Ingredient/INCI Name	% w/w				
	Control	A	B	C	D
Phase A					
Solaveil™ HTP1 (Titanium Dioxide (and) Alumina (and) Stearic Acid)[1]	15.00	15.00	15.00	15.00	10.00
Avobenzone (Butyl Methoxydibenzylmethane)[2]	0	3.00	0	0	0
Solaveil CZ-300 (Zinc Oxide (and) Caprylic/Capric Triglyceride (and) Polyhdroxystearic Acid)[1]	0	0	5.00	10.00	15.00
Crodamol™ EO (Ethyl Oleate)[1]	18.00	18.00	18.00	18.00	18.00
Cromollient™ DP3A (Di-PPG-3 Myristyl Ether Adipate)[1]	1.80	1.80	1.80	1.80	1.80
Span 120 (Sorbitan Isostearate)[1]	1.80	1.80	1.80	1.80	1.80
Crodamol GTCC (Caprylic Capric Triglyceride)[1]	6.40	6.40	6.40	6.40	6.40
Cithrol PGTL (Tri(polyglyceryl-3/Lauryl) Hydrogenated Trilinoleate)[1]	3.00	3.00	3.00	3.00	3.00
Dermosoft® GMCY (Glycyl Caprylate)[3]	1.00	1.00	1.00	1.00	1.00
NG Shea Unsaponifiable (Butyrospermum Parkii Butter)[1]	1.00	1.00	1.00	1.00	1.00
Phase B					
Water Deionised (Aqua)	To 100	To 100	To 100	To 100	To 100
Pricerine™ 9091 (Glycerine)[1]	3.00	3.00	3.00	3.00	3.00
Magnesium Sulphate Heptahydrate	0.70	0.70	0.70	0.70	0.70

Suppliers: **1: Croda** 2. Jeen International Coporation 3. Dr Straetmans Developed in US (please check as ingredient availability can vary by region)

Table 4: Formulation used to determine how to achieve UVA claims (Formulation Reference No: 360-03)

The results for this formulation are shown in Table 5. Formulation A which uses Avobenzone (BMDM) at 3% comfortably achieves the UVA requirements for Europe and indicates it is possible to include a lower proportion of Avobenzone (BMDM). Formulations B, C and D which include Solaveil CZ show that a ratio of 3:2 of Solaveil CZ and **Solaveil HTP1** is required to meet the European and the proposed US requirements for UVA. As Solaveil CZ-300 is a 60% dispersion of Zinc Oxide, this equates to approximately a 1:1 ratio of ZnO to TiO_2.

It should be noted however that UVA (and indeed SPF) efficacy is highly formulation dependant. It has already been shown, in Table 2 that it is not always necessary to include a UVA filter, but these guidelines serve as an indication of what combinations may be required to comfortably achieve UVA requirements that do not rely so much on formulation variability.

Formulation	A Avobenzone	B Solaveil CZ-300	C Solaveil CZ-300	D Solaveil CZ-300
Ratio (UVA filter: **Solaveil HTP1**)	1 Avobenzone: 5 Solaveil HTP1	1 Solaveil CZ: 3 Solaveil HTP1	2 Solaveil CZ: 3 Solaveil HTP1	3 Solaveil CZ: 2 Solaveil HTP1
Percentage of UVA Filter	3%	5%	10%	15%
Percentage of **Solaveil HTP1**	15%	15%	15%	10%
In Vitro SPF	33	37	35	31
Labelled SPF	30	30	30	30
In Vitro UVA PF (ISO 24443)	12.16	8.41	8.82	10.01
Critical Wavelength	380	377	376	375
European UVA Requirement	Yes	No	No	Yes
UVA1/UV	0.87	Not tested	Not tested	0.75
FDA Broadspectrum	Yes	No	No	Yes
PA prediction (based on *In Vitro* UVA PF)	PA +++	PA +++	PA +++	PA +++

