

## Skin Lighteners or Bleaches

Some knowledge of the physiology and biochemistry of skin colour and particularly of the pigmentation processes is essential for an appreciation of the mode of action of skin lightening agents. Readers are referred to Chapter 1 and to several authors providing excellent and detailed reviews of this subject<sup>1-8</sup> but a brief summary follows of our current understanding of the processes involved.

### Colour of the Skin

Three main factors combine to give skin its colour. The dermis and epidermal cells provide a background natural yellowish-white colour, the dominance of which depends to some degree on skin thickness. The superficial blood vessels of the skin contribute a red to blue tone, the intensity depending upon the number and state of dilation of the blood vessels and their nearness to the surface and the colour upon the degree of oxygenation of the blood. By far the most important contribution, however, is that of the pigments carotene and, most important of all, the brown to black melanins which are principally responsible for racial colour differences.

Melanin is synthesized in dendritic cells known as melanocytes which are normally found in the epidermal basal layer. Within the melanocytes melanin is bound to a protein matrix to form melanosomes. The melanocytes transfer their melanosomes to surrounding keratinocytes and, having lost their melanin, these cells presumably migrate upwards through the epidermis.

The control of melanin production is due both to the direct stimulatory effect of ultraviolet light and to a hormone, the melanocyte stimulatory hormone (MSH), secreted by the anterior pituitary gland. Oestrogens also exert a localized effect which is especially evident during pregnancy.

It seems extraordinary that it was only in the late 1960s that we began to understand why Negroid skin was black and Caucasoid white. The number of melanocytes is similar in both. Black skin is produced by increased melanocyte activity associated with the production of melanosomes which are larger than those in white skin. Negroid melanosomes are usually disposed individually in keratinocytes whereas those in Caucasoids are usually complexed. Furthermore, Negro skin usually shows melanin granules as high as the horny layer whereas in Europeans melanin is seldom detectable above the epidermal basal layer, owing, it is suggested, to its chemical reduction to a leuco-base which may be reoxidized by exposure to sunlight (immediate pre-tanning). For an excellent review of this subject see Hunter.<sup>9</sup>

Roberts,<sup>9</sup> who studied the geographical and racial distribution of human skin pigmentation, found a close correlation with geographical factors, suggesting a very strong adaptive role. He concluded that protection against ultraviolet radiation in areas where this is intense, and increased synthesis of vitamin D where there is minimal ultraviolet radiation, provide the two most important selective roles. The protective function of melanin also seems to be two-fold. Firstly, in the short term it protects the deeper layers of the dermis against immediate damage by ultraviolet radiation and secondly, in the long term it affords protection against cancer.

Weiner,<sup>10</sup> Gibson,<sup>11</sup> Yuasa *et al.*<sup>12</sup> and Curry<sup>13</sup> describe instruments and procedures for measuring skin colour.

### Chemistry of Melanin

The metabolic pathways leading to the production of melanin have been described by Riley.<sup>14</sup> The melanins are quinonoid polymers of somewhat uncertain structure, there being two main subdivisions: phaeomelanins, yellowish and reddish-brown pigments containing sulphur; and eumelanins, black or brown insoluble pigments derived from the polymerization of tyrosine oxidation products. Riley surveys the evidence that two types of oxidation are involved, namely oxygen addition to monophenols (cresolase activity) and dehydrogenation of diphenols (catecholase activity). Both processes form highly reactive intermediate quinones which may be important in cell metabolism.

A simplistic representation of the sequence of reactions believed to be involved in the conversion of tyrosine to melanin is given in Chapter 23 and the subject is discussed more fully by Mason,<sup>15</sup> Lerner and Fitzpatrick,<sup>16</sup> Lorincz,<sup>2</sup> Fitzpatrick, Brunet and Kukita,<sup>17</sup> Nicolaus and Piatelli<sup>18</sup> and Seiji, Bileck and Fitzpatrick<sup>19</sup>—not forgetting the early pioneer work of Raper and his co-workers.<sup>20</sup>

### Mechanism of Depigmentation

The processes involved in the production and transfer of pigment granules can be interfered with and Bleehen<sup>21</sup> has summarized the possible modes of action of compounds which may do this. The compounds may:

- selectively destroy the melanocytes;
- inhibit the formation of melanosomes and alter their structure;
- inhibit the biosynthesis of tyrosinase;
- inhibit the formation of melanin;
- interfere with the transfer of melanosomes;
- have a chemical effect on melanin or enhance the degradation of melanosomes in keratinocytes.

A number of substituted phenols have been shown to have a specific melanocytotoxic effect: 4-isopropylcatechol etc. (Bleehen *et al.*<sup>22,23</sup>); hydroxyanisole (Riley<sup>24</sup>); hydroquinone monoethyl ether (Frenk and Ott<sup>25</sup>). Hydroquinone produces similar toxic effects on functional melanocytes affecting not only the formation, melanization and degradation of melanosomes but also producing membranous cytoplasmic disruption. Studies on hydroquinone monomethyl



ether and 4-isopropylcatechol<sup>23,24</sup> suggest that these compounds are converted by tyrosinase to highly toxic oxidation products, probably as free radicals, which then initiate a chain reaction of lipid peroxidation with consequent irreversible damage to the lipo-protein membranes of the melanocyte, producing death of the cell.

### *Products for Skin Lightening*

The foregoing suggests two ways for lightening the skin colour by reducing pigmentation: decolorize the melanin already present and/or prevent new melanin from being formed. Present cosmetic practice usually achieves both of these objectives to varying degrees.

Negro skin differs from white skin in having substantial amounts of melanin pigment in the outer horny layer; this may be bleached either by oxidation with, for example, hydrogen peroxide, or more usually chemically reduced to its leuco-form which is colourless by the use of, for example, hydroquinone.

This epidermal layer will slowly be replaced by the natural process of keratinization. The leuco or reduced form of melanin is susceptible to reoxidation by ultraviolet light and therefore the presence of a sunscreening agent in a topical skin lightening preparation is highly desirable. The formation of new melanin in the basal layers of the skin may be inhibited by the application of suitable agents with the result that the newly generated epidermis has a lower pigment content and is therefore lighter in colour.

The scientific evaluation of the effectiveness of skin lightening preparations is complex and readers are referred to the work of Curry.<sup>13</sup>

## **Skin Lightening Agents and Formulations**

### *Opaque Covering Agents*

Suitable make-up products can achieve a remarkable temporary change in the colour of the skin and therefore should be mentioned here. The more effective products may be marketed especially as blemish covers to hide skin colour imperfections, to be used under normal make-up. In a wider sense, normal make-up with a higher than average content of white or pale pigments such as titanium dioxide, zinc oxide, talc, kaolin and bismuth pigments may be used. These products are more fully discussed elsewhere in this volume.

### *Oxidizing Agents*

Creams containing hydrogen peroxide have had limited use in the past as skin bleaches.

A series of patents taken out by Fellows<sup>26</sup> claim a two-solution product, one containing hydroquinone monobenzyl ether, a non-irritant solvent such as octyl acetate and a lanolin absorption base, and the other sodium hypochlorite solution. The two are mixed before use and the mixture is claimed to be stable for about a week.

### *Mercury Compounds*

The first active material, used over many years, was some form of mercury salt. Red mercuric oxide and mercurous chloride have been used, but mercuric chloride and ammoniated mercury give the most effective preparations.

Mercuric chloride is generally used in lotion form but, owing to the generation of hydrochloric acid when the salt reacts with the skin, causes sloughing of the epidermis. Ammoniated mercury— $\text{NH}_2\cdot\text{HgCl}$ —does not cause the same degree of exfoliation of the skin. Ammoniated mercury is thought to inhibit tyrosinase, possibly by replacing copper which is required for tyrosinase action, but there have always been doubts about its toxicity potential, despite claims by manufacturers that complaints were of the order of one per hundred thousand of sales.<sup>27</sup> In the United Kingdom, ammoniated mercury appears in the list of poisons and toxic substances which may be sold only by pharmacists and then with prescribed labels about toxicity. Furthermore, the UK Cosmetic Product Regulations 1978<sup>28</sup> prohibit the use of mercury compounds (other than specific exceptions) in cosmetics. This follows upon a similar prohibition within the EEC where mercury compounds are cited in Annex II, the list of prohibited substances, in the EEC Directive on cosmetics.<sup>29</sup> Following a study of the potential hazards of topically applied mercury compounds, the American Food and Drug Administration issued a statement of policy<sup>30</sup> which ruled that, because of the known hazards of mercury and its questionable effectiveness, there was no justification for the use of mercury in skin-bleaching preparations.

Marzulli and Brown<sup>31</sup> found significant skin penetration of mercury ( $^{203}\text{Hg}$ -labelled) from skin bleach creams containing 1 per cent and 3 per cent ammoniated mercury. All subjects showed symptoms consistent with mercurialism. They concluded that 'although (penetration is) extremely slow, over a long period of time without excretion, it can build up to significant levels'.

Barr, Woodger and Rees<sup>32</sup> presented evidence for renal damage in young healthy African women using mercury-containing skin lightening creams some of which contained 5–10 per cent mercury ammonium chloride.

In a modern context, ammoniated mercury can scarcely be considered to be a suitable material for use in a cosmetic product which will be applied regularly, day by day, despite its existence in pharmacopoeial formulae.

### *Hydroquinone*

The most favoured material for use in skin bleach preparations currently is hydroquinone which was reported by Spencer<sup>33</sup> to be effective at 1½–2 per cent in a vanishing cream in producing a temporary lightening of skin colour. Spencer found that a concentration of 5 per cent was liable to cause redness and burning. No sensitization was found. The bleaching action is slow and only becomes noticeable after application for some weeks or even months. It disappears when use of the product is discontinued. Despite Spencer's findings, a level of 5 per cent has commonly been used and some preparations contained 8–10 per cent. However, the EEC Directive of 1976<sup>29</sup> and the Cosmetic Productions Regulations 1978 (UK)<sup>28</sup> limit the permitted level to 2 per cent with specific labelling requirements.

Hydroquinone has been 'found to be safe and effective (for skin lightening) by a Food and Drug Administration (USA) expert medical panel'.<sup>34</sup> However, the panel recommended that all such products carry a level warning that exposure to sunlight can rapidly reverse the lightening effect.

Oettel<sup>35</sup> noted in 1936 that when hydroquinone was fed to black-haired cats their coats turned grey after 6–8 weeks. However the depigmenting effect of



hydroquinone in man's skin was discovered by chance. It was found that a sunscreen cream containing it was being bought mainly for its skin bleaching effect.<sup>36</sup> Spencer<sup>37</sup> and Fitzpatrick *et al.*<sup>38</sup> have attested to the effectiveness of hydroquinone as a depigmenting agent for human skin.

The mechanism of action of hydroquinone was studied by Denton, Lerner and Fitzpatrick<sup>39</sup> who found that it could completely inhibit the enzymatic oxidation of tyrosine to 3,4-dihydroxyphenylalanine (dopa). Iijima and Watanabe,<sup>40</sup> on the other hand, found that hydroquinone inhibits the histochemical dopa reaction and postulated its direct action on tyrosinase.

Hydroquinone is a white crystalline material, melting point 170-1°C, soluble in water (1:14), freely soluble in alcohol and ether. Solutions become brown on exposure to air as a result of oxidation and must be stabilized. The oxidation is very rapid if the solution is alkaline.

In formulation, the usual stabilizers are sodium sulphite or metabisulphite, often with the addition of a little ascorbic acid. Compositions should preferably be slightly acid (pH 4-6). Manufacturing equipment must be stainless steel or glass-lined to avoid discoloration, and contact with air minimized.

Two formulae published many years ago by the then Goldschmidt Chemical Corporation still provide good basic products:

<i>Hydroquinone bleach cream</i> <sup>41</sup>	(1)
	<i>per cent</i>
Lexemul AS	15.0
Lexate TA	6.0
Stearyl alcohol	3.0
Silicone oil	1.0
Propyl paraben	0.1
Water, deionized	67.8
Propylene glycol	5.0
Hydroquinone	2.0
Sodium metabisulphite	0.05
Ascorbic acid	0.05

<i>Hydroquinone lotion</i> <sup>41</sup>	(2)
	<i>per cent</i>
Lexemul AS	5.0
Mineral oil light N.F.	2.5
Isopropyl myristate	2.5
Cetyl alcohol	1.0
Brij 35	0.9
Deionized water	79.85
Propylene glycol	5.0
Sodium lauryl sulphate 30%	0.9
Sodium metabisulphite	0.15
Ascorbic acid	0.1
Hydroquinone	2.0
Citric acid	0.1

*Procedure:* Add the oil phase to the water phase at 75°-80°C with high shear mixing. When a smooth emulsion has been formed, transfer to low shear

mixing. Cool with stirring and at 55°–50°C add, in order, sodium metabisulphite, ascorbic acid, hydroquinone and citric acid. Perfume at 40°C; the perfume should not react with hydroquinone or bisulphite.

Shevlin<sup>42</sup> published the following formula for a medium viscosity lotion which will function as a sunscreen lotion as well as lightening and evening out skin tone.

<i>Oil-in-water toning lotion for black skin</i> <sup>42</sup>		(3)
		<i>per cent</i>
Veegum K		1.5
Titanium dioxide		0.2
Cetyl alcohol		1.8
Polychol 5		0.6
Glycerin		5.0
Uvinul D50		1.0
Sodium myristyl sulphate		1.0
Hydroquinone		3.0
Perfume		1.0
Sodium metabisulphite		0.2
Aluminium chlorhydroxyallantoinate		0.2
Water	to	100.0

A soft cream with a dry emollience may be formulated as follows:

<i>Skin lightening cream</i> <sup>43</sup>		(4)
		<i>per cent</i>
A	Arlacel 165	8.0
	Crodamol CSP	8.0
	Liquid base CB.3929	4.0
	Crodalan IPL	2.0
	Polychol 15	3.0
	Cetyl alcohol	1.0
	Nipasol M	0.05
B	Propylene glycol	5.0
	Kelzan	2.0
	Nipagin M	0.15
	Deionized water	64.4
C	Sodium metabisulphite	0.1
	Ascorbic acid	0.1
	Hydroquinone	2.0
	Perfume	0.2 or <i>q.s.</i>

*Procedure:* Heat A to 72°C; disperse the Kelzan in B and heat to 70°C. Add A to B with stirring until emulsified. Cool with stirring, adding the sodium metabisulphite, ascorbic acid and then the hydroquinone at 55°–50°C. Add the perfume at about 40°C. Adjust the pH value if necessary to 5.0–5.5 with citric acid.

The composition in example 5 gives added protection against exposure to sunlight



<i>Skin lightening cream</i> <sup>43</sup>		(5)
		<i>per cent</i>
A	Arlacel 165	6.0
	Estol 1461	6.0
	Isopropyl palmitate	2.0
	Ceto-stearyl alcohol	3.0
	Silicone fluid DC.200/350 cS	1.0
	Crodamol CSP	3.0
	Tiona G	0.2
	Nipasol M	0.1
	Aduvex 2211	0.2
B	Propylene glycol	5.0
	Allantoin	0.2
	Empicol LZ powder	0.3
	Deionized water	70.55
C	Sodium metabisulphite	0.15
	Ascorbic acid	0.1
	Hydroquinone	2.0
	Perfume	0.2 or <i>q.s.</i>

*Procedure:* As described for example 4.

In 1974 Kligman took out a patent<sup>44</sup> claiming synergistic compositions for skin depigmentation comprising a mixture of hydroquinone, retinoic acid and a corticosteroid, for example dexamethasone. Subsequently, Kligman and Willis<sup>45</sup> showed that a composition containing vitamin A acid (tretinoin), hydroquinone and a corticosteroid could bring about complete loss of melanin from normal black skin and was highly beneficial in disorders of hyperpigmentation, notably melasma, freckles and excess pigmentation following inflammation. Each of the three components was essential for effectiveness and their relative parts in the depigmentation process were conjectured. Mills and Kligman<sup>46</sup> reported further clinical experiences with the cream shown in example 6. Black and hyperpigmented skin is much more readily lightened than white skin, which makes this composition especially useful for treating hyperpigmentation disorders.

		(6)
		<i>per cent</i>
	Tretinoin	0.1
	Hydroquinone	5.0
	Dexamethasone	0.1
	Hydrophilic ointment USP	to 100.0

Bleehen<sup>21</sup> reported the successful clinical use of a similar composition:

		(7)
		<i>per cent</i>
	Hydroquinone	5.0
	Hydrocortisone B.P.	1.0
	Retinoic acid	0.1
	Butylated hydroxytoluene	0.05
	Polyethylene glycol 300	47.0
	Methylated Spirit 70 o.n.	to 100.0

A series of patents taken out by Fellows<sup>26</sup> for a skin lightening preparation containing hydroquinone monobenzyl ether and an oxidizing agent were mentioned earlier.

#### *Catechol and its Derivatives*

Extensive screening tests by Chavin and Schlesinger<sup>66,67</sup> showed that catechol and some derivatives caused destruction of the pigment cells though with a lesser effect than hydroquinone.

Bleehen *et al.*<sup>22</sup> investigated thirty-three compounds, including catechol and derivatives, and found 4-isopropyl catechol to be a most potent depigmentating agent. However, levels of 3 per cent or more proved to be irritant to the skin and a sensitizer.<sup>21</sup>

4-Tert-butyl catechol may also cause skin depigmentation in man.<sup>54,68</sup>

A patent<sup>69</sup> covers the use of catechol, methyl- and carboxy-substituted catechol in a sunscreensing composition. Another patent<sup>70</sup> claims a depigmenting composition containing a catechol substituted in the 4-position, for example 4-isopropyl catechol.

#### *Ascorbic Acid and its Derivatives*

Ascorbic acid itself has been listed<sup>21</sup> as an active constituent of skin bleaching preparations but its use is normally confined to that of an antioxidant stabilizer in hydroquinone-based products, where it assists in the inhibition of browning of creams.

Rovesti<sup>71</sup> reported on the successful use of creams containing 3 per cent and 5 per cent ascorbyl oleate for bleaching freckles in human skin, resulting also in a marked improvement in the condition of the skin, which became softer and more supple.

Takashima *et al.*<sup>72</sup> reviewed the chemistry of a number of esters of ascorbic acid and studied the stability of a few when incorporated into cosmetic creams. One in particular, the magnesium salt of ascorbic acid-3-phosphate, was shown to have a bleaching effect on pigmentation in human skin and to be useful in clinical practice.

A patent<sup>51</sup> described the use of a composition containing hydroquinone, ascorbyl palmitate and 2,2',4,4'-tetrahydroxybenzophenone for skin bleaching.

#### *Other Depigmenting Materials*

Chavin and Schlesinger<sup>66,67</sup> found that, among other materials, several mercapto-amines were potent depigmenting agents in black goldfish. Two of these, 2-mercaptoethylamine hydrochloride and N-(2-mercaptoethyl)-dimethylamine hydrochloride, were potent depigmenting agents when applied to the skin of black guinea-pigs.<sup>73</sup> However, both are very malodorous and therefore unusable in man. See also Bleehen *et al.*<sup>22</sup> and Frenk.<sup>57</sup>

The use of mercapto-amines has been patented by Scherico<sup>74</sup> and by Marly.<sup>75</sup>

A method for the treatment of hyperpigmentation, described in a US Patent<sup>76</sup> granted to the Schering Corporation, entails the topical application to the affected area 1-4 times daily of a preparation comprising between 0.1 and 10 per cent of either *p*-amino-benzenesulphonic acid or its alkaline or alkaline-earth metal salts. It is claimed that such treatment will result in depigmentation of



Bristol-Myers Co. have taken out a patent<sup>47</sup> for a synergistic skin bleaching composition containing hydroquinone or an ether thereof, a skin irritant exfoliating agent, and an anti-inflammation corticosteroid, for example retinoic acid and fluorometholone, in a vanishing cream base.

In 1945, the Schering Corporation was granted two patents<sup>48,49</sup> for sunscreen preparations based on hydroquinone stabilized with laevo-ascorbic acid in a vanishing cream base. In 1973, Reckitt and Colman Products patented<sup>50</sup> a skin lightening preparation containing hydroquinone in, for example, glycerol; and Unilever Ltd took out a patent<sup>51</sup> for a product containing hydroquinone, ascorbyl palmitate and 2,2',4,4'-tetrahydroxybenzophenone. In 1979, Helena Rubinstein was granted a patent<sup>52</sup> for a composition based on stabilized hydroquinone in a moisturizing base which also contains a sunscreen agent (amyl *p*-dimethylaminobenzoate).

#### *Hydroquinone Monomethyl and Monoethyl Ether*

Hydroquinone monomethyl ether (usually called hydroxyanisole in the clinical literature) has been extensively studied by Riley<sup>24,53</sup> whose work has been usefully reviewed by Hemsworth.<sup>54</sup> Brun<sup>55</sup> reported it to be a more rapid skin depigmenting agent than the monobenzyl ether while Sidi, Bourgeois-Spinasse and Planat<sup>56</sup> found the reverse.

Hydroquinone monoethyl ether is a potent depigmenting agent<sup>54</sup> and its mode of action has been studied by Frenk<sup>57</sup> and Frenk and Ott.<sup>58</sup> Prolonged treatment with the monoethyl ether causes irreversible depigmentation comparable to that observed in man after using the monobenzyl ether.<sup>54</sup> The unpredictability of its long-term effects has discouraged its use as a skin lightener.

Although no secondary effects have apparently been reported from the use of the monomethyl ether, the undesirable effects from both the monoethyl and the monobenzyl ether do not commend its general use.

#### *Hydroquinone Monobenzyl Ether*

The skin depigmenting activity of hydroquinone monobenzyl ether was first reported by Oliver, Schwartz and Warren<sup>59</sup> as an occupational disease in rubber workers. This activity has been confirmed, notably by Denton, Lerner and Fitzpatrick,<sup>39</sup> Dorsey,<sup>60</sup> Sidi, Bourgeois-Spinasse and Planat,<sup>56</sup> Becker and Spencer,<sup>61</sup> and Mosher, Parrish and Fitzpatrick.<sup>62</sup>

Calnan<sup>63</sup> reported findings in South Africa where skin lightening preparations are very widely used; Dogliotti *et al.*<sup>64</sup> found a sharp increase in the number of cases of leuco-melanoderma traceable to skin lightening creams following a formulation change from ammoniated mercury to the monobenzyl ether of hydroquinone (2 per cent), to which salicylic acid (2 per cent) was often added; Bentley-Philips and Bayles<sup>65</sup> stated that hydroquinone is much safer than the monobenzyl ether, which has now been prohibited in South Africa.

The unpredictable results obtained with this compound, its liability to cause dermatitis, sensitization and sometimes irreversible depigmentation, preclude its general use. The EEC Directive 1976<sup>29</sup> and the UK Cosmetic Products Regulations 1978<sup>28</sup> both prohibit the use of hydroquinone monobenzyl ether in cosmetics.

highly localized areas. The patent specification contains several example formulae.

A patent granted to Lever Brothers (USA)<sup>77</sup> claims a composition for simultaneously lightening skin and protecting it from the sun's rays, containing 0.2–10 per cent niacin and 0.1–10 per cent urocanic acid. A similar patent has been granted to Unilever Ltd (UK).<sup>78</sup>

### The Natural Way

Limited skin lightening effects can be achieved with natural materials which have been used for centuries and which are useful for bleaching out fading suntan and freckles. Among the materials used are cucumber juice, lemon and lime juice, buttermilk, crushed strawberries and fresh horseradish. Some interesting preparations have been described by Buchman<sup>79</sup> and Maxwell-Hudson.<sup>80</sup>

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## Face Packs and Masks

### Introduction

The use of face packs by women dates back to early antiquity when some of the earths used in them were credited with almost miraculous healing powers. These preparations are applied to the face in the form of liquids or pastes. They are then allowed to dry or to set with the object of improving the appearance of the skin, by producing a transient tightening effect as well as by cleansing the skin.

