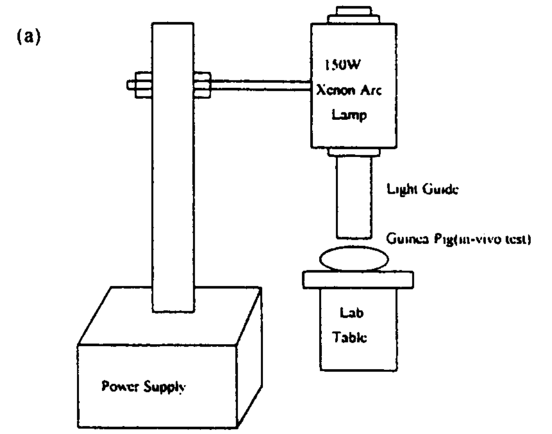
Start mixing- oil phase at 1500~2000rpm while adding water phase slowly. Depending on sample size, the addition of water phase should be 10~15 min.

3). Homogenize. Start cooling and continue mixing with propeller stirrer speed 15 rpm.

4). When temperature drops below 45°C added additives(D) mix after each addition to get uniform mixture.

5) At 30°C stop mixing, take sample for QC and when approved, pack the product.

(a)



(b)

$$\text{SPF value} = \frac{\text{T(the time taken to achieve the MED when the screen product was applied)}}{\text{to(the time taken to achieve the MED when the screen product was not applied)}}$$

Irradiance (without sunscreens) : 6.7×10^{-4} W/cm^2

Exposure time : 1min.(to)

Accumulated radiation exposure : 45mJ/cm^2

Figure 1. Schematic diagram representation of the equipment used(a) and the calculation of SPF values by in vivo method(b).

(a)

$$SPF = \frac{\sum_{290}^{400} E\lambda \times E\lambda\ \lambda}{\sum_{290}^{400} (E\lambda \times E\lambda / MPF\lambda)}$$

$$MPF\lambda = 1/T\lambda = Tref/Trun$$

$$MPF\lambda = \sum_{i=1}^{n} (MPF\lambda)i/n(n=18)$$

(b)

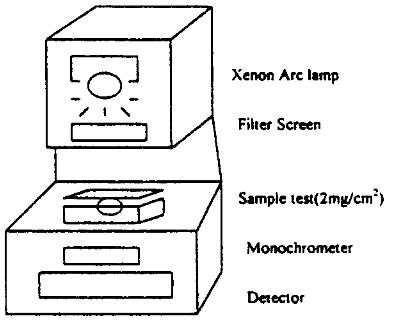


Figure 2. Calculation formula of SPF by the SPF 290-Analyzer(a). Schematic diagram representation of the SPF 290-Analyzer(b).

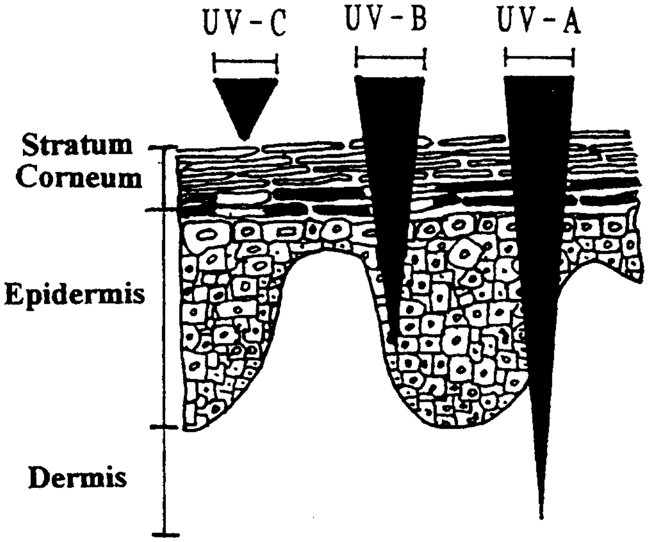


Figure 3. Schematic diagram of UV transmittance on the Skin.