Table 5: UVA results of filter combinations to achieve UVA claims

Solaveil Harmony Demonstration Prototype		CH0105
Ingredient/INCI Name	**Functionality**	**% w/w**
Part A		
Water Deionised (Aqua)	-	To 100
Pricerine™ 9091 (Glycerin)[1]	Humectant	3.00
Magnesium Sulfate Heptahydrate[3]	Stabilizer	0.70
Phase B		
Crodamol™ EO (Ethyl Oleate)[1]	Emollient	18.00
Solaveil HTP1 (Titanium Dioxide (and) Alumina (and) Stearic Acid)[1]	UV filter	15.00
Crodamol™ GTCC (Caprylic/Capric Triglyceride)[1]	Emollient	6.40
Cithrol PGTL (Tri(Polyglyceryl-3/Lauryl) Hydrogenated Triinoleate)[1]	W/O emulsifier	3.00
Cromollient™ DP3A (Di-PPG-3 Myristyl Ether Adipate)[1]	Rich emollient	1.80
Span™ 120 (Sorbitan Isostearate)[1]	Dispersing agent	1.80
Dermosoft® GMCY (Glyceryl Caprylate)[4]	Antimicrobial booster	1.00
NG Shea unsaponifiable (Butyrospermum Parkii (Shea Butter) Unsaponifiables)[2]	Calming, moisturising, nourishing & protective active	1.00

Suppliers: **1: Croda 2: Sederma** 3: Sigma Aldrich 4: Dr. Straetmans GmbH

Procedure:

Combine Part B, excluding **Solaveil HTP1** and Dermosoft GMCY, and use minimal heat to make homogenous. Leave to cool, and once Part B is below 40 °C add **Solaveil HTP1** using Silverson homogenizer at a high shear (8000-10000 rpms) for 2-3 mins and then add Dermosoft GMCY and stir until homogenous. Combine Part A and stir until homogenous. Slowly add Part A to Part B using Silverson homogenizer at 10,000rpm for 4-5 minutes per 200g batch size. Stir slowly until homogenous.

Appearance: White to off-white cream	
pH: NA	Viscosity: 33,000 cps ± 10% (T-C spindle, 10rpm, 1 min.)
Stability: 1 month at 50 ºC, 12 weeks at RT, 40 ºC, 45 ºC, and 5 x - 24 hour freeze-thaw cycles	
In vivo SPF: (ISO 24444:2012, 3 subjects, Eurofins CRL Cosmetics Inc., USA): 27	
In vitro: Critical Wavelength (FDA 2011, Croda, US): 377nm	
In vivo: UVA PF (ISO 24443:2012): 7.2 (sd = 0.23)	
UVA I/UV ratio: 0.69	

Reference: RS360-02-85

Dermasoft GMCY is a registered trademark of Dr. Straetmans GmbH

This formulation was developed in North America. Contact your local sales representative with enquiries as ingredient availability can vary by region.

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Perfectly Balanced

Solaveil™ HTP1

Balancing an effective particle size with appealing aesthetics, our latest innovation brings perfect harmony to your skin protection products. **Solaveil HTP1** is a sunscreen grade Titanium Dioxide (TiO_2) powder and is our first product available from the **Solaveil Harmony** range.

Solaveil HTP1 has been developed using Croda's patent protected technology. Its particle size has been carefully balanced to provide minimal whitening on skin, whilst also being large enough to be suitable for natural and COSMOS approved formulations.

This mineral sunscreen active is approved worldwide and is inherently mild and safe, making it ideal for products for babies and sensitive skin.

