LONG LASTING SUNSCREEN, SPF=8

SUGGESTED FORMULATION Ref. 998PBE

COMPOSITION:

PHASE A:	PARSOL MCX [1]	6,00 %
	INCI: Octyl Methoxycinnamate	
	ONDINA 15 [2]	5.00 %
	INCI: Paraffinum Liquidum	
	IMEX IPP90 [3]	4,00 %
	INCI: Isopropyl Palmitate	
	CETIOL 868 [4]	3,00 %
	INCI: Octyl Stearate	
	EUMULGIN B1 [5]	2,50 %
	INCI: Ceteareth-12	
	EUMULGIN B2 [6]	2,50 %
	INCI: Ceteareth-20	
	ABIL 350 [7]	2,00 %
	INCI: Dimethicone	
	IMEX STEARIC ACID CF [8]	2,00 %
	INCI: Stearic Acid	
	PARSOL 1789 [9]	1,50 %
	INCI: Butyl Methoxydibenzoylmethane	
	CHOLESTEROL NF [10]	1,20 %
	INCI: Cholesterol	
PHASE B:	DEMINERALISED WATER	66,00 %
	INCI: Aqua	
	IMEX GLYCERIN USP [11]	3,00 %
	INCI: Glycerin	
	PHENONIP [12]	0,70 %
	INCI: Phenoxyethanol, Methylparaben, Ethylparaben, Propylparaben, Isobutylparaben, Butylparaben	
	TRIETHANOLAMINE [13]	0,20 %
	INCI: Triethanolamine	

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d,l-PANTHENOL [14] INCI : Panthenol	0,20 %
ALLANTOIN [15] INCI: Allantoin	0,10 %
NIPAGUARD DCB [16] INCI : Phenoxyethanol, Methyldibromoglutaro-nitril	0,05 %
TRISODIUM EDTA INCI: Trisodium EDTA	0,05 %

SUPPLIERS

[1] ROCHE VITAMINS & FINE CHEMICALS
[2] SHELL
[3] INTERMED
[4] COGNIS
[5] COGNIS
[6] COGNIS
[7] GOLDSCHMIDT SKW
[8] INTERMED
[9] ROCHE VITAMINS & FINE CHEMICALS
[10] DISHMAN NETHERLANDS
[11] INTERMED
[12] NIPA LABORATORIES
[13] BASF
[14] ROCHE VITAMINS & FINE CHEMICALS
[15] CLARIANT
[16] NIPA LABORATORIES

MANUFACTURING PROCEDURE

Phase A (the oil phase) is made by heating to 70ºC while mixing gently. The temperature is maintained at 70°C. The oil phase is cloudy due to the presence of self-assemblies of the emulsifiers (ceteareth-12, ceteareth-20 & cholesterol). The liquid crystalline structure is formed at elevated temperature, and is existent up to 120°C; above 120°C (the clarification point) the oil phase becomes clear.
The liquid crystalline phase can be clearly observed using a microscope equipped with a polarisation filter.

Phase B is made by mixing the ingredients except panthenol and allantoin (products are sensitive to excessive heating) and heating to 90°C until the preservatives have been dissolved. Phase B is cooled to 70°C and panthenol & allantoin are added. The mixture is stirred until a clear liquid mixture is obtained while maintaining the temperature at 70°C.

Trisodium EDTA is an essential ingredient as the use of butyl methoxydibenzoylmethane in the formulation may result in severe discoloration due to complex formation with e.g. Fe^{2+}/Fe^{3+} ions or other transition metal ions.

Phase A is added to phase B at 70°C while stirring gently with an anchor mixer. A (low viscous) pre-emulsion is formed immediately. After 5 minutes the pre-emulsion is homogenized during 45 seconds at 1500 rpm using a Silverson mixer. Under laboratory conditions a higher velocity may be used for homogenisation.

After homogenisation the emulsion is stirred with an anchor mixer while cooling the emulsion at a gradient if 1°C/minute. Eventually re-homogenisation at 40ºC may be useful if the particle size and particle size distribution does not yet meet the requirements ($<d> = 20 \pm 5$ µm). The viscosity gradually builds up in time. Optionally a fragrance of choice may be applied.