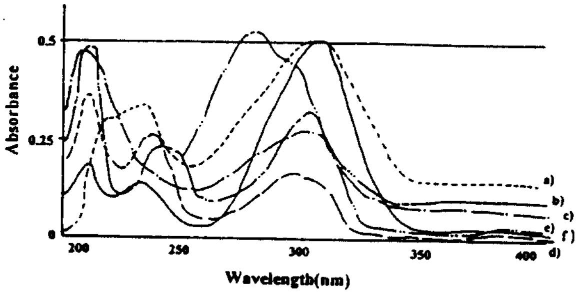


Figure 4a. Manifold UV-B zone spectra in chemicals.[a):Octyl methoxy-cinnamate(5.12ppm), b):DEA-methoxycinnamate(5.35ppm), c):Octyldimethyl PABA(5.20ppm), d):2-phenylbenzimidazolle-5-sulphonic acid(5.66ppm)[2-PBISA], e):Homosalate(5.45ppm), f):Octocrylene (5.1ppm)]

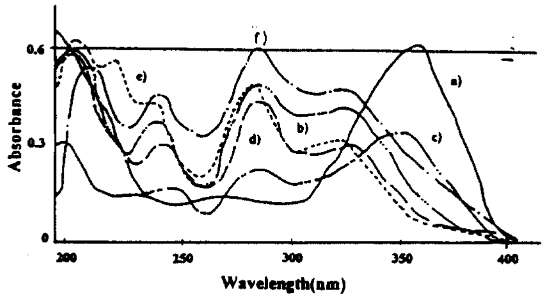


Figure 4b. Manifold UV-A zone spectra in chemicals.[a):Butyl methoxydi-benzoylmethane(5.18ppm), b):Benzophenone-1(5.10ppm), c):Benzophenone-2(10ppm), d):Benzophenone-3(5.12ppm), e): Benzophenone-4(5.18ppm), f):Benzophenone-12(5.00ppm)]

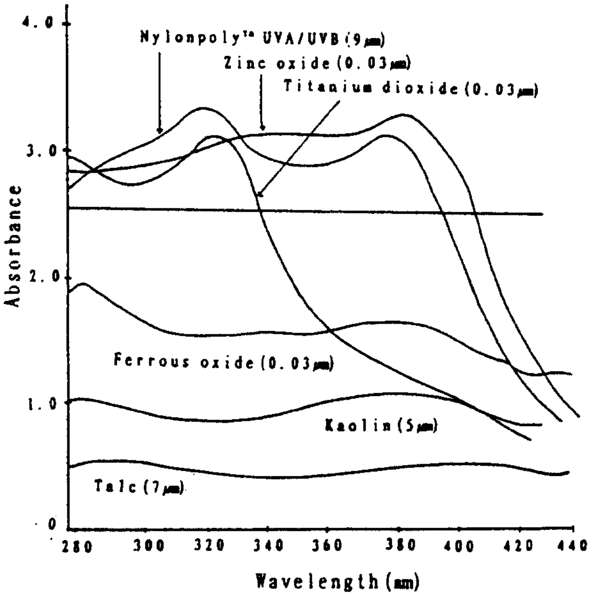


Figure 4c. Effectiveness of UV protection about the UV scattering pigments.

Figure 5: see page 13

Figure 6. Cytotoxicity measurement of UV-B interceptors: a)Octocrylene, b)Octyldimethyl PABA, c)2-PBISA, d)DEA-methoxycinnamate, e)Homosalate, f)OMC.