Their present vogue may be ascribed to their combined psychological and cleansing effect. The warmth and tightening effect resulting from their application produce the stimulating sensation of a rejuvenated face, while the colloidal and adsorptive clays and earth which are present in some packs will adsorb grease and dirt from the facial skin. When they are eventually removed from the face, skin debris and blackheads may be removed simultaneously.

Such a preparation should possess the following properties:

- (1) It should be a smooth paste without gritty particles and without an 'earthy' or other objectionable odour.
- (2) Applied to the face it should dry out rapidly to form an adherent coating on the skin but this coating should be capable of subsequently being removed either by peeling off the face or by gentle washing without producing any pain.
- (3) It should produce a definite sensation of tightening of the skin after application.
- (4) It should produce a significant cleansing of the skin.
- (5) It must be dermatologically innocuous and non-toxic.

There are five basic systems which give products satisfying the above characteristics. These are based on wax, rubber, vinyl resins, hydrocolloids and earth respectively.

### Wax-based Systems

Wax-based masks generally consist merely of paraffin wax of a suitable melting point, or may be mixtures of wax with the addition of a little petroleum jelly and polar materials such as cetyl and stearyl alcohols. The use of microcrystalline waxes may assist the continuity of a suitable 'wax mask'.

These products are solid at room temperature and for use have to be melted and brushed on hot. When the waxes harden, a sensation of tightness is felt. As the wax film forms a moisture-proof barrier, profuse perspiration is induced which helps to flush dirt and impurities from the follicular openings in the skin surface.

A little rubber latex may be included with the waxes to assist in the ease of removal. Even application can be facilitated by formulating the wax blend so that in the molten state—a few degrees higher than body temperature—the product is a thixotropic semi-solid. This can be achieved by the incorporation of a little organophilic bentonite. The following formula illustrates the use of this material:

	(1) <i>per cent</i>
Microcrystalline wax	13.0
Paraffin wax	60.0
Cetyl alcohol	5.0
Mineral oil	20.0
Bentone 38	1.4
Isopropyl alcohol	0.6

### Rubber-based Systems

Mention must also be made of face packs mainly based on rubber latex. After drying out, these packs form a continuous, elastic and water-impermeable film on the face. By interfering with normal skin respiration, the film causes heat to be retained by the skin with a resulting rise in the temperature and increased blood circulation. Rubber masks are removed from the skin very easily, for example by mere pulling. After the removal, slight plumping of the skin becomes noticeable. This effect, however, is transient and disappears after skin respiration has been restored to normal.

Example 2 illustrates the composition of face packs based on rubber latex.

	(2) <i>per cent</i>
Latex emulsion	25
Sorbitol	5
Methylcellulose (low viscosity)	10
Kaolin	3
Borax	1
Water	56
Preservative	<i>q.s.</i>

### Vinyl-based Systems

Vinyl-based systems are generally based on polyvinyl alcohol or vinyl acetate resin as film-former. The use of polyvinyl alcohol in face packs is illustrated by example 3.



	(3) per cent
Veegum	0.5
Kaolin	0.5
Titanium dioxide	0.3
PVA	12.0
Propylene glycol	8.0
Ethanol	20.0
Water	58.7

*Procedure:* Dissolve the Veegum in the water with rapid agitation and heat to 80°C. Add the kaolin and titanium dioxide. Mix the PVA and propylene glycol together, heat to 80°C, add to the Veegum mixture and homogenize. Cool the mixture to 40°C and very slowly add the alcohol.

The inorganic matter can be adapted to absorb both oleophilic and hydrophilic soil by use of nylon powders coated with titanium dioxide. Formulae quoted by Toida *et al.*<sup>1</sup> include:

	(4) per cent
Polyvinyl alcohol	14.00
Titanium dioxide/nylon 12 (1:7)	3.00
Glycerin	3.50
Preservatives	0.10
Polyoxyethylene sorbitan monolaurate (20 EO)	0.50
Water	to 100.00

### Hydrocolloid-based Systems

Hydrocolloid-based systems can be presented either as high-viscosity sols which after application lose water and form a flexible gel film, or as solid gels which are melted before application to the face. The sensation of tightness is produced by the eventual shrinking of the gel on loss of further moisture. A variety of gums can be used; these include gum tragacanth, gelatin, casein, carrageen gum, sodium carboxymethyl cellulose, acacia and guar gum, as well as polyvinylpyrrolidone and many others. The film may be plasticized by the inclusion of humectants such as glycerol, propylene glycol or sorbitol.

The viscosity of liquid masks based on hydrocolloids can be varied considerably depending on the colloid used and its concentration in the face pack.

Masks based on hydrocolloids are sometimes preferred to masks based on earths and clays because they are easier to apply and because they dry more rapidly. Their cleansing action, however, is somewhat inferior because they do not contain a sufficient amount of solids to adsorb any dirt.

More rapid drying may be obtained by the incorporation of ethyl alcohol as part solvent. This restricts the choice of hydrocolloid somewhat. However, certain grades of methylcellulose, Carbopol 934 (Goodrich Chemical Co.) and polyvinylpyrrolidone are soluble in aqueous alcohol and can be used in alcoholic masks.

Face masks based on hydrocolloid systems may also contain small quantities of finely divided solids which act as opacifiers and sometimes facilitate applica-

tion. Kaolin and bentonite may be used, for example, but in amounts preferably not exceeding 5 per cent since, if too much particulate matter is added, a discontinuous film which may lack mechanical strength will be formed.

The preparation of such face packs usually proceeds along the following lines. The preservative is dissolved in the appropriate amount of water, and humectant is added with stirring. The hydrocolloid is then sprinkled in slowly with continuous agitation to prevent the formation of aggregates which are difficult to disperse, if necessary with heating to assist solution. When dispersion has been completed and the hydrocolloid particles have started to swell, it is usual to reduce the rate of agitation so as to avoid undue aeration and the retention of air bubbles in the viscous solution or gel.

Janistyn<sup>2</sup> gave the following formula for a 'gelanthum' mask containing gelatin and tragacanth:

	(5) per cent
Gum tragacanth (best)	2.2
Glycerin	2.5
Gelatin (white)	2.3
Water	90.5
Zinc oxide	2.5

For a 'gelanthum mask with honey', Janistyn suggested the replacement of about 4.5 parts of the water in the above formula by honey. He also gave a formula for a casein-based mask:

	(6) parts
Casein (best quality)	20.0
Borax (powdered)	0.5
Glycerin	5.0
Water (distilled)	100.0
<i>p</i> -Hydroxybenzoic acid propyl ester	0.1

*Procedure:* Moisten the casein with the glycerin and dissolve in the aqueous borax solution (containing also the preservative) with the aid of heat.

Winter<sup>3</sup> suggested the following composition for a face pack:

	(7) parts
Gelatin	10.00
Water	50.0
Camphor (dissolved in a little alcohol)	0.05
Zinc oxide	3.00
Kaolin	5.00
Titanium dioxide	2.00

*Procedure:* First moisten the gum with glycerin, then add the gelatin and water, raising the temperature. (Solids should be mixed in the hot solution and their dispersion is facilitated by first moistening them with a drop of glycerin/or of surface-active agent.)



The preparations given in examples 6 and 7 are heated before use and applied in a warm state.

The use of alcohol as part solvent is shown in example 8.

	(8) per cent
Carbopol 940	2.0
Water	42.0
Alcohol	50.0
Glycerol	2.0
Di-isopropanolamine	4.0

The use of polyvinylpyrrolidone in face packs is illustrated by a model formula quoted from a technical bulletin<sup>4</sup>:

	(9) per cent
PVP K-15	3.0
Methylcellulose (low viscosity)	9.0
Glycerin	7.5
Water	80.5
Insoluble opacifiers, perfumes, preservative	q.s.

### Earth-based Systems (Argillaceous Masks)

Earth-based systems are often referred to as paste masks. They include clay facial packs and the so-called mud packs and usually contain a high percentage of solids.

These products can either be presented in bulk, or packed in sachets, for mixing with water when required, or they can be presented ready mixed for use. In the latter case it is advisable to presterilize by the use of heat or ethylene oxide and to incorporate a suitable preservative as many of the natural earths are heavily contaminated with micro-organisms.

As the mask dries on the face it hardens and contracts, giving the sensation of mechanical astringency. The presence of absorbent clays such as bentonite produces a genuine cleaning effect, particularly on very greasy skins.

China clay, colloidal kaolin, fuller's earth, bentonite, etc., may be used as the 'argillaceous' material, the choice depending in part on the criteria which it is proposed to apply to the finished product.

If the colour of fuller's earth or bentonite is considered objectionable, this difficulty may be met by blending with kaolin, and/or the addition of zinc oxide or titanium dioxide.

*Bentonite* is a colloidal clay derived from volcanic ash found in certain parts of the United States, which is characterized by its strong affinity for water and its thixotropic properties. Certain bentonites will absorb up to fifteen times their volume of water, this property being greatly increased by the addition of a small quantity of magnesium oxide, or some other substance possessing a similar pH.

The wide variations in the analysis of bentonites reported in the literature<sup>5</sup> stem from the considerable variations in different beds in the Benton formation and even in different strata in the same beds.

The consistency of bentonite gels will vary with concentration and will be considerably influenced by the pH of the gels. According to Griffon,<sup>6</sup> a gel containing 6 per cent bentonite has the consistency of glycerin, while a 20 per cent gel has the consistency of lanolin.

Bentonite gels have been described as soothing to the skin,<sup>7</sup> and have been claimed to be of value in the treatment of eczema, abscesses, sores and wounds.<sup>8</sup> They have been used in a number of dermatological preparations.<sup>9-11</sup>

The nature of kaolin and its purification to a quality suitable for cosmetic purposes are described in Chapter 18. This type of electrolytically purified kaolin is equally suitable for use in face packs because of its qualities of fineness, softness, moisture adsorption and easy spreading.

Hydrocolloids such as the carrageen gums may be added to stabilize the suspension of solids while contributing to the mechanical strength of the dried film. Again, plasticizers such as glycerin may be added. Special attributes may be conferred by adding additional ingredients such as sulphur (see Chapter 9), astringents, bleaching agents, acids, etc.

The following formulae illustrate this type of product:

<i>All-purpose masks</i>	(10) <i>per cent</i>
Kaolin	35.0
Bentonite	5.0
Cetyl alcohol	2.0
Sodium lauryl sulphate	0.1
Glycerin	10.0
Nipagin M	0.1
Perfume	<i>q.s.</i>
Water	to 100.0

	(11) <i>per cent</i>
Glyceryl monostearate	3.0
Lanolin oil	2.0
Sodium lauryl sulphate	2.0
Veegum	8.0
Kaolin	10.0
Propylene glycol	7.0
Titanium dioxide	4.0
Ethanol	6.0
Isopropyl myristate	2.0
Water	56.0

	(12) <i>per cent</i>
Water	78.7
Nipagin M	0.2
Nipagin P	0.1
Titanium dioxide	1.0
Arlacel 83	0.2
Tween 60	0.3
Kaolin	9.5



*Procedure:* The cream emulsion is formed at 80°C, with half the water content. The kaolin is dispersed in the remaining water, heated to 80°C, and added to the emulsion.

Winter<sup>3</sup> gives the following two formulations for face packs for dry and greasy skins respectively:

<i>Face pack for dry skin</i>	(13)
	<i>parts</i>
Kaolin	80.0
Starch	10.0
Cold cream	20.0
Cetyl alcohol	2.0
Hydrophilic oil	5.0
Water, boric water or infusions	<i>q.s.</i>

*Procedure:* Melt the cold cream and cetyl alcohol in warm water. Next add the oil, the powders and then the water or other aqueous matter.

<i>Face pack for greasy skin</i>	(14)
	<i>parts</i>
Kaolin	80.0
Magnesium carbonate	15.0
Starch	5.0
Tragacanth gum (powdered)	1.0
Water	<i>q.s.</i>

A so-called 'oxygenated' face mask, based on kaolin and quoted by Bergwein<sup>12</sup> has the following composition:

	(15)
	<i>parts</i>
Colloidal kaolin	800
Salicylic acid	20
Aluminium lactate	5
Magnesium peroxide	200

'Oxygenated' masks have been recommended for use on oily skin, sallow skin or skin blemishes. However, because their use may cause irritation of sensitive skin, a preliminary test should be carried out on a small area of the skin.

### Anti-wrinkle Preparations

Face packs are usually left on the face for about 10–25 minutes, to allow most of the water to evaporate and the resulting film to contract and harden, after which they are removed.

The so-called anti-wrinkle preparations which appeared on the market in the early 1960s are based on bovine serum albumin, and form an 'invisible' mask over the skin. They are allowed to remain in contact with the facial skin for about 6–8 hours, that is, for the period of time during which they remain effective, and are then removed by washing.

In the manufacture of bovine albumin, sodium citrate is added to fresh blood to prevent its coagulation, and the blood is centrifuged to remove the blood cells. The anticoagulant is then neutralized and the serum is defibrinated and spray dried. The resulting product is light in colour and completely water-soluble; its albumin content is about 80–95 per cent. Bovine albumin is available from several sources in three forms: as a 15 per cent sterile solution containing a suitable preservative and ready for immediate use without dilution; as a 30 per cent solution to be diluted with an equal volume of water prior to use; and also in the form of a freeze-dried powder which before use is reconstituted with water. The smoothing effect which follows the application of these preparations would seem to be a purely physical one entailing the 'filling-in' of facial wrinkles and the formation of a tight occlusive film which stretches the skin. How long this effect lasts will largely depend on how much the facial muscles are used. This has been clearly shown during investigations carried out by Kligman and Papa.<sup>13</sup>

Although bovine serum was the basis of most of the original preparations, there would appear to be no good reason why experimentation should not produce similar results from other forms of albumin such as egg white or even soluble casein.

In fact, a wrinkle-smoothing composition disclosed in an American patent<sup>14</sup> contains proteins obtained from cow's milk whey, namely  $\alpha$ -lactalbumin and some  $\beta$ -lactoglobulin. The film produced with this composition is claimed to be effective for about four hours and can be re-activated by moistening it with a little water. Both the component proteins are undenatured and water-soluble, are present in the aqueous composition in a ratio of 4:1 and may constitute 10–30 per cent by weight of the total composition. Water may be present within a range of 70–90 per cent by weight, together with 2–4 per cent by weight (of the composition) of a non-toxic plasticizer, such as glycerin or propylene glycol, to increase the flexibility of the dried films. A preservative must also be included; it should be both bacteriostatic and fungistatic and must not precipitate the lactalbumin or the lactoglobulin. The pH of the final preparation is maintained within the 5–7 range.

Apart from the ability to re-activate films produced, other advantages claimed for this preparation are that when it is dry it is not shining in appearance, that it does not flake away after drying and that no make-up is required to conceal the film.

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## Face Powders and Make-up

### FACE POWDER

#### Function and Properties

The function of face powder is to impart a smooth finish to the skin, masking minor visible imperfections and any shine due to moisture or grease either from perspiration or from preparations used on the skin. The object appears to be to make the skin look as though it would be pleasant to touch. The degree of opacity of the powder can vary from opaque and matt, as for example a clown's make-up, to almost transparent, which will have a type of shine due to the powder itself. Neither extreme is favoured, but between the limits, the pendulum of fashion will swing from time to time. Whatever the finish, it must possess reasonable lasting properties to avoid the need for frequent re-powdering, that is it must adhere to the skin, and be reasonably resistant to the mixed secretions of the skin. Finally, it should serve as a vehicle for a pleasing odour to be disseminated by intimate contact of perfume-laden particles over a warm and relatively large area.

No single substance possesses all the desired properties—covering power, slip absorbency, adhesiveness and bloom—hence a modern face powder is a blend of several constituents each one chosen for some specific quality. The various properties will now be considered in greater detail together with some of the principal ingredients, arranged according to their various functional contributions to the powder base. These properties are included in the section on face powders mainly for historical reasons. The popularity of face powders has declined considerably in recent years in favour of compact powders, foundation and liquid make-up. However, the materials and principles involved are equally applicable to these products so a careful study of the following sections will benefit the formulator in his or her work on more sophisticated products.

#### Covering Power

Good covering power is a very desirable attribute of face powders, its object being to conceal various defects of the facial skin including scars, blemishes, enlarged pores and excessive shine.

Titanium dioxide, zinc oxide, kaolin and magnesium oxide are the materials used to enhance the covering power of face powders.

#### *Titanium Dioxide*

Titanium dioxide has considerably greater covering power *per se* than zinc oxide, about 1.6 times that of the latter in air and about 2.9 times the latter in



petroleum jelly. On a moist greasy skin, its covering power relative to zinc oxide is probably of the order of 2.5 times. Titanium dioxide is not astringent but is physiologically inert and may be found, in any rare cases of allergy to zinc compounds or in cases of dry skin, preferable to zinc oxide. Its sun-screening properties are, however, inferior to those of zinc oxide.

Difficulties sometimes encountered in blending titanium dioxide with other powder constituents may be overcome by using it in conjunction with zinc oxide.

### Zinc Oxide

Zinc oxide is the other metallic oxide that is frequently employed in face powders to accentuate their covering power. It is also mildly astringent, mildly antiseptic and has soothing properties. Because of the last property it has been used in the therapy of minor skin irritations. It has been considered to give satisfactory covering power in powder formulations at a level of 15–25 per cent.

The measurement of the covering power of a pigment has been a controversial subject for a long time, since by varying the test conditions one can obtain widely different values.

Grady,<sup>1</sup> who investigated the characteristics of zinc oxide when used in face powders, also calculated the covering power of several face powder ingredients from the refractive indices of the pigments and of the various media in which they might be used for cosmetics. The values obtained are listed in Table 18.1. It will be noted that in the table the covering power of zinc oxide in each medium has been arbitrarily designated as 100; in fact, it decreases progressively from 100 to 37 to 21 as zinc oxide is placed in air, water and petrolatum respectively.

Table 18.1 Calculated Covering Power of Pigments

Pigment	Refractive index	Relative covering power		
		In air ( $n = 1.00$ )	In water ( $n = 1.33$ )	In petrolatum ( $n = 1.475$ )
TiO <sub>2</sub>	2.52	166	232	292
Zinc oxide	2.008	100	100	100
Chalk	1.658	55	29	15
Talc	1.589	46	19	6

As well as the medium surrounding the pigment, it is necessary to consider the particle size. A reduction in particle size will obviously allow the material to be spread more thinly and thus give increased physical cover. At the same time, reduction in particle size is, in general, accompanied by increased light scatter, thus increasing the opacity of the powder and hence the optical covering power.

However, there is a limit, and a curve for light transmission for zinc oxide in water, published by Grady (Figure 18.1), shows a sharp increase of transmission (that is, decrease in opacity) below  $0.25 \mu\text{m}$  when the particles have become small in comparison with the wavelength of light.

It should also be borne in mind that the covering power of a face powder will decrease as it absorbs moisture and sebum from the skin. However, the

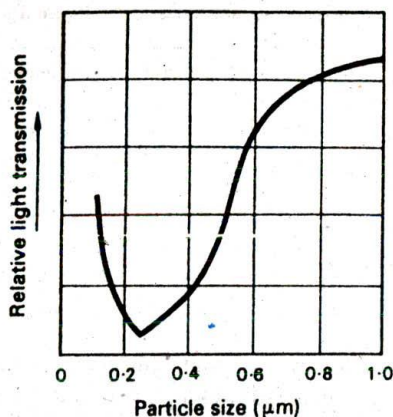


Figure 18.1 Calibration curve relating particle size to relative light transmission<sup>1</sup>

same conditions, pigments with a high refractive index will lose proportionally less opacity than materials of low refractive index, as shown in Table 18.2. This once again emphasizes the desirability of using materials of high refractive index in face powders.

Table 18.2 Relative Covering Power Retained after Wetting Dry Pigment

Pigment	With water (%)	With petrolatum (%)
TiO <sub>2</sub>	51	37
Zinc oxide	37	21
Chalk	20	5
Talc	15	3

Grady also drew attention to the suncreening action of zinc oxide which cuts off the ultraviolet more sharply than any other white pigment used in face powders. He considered, therefore, that zinc oxide and to a lesser degree titanium dioxide should be useful in preventing sunburn. The ultraviolet transmission characteristics of various pigments are listed in Table 18.3.

It is interesting to note that in the experiments carried out by Luckiesh *et al.*<sup>2</sup> on behalf of the US Army Air Force in December 1942 on protective skin coatings for the prevention of sunburn, zinc oxide was found to be of definite value in preventing sunburn whereas titanium dioxide was not found to be a very dependable protective judged on a specimen containing 20 per cent of this substance in petroleum jelly. In addition to titanium dioxide and zinc oxide, some grades of kaolin have a good covering power.

Various published articles have sought to correlate the weight of various face powder constituents with their covering properties or opacities. However, it is



Table 18.3 Ultraviolet Transmission Characteristics of Pigments<sup>1</sup>

Pigment	Per cent transmitted of wavelengths					
	435.8 nm	404.7 nm	365.5 nm	334.2 nm	313.1 nm	302.3 nm
Zinc oxide	46	40	0	0	0	0
TiO <sub>2</sub>	35	32	18	6	0.5	0
China clay	63	61	59	57	55	54
Chalk	87	86	84	82	80	79
Talc	90	90	90	89	88	87

not the weight with which the cosmetician is concerned, but the volume, inasmuch as a woman dips her puff into a powder and takes out a volume which depends partially on the adherent properties of the powder, on the size and type of the puff employed and the depth and pressure with which the puff is applied to the face powder container.

To get a very rough idea of the average relative grade of opacity which prevails in cosmetic materials, the opacities of chalk, kaolin, magnesium stearate, rich starch, talc, titanium dioxide, zinc oxide, and zinc stearate were determined by applying these materials by means of a swansdown face powder puff to similar areas of black velvet paper. The whiteness or opacity produced on the smooth black adherent surface was (a) estimated visually and (b) recorded photoelectrically. For the materials employed, the only particle size specification was that they should all pass completely through a 200 mesh sieve. The order of opacity (starting with the least opaque) was found to be as follows:

- Talc
- Rice starch
- Magnesium stearate
- Chalk (light, precipitated)
- Zinc stearate
- Kaolin
- Zinc oxide
- Titanium dioxide

From what has already been said it will be apparent that to get a true comparison between the relative covering properties of the various face powder constituents it would be necessary to separate similar size particle fractions of each constituent and subject them to exact tests. However, the conditions of such a test, although scientifically more exact, would be so different from the conditions ruling in face powder formulation as to have little actual practical value. The opacities of various finished face powder formulae may be tested approximately by the above method. It should always be remembered that the criterion of effectiveness of a face powder in respect of its water absorption, grease absorption, and covering properties or opacity is judged by the interval elapsing between powdering and repowdering the face; and that a woman judges this by the evidence of shine, which is a complex phenomenon not related to any scientific test and often dependent upon a woman's skin, type of foundation

cream used, and occupation. These facts should be adequately considered by consumer trials, properly carried out and statistically evaluated. Such consumer trials must be carried out scientifically, as ill-conceived trials can be made to prove anything and often prove nothing.

