

Moulding techniques in lipstick manufacture: a comparative evaluation

A. C. DWECK and C. A. M. BURNHAM, Smith & Nephew Cosmetics, Surbiton, Surrey

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Synopsis

This paper examines two methods of lipstick bulk manufacture: one via a direct method and the other via stock concentrates.

The paper continues with a comparison of two manufactured bulks moulded in three different ways – first by split moulding, secondly by Rotamoulding[®], and finally by Ejectoret[®] moulding.

Full consideration is paid to time, labour and cost standards of each approach and the resultant moulding examined using some novel physical testing methods. The results of these tests are statistically analysed.

Finally, on the basis of the gathered data and photomicrographical work a theoretical lipstick structure is proposed by which the results may be explained.

Etude comparative des techniques de moulage dans la fabrication des rouges à lèvres

Résumé

L'article présente deux méthodes de fabrication des masses de rouge à lèvres: l'une basée sur la préparation de concentrés monochromatiques, l'autre sur le broyage direct des pigments dans le mélange des huiles et des cires. Ces deux méthodes ont été utilisées pour fabriquer des masses de rouges à lèvres brillantes, transparentes et nacrées. Les masses ont été coulées en raisins selon trois procédés:

- le système traditionnel et manuel de coulage dans des moules à fente
- le système rotatif 'Rotamould'
- le système 'Ejectoret'

On a étudié les avantages et les désavantages, ainsi que le rendement et les coûts de production de ces deux procédés de fabrication et des trois techniques de moulage. La résistance à la repture et la dureté au pénétromètre des raisins ont été testés sur une période de trois mois. Après avoir gelé les raisins, on les a cassés de façon à permettre l'examen visuel de leur structure. On décrit un appareil original permettant de mesurer la quantité de rouge à lèvres déposée par étalement dans des conditions reproduisant approximativement celles de l'utilisation.

Sur la base de ces diverses observations et d'une étude bibliographique, on a proposé une structure théorique du rouge à lèvres.

Des différences significatives de résistance à la repture et de dureté ont été relevées entre les rouges à lèvres préparés selon les deux procédés de fabrication lorsque la teneur en pigments était élevée et la vitesse de refroidissement lente.

On a mis en évidence des corrélations entre la dureté et la résistance à la rupture et entre la dureté et la quantité déposée par étalement. On a montré que d'une façon générale, la structure des raisins était influencée par la vitesse de refroidissement et que

ceux coulés selon le procédé 'Ejectoret' avaient une structure amorphe durcissant avec le temps. Les raisins coulés selon le système traditionnel ou le système 'Rotamould' ont une structure plus stable. On a également relevé des différences de structure entre les raisins coulés dans les différentes zones d'un moule. La méthode de fabrication directe est plus onéreuse à mettre en oeuvre mais elle réduit le temps de fabrication car elle nécessite un nombre réduit de corrections de couleur.

Le système de moulage 'Ejectoret' est également plus coûteux à l'installation, mais il permet une production plus élevée et la réduction du nombre de raisins défectueux. Le système traditionnel est moins onéreux mais il nécessite une main d'oeuvre qualifiée, à un rendement moindre et donne le plus grand nombre de raisins défectueux. Cependant, une fois que les différents paramètres ont été bien définis, toutes les méthodes de fabrication et de moulage permettent d'obtenir des rouges à lèvres de qualité convenable.

INTRODUCTION

Lipsticks form an intrinsic part of most cosmetic companies ranges, and it is surprising, upon a literature survey, to find very little in the way of published material on the subject over the past 30 years (1-4).

However, lipstick production consumes a high tonnage of raw materials each year and provides an interesting subject for discussion in relation to its manufacture, moulding and resultant structure.

This paper intends to examine two methods of manufacture (though there are many other possibilities (5, 6)) and using the bulk from each uses three moulding techniques.

The mouldings or bullets from each test were critically examined using physical test methods for pay-off, hardness and stick strength, the information collated and statistically analysed. Full account was also taken of the capital expenditure, labour cost and time factors involved in each section of production.

METHODS OF MANUFACTURE

Two methods were examined, which will be called the 'stock concentrate method' and the 'direct method' (Fig. 1).

The stock concentrate method

This method involves the production of a stock concentrate of colour in a castor oil base, together with an oil bound preservative such as propyl paraben.

The pigment to binder (in this case castor oil) grinding ratio is calculated or requested from the supplier, in order to achieve maximum working on the pigment and hence maximum colour development.

The colour and castor oil are premixed using a prop. or shear headed mixer to give an even slurry or dispersion, which is then fed into one of several alternative mills: (i) roller mill; (ii) sand mill; (iii) corundum mill; (iv) ball or pebble mill. There are many views on which method is best (3, 6-10).