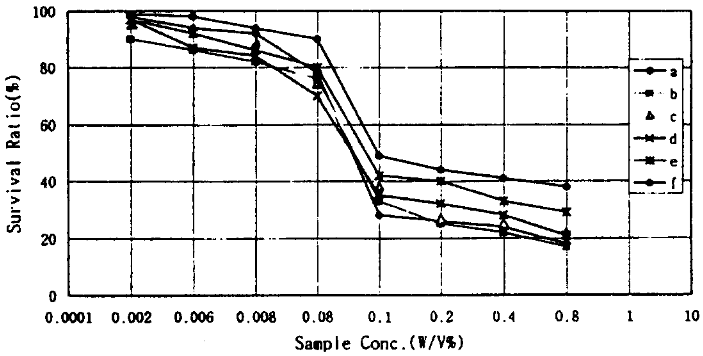
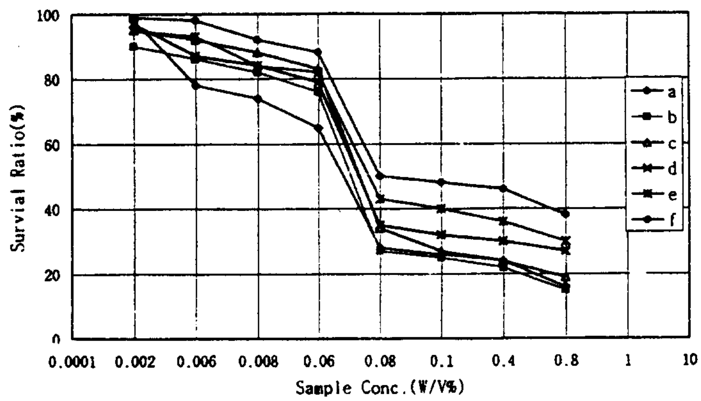


Figure 7. Cytotoxicity measurement of UV-A interceptors: a)B-1, b)B-12, c)B-2, d)B-3, e)B-4, f)BMDM.



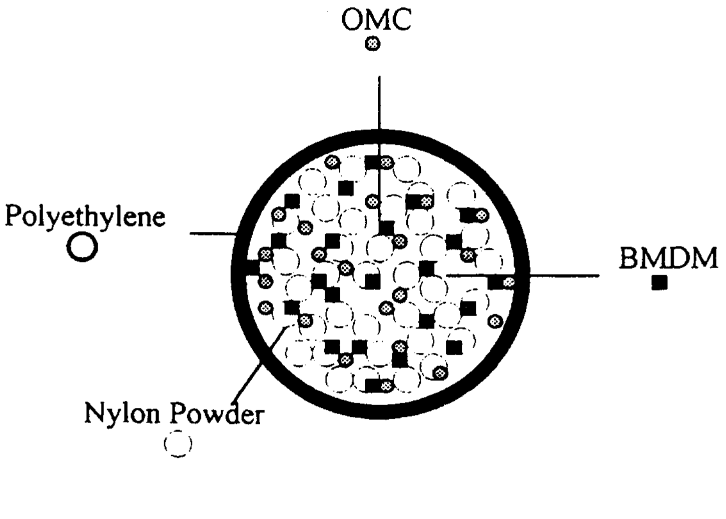


Figure 8. Schematic diagram of a Nylonpoly TM UVA/UVB.

Figure 9. Optimum mixture ratio of ZnO to TiO2.

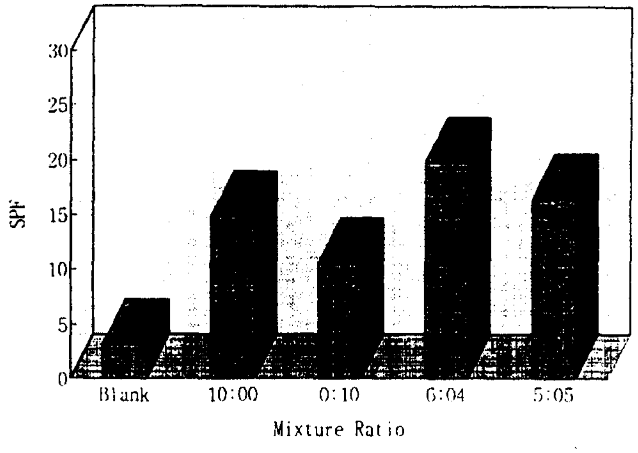


Figure 10. SPF of ZnO:TiO2:Nylonpoly TM UVA/UVB.