### Absorbency

The second important function of face powders is to eliminate shiny skin in certain facial areas by absorbing sebaceous secretions and perspiration. The prime requirement of a material for this purpose is a high absorptive capacity. The components of face powders which confer this property are colloidal kaolin, starch, precipitated chalk and magnesium carbonate.

The water absorbent properties of face powders or face powder constituents may be determined by the method of Hewitt,<sup>3</sup> in which a known weight of the powder is shaken with excess water and filtered under a standard pressure through a Buchner funnel until no more water emerges. The wet powder is then transferred to a weighed, stoppered weighing bottle and the increase in weight determined. Methods involving the addition of water from a burette, until the powder becomes semi-fluid, are open to the objection that different observers do not obtain concordant results and the end point is not easily determined.

Water absorption is by no means the main characteristic required in a powder; it must also be absorbent for grease. If a person's face is inclined to dryness a more greasy foundation is usually employed. A powder which is not grease-absorbent will show a shiny nose or face which will necessitate re-powdering. Constituents of higher opacity such as zinc and titanium oxides tend to mask greasiness, while starch, chalks and kaolin absorb only a certain amount of grease.

### Colloidal Kaolin

Kaolin, a hydrated aluminium silicate, is a naturally occurring compound. According to Halpern *et al.*,<sup>4</sup> kaolin is not a primary mineral but is a generic term applied to several hydrated aluminium silicates. Not all aluminium silicates, however, may be called kaolin. On the basis of X-ray and physical studies, Ross and Kerr<sup>5</sup> established that three different groups of clay are classified as kaolin. These clays (kaolinite, nacrite and dickite) have essentially the same formula ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ). Purified grades of kaolin that are light in colour and free from grit and water-soluble impurities should be used for face powders; the most suitable is electrolytically purified kaolin. Ordinary china clay is obtained by elutriation and on microscopic examination mica, quartz and feldspar are readily discernible. Pharmaceutical grades of kaolin are obtained by a peptizing process in which the clay is suspended in water containing a suitable electrolyte (for example sodium pyrophosphate) which confers an electrical charge upon the clay and keeps the finer particles in suspension. Removal of the suspension of fine particles, followed by removal of the electric charge (by addition of another electrolyte or by means of an electric field), yields the finest forms of kaolin. One such grade is known as Osmokaolin.

Colloidal kaolin is used in face powders primarily because its high moisture absorption capacity enables it to absorb perspiration. It has also good covering



power, excellent grease-resisting properties and it imparts greater skin adhesion properties to the finished product than does talc. Its relatively high density makes it a useful material for controlling the bulking properties of the powders in which it is used. It also helps to reduce the shine of talc which is present. However, it lacks slip, and is inclined to be somewhat harsh. Its proportion in face powders should therefore not exceed 30 per cent.

#### *Starch and Modified Starches*

At one time rice starch was used almost exclusively as the base of face powder formulae on account of its excellent absorptive properties, good covering power and the smoothness it imparted to the skin. The latter property was closely related to its small particle size, the average diameter of rice starch granules being 3–8  $\mu\text{m}$ . However, objections were raised to the use of starch because of its tendency to cake when exposed to a humid atmosphere or in the presence of excessive skin secretions. McDonough<sup>6</sup> asserted that it readily forms a sticky paste when wet, clogging the pores, and that it is an ideal nutrient, when moist, for bacteria. In addition it coats the hair shaft and so accentuates the downy hair, otherwise unnoticeable, on a woman's face. It was also claimed that because of the tendency of starch to favour bacterial growth, it could give rise to skin irritation when in contact with the skin for any length of time. These assertions led eventually to the replacement of rice starch by talc as the powder base in face powders. However, it must be said that when any degree of bloom is required, there are few materials which can surpass starch.

Decomposition can be reduced in many cases by the addition of perfume; mention of clogged pores refers not to pores but to the openings of the hair follicles. (Pore openings are invisible by ordinary inspection.) There is no proof that starch can cause clogging of such openings.

Special grades of treated starch which will not swell up or agglutinate in the presence of moisture have been developed for the cosmetics industry. For example, ANM starch powders (Neckar-Chemie, GmbH) are starch ethers which are produced by reacting the hydroxyl groups of the starch molecule with tetramethylacetylenediurea. These materials are claimed to have a good slip, good adhesive properties and covering power, and a high absorptive capacity for both water and oil. They are chemically inert and are also claimed to have some bactericidal properties by virtue of their small formaldehyde content. The ANM Rice 'K' grade was claimed, unlike untreated rice starch, not to swell in the presence of moisture or perspiration, and not to give rise to enlarged pores and bacterial decomposition. As in the case of untreated rich starch, it is said to confer a peach-like bloom to the skin, and to be superior to talc in terms of covering power.

#### *Microcrystalline Cellulose (Avicel)*

Avicel is a microcrystalline cellulose from the FMC Corporation.

It is interesting to note in this connection that in 1966 a powder was launched which, unlike conventional face powders consisting of talc or starch, was claimed to contain microporous cellulose derived from the centre of the corn cob, with an oil absorption rate many times higher than other powders.

*Precipitated Calcium Carbonate*

Precipitated chalk is yet another material that has been used in face powders because of its excellent absorption characteristics. Like kaolin it is also used to remove some of the inherent shine of talc. It has, however, a deleterious effect on the slip of the product and tends to impart an undesirable dry feel. Consequently (unless it is one of the special grades available) it should not be used in face powder formulations in amounts greater than 15 per cent.

The special grades of precipitated chalk are exceptionally fine and accurately balanced to prevent harshness. They possess good absorption and grease-resisting properties and are available in different densities, depending on the purpose intended. When such grades are used, it is possible to use considerably greater amounts of precipitated chalk than specified above. A specially treated grade of precipitated chalk is also available which is claimed to be unaffected in terms of its absorptive power for oils and grease, which does not dry the skin, and which is claimed to be particularly adhesive. These materials are guaranteed to conform to the USP specification for lead, arsenic, etc.

*Magnesium Carbonate*

Magnesium carbonate is a highly absorbent constituent of face powder formulations. Its absorbent power is about three times as great as that of precipitated chalk and its tendency to dry the skin correspondingly greater. Magnesium carbonate confers fluffiness to face powders and helps to prevent 'balling'. Light magnesium carbonate is the preferred substance for incorporating and maturing the selected perfume. It is subsequently blended with the bulk of the powder; 5 per cent magnesium carbonate is ample for this purpose.

*Plastics*

Powder bases made from plastics have been developed for use on the skin. These powders are available both in the form of solid spherical particles and in the form of a crushed foam. An example of the latter is 'Oracid'—a rigid urea-formaldehyde foam. Tables 18.4 and 18.5<sup>7</sup> show oil absorption and water

Table 18.4 Oil Absorption Capacities of Powder Bases<sup>7</sup>

Substance	Oil take-up (ml per g of substance)	Saturation time (min)
Oracid (urea-formaldehyde foam)	11.11	15
Aerosil	6.00	15
Magnesium carbonate	5.40	15
Magnesium oxide	3.30	15
Kieselguhr	2.80	15
Kaolin	2.70	15
Talc	2.50	15
Rice starch	2.10	15
Zinc stearate	0.40	15



Table 18.5 Water Absorption Capacities of Powder Bases<sup>7</sup>

Substance	Water take-up (ml per g of substance)	Saturation time (min)
Oracid (urea-formaldehyde foam)	16.60	30
Aerosil	8.70	45
Magnesium carbonate	4.03	28
Kieselguhr	3.20	12
Magnesium oxide	2.60	20
Titanium dioxide	2.30	30
Kaolin	1.50	5
Talc	1.40	10
Zinc oxide	1.10	18
Rice starch	0.75	15
Zinc stearate	0.05	120

absorption capacities of various powder bases, including Oracid, in terms of ml absorbed and the time taken for the saturation value to be reached.

Use of finely divided, highly crystalline, high density polyethylene as a substitute for talc in cosmetic powders has been described in a US patent.<sup>8</sup> Preparations containing polyethylene are claimed to be non-irritant and to have good adhesion, covering power and absorbency. The average particle size of the polyethylene used in cosmetic powders should preferably be not larger than 44  $\mu\text{m}$ . For coloured compositions, for example powder rouge, the dye should preferably be incorporated in the molten polymer, rather than mixed with dry powder constituents.

Cosmetic powder preparations in solid, liquid or slurry form based on finely divided polymeric polyesters are claimed in a British patent.<sup>9</sup> These preparations are claimed to spread easily and to adhere tenaciously to the skin, giving a velvety matt finish. The polymers employed are high-molecular-weight polymeric linear polyesters such polyethylene terephthalate and isophthalates, or a copolymer of these two monomers, with a preferable average particle size of 1–10  $\mu\text{m}$ . Cosmetic powder preparations based on mixtures of these polymeric particles preferably also contain one or more of the usual additives of cosmetic powders such as talc, kaolin, zinc oxide and metallic soaps to improve spreading characteristics, slip, adhesion and absorptive capacity for oily secretions and perspiration.

Polystyrene microspheres are a further example of polymeric materials developed for use in cosmetic powders.<sup>10</sup>

### Slip

Slip is the quality of easy spreading and application of powder to produce a characteristic smooth feeling on the skin.

Slip is mainly imparted by talc and also by metallic soaps such as zinc stearate and

### Talc

Talc is a hydrated magnesium silicate to which the formula of  $3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$  has been assigned. In fact, the Mg/Si ratio appears to vary.

Talc may be obtained from Italy, France, Norway, India, Spain, USA, Australia, China, Egypt and Japan. Of these, the Italian, French, American, Australian and some Indian and Chinese grades can be used for face powders and compact powders. The grinding of raw talc is an important parameter in determining its suitability for make-up products. Talc must be very white and bright to allow products to cover the wide range demanded by the modern consumer and this can only be achieved by having the correct grinding process. Products can always be 'dulled off' with the use of colours but, to produce a 'bright' product, the talc must be bright in the first place. This phenomenon cannot be introduced by the use of other materials. One may even have to use two different types of talc to cover all products, for example one grade for make-up products and another cheaper grade for talcum powders, etc. Whatever the source of the talc it must be free from asbestos or 'amphibole material' and care must be taken with lower grade materials, since talc from certain countries may contain tetanus spores. In such cases it is essential that the talc is adequately sterilized (see also Chapter 8).

Talc is the major component of face powders, and in high class products it may be used in amounts of up to 70 per cent or more. Its main function in such powders is to impart to them slip and good adhesion. However, the covering power and the moisture absorbing capacity of talc are low, and it must therefore be combined with other powders to modify these deficiencies. Apart from its use in face powders, talc is, of course, used in talcum powders, baby powders and antiperspirant sticks, all of which are discussed under their appropriate headings (Chapters 7, 8 and 10).

The most suitable physical form of talc for use in cosmetics is the foliated variety, the flat platelets of which slide readily over each other, thus accounting for the high slip characteristics of the product.

In the USA, the standards of the Cosmetic, Toiletry and Fragrance Association stipulate that talc should be free from impurities such as carbonates and water-soluble iron and be neutral to litmus paper, so as to prevent any deterioration of colour and perfume in the finished product. In order to ensure the application of a smooth and even film, the talc used should also be free of any gritty particles and shiny specks of mica, and the bulk of it should pass through a standard 200 mesh sieve. It should also be free of asbestos, this requirement being emphasized in the USA by the OSHA regulations concerning asbestos-containing materials; a review of properties and specifications is given by Grexa and Parmentier.<sup>11</sup>

### Adhesion

Adhesion is another important property of face powder constituents, determining how well the powder will cling to the face.

The property of adhesion is imparted to face powders by the inclusion of talc and some water-insoluble metallic soaps of stearic acid, such as zinc and magnesium stearates. The latter are used in face powders in amounts ranging



between 3 and 10 per cent. In addition to increasing the adhesion of the powder to the skin, they also render the ultimate product soft and fluffy and furthermore impart to powders some water-repellent characteristics.

The adhesion of powders to the skin can also be improved by the incorporation of certain emollients such as cetyl or stearyl alcohols and glyceryl monostearate, usually in amounts varying between 0.5 and 1.5 per cent.

Various proprietary preparations have been marketed from time to time, having as their object the improvement of the adhesiveness of face powders. One British patent<sup>12</sup> describes the use of the zinc or magnesium salts of fatty acids containing an uneven number of carbon atoms, for example undecylic acid, such a base being employed in a proportion of 5–10 per cent in the finished powder. Many manufacturers prefer to correct any lack of adhesiveness in their product either by increasing the amount of zinc or magnesium stearates incorporated or by including in the powder mixture 2 per cent of petroleum jelly, mineral oil or cetyl alcohol. Further variations can be made using encapsulated mineral oil, especially when one is trying to produce a very light fluffy powder and yet achieve good adhesion.

Among other materials which have been suggested, mention may be made of powdered kapok proposed by Varma<sup>13</sup> as a potential face powder ingredient. Powder bases made from plastics, which were referred to earlier, have also been claimed to give improved adhesion.

#### *Powdered Silica and Silicates*

Very finely divided pure silica has been introduced for cosmetic purposes, for example Neosyl (Crosfield) and Aerosil (Degussa). It is claimed that the use of this substance obviates the need for zinc and magnesium stearates. The addition of 10 per cent to an ordinary powder mix exerts a marked effect in increasing its fluffiness. Up to 20 per cent may be used in a face powder or 30 per cent or more in talcum and baby powders. The incorporation of a small amount of such an ultrafine silica is very efficient as an anticaking agent in body powders.

In addition to silica, a number of ultrafine synthetic silicates which have extremely high oil and water absorption properties may also be incorporated into face powders.

#### **Bloom**

The materials chiefly used to impart bloom, the requirement for which may vary according to fashion, are chalk, rice starch and prepared starch. These have been described above.

#### *Powdered Silk*

A raw material for face powders (and other cosmetic preparations) which, apart from any unique or desirable properties it may have, will provide opportunities for the advertising agencies to produce rapturous copy writing, is powdered silk.

A British patent<sup>14</sup> describes a process for its preparation. Two further patents<sup>15,16</sup> have been taken out dealing respectively with a technique for pulverizing silk for producing silk powder and a method for breaking down silk by boiling successively in sulphuric and boric acids.

Powdered silk was also discussed in *Schimmel Briefs*,<sup>17</sup> where it was pointed out that raw silk consists of fibroin, a protein fibre, covered with a coating of a gummy material called sericin. The latter consists mainly of albuminoid substances with small amounts of fatty acids, resin and colouring matter. The silk is, therefore, first degummed by the conventional processes used in the textile industry, then treated with acid or alkali in order to bring about a partial hydrolysis of the protein molecules. At an appropriate stage it is washed and dried; and finally reduced to an impalpable powder by grinding.

It has been claimed<sup>18</sup> that the physical characteristics of silk powder make it well suited to serve as an ingredient of face powders, that it spreads easily and adheres tenaciously to the skin, producing a velvet matt finish, and that it possesses a very high absorbent power in that it will absorb as much as three times its volume of water and still retain the appearance of a powder. The addition of large amounts of such materials as kaolin or chalk is not advised, as it is claimed that they tend to make the final product too dense and compact.

### Colour

Inorganic and organic pigments and organic lakes have all been used to confer colour to face powders. Water-soluble or oil-soluble dyestuffs should be avoided because of the danger of colour bleeding after application due to solubilization by sweat and lipid secretions. Inorganic pigments include natural and synthetic iron oxides which give yellows, reds, browns and black; ultramarines which give green and blue, and chrome hydrate and chrome oxide which give green.

Within the EEC all colours used in cosmetic products, together with their purity limits, are governed by the Cosmetics Directive of 1976.<sup>19</sup> In the USA the Food and Drug Administration controls the use of colours in cosmetics, but specifies purity limits only for the organic colours. However, it is recognized that inorganic pigments should be produced to a 'certifiable standard' which usually refers to heavy metal content and the Cosmetic, Toiletries and Fragrance Association have issued standards for these materials, for example iron oxides, that are in line with the Food and Drug Administration standards.

There is a considerable activity on the part of the Food and Drug Administration in the USA in regulating the materials to be used in, *inter alia*, toilet preparations. This involves not only the well known ranges of FD&C, D&C, and D&C (Ext.) colours, but also many inorganic materials both coloured and uncoloured which have been mentioned above either as additives (colours) or main materials. Present use is permitted pending the consideration by the FDA of applications for materials to be specifically on the permitted list.

Because of different proprietary names and numbers for various colouring matters, local regulations—as for example the use only of colouring matters certified by the FDA in the USA—and different opinions among firms themselves as to what particular combination of colours gives their 'particular' rachel, peach, tango, etc., no attempt has been made to list the multifarious colouring matters available. Useful information may be obtained by consulting the US Colour Regulations and the Colour Index<sup>20</sup> (see also Harry's *Cosmetic Materials*,<sup>21</sup> wherein ninety pages are devoted to listing the properties of these colouring matters). From a description of their solubilities, etc., it will be



possible to rule out a number of these dyes; of the remainder belonging to the colour classification desired, a number will be found to be obtainable of similar composition in other countries<sup>22</sup> but without, in every case, the guarantee of various metallic and other impurity limits. The task of the formulator will often be simplified by consulting dyestuff manufacturers who will readily advise on suitable colouring matters, oxides and earths and give advice concerning proportions for various colour matches which may be required. Suitable blends of colours are readily available conforming to local regulations, and it is well worth while to evaluate them in one's own formulations before spending too much time on colour blending. The colouring of face powders has been discussed by Anstead.<sup>23</sup>

The choice of colour is usually a matter of taste. At one time it was considered, that 'naturelle' (clear pink) shades were suitable for blondes, and 'rachel' (more cream-yellow) shades for brunettes. Later, perhaps because of more outdoor exposure, it was realized that the natural skin colour tends more to a cream colour, and that the old 'naturelle' now suits only a few complexions which by reason of their transparent skin tend naturally toward blue. Conversely, florid complexions may be toned down by pale bluish green powders. The majority of the present-day range of colours are based on a cream-yellow-brown range.

As well as the actual complexion itself, varying colours of hair and dress all affect the apparent tint, which probably accounts for the multitude of colours in demand (see also Chapter 20).

It is advisable to keep colour formulations as simple as possible so that matching with fresh batches of raw material is made easier. The colour effect produced by a powder applied to the skin will depend *inter alia* upon the opacity of both tinted and white pigments, their particle size, the degree of dispersion, the thickness of the applied film and the colour of the skin.

The colour of the thin film of pigment (the undertone) may be different from the colour effect given by the powder viewed in bulk (the mass tone). It is thus important that the formulator assesses the performance of the product applied to the skin.<sup>24</sup>

## Perfume

The importance of the perfume on the sales appeal of the product cannot be over-emphasized. Usually the powders are perfumed very lightly. The odour of the face powder must be fragrant and pleasant, and a preference is shown today for either a flowery fragrance or that of a synthetic bouquet. Unless the manufacturer has had wide experience of perfume manufacture he will be well advised to purchase the perfume from a reputable perfume manufacturer.

The compatibility of perfume with other constituents of the product must be carefully checked. Talc, for example, usually contains a little free lime, magnesia or iron which may adversely affect the perfume depending on the amount of these substances present. The perfume may also be affected by precipitated chalk, by kaolin, magnesium carbonate or a metal stearate, if these contain impurities, or indeed by some of the pigments used in colouring the powders. Finally, it should be remembered that the perfume note in a powder will be

different from that, for example, in an alcoholic solution, particularly in the case of floral bouquets, and any tests on perfume acceptance must be carried out on the final product.

### Formulation

The chemist familiar with the properties and functions of the various powder constituents and with the sources of supply will have no difficulties in formulating a satisfactory product. He should, however, be given details of the type of market for which the product is intended, the advertising story to be used and properties to be highlighted. He will then be able to judge what proportions of which constituents to use, and which materials to avoid in order to produce a suitable formula. Thus, for powders with a good covering power, he will use a higher proportion of either zinc oxide or titanium dioxide; for increased absorbency, the proportion of magnesium carbonate may well be raised at the expense of talc, and where a powder with good adhesion is required the amount of zinc or magnesium stearate may have to be increased.

Multifarious variations in formulae could be listed, many of which, under laboratory conditions, show slight differences in respect to opacity, slip, absorbency, water-resistance and grease-resistance, etc., but experience shows that many of these variations cannot be detected by the average woman under normal conditions of use. The following formulae exemplify various types of face powder, the variations in which are sufficient to be detectable.

The powder given in example 1 is very transparent, that is it is a 'light' powder, and is favoured by persons who wish to impart some colour and bloom to the face without appearing to be 'made up'. The starch may be replaced if desired with precipitated chalk.

	(1)
	<i>per cent</i>
Talc	80.0
Zinc oxide	5.0
Zinc stearate	5.0
Rice starch	10.0
Perfume, colour	<i>q.s.</i>

Example 2 has high opacity and gives a very definite opaque matt finish which tends to hide minor skin defects. It is more popular with certain people who like to have a definite powdered appearance without being over-powdered.

	(2)
	<i>per cent</i>
Talc	30.0
Zinc oxide	24.0
Zinc stearate	6.0
Precipitated chalk	40.0
Perfume, colour	<i>q.s.</i>



In between these two types of powder there are a number of popular variations such as the following:

(3)	
<i>per cent</i>	
Talc	65.0
Precipitated chalk	10.0
Zinc oxide	20.0
Zinc stearate	5.0
Perfume, colour	<i>q.s.</i>

(4)	
<i>per cent</i>	
Talc	60.0
Kaolin	20.0
Zinc oxide	15.0
Zinc stearate	5.0
Perfume, colour	<i>q.s.</i>

As previously stated, both starch and precipitated chalk tend to impart bloom; example 5 is a powder of medium weight, that is medium opacity or coverage and bloom.

(5)	
<i>per cent</i>	
Talc	50.0
Rice starch	15.0
Precipitated chalk	15.0
Zinc oxide	15.0
Zinc stearate	5.0

If it is desired to obtain maximum coverage and still maintain a high talc content, this may be achieved by replacing the zinc oxide in any of these formulae by about one-quarter of its weight of titanium dioxide.

The following formula is prepared from precipitated chalk and a high proportion of zinc stearate is incorporated. To provide some grease-resistance, kaolin and titanium dioxide are employed.

(6)	
<i>per cent</i>	
Waterproof chalk base	50.0
Zinc or magnesium stearate	10.0
Kaolin	20.0
Titanium dioxide	6.0
Talc	14.0
Perfume, colour	<i>q.s.</i>

Jannaway<sup>25</sup> states that the following formula gives a good medium powder, characterized by excellent slip, absorbency, adequate coverage, good velvety feel and adherence—and an indefinable improvement in mattness, etc., which is

attributed to the rice starch:

	(7) <i>per cent</i>
Zinc oxide	16.0
Talc	37.0
Zinc stearate	5.0
Precipitated chalk (light)	18.0
Rice starch	8.0
Kaolin (best cosmetic grade)	16.0

Winter<sup>26</sup> has described a special kind of 'fatty powder' which he states is much favoured by persons afflicted with a rough or dry skin:

	(8) <i>parts</i>
Vaseline	50
White beeswax	40
Petroleum jelly	40
Stearin	20
Glyceryl monostearate	75

*Procedure:* Melt the above fatty materials together and add, while stirring constantly, 500 parts of hot water. Continue to stir until the emulsion has formed, then add 1000 parts of talc. Knead, allow to dry, rub to powder, pass through a sieve and perfume.