The emulsion is further cooled to room temperature and allowed to age during 24 hours. Upon ageing the viscosity markedly increases to obtain a soft cream. The cream is transferred to as suitable container.

An interesting alternative procedure is to dissolve the emulsifier combination (ceteareth-12 & ceteareth-20) in the water phase while heating to 90°C, in the presence of cholesterol. A bluish opaque, viscous product is obtained, the colour being characteristic for the presence of a liquid crystalline phase. In this way the liquid crystalline phase generated is more abundant. However, the final emulsion is significantly more sensitive to high shear and electrolytes. Also the sensorial properties are different, more appreciated by the test panel.

The remainder of the oil phase is subsequently emulsified and the emulsion is stirred with an anchor mixer at low velocity while cooling to room temperature with a small temperature gradient. When (neutralised) stearic acid is used, in principle no hydrocolloid or long chain fatty alcohol is required for stabilisation. In case of stability problems the use of xanthan gum or behenyl alcohol is suggested; these ingredients are well-known to stabilise shear-sensitive liquid crystalline matrices.

DESCRIPTION AND SUGGESTIONS FOR MODIFICATION

All amphiphilic molecules, emulsifiers, have surface active properties, and form self-assembles (micelles) in a suitable solvent or solvent mixture. These micelles may be spherical or rod-like, but in a number many cases the molecules may position themselves in a double layer. This double layer is also identified as a nematic liquid crystalline phase. Its presence can be demonstrated using optical light microscopy using polarised light. Cholesterol is build into the LCM as a spacer enabling to monitor its porosity. The emulsifier combination also greatly determines the rheological properties of the cream.

The cream has been formulated to provide long term protection to the user to the undesired effects of UV-B radiation using two UV filters octyl methoxycinnamate and butyl methoxydibenzoylmethane. According to EC Legislation octyl methoxycinnamate may be used to a maximum of 10%, while butyl methoxydibenzoylmethane may be used to a maximum of 5%. The SPF value has *in vitro* been determined as:

8±1	residence time: 30 minutes
6±1	residence time: 2 hours
5±1	residence time: 5 hours

Other UV filters may be applied as well, although no SPF data are available. In addition, the carrier system of emollients may be adjusted, although it should be noted that the solubility butyl methoxydibenzoylmethane is limited. Especially polar lids such as $C_{12\text{-}15}$ alkyl benzoate, octyl palmitate or isopropyl isostearate are suitable solvents for this filter.

The rheology of the emulsion may be modified in terms of increasing/decreasing viscosity, thixotropy and or elasticity. The usual rheological additives are applicable, such as fatty alcohols, glycerol monoesters, cellulosics, natural gums and synthetic rheological additives such as carbomers, acrylates and acrylate copolymers.

SENSORIAL EVALUATION AND STABILITY TESTING

The obtained soft cream emulsion is glossy and white and is pleasant upon application to the skin. The emulsion spreads easily and is smooth on application. The emulsion does not quickly denature on the skin. A modest residual skin feel remains due to the time required for the liquid crystalline matrix to be absorbed by the naturally occurring skin lipids.

The emulsion was tested for three months at 40ºC for stability, and was subjected to a freeze-thaw test. The emulsion did not show phase separation and the rheological parameters remained unchanged. Quantitative rheology showed that the emulsion is stable for three years at ambient temperature.

A fragrance may optionally be used as the emulsion has a gentle, not disagreeable, own odour. As oxidation sensitive products are absent there is no need for the use of an anti-oxidant. However, it was shown that the emulsion accepts vitamin E (tocopheryl acetate; 0,2-1,0%), enabling to also contribute to the cosmetic performance of the cream for sun care application: tocopheryl acetate is a well-known and appreciated radical scavenger. Other radical scavengers may be used as well (e.g. rosmarinic acid, β-glucan).

The microbiological status of the cream was tested using a plate count. The emulsion was also subjected to a challenge test using Pseudomonas aeruginosa, Staphylococcus aureus, Candida albicans and Aspergillus niger according to the protocol of the EP 1997 at an initial inoculum level of 10^6-10^7. It was observed that after

2 days the number of colony forming units was < 10 CFU/g for 3 species. After 7 days also Aspergillus niger was reduced to < 10 CFU/g. The results are considered satisfactory.