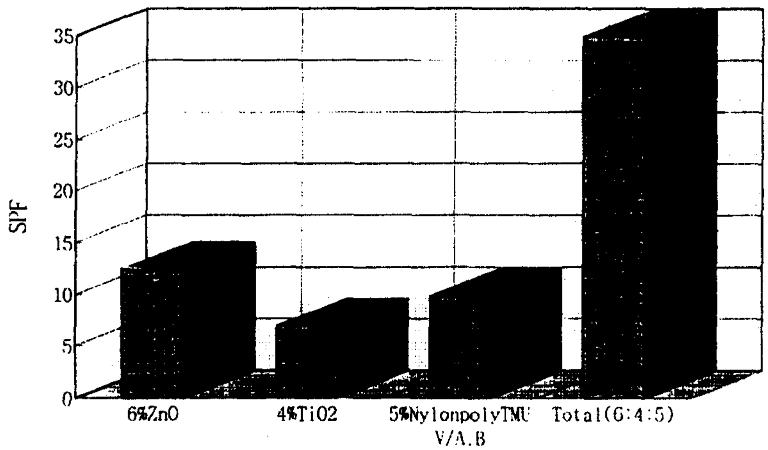
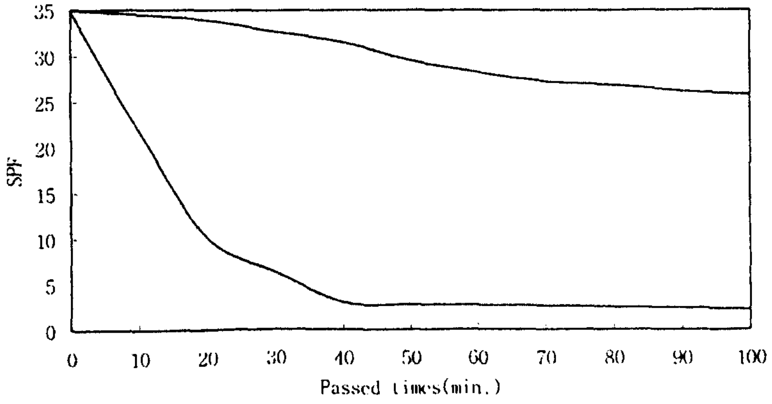
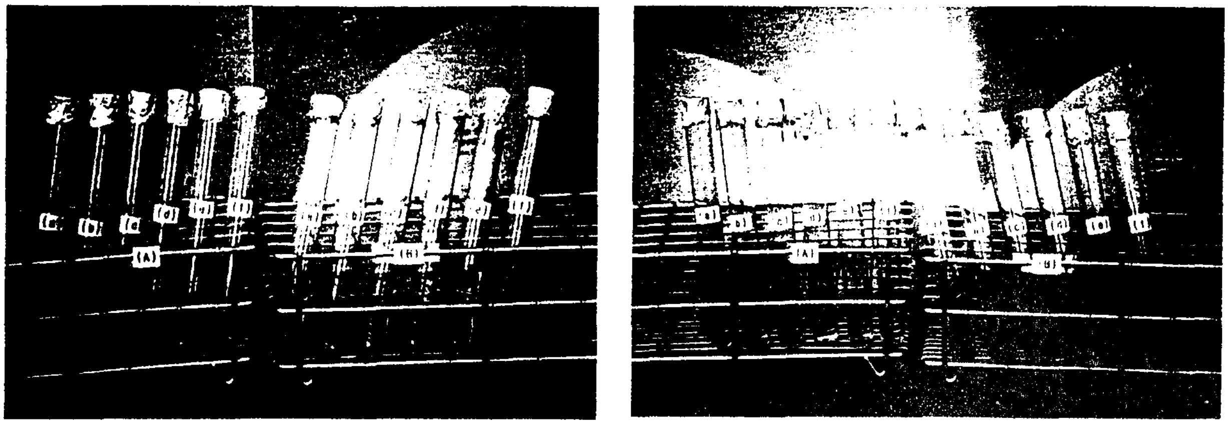