Other formulations are given by Keithler<sup>27</sup> and Hilfer.<sup>28</sup>

The modern trend with face powder is to apply it over foundation to achieve special effects, for example, matt or shimmer. The following complete formula illustrates a light shimmering effect:

	(9) <i>per cent</i>
Talc	77.00
Zinc stearate	5.00
Zinc oxide	2.00
Kaolin	5.00
Mica	10.00
Red iron oxide	0.36
Yellow iron oxide	0.36
Black iron oxide	0.03
Perfume	0.25

### Manufacture

Mixing of the ingredients in face powders is usually carried out in a horizontal mixer with a screw agitator. If laves are used they may be mixed with a small quantity of one of the constituents, chalk, zinc oxide or talc, and the colour concentrate so formed mixed with the main bulk. If water-soluble or alcohol-soluble dyes must be used they are best sprayed on to the mix or alternatively on



to one of the constituents which possesses good absorbency such as chalk, kaolin or magnesium carbonate, this being then dried and mixed into the main bulk.

Machines are available which mix, sift and spray the perfume automatically. One method would be to add the perfume by a meter pump feeding a long tube with a multitude of small holes fitted along the top of the blender (for example, on to magnesium carbonate or chalk in the ribbon blender) before mixing with the remainder of the powder.

The use of water-soluble or oil-soluble colours should be avoided in a face powder since they lead to streaking, darkening and staining of the skin when applied over ordinary make-up. Chilson<sup>28</sup> has drawn attention to the pebble mill method of mixing which obviates the making of a colour base. All the ingredients, including the colour and perfume, are milled together in a pebble mill for six hours, discharged and then sifted. He states that such a mill delivers an excellent product and is widely employed in preference to mixers. This method, however, is too slow to be recommended for large-scale production. Micropulverizers are being increasingly employed since the material obtained is finely ground and uniformly mixed and only a rough pre-mixing is required. Various other pulverizers such as disintegrators, hammer mills, attrition mills, etc., may also be used.

Pin-disc mills give good results, especially in respect of colour dispersion, provided that the material is first given a rough pre-mix. Such mills are often applied in conjunction with a turbine sifter.

The Air Spun Process employed by Coty Inc. is described briefly by deNavarre<sup>30</sup> as follows:

... Purified and cold air, under great pressure (100 psi; 700 kPa) is permitted to rush in a continuous stream into a closed drum-shaped chamber. This chamber or mill is called a micronizer because it reduces particles to micron ( $\mu\text{m}$ ) size. The speed of the air stream, the manner in which it is directed and the shape of the chamber itself, cause air to revolve about this chamber at a rate somewhat in excess of 1000 mph. In this super cyclone all the ingredients are hurled against each other until they reduce themselves to the desired size and fluffiness. When this is attained such particles are emitted through a central exit while the heavier particles are forced to remain. It is claimed that the particle size was selected after numerous experiments had shown it to be the best for the appearance, effect and adhesiveness of a face powder.

Apart from this process, various physical methods exist for preparing powders of a desired particle size range. These depend upon elutriation by means of air or water. Control of such separated fractions may be carried out by microscopic examination of the fractions, but this method involves the visual or photomicrographic inspection of a very large number of fields in the microscope and is tedious. Various sedimentation tests, some based upon passage of a beam of light through the sedimentation column into a photoelectric cell, have also been suggested. One of the simplest methods for the cosmetician is that based upon the principles of Stokes's Law, described by Hinkley.<sup>31</sup> A useful variation is the method of Andreason in which volumes of the suspension, after definite times of settling (calculated from Stokes's Law) are pipetted into a tared dish, evapo-

rated to dryness and weighed. Various air permeability methods are also widely used.

The most modern method of particle size analysis applicable to cosmetic powders entails the use of a Coulter Counter. A paper dealing with the subject was presented by Wood and Lines.<sup>32</sup>

Packaging is usually carried out by automatic vacuum filling devices, which minimize dusting.

In choosing powder boxes, care should be taken to see that these are prepared with an odourless glue, otherwise the fragrance of the powder will be ruined in storage.

## COMPACT POWDER

Because they are convenient to use, compact powders enjoy wide popularity. They are nowadays prepared by either a damp or a dry compression process. The moulding process used originally, which entailed the use of plaster of Paris, has fallen into disuse.

In the damp process, the powder, intimately mixed with a suitable binding agent, is milled to the requisite plasticity, compressed into suitable containers, usually metal godets, and dried for the requisite period in a current of warm air. In the dry process the mass is subjected to compression without being wetted to any appreciable extent. This process, although difficult to achieve satisfactorily at first, is probably the best to use for manufacturing compacts on a large scale, because it can be rigidly adhered to once the mix and suitable conditions have been determined. Presses available for the manufacture of compact powders may be of the hydraulic or reciprocating mechanical type, varying in size, operating pressures and output. They range from foot operated presses producing one cake at a time, to fully automatic presses which may produce up to 60 units per minute.

The requirement of good covering power, adhesion and uniformity in compositions mentioned in respect of conventional face powders also applies to compact powders. The latter should, in addition, be easy to remove from the cake for application by means of a powder puff, without crumbling or breaking during handling; this requirement is met by conferring adequate binding properties to the powder mixture to be compressed. Furthermore, the powders used for compacts should be free-flowing so that they do not adhere to punches or dies during compression; otherwise, air pockets will be formed which will result in an uneven compression and cause the cakes to break. From this it can be seen that one of the main aims during manufacture of compact powders is to ensure that the compressed cakes are of uniform density.

The main difference between loose powders and compact powders lies in their binding properties. If these are inadequate, the compressed cakes are liable to crumble easily following compression. If they are excessive, the cake will form lumps and go greasy on application. Thus, satisfactory binding properties are essential for trouble-free compression and the production of good quality cakes over long manufacturing periods.



The actual pressing process can also affect the shade of the product so quality control can be a problem. In fact, if large volumes are anticipated it is always best to conduct an extended manufacturing trial rather than go straight from the laboratory bench into full-scale production.

The composition of compact powders is generally very similar to that of loose powders. The differences which exist arise from the need to meet the requirement of greater cohesion and are largely evident in terms of percentages of some of the components present. In compact powders, colloidal kaolin, zinc oxide and metallic stearates are usually present at a higher level than in loose face powders, and starch is sometimes incorporated to facilitate compression. If the powders are not sufficiently binding they will require the addition of a binding agent to improve their cohesion so that on compression a firm cake is produced. Water-soluble and water-insoluble binding agents may be used. The former are natural and synthetic gums which are used in amounts ranging from 0.1 per cent to about 3 per cent by weight of the product, and are usually mixed with component powders in the form of a 5–10 per cent aqueous solution. A very much favoured binding agent for this purpose is low-viscosity carboxymethylcellulose. A small amount of a humectant is usually added to the solutions. If a water-insoluble binding agent is employed, for example glyceryl monostearate, cetyl or stearyl alcohols, isopropyl esters of fatty acids, lanolin and its derivatives or ozokerite, paraffin wax and microcrystalline waxes, it is preferable to use it in the form of an oil-in-water emulsion so that it is uniformly distributed throughout the product.

In the dry compacting process, it is usual to employ zinc or magnesium stearates at a level of 5–15 per cent by weight, as well as a lubricant such as mineral oil in similar proportions.

Considerable changes have taken place in formulation during the last forty years or so; thus in 1932 Winter<sup>33</sup> recommended an ammonia–stearin–starch compound containing white petrolatum, ammonia, starch and stearin (example 10) for the manufacture of compact powder and rouge.

	(10)
Stearin	100 g
White petrolatum	20 g
Ammonium hydroxide solution, 0.97 s.g.	50 cm <sup>3</sup>
Rice or maize starch	250 g

*Procedure:* Melt the stearin and the petrolatum together. Add the ammonium hydroxide solution and stir thoroughly while hot. Add the starch to the warm mixture with vigorous stirring (during the addition, thick lumps will form in the starch powder; with vigorous stirring and pressing, these break down and mix with the starch). Rub the resulting crumbly mass in a mortar and then pass it through a 70–80 mesh sieve. Cool well before sifting.

This compound is added, in a proportion of 13–15 per cent, to the powder base together with the colouring matter, and the mixture is subjected to pressure. It is stated that such pressing should not be carried out suddenly but by gradually increasing the pressure. Experiments have fully confirmed this opinion. Unless a little time is given for the air to escape gradually, it becomes entrapped in the compact with disastrous results.

As mentioned previously, rice starch has been used in compact powder manufacture to facilitate compression of the powders. There has, however, been some controversy regarding the maximum permissible proportion of starch in a compact. On one hand, views were expressed to the effect that the starch content should be low, otherwise there is a tendency to produce hard cakes and to make the removal of the powder, when the puff is applied, more difficult.<sup>34</sup> One formula quoted included only 2.5 per cent of rice starch.<sup>35</sup> Winter, on the other hand, considered that starch acts as a good binding agent and recommended a base containing about 13 per cent of starch.<sup>33</sup> In yet another article, up to 20 per cent of starch was regarded as helpful in binding a compact.<sup>36</sup>

Winter suggested the powder base mixture given in example 11, to which may be added 13–15 per cent of stearin-starch and the required colouring matters. Water is added to produce a dough-like paste which is dried, ground and passed through a 120 mesh sieve and the mass compressed into a suitable metal case or godet.

	(11) <i>per cent</i>
Talc	26.7
Kaolin	56.7
Zinc oxide	3.3
Rice starch	13.3

Modern manufacturing procedures and compositions vary appreciably from those just described. In the case of the damp process, any colours to be used are first ground with the powder constituents, and the resulting mixture is passed through a sieve. The powder is then moistened with the binder solution or emulsion and perfume is incorporated. After a thorough mixing, the blend is sieved once more, for example through a 60 mesh sieve. The produce is then dried at room temperature or in warm air, provided that the temperature used does not exceed that at which the perfume will volatilize. The product is then compressed and placed in suitable containers.

Example 12 gives a formula to which the damp compression process is applicable.

	(12) <i>per cent</i>
Kaolin	20.0
Zinc oxide	15.0
Precipitated chalk	25.0
Talc	32.0
Compact binder (e.g. soap)	8.0
Perfume, colour	<i>q.s.</i>

The second of the two modern compacting processes, that is the dry compression method, is particularly suitable for mass production, but necessitates the use of higher pressures than are employed in the damp compression process. The manufacture of a compressed face powder cake by the dry compression process has been described in some detail in a US patent<sup>37</sup>. In an



embodiment example of this patent the following composition and procedure were given:

	(13) per cent
Talcum	61.25
Sodium lauryl sulphate	0.75
Titanium dioxide	7.50
Zinc stearate	11.25
Inorganic pigments	1.00
Mineral oil	4.50
Spermaceti	3.00
Cetyl alcohol	1.50
Lanolin	1.00
Glycerin	7.50
Hexachlorophene	0.25
Alkyldimethylbenzyl ammonium chloride—50%	0.20
Methyl- <i>p</i> -hydroxybenzoate	0.09
Propyl- <i>p</i> -hydroxybenzoate	0.09
Perfume	0.12

*Procedure:* Talc, sodium lauryl sulphate, titanium dioxide, zinc stearate and inorganic pigments are first mixed together in a ribbon mixer for about an hour. Next, hexachlorophene, quaternary ammonium base and preservative, thoroughly premixed, are added to the mixer and the batch is mixed for about two hours, before passing through a micro-pulverizer using a no. 0.013 screen. The temperature of the material should not be allowed to rise more than 10°C above room temperature during this operation. After cooling, the powder blend is repressed through the micro-pulverizer. The mineral oil, spermaceti, lanolin and glycerin, mixed with heating until liquid, are then sprayed into the dry batch which is mixed in the ribbon mixer for a further half hour. The batch is passed through a comminuter and once again through a pulverizer, using a 3/16 in screen and taking the same precautions about temperature as before. When cool, the powder is passed through the micro-pulverizer using a no. 0.027 screen, and finally, having been cooled once more, is passed through a 40 mesh screen, ensuring that no heat is generated during this final screening. The resulting material is filled into containers and pressure is applied, for example 40–50 psi (300 kPa), to convert the powder into cake. It is usually advisable to keep the powders for several days in a suitably humid atmosphere before pressing, to facilitate the escape of any trapped air, and to ensure that the powder blend will not be too dry when compressed. During the dry compression process, it is usual to apply a small pressure initially to squeeze out the air, and thus avoid the formation of air pockets in the powder cakes. Pressure is then gradually increased up to 150 psi or even more (1000+kPa) before the die is removed from the surface of the cake. It is possible, however, with careful formulation and preparation of the powder to press the cakes with immediate pressures of up to 600 psi (4 MPa) and outputs approaching two thousand units per hour.

## CAKE MAKE-UP

The modern cake make-up originates from theatrical grease-paint and has acquired popularity because of its ease of application and stability and also

because more product can be applied to the face and thus deeper shades and effects can be achieved. As in the case of compact powders, the cake make-up is also made from talc, kaolin, zinc oxide and precipitated chalk, but it contains, additionally, inorganic pigments such as titanium dioxide and iron oxides. Humectants such as sorbitol or propylene glycol may also be incorporated together with other additives such as sorbitan sesquioleate, lanolin or mineral oil and perfume. Humectants and other liquid constituents are usually combined and sprayed on to the powder constituents while these are mixed in the ribbon mixer. The resulting blend is granulated and finally compressed.

Patents<sup>38,39</sup> granted in the 1930s claimed a product in cake form which, when applied to the face using a damp sponge, dried to form a coherent water-repellent film of powder. The advantage of this product is that no foundation cream is required and make-up can be retouched very quickly and conveniently.

A patent by Max Factor<sup>40</sup> describes a dry cake-form make-up which can be applied with a moistened pad or sponge. The cake contains oily and waxy ingredients (0.8–24 per cent), a water-soluble dispersing agent (1–13 per cent) and fillers (35–80 per cent) and pigments (12–50 per cent) whose particles are coated with the oils and waxes to make them water-repellent. The make-up is prepared by adding the fillers (talc, chalk) and pigments (zinc, titanium and ferric oxides) to the oils and waxes dispersed in water, drying the mixture so formed, pulverizing the product, and compressing it into cake form.

Products made to this style of formula can be prepared without drying by using a much higher ratio of powder to emulsion, for example approximately 12 to 1, and mixing with a Beken Planetex or Duplex mixer. The mixing operation takes between 30 and 60 minutes and the mix is compacted after granulating through a fine screen.

The following formulae can be made in this fashion:

	(14)
	<i>parts</i>
Powder base (perfumed)	100
Emulsion:	
Stearic acid	34.5
Mineral oil	21.5
Triethanolamine	10.5
Water	33.5
	} 8.5

For deep shades with good coverage, zinc oxide should be replaced by titanium dioxide (this also facilitates better pressing characteristics):

	(15)
<i>Colour mix:</i>	<i>per cent</i>
Talc	89.75
Titanium dioxide	9.00
Yellow iron oxide	0.75
Red iron oxide	0.40
Black iron oxide	0.10



<i>Base:</i>	<i>per cent</i>
Stearic acid	15.0
Acetylated lanolin	3.0
Stearyl alcohol	3.0
Glyceryl monostearate (self-emulsifying)	2.2
Sulphonated castor oil	1.2
Triethanolamine	3.0
Mineral oil	35.0
Polyethylene glycol	10.0
Water	27.6
<hr/>	
<i>Completion ratio:</i>	
Base	20.0
Colour mix	79.8
Perfume	0.2

Pressing of this product can be very rapid as for compact powders, although it is important to check that there is consistent dispersion of the base in the colour mix throughout long manufacturing runs.

A prototype cake make-up formula suggested in a cosmetic formulary of Atlas Industries<sup>41</sup> had the composition given in example 16.

	(16)
<i>Pigment blend:</i>	<i>per cent</i>
Talc	60.0
Kaolin	20.0
Zinc oxide	10.0
Titanium dioxide	5.0
Calcium carbonate, light	5.0
Iron oxide colours	<i>q.s.</i>
<hr/>	
<i>Completed formula:</i>	
Pigment blend	81.4
Arlex (sorbitol)	4.1
Propylene glycol	2.4
Arlacel C (sorbitan sesquioleate)	9.7
Mineral oil or lanolin	2.4
Perfume, preservative	<i>q.s.</i>

*Procedure:* Mix the Arlex (sorbitol) with the propylene glycol and preservative and spray this mixture into the powder while it is being mixed in a dough mixer. Mix the Arlex C and mineral oil. If lanolin is used, melt the lanolin with the Arlacel C. Add the perfume and spray on to the powdered mass which is agitated in the dough mixer. Transfer to a Fitzpatrick tablet granulator or an equivalent machine. After granulation, the mass is ready for performing and compression. The same procedure is employed as in the case of compact powders, that is the initial application of a relatively light pressure (about 25–50 psi; 250 kPa) to remove air, followed by the application of a higher pressure of 100–150 psi or more (1000 + kPa).

#### *Application of Cake Make-up*

Maurice Seiderman, a pioneer of make-up in the motion picture industry, recommended the application of cake make-up in the following manner: 'To

apply cake make-up correctly, wet and squeeze out a sponge and rub it lightly over the face. Smooth the cake make-up on the face and before it dries blend in the rouge. Carefully squeeze all water out of the sponge and rub it lightly over the face until the make-up is dry.' Macias-Sarria<sup>42</sup> adds that people who do not like a heavy make-up should blot off the excess of water with a dry towel or facial tissue.

It is claimed that cake make-up is satisfactory for the younger woman; in the case of persons in the later thirties, and even younger persons who suffer from dry skin, it is found to be rather drying. To cater for this market, to coincide with the general trend towards more greasy make-up as exemplified by many foundation creams and possibly also with a view to cashing in on the flat type of pack in which cake make-up is packed, a greasy type of compact containing pigments of high covering power in a waxy base has been marketed.

Such products appear to be poured instead of being compressed in the usual manner. Wetting agents may be incorporated so that these products may be removed with a damp sponge; alternatively, silicone oils may be used to improve the spreading characteristics and make the product suitable for finger-tip application.

## MAKE-UP CREAM

Foundation make-up preparations in cream form are essentially suspensions of pigments in an emulsified lotion. The addition of the pigment is usually made at about 50°-55°C as the emulsion is slowly cooled with agitation. Janistyn quoted the following formula for a cream make-up.<sup>43</sup>

	(17) <i>parts</i>
Glycerol mono- and distearate (pure)	2.0
PEG 400 monostearate	1.0
Stearic acid	11.5
Cetyl alcohol	0.5
Isopropyl myristate or palmitate	2.0
Propylene glycol	12.0
Sorbitol syrup	2.5
Preservative	0.1
Titanium dioxide	2.2
Talc	8.0
Colour pigments	1.0
Water	57.4

The suspension is usually homogenized and milled.

## LIQUID POWDER

So-called 'liquid powders' have sometimes been used as a base for ordinary powder or to replace such powder for evening wear, dances or similar occasions. They have also included a theatrical product, 'wet white', which was used for



whitening the neck and arms. Its basic components were zinc oxide and bismuth oxychloride, subnitrate and carbonate incorporated into a liquid consisting of a mixture of glycerin and water in varying proportions. Glycerin was employed in amounts of up to 30 per cent and the water was often replaced by triple rose water or other fragrant waters; starch was also used sometimes to suspend heavier constituents. To prepare 'wet white' the constituent powders were mixed and glycerin was added. Surfactants could also be included to assist in dispersing the powders.

A 'wet white' formula on these lines, quoted by Poucher,<sup>44</sup> is given in example 18.

	(18) <i>per cent</i>
Bismuth subnitrate	5.0
Starch	5.0
Zinc oxide	10.0
Glycerin	15.0
Rose water	65.0

Other more modern formulae omit the bismuth salt and starch, and employ more pigments, fillers and colouring materials to simulate various shades of flesh colour. Examples 19 and 20 will serve as a basis for experiments:

	(19) <i>per cent</i>
Zinc oxide	3.0
Chalk (precipitated)	15.0
Kaolin	2.0
Colouring matter	<i>q.s.</i>
Glycerin	15.0
Water	65.0
Preservative, perfume	<i>q.s.</i>

	(20) <i>per cent</i>
Zinc oxide	10.0
Titanium oxide	10.0
Talc	10.0
Colouring matter	<i>q.s.</i>
Gum tragacanth (0.5% solution)	25.0
Glycerin	15.0
Water	30.0
Preservative, perfume	<i>q.s.</i>

Janistyn<sup>43</sup> quoted a liquid powder formulation of the following composition:

	(21) <i>per cent</i>
Sodium carrageenate (medium or high viscosity)	2.0
<i>n</i> -Propyl alcohol	2.0
Propylene glycol	5.0

	<i>per cent</i>
Water	68.5
Veegum HV	0.5
Talc	10.0
Magnesium carbonate	4.0
Titanium dioxide	1.0
Colour pigments	7.0
Perfume	<i>q.s.</i>

*Procedure:* Wet the carrageenane with propyl alcohol and then dissolve it in the propylene glycol-water mixture. Veegum is then dispersed in the solution, followed by the addition of mixed pigments.

### *Cosmetic Stockings*

This type of aqueous make-up was used during World War II as a leg make-up or liquid cosmetic stocking. The requirement was that the appearance of a treated leg should be very similar to a leg encased in a normal stocking. It was essential that the preparation should not wash away in the rain, nor rub off on to clothing, yet should be easily removable by washing with soap and water.

A basic formula upon which to elaborate a liquid 'cosmetic stocking' is given in example 22.

	(22) <i>per cent</i>
Zinc oxide	6.0
Chalk (precipitated)	16.0
Methylcellulose	0.5
Glycerin	16.0
Water	61.5
Colouring matter, preservative, perfume	<i>q.s.</i>

More opacity in the finished make-up may be obtained by increasing the proportion of zinc oxide and/or including titanium dioxide, but an obviously artificial effect is undesirable. A little alcohol may be included in order to accelerate drying; the viscosity may be varied by the use of different viscosity grades of methyl cellulose or other cellulosic film-forming material; alternatively alginates or gum mucilages may be included, but care must be taken that the finished preparation is not sticky.

To increase the time during which the pigmented powder remains in suspension (after shaking) while the preparation is being applied to the leg, a few per cent of bentonite or other similar clays may be incorporated.

A certain amount of glycerin or similar non-evaporating and foundation-forming substance is desirable as this improves the adherence of the film of powder to the leg.

Colouring matters usually consist of mixtures of harmless yellow, red, and brown pigments according to the shade desired; in addition some soluble dyes may be included to produce a slight staining effect on the leg and also minimize the appearance of separation between the aqueous and powder phase in the container.



The following formula appeared in the *American Perfumer*:<sup>45</sup>

	(23) per cent
Zinc stearate	2.0
Titanium dioxide	3.5
Colloidal aluminium-magnesium silicate gel (Veegum)	20.0
Isopropyl alcohol	6.0
Umber	0.5
Yellow oxide	2.5
Red oxide	2.5
Propylene glycol	1.0
Methylcellulose (1500 viscosity grade)	0.5
Water, perfume, preservative	to 100.0

### LIQUID MAKE-UP

Liquid make-up for cosmetic use is another development of the wetted powder which consists essentially of pigments dispersed in a viscous base.

The early liquid make-up preparations were suspensions of pigments in an aqueous alcoholic solution, which required vigorous shaking prior to use to ensure uniform distribution of the product during application.

The basic problem in the preparation of more elegant products of this type is to prevent the sedimentation of constituent pigments by dispersing them in a hydrocolloid base or in a liquid emulsion. The hydrocolloids used for thickening the preparations may be selected from cellulose derivatives, carrageenates, Carbopol 934 or 941, Veegum and others.

The pigments used in liquid make-up preparations are the usual components of powder bases such as talc, kaolin, zinc oxide, titanium dioxide, calcium or magnesium carbonates and others.