- Balanced particle size TiO_2
- Minimal whitening on skin
- Large enough for natural formulations
- Approved worldwide
- Mild and safe

Applications: Skin care, sun care, colour cosmetics

Physical Form: White powder
Product Category: Metal Oxide
INCI Name: Titanium Dioxide (and) Stearic Acid (and) Alumina
Use levels: 1-25%
Product Datasheet: DS-2504

Solaveil™ physical shield

CRODA

Fundamental Properties

Mineral Source

Solaveils, including the new **Solaveil Harmony (Solaveil HTP1)**, are comprised of "nature identical minerals" obtained by transformation of inorganic compounds mined from the earth and then processed to obtain clean and safe UV filters. Organic (chemical) sunscreens are fossil derived in origin and are entirely synthetic chemical compounds, in contrast to inorganic sunscreens, like Solaveil, which are not chemically synthesised, but instead are modified by applying coatings to improve performance and formulation.

Broad Spectrum Protection

Organic sunscreens work by absorption of UV light. Mineral inorganic sunscreens, by contrast, attenuate UV by two mechanisms: absorption and scattering. Because of these different mechanisms, mineral sunscreens attenuate UV over a broader wavelength range, and this is one of the key advantages of these materials over organic sunscreens. The UV/Visible absorption spectrum of **Solaveil Harmony (Solaveil HTP1)** is shown in Figure 2. This has been compared with Croda's transparent grade of TiO_2 (Solaveil Clarus), against its larger particle size TiO_2 (Solaveil SpeXtra). As you can see **Solaveil Harmony** falls in between Solaveil Clarus and Solaveil SpeXtra.

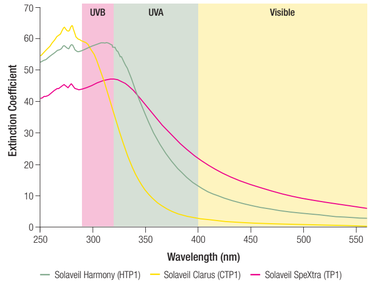

Figure 1: UV/Visible Spectrum of Solaveil Harmony (HTP1)

Long Lasting UV Protection

Whereas some organic filters can break down in the presence of UV light, inorganic sunscreens such as **Solaveil Harmony** are photostable and will not degrade or oxidise, maintaining SPF for as long as the particles remain on the skin.

Mild and Safe

TiO_2 is well-known as a safe material, extensive safety reviews have been conducted by the US Food & Drug Administration (FDA)[1] and the EU Scientific Committee for Consumer Safety (SCCS)[2] and these reviews have concluded that whatever its size (nanomaterial or not) TiO_2 does not penetrate the skin (the FDA classify them as GRASE (Generally Recognised as Safe & Effective Category 1). TiO_2 also has a very low potential for skin irritation, making **Solaveil Harmony** a great choice for formulators working on products for any skin type including babies, children and sensitive skin.

Particle Size

Solaveil Harmony has been optimised to give a particle size greater than 100nm. Measurement of particle sizes in the sub-micron range is problematical and results can vary enormously depending upon the technique, which should therefore always be quoted alongside any measurements. For measurement of particle size of inorganic sunscreens, we have found that the most reliable techniques are centrifuge methods as they provide better resolution for smaller sized particles. We currently use the X-Ray Disc Centrifuge (XRDC) as our preferred measurement technique, but also measure particle size by Dynamic Light Scattering (DLS). Furthermore, mean particle size can be quoted by volume (mass fraction distribution), or by number distribution.

Measurement of mean particle size of **Solaveil Harmony (Solaveil HTP1)** by XRDC by volume gives a result of 160nm. The result by number distribution, and particle size measurements by DLS, are available from Croda on request.

Table 1 has been provided as a summary to show how the particle size of Solaveil HTP1 relates to the definition of "nano" according to Registration, Evaluation, Authorisation and Restriction of Chemicals (REACh)[3] and Cosmetics Europe[4]. For REACh 50% or more of the particles in the number size distribution should have one or more external dimensions in the size range 1nm-100nm to be considered "nano". For Cosmetics Europe a threshold of 10% of the mass fraction (volume) of a material should be used in order to determine whether a given material should be considered as a nanomaterial or not. Based on both the REACh definition of "nano" and the Cosmetics Europe interpretation of "nano", **Solaveil HTP1** would be considered to be "non-nano".