Figure 11. SPF of W/S type and O/W type by Passed times.





a)Octocrylene, b)Homosalate, c)2-PBISA, d)Octyl dimethyl-PABA, e)DEA-methoxycinnamate, f) OMC ; UV-B Interceptors

a)B-1, b)B-2, c)B-3, d)B-4, e)B-12, f)BMDM ; UV-A Interceptors

Figure 5. Stability of Chemucal UV agents in paraffin oil (each samples 3% soln.)

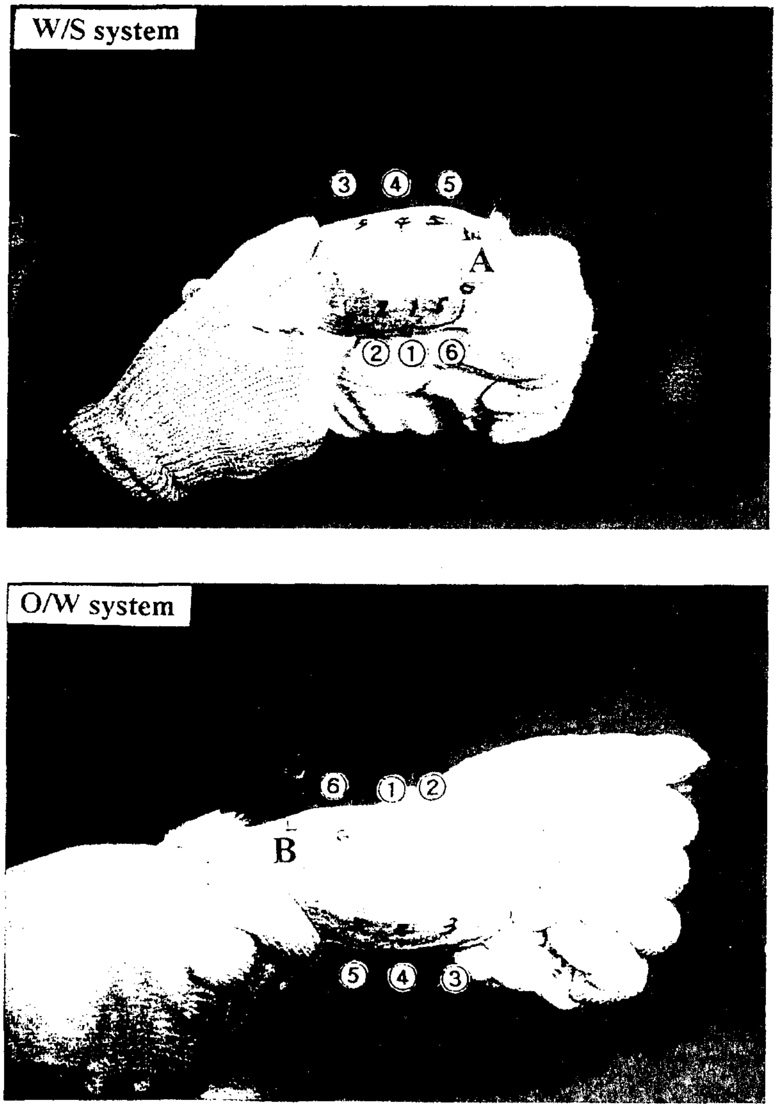


Figure 12. SPF of W/S system and O/W system by Guinea pig.