In emulsified products, raw materials used included propylene glycol monostearate, glyceryl monostearate, fatty alcohols such as cetyl or oleyl alcohols, isopropyl myristate, lanolin and its derivatives, polyethylene glycols, humectants and others; in general they resemble the make-up cream described in example 17 above.

A liquid make-up formula quoted by Shansky<sup>46</sup> is given in example 24. Viscosity may be adjusted by varying the amount of gum and bentonite.

	(24) parts
Propylene glycol	4.40
Polyethylene glycol 400 monostearate	1.92
Preservative	0.32
Gum tragacanth (0.175% solution)	76.68
Bentonite	0.96
White oil	1.20
Oleyl alcohol	6.72
Stearic acid	4.20
Triethanolamine	1.92
Perfume	q.s.
Titanium dioxide plus powdered pigments	q.s.

Pigments and titanium dioxide are usually in the formula at a level of 5–10 per cent, as in example 25 (the actual amount will depend on the shade desired).

	(25) per cent
Isopropyl lanolate	3.50
Isopropyl myristate	4.20
Squalane	1.40
Purcellin oil	2.10
Mineral oil	12.80
Sorbitan oleate	1.00
Veegum (5% solution)	30.00
Propylene glycol	8.00
CMC (1% solution)	20.60
Polyoxyethylene (20) sorbitan mono-oleate	4.00
Water	6.04
Titanium dioxide	4.60
Yellow iron oxide	0.56
Red iron oxide	0.55
Black iron oxide	0.10
Perfume	0.25
Preservative	0.30

## STICK MAKE-UP

Liquid make-up products are by far the most acceptable products for face make-up, mainly due to their light application which suits the modern style and their convenience of use (bottle or tube). However, there is a small but significant proportion of the market who prefer a heavier make-up but with the convenience factor as well. This has led to the development of stick make-up, which in essence is a dispersion of pigments in a wax base. A typical formula is shown in example 26.

	(26) per cent
Mineral oil	47.65
Paraffin wax	3.50
Beeswax	1.50
Carnauba wax	4.00
Kaolin	9.00
Titanium dioxide	30.00
Yellow iron oxide	2.50
Red iron oxide	1.50
Black iron oxide	0.30
Perfume	0.05

*Procedure:* Mix the oils and waxes together and heat until a clear solution is obtained. Mix in the colours and pigments gradually with a high-speed Silverson-type mixer. Shade, dispersion and setting point should be checked prior to pouring the product into the appropriate containers.



This stick concept can be extended further to give the cover-up product, used to disguise birthmarks and blemishes. The product is heavily pigmented and is applied like a lipstick.

	(27) per cent
Lanolin alcohols	2.8
Ozokerite wax	8.0
Paraffin wax	6.0
Mineral oil	20.2
Isopropyl myristate	10.0
Lanolin	2.8
Titanium dioxide	36.8
Kaolin	8.0
Yellow iron oxide	2.5
Black iron oxide	0.6
Red iron oxide	2.0
Perfume	0.3

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## Coloured Make-up Preparations

### LIPSTICK

#### Introduction

Lipsticks, the lip cosmetics moulded into sticks, are essentially dispersions of colouring matter in a base consisting of a suitable blend of oils, fats and waxes.

Lipstick is used to impart an attractive colour and appearance to the lips, accentuating their good points and disguising any bad ones. Narrow bad-tempered lips may be widened, and broad sensual lips made to appear narrower by its use. In fact, if applied intelligently it is capable of entirely altering the apparent facial characteristics.

Since lips are considered to be more alluring when they possess a slightly moist appearance, this is always achieved by the use of a greasy base which also exerts an emollient action.

There is no doubt that the wide use of lipstick among women has led to a decrease in cracked and chapped lips, the crevices of which were always liable to bacterial infection. In addition, as in the case of many other cosmetics, it exerts a psychological effect difficult to assess, and induces a feeling of mental comfort.

#### *Characteristics Required in a Lipstick*

A good lipstick should have the following characteristics.

- (1) It should have an attractive appearance, that is, a smooth surface of uniform colour, free from defects such as pinholes or grittiness due to colour or crystal aggregates. This should be retained during its shelf life and usage life—it should not exude oil, develop a bloom, flake, cake, harden, soften, crumble nor become brittle over the range of temperatures likely to be experienced.
- (2) It should be innocuous, both dermatologically and if ingested.
- (3) It should be easy to apply, giving a film on the lips that is neither excessively greasy nor too dry, that is reasonably permanent but capable of deliberate removal, and which has a stable colour.

It will be realized that a system of colouring matter dispersed in a plastic fatty medium is one most likely to satisfy the above requirements.

#### Ingredients of Lipsticks

##### *Colouring Materials*

The colour of a lipstick is one of the major selling points, but it is one which can only be dealt with in general terms, since the precise shades are dictated by

ephemeral fashion. It is usual for the colour to contain some measure of red and this allows shades ranging between orange-yellow and purple-blue, although even greens are not unknown. Depth of colour and opacity are also variable and during periods when the fashion trend was to a 'no make-up' look, uncoloured lipstick base of high gloss has been seen under the name 'lip gloss'. 'Lip gleams' containing pearlescent materials are also known, and occasionally sticks with some degree of gold or silver lustre achieved by the use of finely divided metal (coloured aluminium) are presented. However, the main accent in this chapter will be on the predominantly red conventional shades, since these involve the basic principles of lipstick formulation.

The colour is imparted to the lips in two ways: (a) by staining the skin, which requires a dyestuff in solution, capable of penetrating the outer surface of the lips; (b) by covering the lips with a coloured layer which serves to hide any surface roughness and give a smooth appearance. This second requirement is met by insoluble dyes and pigments which make the film more or less opaque.

Typical proportions for the colours in a lipstick are as follows:

	<i>per cent</i>
Staining dyes (bromo acids)	½-3
Oil-soluble pigment	2
Insoluble pigment	8-10
Titanium dioxide	1-4

**Staining Dyes.** The most widely used staining dyes are water-soluble eosin and other halogenated derivatives of fluorescein which are generally referred to collectively as 'bromoacids', a term originally applied to acid eosin, tetrabromofluorescein.

*Eosin*, also known as D&C Red No. 21, is an insoluble orange compound which changes to an intense red salt when the pH value is above 4. When applied to the lips in the acid form, it produces a relatively indelible purple red stain on neutralization by the lip tissue.

Other halogenated fluoresceins can be used to give different staining colours and varying degrees of indelibility. Thus D&C Red No. 27 (tetrachlorotetrabromofluorescein) produces a brilliant bluish red stain, and D&C Orange No. 5 (dibromofluorescein) a yellow red, which often used in conjunction with D&C Red No. 21. D&C Orange No. 10 (di-iodofluorescein) is another derivative frequently used.

Unfortunately, eosin and some of its derivatives can give rise to sensitization or photosensitization, leading to cheilitis (inflammation of the red portion of the lips) or more general allergic reactions. Whether this is due to the bromoacid *per se*, or to impurities contained therein or even to the perfume in the lipstick is by no means clear, but the fact that it does occur with a small proportion of lipstick users (coupled with the facts that the skin of the lips is devoid of a horny layer, and that there is a possibility of ingestion of lipstick) has focussed attention on permissible colours. The USA and the EEC countries have defined lists of permitted colours, but these do not always coincide. Thus, for example, FD&C Red No. 2 is now banned in the USA whereas it is permitted in the EEC. The situation becomes even more confused when EEC directives clash with local



laws. For example, D&C Orange No. 5 has been banned for some time in Germany, but is allowed for mucous membrane products by the EEC Cosmetics Directive. Incidentally, D&C Orange No. 5 is allowed in the USA with certain restrictions (see below). Formulators should check very carefully therefore to determine the use of the various colours in the individual countries in which the lip products are to be sold. A brief summary of the status of the most popular colours as used in the USA and the EEC is given in Table 19.1.

**Table 19.1 Status of Colouring Materials for Lipsticks in the EEC and the USA**

Colour index no.	Common name	Status under EEC Cosmetics Directive	Status under US FDA
12085	D&C Red 36	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81; 3% allowed in lip products
15585	D&C Red 8 (Na)	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81; 3% allowed in lip products
15630	D&C Red 10 (Na)	Allowed: Annex III Part 2	Delisted 13 Dec 77; contains $\beta$ -naphthylamine
15630 (Ca)	D&C Red 11 (Ca)		
15630 (Ba)	D&C Red 12 (Ba)	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81 for ingested and external use
15630 (Sr)	D&C Red 13 (Sr)		
15850	D&C Red 7 (Ca)	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81 for ingested and external use
16185	FD&C Red 2	Allowed: Annex III Part 2	Delisted 2 Dec 76
45170	D&C Red 19	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81; 1.3% max. allowed in lip products
15880	D&C Red 34	Allowed: Annex III Part 2	Permanently listed for external use
45370	D&C Orange 5	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81; 6% allowed in lipsticks
45380	D&C Red 21	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81 for ingested and external use
45410	D&C Red 27	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81 for ingested and external use
45425	D&C Orange 10	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81 for external use only

Table 19.1 (cont.)

Colour index no.	Common name	Status under EEC Cosmetics Directive	Status under US FDA
45430	FD&C Red 3	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81 for ingested and external use
77491	Iron oxide	Allowed: Annex III Part 2	Provisionally listed for ingested/external and eye area use
12075	D&C Orange 17	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81; 5% max. allowed for lip products
15510	D&C Orange 4	Allowed: Annex III Part 2	Permanently listed for external use
15985	FD&C Yellow 6	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81 for ingested and external use
19140	FD&C Yellow 5	Allowed: Annex III Part 2	Provisionally listed 31 Jan 81 for ingested and external use
77891	Titanium Dioxide	Allowed: Annex III Part 2	Permanently listed for ingested/external and eye area use
26100	D&C Red 17	Provisionally allowed: Annex IV Part 2	Permanently listed for external use
77163	Bismuth oxychloride	Provisionally allowed: Annex IV Part 2	Permanently listed for ingested/external and eye area use
15585 (Ba)	D&C Red 9 (Ba)	Provisionally allowed: Annex IV Part 2	Provisionally listed 31 Jan 81; 3% max. allowed in lip products
15800	D&C Red 31	Provisionally allowed: Annex IV Part 2	Permanently listed

Difficulties may be encountered when using bromoacid dyestuffs in obtaining entirely homogeneous dispersions of these dyes in the lipstick mass and this could result in shade variations in lipsticks. Novel compositions have been disclosed<sup>1</sup> which aim to overcome these difficulties and to ensure that the colours of the lipsticks sold are identical with those conferred by them on the skin. These compositions comprise amine salts of bromoacid dyestuffs and fatty substances which are at least partial solvents for such components. The amines employed in



the preparation of these compositions are selected from non-aromatic primary, secondary or tertiary monoamines, in which the groups attached to the nitrogen atom contain at the most 6 carbon atoms. Some of them may contain hydroxy radicals, others may be heterocyclic compounds. Specifically mentioned in patent claims have been triethanolamine, diethanolamine, 2-amino-2-methylpropanediol-1,3, monoisopropanolamine, trihydroxymethylaminomethane, diglycolamine and morpholine.

To prepare these compositions the bromoacids are dispersed in a fatty material, preferably soya lecithin, and the amine is added with mixing. The resulting mixture may be optionally heated and allowed to cool. The amine salts of the bromoacid dyestuffs are then dissolved in a mixture of waxes (for example carnauba wax, beeswax and ozokerite) and oils (for example various lanolin derivatives and vaseline).

*Non-eosin staining dyes* reported by Wilmsmann<sup>2</sup> are an interesting development in view of the restrictions applying to some of the fluorescein derivatives. It is claimed that water-soluble FD&C and D&C dyes, which are useless in lipsticks, when converted to the free sulphoacid form become water-insoluble, lipophilic, and suitable for use as staining dyes in lipsticks, covering a wide colour range.

*Pigments.* Both inorganic and organic pigments and metallic lakes are used to give intensity and variation of colour. When selecting lakes the possibility of reaction with the base, for example soap formation with free fatty acid, must be borne in mind.

*Titanium dioxide*, often used at levels up to 4 per cent, is the most effective white pigment for obtaining pink shades and giving opacity to the film on the lips. However, the use of titanium dioxide requires great care in the grade of material selected (anatase or rutile) and the surface treatment it has received to make it lipophilic, and also in the method of incorporation, if unexpected troubles such as oily exudation, streaking, dullness and coarse texture are to be avoided.

Two D&C colours, D&C Red No. 36 and D&C Orange No. 17, are so insoluble in both water and oil that they may be considered as pigments, although they are not in the form of metallic lakes. Similarly the amount of staining dye used often exceeds the solubility in the base and the insoluble portion will act as a pigment.

*Lakes* of many of the D&C colours with metals such as aluminium, barium, calcium and strontium are potential pigments for lipsticks. However, some strontium and zirconium lakes have to be avoided in most EEC countries, as they are banned. Aluminium lakes are not usually favoured because of their lack of opacity, but this very property would seem to suggest their use in transparent lipsticks.

The following lakes are considered to be the most useful lipstick colorants:

Calcium lakes of D&C Reds Nos<sup>7</sup> 7, 31 and 34

Barium lakes of D&C Red No. 9 and D&C Orange No. 17

Aluminium lakes of D&C Reds Nos 2, 3, and 19 and FD&C Yellows Nos. 5 and 6

When the parent D&C colour is subject to restriction the lakes are also restricted in the same way.

As noted above, pigments and lakes are used at levels between 8 and 10 per cent or perhaps over a wider range, say 5–15 per cent.

Iridescent lipsticks utilize either mica coated with titanium dioxide or bismuth oxychloride at levels of up to 20 per cent, depending upon the effect desired.

### Base

Apart from the colour, the quality of the lipstick during manufacture, storage and use will be determined for the most part by the composition of the fatty base. This quality is largely concerned with the rheology of the mixture at various temperatures. For instance, during manufacture (usually while warm) it must be possible to mill and grind the mass, and to pour and mould it while holding the insoluble colours evenly dispersed without settling. In the moulds it must set quickly with a good surface and good release properties. During shelf life and usage life the stick must remain rigid and stable, and generally in good condition. In use the stick must soften sufficiently in contact with the lips, and be sufficiently thixotropic to spread on the lips to form an adherent film which will not smear nor, ideally, transfer to cups or glasses.

*Dyestuff Solvents.* Although all the base ingredients must contribute to the physical and rheological properties, there is the additional requirement that some part of the base must act as the necessary solvent for the staining dyes. Many of the normal fatty materials which might be considered for use in the base are too non-polar to dissolve the dyestuffs, and it is convenient to consider first those ingredients which do have solvent properties for eosin, and which must form some part of the base.

Table 19.2 details the solubility of eosin in a number of fatty or lipophilic materials, not all of which could be included in lipsticks, and serves as a starting point for considering this type of material. In general terms, vegetable oils have the greater solvent power for eosin but suffer from degradation properties. Mineral oils are more stable but have poorer solvent properties.

*Castor oil* is a traditional material for dissolving bromoacid and it owes this property to its high content of ricinoleic (hydroxyoleic) acid, which is unique among natural oils. Its other properties include a high viscosity, even when warm, which delays pigment settling and the oiliness which helps with gloss and emollience, although too high a quantity causes drag during application and an unpleasant greasy film. As much as 50 per cent has been used, but a better quantity is probably about 25 per cent. The disadvantages of castor oil include an unpleasant taste and potential rancidity.

*Fatty alcohols*, four of which are included in Table 19.2, can be seen to have some solvent power for dyestuffs. Any of those quoted (lauryl—C<sub>12</sub>, myristyl—C<sub>14</sub>, stearyl—C<sub>18</sub>, oleyl—C<sub>18</sub> unsaturated), or cetyl—C<sub>16</sub>, could be used according to the consistency contribution required. However, synthetic isomers of cetyl alcohol, such as the hexadecyl alcohol manufactured by the Enjay Chemical Co., which is actually  $\beta$ -hexyldecyl alcohol, are available. It is claimed that hexadecyl alcohol is a good solvent for bromoacid dyes, that lipsticks



Table 19.2 Solubility of Eosin in Various Solvents<sup>3</sup>

Solvent	Eosin in solution at 20°C (% approx.)
Polyethylene glycol 4000	12.0
Polyethylene glycol 1500	10.0
Polyethylene glycol 400	10.0
Hexa-ethylene glycol	9.0
Phenyl ethyl alcohol	8.0
Diacetone alcohol	6.5
Benzyl alcohol	6.0
Tetraethylene glycol	5.7
Hydroxycitronellal	4.5
Citral	4.5
Triethylene glycol	4.0
Acetone	3.5-4.0
Diphenyl ketone	3.5-4.0
Diethylene glycol	2.5
Terpineol	1.9
Ethylene ricinoleate	1.9
Cyclohexanol	1.6
Ethyl ricinoleate acetate	1.4
Oleyl alcohol	1.0
Ethylene glycol	1.0
Lauryl alcohol	0.75
Myristyl alcohol	0.57
Stearyl alcohol	0.5
Glycol oleate acetate	0.4
Cocœa butter	0.35
Lauric acid	0.3
Myristic acid	0.3
Cetyl acetate	0.3
Ethyl oleate	0.3
Castor oil	0.2-1.7
Ethyl stearate	0.2
Glycol oleate	0.1

Table 19.3 Solubility of Eosin in Various Compounds

Compound	Solubility (% at 20°C)
Isopropyl myristate	0.2
Oleyl alcohol	1.0
Diethyl sebacate	1.3
Di-isopropyl adipate	1.4
Propylene glycol	1.6

containing it can be applied with very little drag and do not bleed or smear and that its presence lessens any tendency to develop an unpleasant taste during storage.

*Esters* of various kinds have been proposed, including lower alkyl esters of fatty acids and dibasic acids such as adipic and sebacic, short chain-length acid esters of fatty alcohols, mono-, di- and mixed esters of glycol or glycerol. They have no specific virtues other than that they are lipophilic liquids of low oiliness giving lubricating and emollient effects with some dye solvent properties (see Tables 19.2 and 19.3). If too much is used the stick may sweat.

*Glycols* with two hydroxy groups are more polar than (for example) the fatty alcohols and might be expected to be better dye solvents; Table 19.2 shows this to be so. However, glycols are not particularly miscible with fatty materials and are of little importance.

*Polyethylene glycols* (Carbowaxes) also have good dyestuff solvent power (see Table 19.2). The solvent power correlates to some extent with water solubility and this is a detraction. However, correctly chosen, these materials would seem to have considerable possibilities in lipstick formulation.

*Monoalkanolamides*, for example Loramine Wax 101,<sup>4</sup> have been claimed to have good dye-dissolving properties and to have an advantage in that they have no action on the plastics material of the lipstick cases. It is suggested that dye can be pre-dissolved in Loramine Wax and kept as a concentrate for more convenient dispensing when required.

*Other solvents* proposed for bromoacids include Polychol 5 and Volpo N.3 (Croda Ltd); Polychol 5 is an ethylene oxide derivative of lanolin alcohols, while Volpo N.3 is a polyoxethylene oleyl ether. Both compounds are compatible with oleyl alcohol (for example Novol) and castor oil, and can be used in combination as a bromoacid solvent mixture consisting, for example, of

	<i>parts by weight</i>
Polychol 5 or Volpo N.3	10-20
Oleyl alcohol	20-10
Castor oil	40

to solubilize 1-3 parts by weight of bromoacid dyes.

Wetting agents, once used to some extent to 'solubilize' the dyestuff, nowadays find no application in lipsticks.

*Other Base Ingredients.* It will have been observed that few of the ingredients quoted so far have had the high melting points or hardness which are required to give satisfactory moulding properties, that is, quick setting and good release with a glossy surface and a rigid stick. This function is generally fulfilled by the inclusion of waxes or wax-like materials.<sup>5</sup> The importance of melting point as a criterion is emphasized by Gouvea,<sup>6</sup> who achieves the desired melting characteristics by working to a 'carnauba equivalent'.

*Carnauba wax* is a very hard vegetable wax used for raising the melting point, imparting rigidity and hardness and providing contraction properties in the moulding process.

*Candelilla* is another hard vegetable wax serving the same functions as carnauba wax but has a lower melting point and is less brittle.



*Amorphous hydrocarbon waxes* in mineral oil, for example ozokerite wax, give a short-fibred texture to the product.

*Petroleum-based waxes*, for example microcrystalline wax, are also used to modify the rheology of the product.

*Beeswax* is the traditional stiffening agent for castor oil, but it can give a grainy and dull effect if used in large quantities.

*Cocoa butter* might be thought an ideal material owing to its sharp melting point just below the human body temperature, which makes it so useful in other products. However, it cannot be used alone since it does not have all the required properties, and used in too large quantities it will cause the stick to 'bloom'.

Other more or less wax-like materials are hydrogenated vegetable oils which are more solid and less prone to rancidity than the unhardened oils. Of particular interest is hydrogenated castor oil, which in addition to wax-like properties still retains eosin solvent properties. Some other softer materials will be found to be of use in lipstick manufacture.

*Lanolin and lanolin absorption bases* are very useful ingredients up to about 10 per cent by virtue of their emollient properties. They are claimed to have eosin solvent properties and act as binding agents for the other ingredients, tending to minimize sweating and cracking of the stick and acting as plasticizers. Absorption bases in particular are recommended to enhance the gloss on the lips.

*Petroleum jelly* and the more viscous *paraffin oils* may be used to adjust consistency, act as lubricants and improve spreading properties. Large amounts tend to impair the adhesion properties and can be difficult to blend if much polar material such as castor oil is present.

*Lecithin* is another possible component which acts as a dispersing agent for pigments, in addition to facilitating the application of the lipstick and improving the adhesion to the lips.

*Silicone waxes* are included in improved lipstick compositions disclosed by Dow Corning.<sup>7</sup> These comprise essentially a wax (of which at least 15 per cent by weight is a silicone wax), a cosmetic solvent and a colouring agent. The preferred silicone wax compounds include organosilicon block copolymers, hydrocarbon silicone copolymers, and silphenylene copolymers.

The silicone waxes are largely insoluble in water, ethanol and organic fats and oils, and the compositions containing them have improved viscosity, stability and a sharper melting point. They are said to be superior to compositions with no silicone wax, retaining their form plus all the properties of the silicone wax over a wider temperature range. They are also said to have good shelf stability.

From the foregoing description of the properties of the various materials it will be seen that no one or two materials are able to provide all the properties and qualities required in a lipstick; this serves to explain the almost invariable complexity of lipstick formulae, which is evident in the examples given below.

### *Perfumes*

Special attention must be paid to the choice of perfume, which is frequently used in relatively high amounts (2-4 per cent), from the point of view of consumer

acceptance and freedom from irritation. The perfuming of lipsticks was discussed by Vasic<sup>8</sup> and was also the subject of an article in *Dragoco Report*.<sup>9</sup>

Perfumes selected should mask the fatty odour note of the base and should be non-irritant to the lips. Since the consumer is likely to apprehend the perfume in the mouth as well as the nose the flavour must be considered as well as the odour. Perfumes should be stable and compatible with the other constituents of the lipstick base.

The preferred perfumes are of the light floral or light sophisticated type with no single essential oil predominating. Rose alcohols and esters are often used as well as other essential oils, preferably terpeneless, such as aniseed, cinnamon, clove, lemon, orange and tangerine, although definite fruity flavours have not proved very popular when tried in lipsticks. Geranium, patchouli and petitgrain oils are not considered to be suitable components of lipstick perfumes.

### Example Formulations

The following examples illustrate some of the many lipstick formulations in existence.