X-ray Disc Centrifuge		Dynamic Light Scattering	
<10% by Volume (1-100nm)	<50% by Number (1-100nm)	<10% by Volume (1-100nm)	<50% by Number (1-100nm)
"non-nano"	"non-nano"	"non-nano"	"non-nano"

Table 1: X-Ray Disc Centrifuge and Dynamic Light Scattering particle size summary for Solaveil HTP1

Regulatory

TiO_2 is globally approved for use in sunscreen formulations. In most countries, the maximum allowed concentration in the final product formulation is 25% w/w active TiO_2; however there is some variation in controls and the appropriate country regulations should be consulted in order to ensure compliance for the final product formulation.

Solaveil Harmony (Solaveil HTP1) is derived from natural sources (mineral origin) and is compliant with ISO 16128 (natural derived mineral) and is approved by COSMOS.

1 https://www.fda.gov/NewsEvents/Newsroom/PressAnnouncements/ucm631736.htm
2 https://ec.europa.eu/health/scientific_committees/consumer_safety/docs/sccs_o_136.pdf
https://ec.europa.eu/health/sites/health/files/scientific_committees/consumer_safety/docs/sccs_o_206.pdf
https://ec.europa.eu/health/scientific_committees/consumer_safety/docs/sccs_o_103.pdf
3 https://ec.europa.eu/environment/chemicals/nanotech/reach-clp/index_en.htm
4 https://docplayer.net/54947113-Cosmetics-europe-nano-guidance-package.html

SPF and UVA Efficacy

Solaveil Harmony (Solaveil HTP1) has been compared to Solaveil SpeXtra and Solaveil Clarus on the basis of three key parameters: SPF efficacy, UVA efficacy and whitening. In these evaluations Solaveil was included at 7.5% TiO_2.

The data show that, in general, all UV filters give similar SPF performance. As is well known, the SPF of a sunscreen product depends a great deal on the formulation, so it is not possible to give definitive rules to say that a certain percentage of an active ingredient will give a certain SPF, however based on the results obtained from this study we can derive the following guideline: every 1% TiO_2 provides approximately 2 SPF units. See Table 2 for full SPF data.

In terms of UVA performance, then there are differences between the UV filter performance. UVA protection levels for Solaveil Clarus (Solaveil CTP1) are low, which is to be expected as it is optimised to be a UVB filter with very high transparency and therefore it has low attenuation in the UVA region, see Figure 1. **Solaveil Harmony (Solaveil HTP1)** and Solaveil SpeXtra (Solaveil XTP1) both give improved UVA performance compared to Solaveil Clarus. They both exceed the current FDA requirements for UVA which specify a critical wavelength of at least 370nm. **Solaveil Harmony (Solaveil HTP1)** has a critical wavelength of 379nm and Solaveil SpeXtra has a critical wavelength of 380nm.

To determine absolute UVA efficacy of each of these products the UVA Protection Factor (UVA PF) was measured according to the ISO 24443:2012 method. In Europe the requirement for sunscreens is that the UVA PF should be at least 1/3 of the labelled SPF, so that the ratio of labelled SPF/UVA PF should be less than or equal to 3.

The results for UVA PF are also shown in Table 2. Solaveil SpeXtra (Solaveil XTP1) gives a slightly higher UVA PF than **Solaveil Harmony** (Solaveil HTP1). Again, this result is to be expected when considering the relative levels of UVA attenuation of each product, shown in Figure 1. For both Solaveil SpeXtra (Solaveil XTP1) and **Solaveil Harmony** (**Solaveil HTP1**), the formulations achieve the European requirements for UVA as the UVA PF is at least one third of the SPF. Please note however that for **Solaveil HTP1** achieving the one third UVA PF to SPF is formulation dependant.