	(1)
	<i>parts</i>
Loramine O.M. 101	20.0
Lanolin	10.0
Cocoa butter	5.5
Refined beeswax	4.0
Ozokerite	18.0
Carnauba wax	4.2
Oleyl alcohol	7.0
Mineral oil (high viscosity)	29.3
Perfume	2.0 or <i>q.s.</i>
Insoluble lake	10.0 or <i>q.s.</i>
Bromo acid (or eosin salt)	2.0

(Cited from *Loramine Wax O.M. 101*, Dutton and Reinisch)

The use of silicones in lipstick base formulations is illustrated by example 2.

	(2)
	<i>per cent</i>
Castor oil or bromoacid solvents	30.0
Mineral oil	15.0
Beeswax	15.0
Paraffin	10.0
Carnauba wax	10.0
Ceresin wax	10.0
Union Carbide L-45 Silicone Fluid (1000 cS)	10.0
Perfume-flavour compound	<i>q.s.</i>

(*Union Carbide Bulletin CSB 45-176 4/64*, Formula C-106)

Two lipstick base formulae containing hexadecyl alcohol as bromoacid solvent, from technical bulletins of the Enjay Chemical Co., are given in examples 3 and 4.



	(3) <i>per cent</i>
Hexadecyl alcohol	26.0
Castor oil	20.0
Propylene glycol monolaurate	15.0
Anhydrous lanolin	2.0
Ceraphyl 28	5.0
Candelilla wax	32.0

*Procedure:* Heat the ingredients together until molten. Add 7 parts of pigment to 92 parts of base, and run the resultant mixture through a heated mill. Add 1 part of perfume and run the final blend into the moulds.

	(4) <i>per cent</i>
Hexadecyl alcohol	44.0
Butyl stearate	2.0
Isopropyl palmitate	3.5
Anhydrous lanolin	7.0
Petrolatum	12.0
Candelilla wax	11.0
Carnauba wax	11.0
Stearic acid, triple pressed	8.0
Cetyl alcohol	1.5
Nordihydroguaiaretic acid (antioxidant)	0.02
Citric acid	0.006

Lipstick formulae using Dehydag products (examples 5-8 below) have been suggested by Henkel.

	(5) <i>per cent</i>
Stearyl alcohol	7.00
Beeswax (bleached)	7.00
Stearic acid	1.75
Paraffin wax (MP 72°C)	12.25
Anhydrous lanolin	2.80
Carnauba wax	2.80
White mineral oil	1.40
Comperlan HS	20.00
Eutanol G	45.00

*In addition:*

Bromoacid	1.50
Pigment colour	6.00

*Procedure:* Dissolve the bromoacid in Comperlan HS on the water bath at approximately 95°C. Blend or dissolve the pigment colour in Eutanol G. Melt the remaining fatty and waxy substances on the water bath and add to the two above-mentioned compounds. Pass the finished mixture through a roller mill while liquid, and finally pour into moulds. The finished sticks are then passed quickly through a small flame.

## Coloured Make-up Preparations

(6)	
	per cent
HD-Eutanol	20.0
Castor oil	24.8
Beeswax (bleached)	5.0
Carnauba wax	8.0
Ozokerite wax (70°-72°C)	11.0
Candelilla wax	3.0
Lanolin, anhydrous	8.0
Liquid paraffin	13.0
Bromoacid	0.2
Pigment colour	7.0
Antioxidant, perfume	q.s.

(7)	
	per cent
Eutanol LST	10.0
Castor oil	47.0
Cetyl alcohol	1.5
Beeswax (bleached)	5.0
Candelilla wax	12.0
Lanolin anhydrous	13.0
Bromoacid	0.2
Pigment colour	11.3
Antioxidant, perfume	q.s.

(8)	
	per cent
Eutanol LST	20.0
HD-Eutanol	14.0
Cetyl alcohol	5.0
Castor oil	20.0
Beeswax (bleached)	6.0
Lanolin, anhydrous	14.0
Ozokerite wax (70°-72°C)	15.0
Carnauba wax	5.0
Bromoacid	1.0
Antioxidant, perfume	q.s.

**Procedure:** Dissolve the bromoacid in the mixture of HD-Eutanol and Eutanol LST at 80°C. Melt the remaining fatty substances on a water bath, add the appropriate pigments and add the whole mixture to the bromoacid solution. Add perfume and antioxidant at 60°C. Pass the prepared mass two or three times through a roller mill, then remelt and pour into moulds at 80°C. For lipsticks without pigments, no rolling is necessary.

A deep-staining lipstick formula put forward by Croda, includes a polyoxyethylene oleyl ether, Volpo N.3 (Croda) as a bromoacid solvent, used in conjunction with oleyl alcohol and castor oil (example 9). The resultant lipstick is said to possess superior gloss (without flaming), plasticity and moulding properties. It is further claimed that the combination of Volpo N.3 and Novol in



the stick provides pigment-dispersing action and helps to prevent titanium dioxide streaking.

	(9) per cent
Volpo N.3	10-20
Bromoacid dyes	1-3
Novol	20-10
Castor oil	40
Candelilla wax	10
Carnauba wax	10
Perfume	q.s.
Insoluble pigments and lakes	q.s.

*Procedure:* Dissolve the bromoacid in the Volpo N.3 with heating to facilitate solution; add the oleyl alcohol to the resulting solution, which remains clear. Now add the castor oil and the waxes at an elevated temperature; it is claimed that the bromoacid will not precipitate. Add the pigments and mill the mass.

Croda have also developed a series of synthetic waxes known as Syncrowaxes, which can be used instead of naturally occurring waxes in stick and salve-based cosmetics. Three lipstick formulae are given below:

	(10) per cent	(11) per cent	(12) per cent
Paraffin wax 145	—	3.0	—
Syncrowax HRC	5.0	—	5.0
Paraffin wax 125/130	—	—	13.0
Syncrowax PRLC (or ERLC)	6.0	6.0	7.5
Syncrowax HGLC	9.0	6.0	7.5
Castor oil	54.4	66.1	57.2
Crodamol IPP	15.0	6.3	—
Mineral oil	—	—	5.0
Timica brilliant gold	—	10.0	—
BHT	0.1	0.1	0.1
Perfume	0.5	0.5	0.5
Colour blend (in castor oil)	10.0	2.0	4.2

(cited from *Syncrowaxes in Cosmetics*, Croda Chemicals Ltd)

*Procedure:* Melt the waxes and oils and heat to 85°C. Remove from heat source; add colour blends, Timica and preservative. Re-warm with stirring to 70°C. De-aerate in 80°C water bath for 20-30 minutes. Add perfume. Pour into slightly warm mould. Leave to set.

It is also claimed that a softer stick with increased pay-off can be produced by replacing some of the PRLC or HGLC with HRC. The Crodamol IPP should be replaced by Crodamol OP or Crodamol ML when the final product is to be packed in polystyrene.

Example 13 is a lipstick based upon a non-self-emulsifying grade of glyceryl monostearate (Tegin 515).

	(13) per cent
Tegin 515	42.0
Castor Oil	36.0
Bromoacid	5.0
Mineral oil	6.0
White petroleum jelly	4.0
Carnauba wax	4.0
Lanolin	3.0

(Cited from *Emulsifiers*, Goldschmidt Chemical Corp.)

Another deep-staining lipstick formula suggested by Croda contains:

	(14) per cent
Polychol 5	20.0
Novol	20.0
Candelilla wax (68°/70°C)	10.0
Carnauba wax (85°C)	10.0
Castor oil	40.0

*In addition:*  
Indelible colours 1-3

Lipstick formulae proposed by Fishbach<sup>10</sup> contained:

	(15) Deep stain lipstick per cent	(16) Creamy lipstick per cent
Castor oil	60.0	65.0
Lanolin	5.0	10.0
Isopropyl myristate	—	5.0
Propylene glycol monoricinoleate	10.0	—
Polyethylene glycol 400	5.0	—
Beeswax	7.0	7.0
Candelilla wax	7.0	7.0
Carnauba wax	3.0	3.0
Ozokerite	3.0	3.0
<i>In addition:</i>		
<i>p</i> -Hydroxybenzoic acid propyl ester	0.2	0.2
Bromoacids	3.0	3.0
Colour lakes and pigments	12.0	12.0

### Manufacture of Lipsticks

Lipstick manufacture is by no means a simple operation. The method employed will depend to some extent on the formulation and the plant available.

In general, the manufacture of lipsticks consists of three stages: (i) the preparation of component blends, that is the oil blend, colour dispersion and



wax blend; (ii) the blending of these intermediates to form the lipstick mass; (iii) the moulding of the lipstick mass into sticks.

### *Preparation and Blending*

The colours, that is the bromoacids, pigments and lakes, may be (a) blended separately with appropriate constituents of the base mixture, or (b) dispersed in the complete lipstick base—a procedure which is seldom employed, except when a proprietary complete base is used.

The object of the operation is to produce a completely homogeneous dispersion of colorants conducive to the preparation of a smooth stick. Although the colours used are usually received in a finely ground form, they nevertheless tend to be difficult to wet with the fatty mixture and it is necessary to employ some type of milling or grinding process. A variety of types of mill—ball mills, sand mills, roller mills, colloid mills, edge or end runner mills and others—has been used. It is desirable that the portion of the base used should be a plastic semi-solid during the milling and it may sometimes be necessary to mill under warm conditions.

The soluble dyes are first blended with the dye-solvent materials, using heat if necessary to secure solution. If solution is complete, this portion can be left on one side while the pigments are dispersed, but if not, the dye blend must at some point be ground with the pigment dispersion.

In preparing the pigment dispersion the colours are first milled with those constituents most likely to wet them, for example lanolin, polyglycol compounds or the like, and only later are the completely non-polar materials added.

The remainder of the base materials, for example the high melting waxes which may not have been used in the dye or pigment blends, are melted and blended together and then blended with the others. In order to secure an intimate mix a final milling operation is desirable.

Jakovics<sup>11</sup> suggested that vacuum processing of the lipstick melt facilitates dispersion of pigments by removing the film of adsorbed gas on the pigment particles, which otherwise acts as a barrier and causes incomplete wetting. It has been established elsewhere that petroleum jelly, for instance, will dissolve 10 per cent air by volume at 90°C but only 5 per cent at 20°C. If, therefore, the mix has been held at high temperature and allowed to absorb its full quota of air and then cools, the air is not released until the viscosity of the mass is too great to allow it to escape completely and it may collect round the pigment particles, displacing the oil and giving the effect of incomplete wetting.

The manufacture of lipsticks, therefore, should be conducted at the lowest temperature convenient for the process and it is very desirable that when mixing is complete the mixture should be transferred to a jacketed closed vessel in which it can be kept fluid and stirred gently while sufficient vacuum is applied to remove all occluded air. When the air has been removed, vacuum is shut off, the mass is stirred and the perfume is added and mixed through.

The mass is then ready for moulding immediately or can be set into blocks which are stored and moulded as required.

Lipstick manufacture in modern plant was also discussed by Daley.<sup>12</sup> In one of the two methods described, the wax blend is prepared and stored in a molten state while all constituent oils are metered into a storage tank fitted with a



suitable mixer. A part of the oil blend is then mixed with the dry pigments in a pre-mix pan attached to a sand mill and the resultant slurry passed through the sand mill into the steam pan fitted with a combined mixer and pump. The remainder of the oil blend is then passed through the pre-mix pan and sand mill to flush any residual colour.

The wax blend is metered into the steam pan simultaneously and thoroughly mixed with the other constituents. The resulting base is tested for shade, perfume and flavour are added, and the lipstick mass is pumped into storage trays, using the mixer as a pump.

### *Moulding*

When required, the lipstick mass is gently remelted in a small jacketed pan and agitated slowly for about 30 minutes, in order to allow any entrapped air to rise to the top and thus prevent pinholing of the finished product. The molten mass is then run into moulds for casting.

Moulds are usually filled to excess to prevent the formation of a depression in the centre of the stick. After they have been allowed to stand to allow this excess to congeal, the latter is scraped off and the mould is then carefully cooled to allow the mass to set, without overcooling which would delay the removal of the stick from the moulds.

The moulds are made of brass and aluminium. They may be of the vertical split type (with a split down the centre to allow for the easy removal of the sticks) or the automatic ejection type. A cooling cabinet is required with the split moulds. A preheating device is stated to be useful in that the moulds can then be raised to a temperature of about 40°C before filling with the lipstick mass, thus avoiding 'flow marks' which would otherwise be visible on the moulded stick. With the water-jacketed automatic ejection mould a cooling cabinet is unnecessary. The jacket is filled with warm water prior to pouring the lipstick mass, and then the cold water is introduced and kept running to chill the lipstick mass. When cooled, the moulds are opened and the sticks are pushed out automatically or with a rubber-covered finger.

It has been stated that no type of automatic ejection mould (whether air-cooled or water-cooled) will produce a good 'bullet'-shaped stick because a small ring or ridge is left near the tip of the stick caused by the thickness of the metal edge of the ejection plunger. It is considered that such moulds are best for the wedge-shaped stick. After moulding, the sticks should be stored for about a week before being filled into the lipstick holders, after which they are subjected to a procedure known as 'flaming', in which the lipstick is passed rapidly through a small gas flame to melt the surface layer, in order to remove any surface spots and to produce a bright, smooth and glossy surface. In large plants the procedure is fully mechanized.

A glazing apparatus for meltable products such as lipsticks was claimed in one British patent,<sup>13</sup> and a process for the manufacture of multi-coloured lipsticks was described in another.<sup>14</sup> A comparison between split moulding and automatic ejection moulding has been drawn by Dweck and Burnham.<sup>15</sup>

To overcome the problem of holding stocks and blending multiple materials, a lipstick base can be purchased ready blended from supply houses. This has



adequate solvent powers for eosin dyes and it is only necessary to dissolve the dyes, mill in the pigments, melt, add perfume and mould.

### Transparent Lipsticks

In a US patent published in 1964<sup>4</sup> and assigned to Yardley of London, a lipstick was described which does not contain any insoluble opaque pigments or lakes, but instead uses soluble or solubilized dyes. This allows light to shine through it, giving it a sparkle. The staining action of these dyes is enhanced by the use of suitable solvents such as a monoalkylolamide of mixed fatty acids (Loramine OM-101) or dipropylene glycol methyl ether.

Water-soluble dyes may be used for additional colour effect or instead of oil-soluble dyes. The compatibility of such dyes with the vehicle may be improved by using anhydrous lower alcohols such as ethanol or isopropanol as co-solvents. If used in amounts between 2 and 10 per cent, these alcohols are claimed to control syneresis, thereby improving the stability of the lipstick.

For moulding lipstick disclosed in the patent, polyamide resins with a molecular weight range of about 2000 to 10 000 are used, or preferably a blend of a solid polyamide resin of molecular weight of 2000–10 000 together with a small proportion of a liquid polyamide resin of a molecular weight of 600–800 to give a solid stick. Representatives of these resins are the Versamids of General Mills Inc. or the Omamids of Olin Mathieson Chemical Corporation.

To ensure that the resulting sticks are not brittle, these materials are used with softening agents such as the lower aliphatic alcohols in conjunction with other polyamide solvents such as fatty acid esters, for example the glycol ester of higher fatty acids, particularly those between C<sub>12</sub> and C<sub>18</sub>. Special mention is made in this connection of propylene glycol monolaurate, polyethylene glycol (400) monolaurate, castor oil, lauryl lactate and fatty alcohols. Four formulations are given in the patent to illustrate transparent lipsticks. In one of the examples, the lipstick composition is as follows:

	(17) per cent
Polyamide resin (average MW 8000)	20.0
Polyamide resin (average MW 600–800)	5.0
Propylene glycol monolaurate	28.0
Castor oil	12.6
D&C Red 21	0.3
Lanolin alcohols	8.0
Dipropylene glycol methyl ether	10.0
Ethoxylated lanolin alcohols (5 mol Et <sub>2</sub> O)	10.1
Anhydrous isopropanol	5.0
Perfume	1.0

### Lip Salves

The lip salve is used, not for decoration, but for protection against exposure to cold, in winter or sub-arctic conditions. The requirement is simply for a fairly substantial, flexible, adherent, moisture-resistant film on the lips and there is no

need for staining dyes and hence none for dye-solvent materials. The base can be made largely from mineral oils, jelly and waxes, but it is necessary to include a proportion of a more hydrophilic material to promote adherence, perfume blending and general properties. In some cases a small amount of antiseptic can be added, and occasionally some users will prefer a coloured salve in which case the colour is provided by a small amount of oil-soluble dye or dispersed lake not necessarily of a staining type. Suitable base formulae are given in examples 18 and 19.

	(18)
	<i>per cent</i>
Paraffin wax	30.0
White petroleum jelly	35.0
Technical white oil	20.0
Beeswax	15.0

	(19)
	<i>per cent</i>
Cetyl alcohol	5.0
HD-Eutanol	30.0
Beeswax, white	25.0
Paraffin wax, 52°C	15.0
Liquid paraffin	25.0

A lip-protective pomade from a Union Carbide Bulletin (formula C-105) is made up as follows:

	(20)
	<i>Parts</i>
A Mineral oil	25.0
Petroleum	7.0
Ceresin wax	24.0
Beeswax	7.0
Atlas G-2859	3.5
Tween 60	0.5
Union Carbide L-45 Silicone Fluid (1000 cS)	5.0
B Water	28.0
Preservative	<i>q.s.</i>
C Perfume	<i>q.s.</i>

*Procedure:* Heat A to 65°C; heat B to 70°C and add to A with stirring. Continue stirring and add perfume at 50°C. Pour into heated moulds.

A lip salve which is used as a decorative item has become known in recent years as a lip gloss. This popular item can be applied to the lips without other make-up or over the normal lipstick. Lip gloss preparations take the form of a stick, or a salve for finger application, or a liquid for roll-on or brush application. These compositions may also contain a pearl pigment to confer a transparent sheen in addition to the normal gloss.



<i>Finger-applied lip gloss</i>	(21)
	<i>per cent</i>
Syncrowax HRC	5.0
Syncrowax HGLC	5.0
Timica silk white	8.0
Fluilan	8.0
Liquid paraffin	74.0
Perfume, antioxidant	<i>q.s.</i>

(Cited from *Syncrowaxes in Cosmetics*, Croda Chemicals Ltd.)

*Procedure:* Melt waxes. Add Fluilan and liquid paraffin. Disperse the Timica with low shear and de-aerate at 65°C in a water bath. Fill off at 50°C.

<i>Liquid lip gloss</i>	(22)
	<i>per cent</i>
Polybutene	30.0
Lanolin oil	20.0
Mineral oil	24.8
Oleyl alcohol	25.0
Saccharin	0.2
Perfume, antioxidant	<i>q.s.</i>

### Liquid Lipsticks

In addition to conventional lipsticks, other methods for applying colour to the lips have also been proposed. Several patents have been issued, dealing for example with lip paint or paste applicator, lipstick brush applicator and others.<sup>16-18</sup> In 1959, a roll-ball lipstick was introduced in the USA.<sup>19</sup>

Liquid lipsticks have been developed with the object of providing more permanent films than can be obtained with conventional lipsticks. These liquid lipsticks consist of alcoholic solutions of alcohol-soluble dyes, suitable film-forming resins and plasticizers. The solvent employed is ethyl alcohol; the film-formers include ethyl cellulose, polyvinyl alcohols and polyvinyl acetate and, as plasticizers, triethyl citrate, dioctyl acetate, methyl abietate or polyethylene glycols have been used. The dyestuffs employed are alcohol-soluble halogenated fluoresceins and also other alcohol-soluble dyes.

The dyes and perfume are dissolved in the alcohol before combining with the film-former and other constituents of the preparation. The resulting solution is then filtered. The disadvantage associated with the use of these alcoholic preparations is 'smarting' produced on the lips on application. Excessive drying of the lips may also result. Persons affected in this way should discontinue the use of these preparations, apply occasionally a 40 per cent solution of aqueous glycerin and use an emollient lip salve:

The manufacture of liquid lipsticks based on ethyl cellulose is covered by a patent<sup>20</sup> from which the following illustrative examples are quoted.

	(23)
	<i>per cent</i>
Ethyl cellulose	3.1
Ethyl alcohol	68.4
Petroleum ether	20.0
Hydrogenated methyl abietate	7.5
Rhodamine	1.0

	(24)
	<i>per cent</i>
Ethyl cellulose	3.06
Bleached wax-free shellac	4.94
Ethyl alcohol	65.00
Petroleum ether	14.66
Hydrogenated methyl abietate	12.00
Fuchsine	0.30
Saccharin	0.04

According to the patent quoted, accessory agents such as perfumes, preservatives, antioxidants, etc., may be added. The preparations are preferably clear and transparent and are of a viscosity ranging between 3 and 500 cP, while best results are obtained in a range between 20 and 50 cP.

Another lipstick formulation put forward by Janistyn<sup>21</sup> had the following composition:

	(25)
	<i>per cent</i>
Ethyl cellulose	1.0
PVA (high viscosity)	3.0
Triethyl citrate	1.0
Alcohol-soluble lanolin	0.5
PEG 400	1.0
Isopropyl alcohol, pure	40.0
Bromoacid dyes	3.0
Flavour	0.5
Ethanol	50.0

## ROUGE

### Introduction

Rouge is one of the oldest types of make-up preparation used to apply colour to the cheeks. The Hittites used cinnabar for this purpose, the ancient Greeks coloured their cheeks with a root and the Romans were known to use a seaweed to impart a rosy tint to pale cheeks.

In Elizabethan days, red ochre, vermilion and cochineal were used as rouge, as well as extracts of sandalwood and brazilwood. Cochineal was still the standard material for rouge in the eighteenth century. In the late nineteenth and early twentieth centuries liquid rouge made from ammonia and carmine was popular, while in theatrical rouges the red pigment carthamine and the aluminium lake of the dye obtained from brazilwood were extensively used to enhance the facial colouring of actors under the glare of footlights on the stage.

In the early 1920s liquid rouges still consisted of carmine and ammonia, whereas grease rouges were composed of carmine dispersed in a tallow and ceresin base. Also available was dry rouge, prepared by mixing carmine solution and eosin with pumice, chalk and gum arabic. Another preparation employed alloxan in a wax base. The precursor of the modern compact rouge at the beginning of the twentieth century was a small book of thin paper leaves coated



with various shades of red and white powder, which were detached and rubbed on the cheeks.

Such are the vagaries of the cosmetics industry that the term 'rouge' has become old-fashioned and somewhat passé. The more modern description currently in vogue is 'blusher', but in basic formulation terms the two are synonymous and, for the sake of continuity, we will continue with the older name. If a distinction had to be made between the two terms, it could be said that a rouge produces a bright red colour, whereas a blusher produces a duller, more brown effect. The preparations are available in liquid, cream and solid forms, of which the pressed powder or dry rouge is the most popular.

### **Dry Rouge (Compact Rouge)**

Compact rouge differs from an ordinary compact powder in being more highly tinted. The desirable properties of compact rouges are therefore virtually identical with those of ordinary compact powders (Chapter 18). The finished product must be smooth and free from grittiness and should be easy to apply; it should have good adhesion to the skin and a good covering power. In addition however, because these products are highly tinted and may be conspicuous if not adequately blended, it is essential that component colours are distributed evenly throughout the product. Care must also be taken to ensure that any component liable to impart undesirable opacity is used in moderate amount so that it has no deleterious effect on the appearance of the product; consequently zinc oxide is used in compact rouges at lower concentrations than in ordinary compact powders. Similar stipulations apply to components such as kaolin, which by virtue of their hygroscopic nature are liable to produce colour streaking in damp weather.

Compact rouges may thus be manufactured broadly along the lines discussed for compact powders in Chapter 18. In addition, grinding and shade-matching stages are included in the manufacture of compact rouges. The component raw materials must be very finely divided to facilitate their intimate blending as a prerequisite to a uniform distribution of component colours. The constituents are, therefore, intimately mixed and simultaneously ground in hammer mills or attrition mills rather than just blended in ribbon mixers. This operation is carried out in the absence of large quantities of liquids, though the presence of a little water or oil will ensure better colour distribution. The binder is either sprayed into the powders while they are being mixed, or incorporated in dry form.

The raw materials used in the manufacture of compact rouges are talc, kaolin, precipitated chalk, magnesium carbonate, titanium dioxide, zinc stearate, inorganic oxides, certified colorants and perfumes. Zinc oxide, used to increase the adhesion of the rouge, confers opacity to the product and is usually employed in amounts of between 5 and 10 per cent.

Titanium dioxide has an appreciably greater covering power than zinc oxide, and it also gives brighter and more stable colour shades.

Metallic stearates are also essential components of compact rouges and improve the adhesion of the products to the skin. They are nowadays used extensively as dry binders for compact rouges. Amounts employed range between 4 and 10 per cent.

*Compact Rouge Formulation*

Typical compact rouge formulations are given in examples 26 and 27.

	(26) <i>per cent</i>
Talc	48
Kaolin	16
Zinc stearate	6
Zinc oxide	5
*Magnesium carbonate	5
Rice starch	10
Titanium dioxide	4
Colours	6

	(27) <i>per cent</i>
Talc	67.5
Zinc stearate	5.0
Titanium dioxide	4.0
Red iron oxide	11.5
Black iron oxide	0.3
D&C Red 9—barium lake	0.2
Mica coated with titanium dioxide	6.8
Perfume	0.2
Base	4.5
<i>Base:</i>	
Beeswax	12.00
Lanolin	2.00
Mineral oil	86.00

The proportion of colouring matter may vary from  $1\frac{1}{2}$  per cent in the case of the lighter shade to some 6 per cent with the deeper shades.

Colour lakes are the most extensively used pigments because of the wide range of shades that they provide; water-soluble and oil-soluble dyes, on the other hand, are not suitable as colorants in compact rouges.

For medium-red shades Keithler<sup>22</sup> suggested the following blend of pigments:

	<i>parts</i>
D&C Red No. 8 Na lake	2.2
D&C Red No. 19 Al lake	2.0
D&C Red No. 10 Na lake	2.0
D&C Red No. 11 Ca lake	1.1
D&C Red No. 34 Ca lake	0.7

D&C Red Nos 10 and 11 are now delisted but can be replaced by D&C Red No. 9—barium lake.

A variety of binding agents may be employed, as already mentioned in connection with compressed powders. For example Winter<sup>23</sup> has suggested 13–15 per cent of a starch–stearin mixture as binding agent, or alternatively a soap–oil mixture of about 3 per cent sodium stearate or triethanolamine stearate



and 2 per cent oil may be used. However, compact rouges with incorporate water-soluble binders are subject to spotting by water. Consequently, water-repellant binders are preferred and particularly dry metallic stearates. If the latter are used, it will be necessary to employ higher pressures during the compression of powders.

Shade matching may be carried out in a ribbon mixer provided that colour blends are readily available in several concentrations.

In compressing rouges, the powder is pressed into godets by being subjected to a gradually increasing pressure.

### Wax-based Rouge

Wax-based rouges are in many respects similar to lipsticks. Formulae for bases resemble the drier type of lipstick; shape is generally similar although frequently of larger diameter (about 2 cm) and colours are generally of a red or pink shade, although ochres and tans and even white are also used to disguise the contours of the face, when the products are known as 'gleamers'. The use of staining dyes of the bromoacid type is inadvisable, owing to the possibility of inducing eosin dermatitis with the greater exposure to light, and this renders it unnecessary to include any materials specifically for their eosin-solvent properties.

The following are examples of formulae for wax-based rouges:

	(28)
	<i>per cent</i>
Candelilla wax	8.6
Carnauba wax	5.4
Beeswax	4.0
Mineral oil	17.0
Lanolin	2.0
Isopropyl myristate	33.0
Inorganic pigments and colour lakes	30.0

	(29)
	<i>per cent</i>
Carnauba wax	10.0
Paraffin wax	2.5
Ozokerite wax	5.0
Petrolatum	4.0
Mineral oil	68.5
Titanium dioxide	1.9
Red iron oxide	1.0
Bismuth oxychloride	7.0
Perfume	0.1

### Cream Rouge

Two types of cream rouge are available: anhydrous products and emulsified products.

*Non-aqueous Cream Rouge*

Although cream rouges are more difficult to apply than compact rouge, it is claimed that their use gives better results, in that there is no obvious line of demarcation when they are correctly applied. This gives a more natural effect.

An effective anhydrous cream rouge may be prepared to the following formula:

	(30)
	<i>per cent</i>
Petroleum jelly	70.0
Kaolin	30.0
Colour, perfume	<i>q.s.</i>

*Procedure:* Grind the colours with the powder constituent, and then mill this into the warmed fatty base; alternatively, mix all the ingredients and mill in a warmed ointment mill.

Hexadecyl alcohol is claimed to impart a smooth and soft feeling to the skin during application and use, and also facilitates the spreading of a cream rouge in which it is present. Such an anhydrous rouge may, for example, be prepared with the composition given in example 31.<sup>24</sup>

	(31)
	<i>per cent</i>
*Hexadecyl alcohol	27.0
Light mineral oil	23.5
Talc	10.0
Ozokerite	10.0
Carnauba wax	6.0
Titanium dioxide	20.0
D&C Red No. 9 lake	3.0
Perfume	0.5

\*Enjay Chemical Co.

*Procedure:* Melt the constituent waxes and blend them with the remaining constituents; pass through a roller mill and pour into containers.

Of the following formulae for cream rouge selected from the technical literature, example 32 is given by Thomssen.<sup>25</sup>

	(32)
	<i>per cent</i>
White beeswax	5.0
Stearic acid	7.0
Cetyl alcohol	3.5
Petroleum jelly (short fibre)	77.0
Mineral oil	7.5
Colours	<i>q.s.</i>



	(33) per cent
Eutanol G	25.0
Castor oil	45.0
Liquid paraffin	4.0
Beeswax, white	2.0
Ozokerite 70°-72°C	5.0
Carnauba wax	6.0
Candelilla wax	5.0
Pigment colours	8.0
Antioxidant, perfume	q.s.

(Cited from *Cosmetic Preparations based on Dehydag Products*, Henkel).

### Emulsions

Emulsified products are either of the cold cream or of the vanishing cream type.

An emulsified form of cream rouge of the cold cream type may be prepared by the interaction of beeswax with an aqueous solution of borax:

	(34) per cent
Lanolin	5.0
Cocoa butter	5.0
Beeswax	14.0
Liquid paraffin	30.0
Cetyl alcohol	1.0
Water	44.2
Borax	0.8
Colour	q.s.

*Procedure:* Disperse the finely powdered colour base with the melted fats, add the hot aqueous solution of borax at about 75°C (both fats and water being at the same temperature to prevent crystallization), mix well and finally mill.

The cream rouge given in example 35 is of the water-in-oil type, the emulsifier being Arlancel C (sorbitan sesquioleate). It is claimed<sup>26</sup> that this formula provides a product which is soft and creamy, and which possesses excellent spreading properties. Because it is of the water-in-oil type it has less tendency to dry out than emulsified creams of the oil-in-water type.

<i>Oil phase</i>	(35) per cent
Arlancel C	2.0
Lanolin (anhydrous)	2.0
Mineral oil 65/75	16.0
Petrolatum	30.0
Preservative	q.s.
<i>Aqueous phase</i>	
*Brilliant red C-10-013	10.0
Arlex	5.0
Water	35.0
Perfume	q.s.

\*Ansbacher

Cream rouge of the vanishing cream type may be prepared as in example 36.

	(36)
	<i>per cent</i>
Stearic acid	15.00
Water	76.32
Potassium hydroxide	0.50
Sodium hydroxide	0.18
Glycerin	8.00
Colour, preservative	<i>q.s.</i>

If water-soluble dyes are used in these emulsion products, they may be dissolved either in a portion of the water or in the glycerin, and added to the main bulk of cream after saponification in the normal manner (see Vanishing Creams, Chapter 4).

In example 37, diglycol stearate is employed as emulsifying agent.

	(37)
	<i>per cent</i>
Diglycol stearate	20.0
Water	70.0
Glycerin	10.0
Water-soluble colour	<i>q.s.</i> (from 0.6 to 1.0)
Preservative	<i>q.s.</i>

The difficulty in all cases where a water-soluble colour is introduced is to prevent evaporation, which leads to a darkening of the cream surface owing to concentration of the colour. When such colours are used it is necessary to incorporate sufficient hygroscopic constituents, such as glycerin, the glycols, etc; *d*-sorbitol would appear to offer interesting characteristics for this purpose, since it possesses a much narrower humectant range than glycerin, that is, it absorbs less moisture in a moist atmosphere, but holds it better in a dry one. A further improvement might be the packing of such products in a tightly capped tube.

In general, however, most of the colours employed in cream rouge are insoluble lakes, although sometimes oil colours are used. In the vanishing cream type, however, these lakes may be supplemented if desired by fluorescein dyestuffs when, as stated above, it is necessary to incorporate a non-volatile solvent.

A further example of a vanishing cream type of rouge, given by Janistyn,<sup>27</sup> has the following composition:

	(38)
	<i>per cent</i>
Cetyl alcohol	2.00
Stearin	18.00
Propylene glycol	4.00
Isopropyl myristate	8.00
Carbowax 4000	4.00
Potassium hydroxide	0.70
Water	54.65
Perfume	0.50
Preservative	0.15
Lakes	8.00



### Liquid Rouge

Liquid rouges appear to maintain a degree of favour and are claimed to yield extraordinarily good results when skilfully applied. The difficulty of applying them has been overcome by fitting a small wick in the neck of the container, the rouge being readily transferred by capillary attraction to the lip or cheek.

The essential requirements of such a preparation are that it has sufficient viscosity to permit easy and even application and that it dries reasonably rapidly.

Aqueous preparations are prepared by dissolving the requisite amount of a suitable water-soluble dye, adding a gum or synthetic thickener to increase the viscosity of the solution and including a small proportion of a suitable wetting agent to promote easy spreading. A little glycerin may be included if desired. In example 39, the methylcellulose may be replaced by 0.4 per cent gum tragacanth.

(39)	
	<i>per cent</i>
Methylcellulose	2.0
Wetting agent	0.1-0.2
Water	to 100.0
Water-soluble red, preservative	<i>q.s.</i>

Another preparation has the following composition:

(40)	
	<i>per cent</i>
Sodium alginate	0.45
Calcium citrate	0.13
Wetting agent (dermatologically acceptable)	0.1-0.2
Water	to 100.0
Water-soluble red, preservative	<i>q.s.</i>

*Procedure:* Dissolve the sodium alginate, dye and wetting agent in about two-thirds of the water, form a slurry of the calcium citrate in the other third portion and add to the alginate solution as soon as thickening occurs on standing. By varying the proportion of the alginate and calcium citrate the viscosity may be altered at will.

In order to shorten the drying time, 10-20 per cent alcohol or more may be added to gum mucilages, but high concentrations must not be used with alginate products otherwise precipitation may occur.

The preparations that have already been mentioned in connection with liquid lipsticks, consisting of a suitable alcohol-soluble dye dissolved in an alcoholic solution of ethyl cellulose or alcohol-soluble resin, with a plasticizer to obtain the requisite flexible film, can also serve as liquid rouge. No wetting agent is necessary, of course, and by the use of suitable dyes more permanent preparations are obtainable than with aqueous mixtures. Since evaporation of alcohol will lead to an increase in the viscosity of the product, it is particularly important that containers should be provided with a well-fitted wick built into, or screwed into, the neck of the container, the whole being enclosed in a screw dust-cap. By this means, ready and even application is obtained with the minimum of care and

with the least possible chance of covering the user's hands with sticky red varnish.

The resultant rouge film is readily removed with a little alcohol, to which may be added if desired a small proportion of a relatively high-boiling solvent to prevent too rapid evaporation of the rouge remover.

Preparations of the type just described are illustrated by the following formula:

	(41) per cent
Tincture of styrax, 5%	38.0
Alcohol, 95%	60.0
Castor oil	2.0
Spirit-soluble red	q.s.

The above solutions are prepared by simple mixture. Proportions of film-forming material, plasticizer and dye may be altered to suit particular requirements.

## EYE MAKE-UP

### Introduction

Eye make-up, too, has been used for thousands of years. The accepted eye make-up used by the women of many ancient civilizations was a black colouring, kohl, based on antimony trisulphide. In addition to kohl, Egyptians used malachite to confer a green tint, while Indian women tinted their eyelids with tsocco which has an antimony base. Chinese and Japanese women used Peruvian bark for eye make-up, and Phoenician women lengthened their eyebrows with a black paste composed of gum arabic, musk, ebony and powdered black insects.

Big eyes were also considered a mark of beauty, and it was the practice of women of these ancient civilizations to make their eyes appear larger and shinier.

Modern eye preparations include mascara, eyeshadow and eyebrow pencils, all of which must be applied sparingly and correctly if the effect is not to be ruined.

These preparations will now be discussed under their respective headings.

### Mascara (Eyelash Cosmetic)

. . . Egyptian women soon discovered that smearing the eyelids with unguent and stibium (that is, powdered antimony) not only eased the pain in the eyes and rested them but also added to the natural beauty of their faces. The whiteness of the whites of the eyes was emphasized by the darkened eyelids and the large dark pupils appeared like black pools in their midst, the whole effect being very striking . . .

(*Dwellers of the Nile*, Sir Wallis Budge)



Mascara is a black pigmented preparation for application to the eyelashes or eyebrows to beautify the eyes. It has been claimed that by correctly applying mascara to darken the eyelashes and to increase their apparent length, the brightness and expressiveness of the eyes is enhanced.

Mascara is marketed in cake (block), cream and liquid forms, and must possess the following characteristics:

- (1) It must be capable of easy and even application.
- (2) It must have no tendency to run and thereby cause smudging.
- (3) It must not cake, causing the eyelashes to stick together.
- (4) It must not dry too rapidly to interfere with the evenness of the application, but should nevertheless dry fairly rapidly and be reasonably permanent once applied.
- (5) It must be neither toxic nor irritant.

Under the US Food, Drug and Cosmetic Act, the use of coal tar colours in preparations to be applied in the area of the eyes is forbidden, even when those colours have been certified for use in cosmetics. The area of the eyes is defined as 'the area bounded by the supra-orbital ridge and the infra-orbital ridge, including the eyebrow, the skin below the eyebrow, the eyelids, the eyelashes, the conjunctival sac of the eye, the eyeball and the soft areolar tissue that lies within the perimeter of the infra-orbital ridge'. Thus defined, the 'prohibited area' extends from the top of the eye socket to the top of the cheekbone.

Thus in the USA only natural colours, that is vegetable colours and inorganic pigments and lakes (usually black and dark brown pigments), provided that these are in a highly purified form, may be employed in eye make-up preparations.

Pure vegetable colours which can be used include chlorophyll, except for the grade containing copper. The same applies to mineral and earth colours, but in the USA suppliers are required to guarantee the absence of certain impurities from their products; their arsenic content must not exceed 2 parts per million.

Black pigments used in eye make-up preparations are now restricted to black iron oxide ( $\text{Fe}_3\text{O}_4$ ). This is sometimes used in conjunction with ultramarine blue to impart blue-black shades.

Umbers (brown ochres), burnt sienna (a mixture of hydrated ferric oxide  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$  with some manganic oxide) and synthetic brown oxides have been used for brown shades, and yellow ochres for yellow shades. For bluish shades soluble oil blue is employed, while for green shades chromium oxides and for red shades carmine, the aluminium lake of cochineal, have been used. Salts of cobalt may be used only if they are insoluble salts and will not react with other ingredients of the preparation to form soluble cobalt compounds. Finely powdered metals, such as silver and aluminium, have been widely employed in eyeshadow and may be used if they consist entirely of the pure metal. If they contain an appreciable content of copper they are considered to be harmful. White pigments such as titanium dioxide and zinc oxide may also be sometimes included to lighten shades.

Colours that may be used within the EEC for mucous membrane products (including eye products) are listed in Annex III Part 2 of the EEC Cosmetics

Directive. A list of provisionally approved colours is detailed in Annex IV Part 2. Both these lists contain coal tar colours, so the formulator must be careful in considering the colour combinations to be used for individual countries, especially for importation into the USA.

#### *Cake (Block) Mascara*

Cake mascara remains a very common form of product, although during the 1960s softer cream or liquid preparations gained in popularity with the development of special container-dispensers with a brush as part of the screw-on cap.

Earlier types of mascara were based on a mixture of soap and carbon black, with sufficient water added to produce a stiff paste which was dried, and then stamped into cakes.

These could be readily applied and were fairly permanent but, since they contained a water-soluble base, tears and even heavy rain were capable of producing black streaks and smudges. Moreover, a trace of soap base in the eye produced extreme irritation. The sodium soaps of coconut and palm oils used originally in these preparations were eventually replaced by the less alkaline and hence less irritant triethanolamine soaps, for example triethanolamine stearate. According to Jewel<sup>28</sup> these products consist essentially of a soap (generally triethanolamine stearate) modified in consistency with oils and waxes, and colour. Modern cakes may be produced by melting together the waxy materials, adding the colour and mixing them in thoroughly to obtain an even distribution. The resultant mixture is then cooled and worked in a heated roller-mill. It is subsequently re-melted in a pan and cast into cakes. A formula has been quoted (example 42) which is claimed to be very similar to a large majority of block mascara preparations sold in the USA.

	(42)
	<i>parts</i>
Stearic acid	27.0
Triethanolamine	12.0
Beeswax	30.0
Carnauba wax	50.0
Colour (bone black)	25.0

Other cake mascara formulae quoted from the technical literature are as follows:<sup>29</sup>

	(43)
	<i>per cent</i>
Triethanolamine stearate	40.0
Paraffin wax	30.0
Beeswax	12.0
Anhydrous lanolin	5.0
Lampblack	13.0



	(44)
	<i>per cent</i>
Glyceryl monostearate	60.0
Paraffin	15.0
Carnauba wax	7.0
Lanolin	8.0
Lampblack	10.0

*Procedure:* The various components are melted, mixed and then cast or extruded to the proper form, or after mixing they may be milled and passed through a warm plodder, after which the mascara strip is cut to the desired lengths.

	(45)
	<i>per cent</i>
Triethanolamine stearate	50.0
Carnauba wax	24.0
Paraffin wax (MP45°C)	12.5
Anhydrous lanolin	4.5
*Silicone fluid L-43	5.0
Carbon black	3.8
Propyl- <i>p</i> -hydroxybenzoate	0.2

\*Union Carbide

With the delisting of carbon black in the USA, even though it is allowed under EEC legislation, the material has become scarce. It can be replaced in most formulations by black iron oxide or with combinations of ultramarine blue and black iron oxide.

### *Cream Mascara*

Cream mascara may be prepared by milling the pigment into a vanishing cream base, or by the use of a suitable oil-soluble dye, but in such cases it is often necessary to include a suitable wetting agent to lower the surface tension, otherwise the colour will not adhere to the brush. The coloured pigment may be incorporated into the base immediately after emulsification has been completed. Agitation is continued while the product is cooling, any entrapped air being allowed to escape. The product is then filled into the tubes.

Alternatively, a previously prepared base is melted in order to incorporate the pigment.

A reliable cream mascara which does not dry up in the tube and which adheres well to the eyelashes can, according to Richardson,<sup>31</sup> be made to the formula given in example 46. A modern equivalent of this formula would employ a synthetic hydrocolloid such as hydroxyethylcellulose to replace the quince seeds.

	(46)
	<i>per cent</i>
Mucilage of quince seeds	35.0
Sugar syrup	35.0
Gum arabic	7.5
Ivory black (or umber)	22.5

*Procedure:* Grind the gum and the ivory black (or umber, etc.) with the syrup (3 parts sugar to 2 parts water) and then add the mucilage containing a preservative, such as methyl-*p*-hydroxybenzoate.

More modern forms of cream mascara are shown in examples 47<sup>31</sup> and 48<sup>32</sup> and also in example 49<sup>24</sup> which is claimed to give an even application on eyelashes by being free from the tendency to run or cake, and also to have good performance properties.

	(47) <i>per cent</i>
Polyethylene glycol 400 stearate	10.0
Diglycol stearate	8.0
Lanolin	3.0
Stearyl alcohol	13.0
Isopropyl palmitate	2.0
Triethanolamine lauryl sulphate	1.5
Water	54.5
Colour	8.0

	(48) <i>per cent</i>
Triethanolamine stearate	45.0
Carnauba wax	15.0
Glyceryl monostearate	5.0
Anhydrous lanolin	10.0
Unbleached beeswax	5.0
Lampblack	20.0

	(49) <i>per cent</i>
Hexadecyl alcohol	7.3
Propylene glycol	9.1
Stearic acid	11.2
Glyceryl monostearate	4.5
Triethanolamine	3.6
Ultramarine blue	9.1
Methyl- <i>p</i> -hydroxybenzoate	0.2
Water	55.0

Another cream mascara formulation includes among its components a silicone fluid;<sup>33</sup> this is easily applied, gives smooth coverage and affords moisture protection:

	(50) <i>per cent</i>
A Stearic acid triple pressed	9.1
Petrolatum MP 43°C	5.5
Mineral oil 65/75	4.1
L-43 Silicone fluid	5.0
B Triethanolamine	2.75
Water	64.45
Pigments	9.1



*Procedure:* Melt A and heat to 60°C. In a separate container heat B to the same temperature. Add B to A while stirring. Incorporate the pigments in a combined mix.

Greasy preparations, more resistant to removal, may be made by incorporating the pigment or dyestuff, about 10 per cent, into a greasy base, which is warmed slightly for application with a fine brush—examples 51 and 52.

	(51) <i>per cent</i>
Beeswax	4.0
Spermaceti	4.0
Cetyl alcohol	2.0
Cocoa butter	6.0
Petroleum jelly	64.0
Oil-soluble blue	20.0
Preservative	<i>q.s.</i>

	(52) <i>per cent</i>
Cocoa butter (odourless)	5.0
Petroleum jelly	50.0
Paraffin wax (uncrystallizable)	5.0
Lampblack	40.0

*Procedure:* Melt and mix.

The development of novel brushes and applicators has led to the introduction of speciality cream-type mascaras. For example, waterproof mascaras are very popular at present and there is a variety of applicators available which allow the lashes to be separated and coated evenly with the product without clogging.

<i>Typical waterproof mascara</i>	(53) <i>per cent</i>
Isoparaffins	54.7
Ozokerite wax	18.0
Carnauba wax	2.5
Aluminium stearate	2.0
Isopropyl myristate	2.5
Mineral oil	0.5
Kaolin	12.8
Black iron oxide	5.8
Yellow iron oxide	1.2

In some formulations the gelling properties of the aluminium stearate are supplemented with other materials such as bentone. The waterproof properties can be enhanced by the addition of polymer-type materials such as polyethylene or polybutene. Small rayon or nylon fibres can also be added to the formula to give lengthening and thickening effects to the eyelashes.

*Liquid Mascara*

Satisfactory liquid mascara formulae have long been available but they did not gain wide consumer acceptance until the advent of the special cylindrical container with a small-diameter brush integral in the screw-on lid. This dispenser enhances the convenience of the quick-drying liquid product and is also convenient to carry in the handbag.