		In-vitro Results
Solaveil Clarus (Solaveil CTP1)	SPF	16
	STD	2
	Critical Wavelength (nm)	365
	UVA/UVB ratio	0.40
	UVA PF (ISO 24443:2012)	Not measured
Solaveil Harmony (Solaveil HTP1)	SPF	14
	STD	2.1
	Critical Wavelength (nm)	379
	UVA/UVB ratio	0.65
	UVA PF (ISO 24443:2012)	6.19
Solaveil SpeXtra (Solaveil XTP1)	SPF	15
	STD	1.5
	Critical Wavelength	380
	UVA/UVB ratio	0.73
	UVA PF (ISO 24443:2012)	7.71

Table 2: In-vitro SPF and UVA results for formulations based on Solaveil Clarus, Solaveil Harmony and Solaveil SpeXtra as the sole active

Appearance on Skin

In addition to the UVB attenuation, and enhanced UVA performance offered by **Solaveil Harmony**, it also offers moderated whitening on skin. A draw down whitening test was conducted to compare the whitening of **Solaveil Harmony (Solaveil HTP1)** to the other Solaveil grades at 7.5% TiO_2. A small quantity of the formulation was pipetted on the top of a black and white contrast card. A 'drawn down' was conducted using a number 2 'K' bar with the application of a small amount of even pressure to get an even film. The sample was then left to dry for 15 minutes, after which photographs were taken, as shown in Image 1.

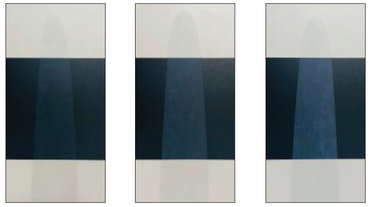

Image 1: Visual whitening comparisons of Solaveil Clarus, Solaveil Harmony and Solaveil SpeXtra on black and white contrast card

Evaluations were also conducted *in-vivo* on skin using the same formulations at 7.5% TiO_2. 0.05g of sample was applied to the subjects. After application the subjects were instructed to rub in the sample 40 times, the skin was left to dry for 5 minutes before images were taken, see Image 2. The results demonstrate that **Solaveil Harmony (HTP1)** shows a level of whitening intermediate between Solaveil Clarus and Solaveil SpeXtra.

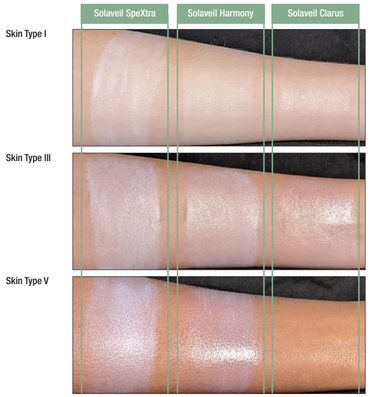

Image 2: Visual whitening comparisons of Solaveil Clarus, Solaveil Harmony and Solaveil SpeXtra on various skin phototypes

Formulating Guidelines

Solaveil Harmony has been designed to be compatible with a wide range of different oil phase ingredients and therefore it can be used in combination with many different emollients. **Solaveil HTP1** powder should be dispersed into a suitable carrier oil prior to formulation. Croda recommends Crodamol™ GTCC, Crodamol ISIS, Crodamol AB, Pripure™ 3759, Crodamol GTIS and Crodamol GTEH/Arlamol HD (50:50) blend. **Solaveil HTP1** produces low viscosity premixes compared to Solaveil CTP1 and Solaveil XTP1 due to a favourable balance of particle size, surface area and coating. The viscosity of TiO_2 and oil premixes for the three Solaveil TiO_2 powders is shown in Table 3. In this experiment the TiO_2 powder was added to the oil at a ratio of 35:65 powder to oil and mixed using a Silverson homogeniser at 4000 rpm for 1 minute. Viscosity measurements were then taken using a RV-05 spindle at 10 rpm for 1 minute.

	Crodamol AB	Crodamol GTCC	Crodamol GTIS	Crodamol ISIS	Pripure 3759	Crodamol GTEH & Arlamol HD (50:50)
Neat Oil	120 cps	200 cps	400 cps	200 cps	200 cps	160 cps
Solaveil CTP1	10400 cps	17000 cps	9000 cps	5200 cps	1200 cps	360 cps
Solaveil XTP1	200 cps	600 cps	5200 cps	1000 cps	400 cps	200 cps
Solaveil HTP1	200 cps	400 cps	2400 cps	600 cps	200 cps	160 cps

Table 3: Viscosity measurements of oil and oil TiO_2 premixes

Solids Loading

With **Solaveil HTP1** a formulator can easily achieve solid loadings up to 15% for oil-in-water and water-in-oil emulsions; anhydrous systems can more easily tolerate higher loadings. It is important to adjust the oil load to assist in dispersing the powder at higher inclusion levels. A ratio of 35:65 of powder to oil has been found to be optimal.

Dispersants

Dispersants can be added to a formulation to aid the dispersion of **Solaveil HTP1** and this is especially useful at high solid loadings. Croda recommends Span 120 at 1.5-2.0% and Cromollient™ DP3A at 1.5-2.0% supported by Crodamol GTTC as a carrier oil.

Homogenisation

When adding **Solaveil HPT1** to the premix oil phase homogenisation is recommended. A Silverson mixer can be used for 3 minutes at 8,000-10,000 rpm or an Ultra Turrax for 10 minutes at 10,000 rpm. Following emulsification homogenisation using Silverson or Ultra Turrax for 3 minutes at 10,000 rpm is recommended. Advised time is for batch sizes of 100 to 200g. Larger batches will require extra time for complete homogenisation.

Heating

Formulation development work has shown that heating is not necessary, **Solaveil HPT1** can be used in cold process systems. It is our general recommendation for Solaveils to avoid heating at high temperatures (80ºC) for prolonged periods as extensive studies have not been conducted under these conditions.

Emulsion Formation

Solaveil HPT1 should be dispersed into a carrier oil and then incorporated into the oil phase of the emulsion. Emulsions have been formulated with a wide variety of water-in-oil and oil-in-water formulations which show good initial compatibility and SPF/UVA efficacy in line with expectations, full stability testing of these systems is ongoing and updates will be made upon completion.

Formulating to Achieve UVA Claims

According to both the FDA and EU regulations sunscreens must provide UVA protection in addition to UVB protection. Used as a single active ingredient, **Solaveil Harmony (Solaveil HTP1)** meets current FDA guidelines for UVA protection (critical wavelength) and in most cases also meets the European requirements for UVA protection of a UVA PF that is at least one third of the labelled SPF (see Table 2).

However, as UVA protection levels are formulation dependant it may be necessary to combine **Solaveil Harmony (Solaveil HTP1)** with UVA filters in order to comfortably achieve the European UVA requirements, and the new proposed FDA guidelines for UVA. In February 2019 the FDA proposed a new criteria for UVA. As well as achieving a critical wavelength of greater than or equal to 370nm, sunscreens must also achieve a UVA1/UV ratio of greater than or equal to 0.70. The levels of UVA protection that can be achieved are low (0.20-0.39), medium (0.40-0.59), high (0.70-0.95) and highest (>0.95).

In order to determine the levels of UVA filters that can be used to achieve the EU and US requirements for UVA a formulation study was conducted. **Solaveil HTP1** was combined with a two of the most commonly used UVA filters. For the organic UVA filter Avobenzone/Butyl Methoxydibenzylmethane (BMDM) was chosen. For the inorganic UVA filters, the Zinc Oxide based mineral filter, Solaveil CZ-300 was chosen. The formulation details are shown in Table 4.