The earlier preparations were prepared by suspending the pigment in a mucilage, as in example 54.

	(54) <i>per cent</i>
Gum tragacanth	0.2
Alcohol	8.0
Water	83.8
Lampblack	8.0
Preservative	<i>q.s.</i>

However, preparations of this type were not very popular, because they tended to be sticky and, being water-soluble, they tended to smudge.

In order to overcome these drawbacks, more modern preparations use an alcoholic solution of a resin in which carbon black is suspended. A little castor oil is often included in such formulae. These dry rapidly, producing a semi-permanent waterproof colour, but are irritant if dropped accidentally into the eye. Such preparations may be illustrated by the following example:

	(55) <i>per cent</i>
Rosin (10% alcohol solution)	3.0
Castor oil	3.0
Ethyl alcohol	84.0
Lampblack	10.0

Other resins, or ethyl cellulose, may replace rosin if desired. Industrial methylated spirit is best avoided in such preparations as it tends to irritate.

**Eyeshadow**

Eyeshadows are marketed in a variety of shades, such as brown, green, blue and others. They are applied to the eyelids in order to produce an attractive 'moist'-looking background for the eyes. This is sometimes enhanced, particularly for evening wear under electric lights, by the inclusion of fine glittering metallic particles to produce a 'silver' or 'gold' effect. For this purpose gold leaf, bronze powder and powdered aluminium may be used. Various pearlescent pigments, for example those based on bismuth oxychloride or mica coated with titanium dioxide may also be used. Eyeshadow preparations are available, for example:

- (i) as anhydrous creams of the liquefying or emulsifying type;
- (ii) in the form of emulsions;



- (iii) as stick eyeshadow;
- (iv) in the form of liquid suspensions or dispersions;
- (v) as pressed powders.

The anhydrous cream form and the stick eyeshadow have been very popular but at present pressed powder eyeshadows are the dominant force in the market place.

### *Cream Eyeshadow*

Cream eyeshadow may be made by mixing first of all the selected colours (and, if pastel shades are required, including in the mixture also a white pigment such as zinc oxide or titanium dioxide) and blending these pigments with petrolatum in a roller mill. This mass is then stirred into the blend of fatty and waxy constituents to be used in the preparation, which have been previously blended by melting them together in a pan. After thorough agitation, the product is then poured into suitable containers while still in liquid form. Alternatively, the pigments in powder form may be stirred directly into the molten mass of fats and waxes, and passed through a roller mill to ensure thorough distribution of colours and remove any lumps from the formulation which may otherwise cause streaking of the product during application. A second mixing operation may follow before the product is poured into the containers. As in the case of mascara, the eyeshadow base should be capable of easy and smooth application. It should also be waterproof to avoid streaking. Sometimes, in order to facilitate liquid filling of the product into containers, the product requires to be kept warm in a jacketed steam-heated pan and, if necessary, gently stirred to prevent the sedimentation of any heavy pigments.

Eyeshadow creams of the anhydrous type may, for example, include cocoa butter, as in the following formulae.

	(56)
	<i>per cent</i>
Cocoa butter (odourless)	2.0
Beeswax	3.0
Spermaceti	5.0
Lanolin	5.0
Petroleum jelly	55.0
Zinc oxide	30.0
Cosmetic lake, preservative	<i>q.s.</i>

	(57)
	<i>per cent</i>
Petroleum jelly	75.0
Cocoa butter (odourless)	8.0
Lanolin	7.0
Cetyl alcohol	3.0
Paraffin wax (uncrystallizable)	7.0
Cosmetic lake, preservative	<i>q.s.</i>

A formula suggested by Miller<sup>34</sup> contained:

	(58) per cent
Bleached beeswax	4.5
Spermaceti	9.5
Lanolin absorption base	13.0
Paraffin of low melting point	73.0

*Procedure:* Mix 85 parts of this base with (say) 12 parts of titanium dioxide and 3 parts of a mineral pigment or earth colour.

To obtain a grey shade, Miller<sup>34</sup> suggested a mixture of 1 part of ultramarine blue, 1 part of lampblack and 2 parts of titanium dioxide: for a brown shade, a mixture of 3 parts of an umber and 1 part of titanium dioxide. He suggested heating the colours with the mixture of fats and waxes (example 58) and passing them through a roller mill. Another eyeshadow cream formula suggested by the same author contained:

	(59) per cent
White petroleum jelly	59.0
Glyceryl monostearate	17.0
Lanolin	4.0
Beeswax	8.0
Candelilla wax	4.0
Pigment	8.0

Emulsion-type eyeshadow creams are produced by mixing suitable pigments into an emulsion and distributing them evenly throughout the base. The products should be filled into containers when cold. Emulsions based on triethanolamine stearate can be used as bases for such preparations (example 60).

	(60) per cent
Stearic acid	1.5
Glyceryl monostearate	1.5
Lanolin	4.0
Isopropyl myristate	5.0
Veegum (5% solution)	30.0
Triethanolamine	0.75
Water	38.25
Propylene glycol	4.0
Ultramarine blue	4.5
Black iron oxide	1.2
Chrome hydrate	0.8
Mica coated with titanium dioxide	8.5

The anhydrous creams and the emulsion products were originally intended for finger-tip application. However, the modern trend is to use special applicators



and the most popular of these is the sponge tip. It is attached to a plastic rod which is usually an integral part of the cap of the container, as in mascara packaging. The container itself can be either polyethylene or PVC and it is important that a good seal is obtained with the cap and the container to prevent the product, especially the emulsion products, from drying out.

### Stick Eyeshadow

The sticks contain a fairly high proportion of waxes such as ceresin, ozokerite or carnauba. A stick eyeshadow suggested by Wetterhahn<sup>35</sup> contained:

	(61) per cent
Castor oil	43
Mineral oil 75/85	6
Hydrogenated cottonseed oil	5
White ceresin MP 76°C	26
Carnauba wax	4
Titanium dioxide	8
Iron oxide ochre shade	4
Iron oxide sienna shade	4

Croda (in *Syncrowaxes in Cosmetics*) have suggested the following formula:

	(62) per cent
Syncrowax HGLC	15.0
Syncrowax HRC	5.0
Mineral oil	35.2
Liquid base CB3896	15.0
PVP	0.8
Colour	9.0
Timica sparkle	20.0

*Procedure:* Melt waxes at 85°/90°C. Add liquid base and mineral oil. Maintain temperature at 80°C. Mill PVP and pigments into molten wax phase. Disperse the Timica with low shear agitation, allow to de-aerate and fill off.

### Pressed Powder Eyeshadow

Pressed powder eyeshadows can be regarded as compact rouge with a different colour system. In general, they contain much higher colour levels than the rouge products and this has to be taken into account when manufacturing the products. For example, with products containing high levels of an iridescent material such as mica coated with titanium dioxide, care must be taken that, in dispersing the product, the platelets are not shattered by the hammer mills. The product is usually applied by means of a sponge-tipped plastic rod, although finger-tip application is popular—a point to remember when the hardness of the product is being determined during manufacture.

Typical formulae illustrating a matt shade (example 63) and a highly iridescent shade (example 64) are given below.

	(63) per cent	(64) per cent
Talc	82.5	41.7
Zinc stearate	6.0	7.0
Ultramarine blue	5.4	—
Black iron oxide	0.1	—
Chrome hydrate	2.0	—
Yellow iron oxide	—	2.0
Mica coated with titanium dioxide	—	40.0
Base (see example 27 above)	4.0	9.3

### Liquid Eyeshadow

Liquid eyeshadows take the form of either a liquid suspension or a liquid dispersion. In the former case the pigments are suspended in a mixture of oils, but they usually settle and have to be shaken before use. The liquid dispersions are prepared, for example, from dilute solutions of alcohol in water thickened with a synthetic gum such as methylcellulose, suitably preserved and containing a wetting agent in which the pigments have been dispersed. However, liquid eyeshadows are not very popular.

### Eyeliner

Eyeliners are preparations for use on eyelids, particularly the upper eyelids, close to the eyelashes, to help to accentuate the expressiveness of eyes. They are available in liquid, cake and pencil form. The compressed powder form is similar in composition to cake mascara. As an example of a dry eyeliner, Fishbach<sup>10</sup> quoted the following composition:

	(65) per cent
Mineral oil	5
Colour	30
Titanium dioxide	5
Talc	60

Liquid eyeliners may be used to apply colour to the tissue around the eyes. Usually a brown colour is considered a good colour for daytime wear.

A liquid eyeliner formula cited in *American Perfumer and Cosmetics*<sup>36</sup> contained:

	(66). per cent
Water	40.00
Methocel HG 60-50V	1.00
Veegum	1.00
Shellac	1.08
Oleic acid	0.50
Triethanolamine	0.40
Water	3.02
Pigment	18.20
Alcohol SD-40	5.00
Water, preservative	to 100.00



A formula which appeared in the *General Aniline Guide to Cosmetics* contained:

	(67) per cent
Veegum (Vanderbilt)	2.5
PVP K-30	2.0
Water	85.5
Pigment	10.0
Preservative	q.s.

### Eyebrow Pencils

Eyebrow pencils may be either of a wax crayon type pigmented black or brown, in which the pigments are present at a higher level than in cream eyeshadow preparations, or they may be in the form of an extruded eyebrow pencil similar to an ordinary lead pencil, in which a crayon-type formulation has been enclosed in a wooden casing. In the latter form they are even more readily applied than the stick type. Eyebrow pencils are used either to accentuate the line of the eyebrows, or to modify their outline after plucking. The pigment is dispersed in a wax base of the lipstick type, in which the proportion of beeswax or other high-melting constituent may be optionally increased to produce a somewhat firmer pencil. However, such sticks must be capable of ready and uniform application and should not be brittle.

	(68) per cent
Hydrogenated castor oil	46.0
Hydrogenated cottonseed oil	12.0
Cocoa butter (odourless)	8.0
Castor oil	8.0
Lanolin absorption base	17.0
Black iron oxide	9.0

If a brown stick is required the black iron oxide may be partially replaced by the red and yellow versions.

An eyebrow pencil formula cited from the literature consisted of:

	(69) per cent
Ozokerite (MP 70°-75°C)	45.0
Unbleached beeswax	24.0
Cocoa butter	22.5
White petrolatum	6.0
Absorption base	1.5
Anhydrous lanolin	1.0
<i>In addition:</i>	
Pigment or lake colours	10.0
Oil-soluble colour	1.0

*Procedure:* Stir the colours into the molten fat phase and mill until uniform. Fill the warm batch into the moulds.

Dehydag Products (Henkel) have suggested the following formula:

	(70)
	per cent
Cetyl alcohol	5.0
Cutina GMS	5.0
HD-Eutanol	20.0
Beeswax, white	2.0
Candelilla wax	6.0
Carnauba wax	6.0
Ozokerite wax	24.0
Castor oil	20.0
Liquid paraffin	2.0
Pigment colour	10.0
Antioxidant	q.s.

Finally, a formula suggested by Keithler<sup>22</sup> may be prepared by combining the following constituents:

	(71)
	per cent
Beeswax	21.0
Carnauba wax	5.0
Paraffin	29.0
Cetyl alcohol	8.0
Vaseline	18.0
Lanolin	9.0
Pigments	10.0

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## The Application of Cosmetics

### Introduction

The use of cosmetic preparations has come a long way since the days when the detectable use of any cosmetic was the sign of an actress or of an 'abandoned woman'. Nowadays there are few women who totally abjure the use of any form of make-up, although the degree of use varies—from a little powder to reduce the shine on the nose, to the face that is such a work of art or craft that apparently every product described in this book must have contributed to it.

The advice offered by beauty columnists and in the various textbooks and cosmetics manufacturers' instructions is apt to prove confusing, but works by Kendall,<sup>1</sup> Anderson<sup>2</sup> and Young<sup>3</sup> are worthy of consultation. In this brief chapter, the technique for the use of beauty preparations has been stripped of all but essentials.

The correct use of cosmetics falls into two parts:

- (1) *Skin care*, which has as its object the maintenance of a soft, supple and clean skin and the prevention of effects due to external causes such as excessive exposure to cold, heat, sun, wind, etc.
- (2) *Decoration* to produce a pleasing appearance by minimizing facial defects of colour or shape and unobtrusively enhancing and directing attention towards better points.

The first depends to a large extent upon the type and condition of the skin. The possessor of a healthy normal skin is fortunate in that it will withstand many treatments and conditions which can have serious effect on skins which are definitely dry or greasy and which demand particular care and treatment. Even the healthy and normal skin may vary from time to time and need particular care to correct any departure from normality.

The second point—the amount of cosmetic decoration or make-up tolerable—is dependent chiefly on social conventions which vary from time to time and from society to society. The general trend for many years has been towards a greater tolerance, although there have been shorter swings in particular social sets to exaggerate effects in various directions; these are generally short-lived, but are assimilated, often in severely modified form, and contribute to the general trend.

### Care and Cleansing of the Skin

It goes without saying that a healthy skin should be a clean skin, and in general a good toilet soap and water is the best way of achieving this cleanliness. A final



rinse with clean cold water is an excellent measure to promote circulation and tone up the skin. At night such cleansing should be followed by the application of a cream of more or less greasy type according to the natural dryness or greasiness of the skin.

The cream should be applied by proper massaging movements, whether or not the massage is carried on for long, because friction increases the supply of blood to the skin and skilful massage assists in keeping the skin supple. Charts designed to show the paths which should be followed in facial massage and application of cosmetics have been prepared by cosmetic manufacturers (see Figure 20.1).

The regular use of a correctly formulated face cream in a suitable manner can prevent the premature (not ultimate) aging of the skin due to external causes and delay the appearance of wrinkles produced by loss of epidermal elasticity and subcutaneous moisture. It cannot, however, prevent the natural aging of the

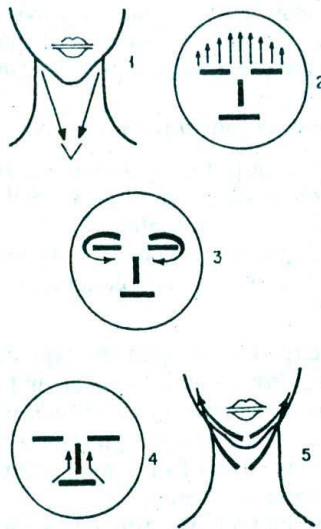


Figure 20.1 Facial massage (Courtesy Harriet Hubbard Ayer)

1. *For the neck*—Using alternately both hands cupped round the neck, firmly massage with downward movements.
2. *For the forehead*—Gently massage the forehead upwards from the eyebrows to the hairline with alternate movements of the hands.
3. *For the eyes*—Both hands working simultaneously, make circles round the eyes, ending by pats on the lower eyelid. Always work in the direction indicated on the diagram.
4. *To smooth the 'smile line'*—Massage with both hands with upward symmetrical movements over the 'smile line', finishing between the corner of the mouth and nostrils.
5. *Against sagging of the lower part of the face*—Both hands working alternately, taking the jaw line between the second and third finger, make upward movements from the point of the chin to the temporal muscle.

skin due to metabolic processes nor wrinkling caused by ill-health, psychogenic factors or certain diseases.

Wrinkles must be differentiated from the lines produced by emotions, for example constant scowling or sneering. The best cure for these is to cultivate a placid disposition.

In the case of excessively dry or greasy skin it may be necessary or advisable to cleanse it without the use of soap. The appropriate choice of material for dry skins would be complexion milk or a more greasy cleansing cream; for greasy skins, non-greasy cleansing lotions based on very mild alkalis such as sodium bicarbonate, or on certain buffered soapless detergents, may be used with advantage. Whatever method is adopted, the cleansing should be efficient and no trace of the day's grime or make-up should remain.

### **Cosmetic Application**

Having secured a clean skin on which to work, the essentials of facial make-up include the application of:

- (i) colour to the cheeks to give a pleasing healthy appearance and, if required, to modify the shape of the face;
- (ii) powder to hide shine or greasiness and confer a matt bloom to the skin;
- (iii) cosmetics in the region of the eyes to enhance the appearance and draw attention to the most attractive feature;
- (iv) colour to the lips, to modify the shape of the mouth, again to attract attention and, by contrast, enhance the whiteness of the teeth and improve the smile.

It will be noted that all the objectives given are basically the natural attributes of youth; a lavish use of cosmetics, or a high degree of contrast, may produce a more formal or 'sophisticated' effect.

A little skin lotion with some astringency or greasiness, as appropriate to the skin condition, should be applied immediately after washing in the morning, using a pledget of cotton wool and smoothing in the same directions as indicated for massage. This should then be followed by the application of a light moisturizer to 'set the skin' ready for the make-up. This should not be applied immediately. The skin appears to require a period of acclimatization to the day, and if make-up is applied immediately on rising it is rarely satisfactory or long-lasting.

### *Foundation*

When applying make-up, the foundation, with a degree of greasiness appropriate to the condition of the skin, is applied first of all, just sufficient to provide an adherent base for the rouge or blusher and powder. It is applied in the same directions as shown in Figure 20.1 for massage, but with more gentle smoothing movements, extending well on to the throat and further if desired, to avoid any lines of demarcation. It also hides skin imperfections and imparts a smooth, even appearance to the skin. In the case of normal or dry skin the foundation is usually a cream, but liquid foundation and cake make-up can equally be



employed. In the case of oily skin, non-oily liquids, cake make-up and medicated lotions are more appropriate.

### *Rouge or Blusher*

The application of rouge comes next. The rouge chosen should match the colour of the natural flush which appears through blushing or exertion. If it is applied correctly, it can accentuate the more attractive features and reduce the less attractive ones. Liquid or cream rouge is applied in sufficient quantity to the most prominent part of the cheek bones, just below the eyes and is spread with the finger tips, roughly into a triangle shape. The exact proportions and position of the triangles depend upon the shape of the face and the impression of length and breadth it is desired to convey. Naturally the edges of the coloured patch should blend easily into the remainder of the face with no hard outline. Powder rouge, however, is applied over the face powder.

### *Powder*

The powder should be applied generously with cotton wool or a clean puff and the excess brushed off with a soft brush. It should be slightly darker than the foundation and have a covering powder appropriate to the type of skin—light for dry skin, and heavy for an oily skin. The correct shade is that which imparts an unobtrusive smooth, but not skiny, look to the skin. Thought should be given, as with all cosmetics, to the lighting under which the make-up will be seen; daylight demands shades nearer to natural, while for artificial light all colours should be a shade or two deeper, and slightly heavier application is advisable. To obtain a very transparent effect, which can look attractive especially for evening wear, two contrasting powders should be used, first a lighter one and then a deeper one. With two shades of powder used simultaneously, the same effect can be achieved as by the location of rouge, namely a change in the apparent shape of the face. Applying a powder of a darker shade across the lower part of the face will appear to shorten a long face. By application of a lighter-shade powder to either side of the jaw line, a heart-shaped face will appear wider.

### *Eye Make-up*

For a long period eye make-up was practised much more discreetly by European or American women than by their Eastern sisters; even after the emancipation of make-up, lips were made up far more prominently than eyes.

However, the pendulum has now swung and all aspects of this most attractive feature of a woman's face are enhanced by the use of the appropriate cosmetics, outdoors by day as well as indoors by night. Thus, there are pencils to improve and enhance the colour and shape of the eyebrows, mascara to colour and lengthen the lashes, eyeshadow to draw attention to and re-shape the eyelids and to enhance the eye colour and eyeliners to outline and emphasize the eye itself. These are applied as follows.

*Pencil.* The eyebrows are first of all tidied, if necessary, by slightly plucking them with tweezers, mainly from below; a thin line should, however, be avoided. The eyebrows are then prolonged deftly towards the temples by means of an eyebrow pencil which should match the natural colour of the eyebrows. In order to obtain

a neat result, a sharpened pencil should be used. The pencil can also be used to outline the upper eyelids just above the eyelashes, again extending the line slightly towards the temple.

*Mascara.* The function of mascara is to increase the natural charm of the features by darkening the eyelashes and increasing their apparent length. It is claimed that, by its judicious use, the brightness and expressiveness of the eyes is enhanced.

Mascara is available in either cake or cream form, and slightly different techniques are employed in their application. Cake mascara is applied by rubbing the wetted brush over the cake until sufficient colour has been imparted to it and until it is almost dry. It is then stroked over the upper lashes only, whereby the colour is concentrated on the tips and outer lashes in order to secure a natural effect. A fresh brush is used when the lashes are dry in order to separate them.

At one time cream mascara had to be applied by means of the fingers, and consequently was much less popular than blocks. However, the advent of a much more convenient applicator, in which the mascara as a thin cream is packed in a cylindrical container and applied by means of a small-diameter cylindrical brush, has increased the popularity of cream mascara to such an extent that it is now the dominant type. The brush, charged with mascara, is withdrawn from the tube and revolved against the outer side of the eyelashes, working away from the eyelid. The brush simultaneously deposits mascara and separates the eyelashes. Black mascara is suitable for black or dark brown eyelashes, while blue mascara when applied to the lashes of blue or grey eyes increases their apparent blueness.

*Eyeshadow.* Eyeshadow is used to impart more depth to the eyes and to intensify their colour. It is available in cream and stick forms, or as a pressed powder.

The shade is governed by the colour of the eyes, and may include colours ranging from black and blue to green and silver. The cream eyeshadow is applied just above the lashes on the centre of the eyelids and smoothed in with an outward movement of the finger tips. Pressed powder eyeshadows are usually sold with a sponge-tipped applicator and this is used to transfer the product from its compact onto the eyelids. Some powdered eyeshadows can also be applied with the finger. For daytime wear, eyeshadow should be applied lightly, and for evening wear the application should be only slightly more heavy. The use of two products at the same time is becoming very fashionable. A dark colour (blue, green or brown) is applied on the eyelid and lighter coloured product or 'high-lighter' (pearlized white or pink) is applied in the gap between the eyelid and eyebrow to merge and tone in with the original colour. Wearers of heavy-framed spectacles should refrain from heavy eye make-up.

*Eye Liner.* According to the instructions issued with one of the many liquid eye liner preparations, the brush provided with it is dipped into the liquid, held as one would a pencil, and then the desired line is drawn in one single brush-stroke. The same instructions also state that an eye opening effect can be achieved by broadening the line towards the centre of the eye.



### Lipstick

The last touch to the make-up, but by no means the least, is the lipstick, which is used to impart colour and an attractive shape and appearance to the lips, and also to protect them. Intelligently used, it is capable of altering the apparent facial characteristics. Thus, by using a coat of darker lipstick with a lighter shade on top, narrow lips can be widened; by other methods wide lips can be narrowed, and the length of the lips can be brought into better proportion to the shape of the face.

Lipstick is best applied by means of a lipstick brush, which after a little practice will be found to give much better control than direct application. The desired mouth shape is first outlined and then filled in. The lips are blotted by biting on a tissue and the lipstick is applied once more. After a few seconds the lips are pressed together without tissue and the make-up is complete. If a more matt effect is desired, the lips should be lightly coated with powder before they are finally pressed together.

The colour of the lipstick can vary more than that of almost any other cosmetic and is chosen by the user not only on the basis of the tone of the skin, but also depending on the colours of the clothes worn. The fashionable colours for the seasons, determined each year by the fashion designers and the fabric manufacturers, lead to definite trends in the colours of lipsticks offered for sale. Thus, for a period, the great majority of lipsticks may have a bluish cast while the prevailing dress fabrics are greens, blues and mauves, whereas at another time an orange cast or pure reds will be needed to match yellow and brown outfits. The intelligent manufacturer will keep abreast of such fashion changes and change his lipstick range accordingly.

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