Generally, the efficiency of milling versus the time of milling must be decided before concluding on which method to use. In efficiency terms the order might run as follows:

$$\text{ball mill} > \text{sand mill} \geq \text{corundum mill} > \text{roller mill}$$

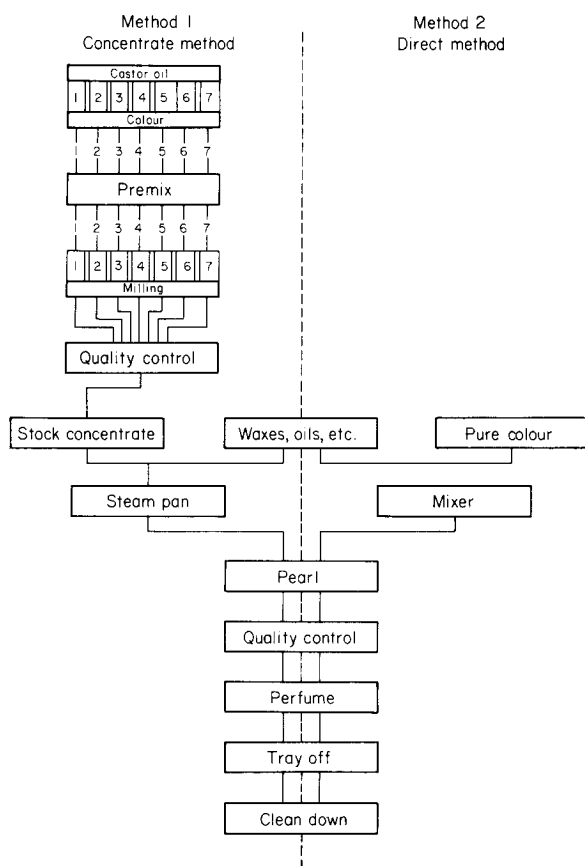


Figure 1. Diagrammatic scheme of two methods of lipstick manufacture.

though in time consideration the order might appear:

sand mill < corundum mill < roller mill \leq ball mill

when clean down times are considered, assuming one piece of equipment to handle all colours, then the corundum mill with changeable stones for each colour is best.

A separate and exclusive machine for titanium dioxide white is vital!

The mill is set to as small a gap as possible, and water cooled, the delivery rate is approximately 30–40 kg/h. Once milling is complete it should be checked against a standard (as a 5% let down in standard white) and the necessary corrections made. Particle size is checked using a Hegmann gauge, which should read off scale.

The waxes are then weighed and dispensed into a steam pan and allowed to melt. The surface of the steam pan can reach temperatures of up to 110°C, so that it is important to keep a close eye on the temperature climb. The advantages of a steam pan over an electrically heated kettle is the speed of control using steam.

Once the waxes have melted the colour concentrates together with any remaining castor oil are dispensed into the steam pan and mixed for 5–10 min using a shear head mixer, the

temperature being maintained at around 85°C. The shear head mixer is preferable to a prop. mixer because it creates less of a vortex, and therefore less entrapment of air. (This type of mixer will not develop the colours further than the corundum stone mill.)

Once the waxes and colour concentrate have been well dispersed, the pearl material (if any) is added, and a stick moulded. The stick is then checked for colour by quality control and colour corrections, if required, are then made.

The perfume is added at 75°C and mixed in for a few minutes, and then the bulk poured off into trays and stored until required.

The direct method of manufacture

The waxes are weighed and dispensed into the mixer and the vessel heated to 85°C. Once the waxes have melted the castor oil is introduced and the vessel lid lowered, and the temperature allowed to fall to 75°C. Mixing is started using anchor blades only.

The pigments and lakes are weighed and dispensed into a suitable container. The mixer is then put under a vacuum of between half and one atmosphere, and the pigments slowly drawn in with the vacuum pump running via the bottom pipe of the mixer. The colloid mill or dispersion head should be running during the addition of the colourants.

Once the pigments have been introduced, the colloid mill and anchor blades continue to run for 30 min whilst maintaining the mixer at a half atmosphere of vacuum. This greatly reduces entrapment of air in the product. After this time the mixers are turned off and the bulk allowed to stand under vacuum for a further 5 min. Any pearl material in the formula would be drawn in under vacuum at this stage. The completion of manufacture would then follow the stock concentrate method detailed previously.

Colours which sometimes give problems through being improperly dispersed, such as D & C Red No. 7 Ca lake or D & C Red No. 9 Ba lake, gave no trouble by this method.

It was perhaps surprising that the amount of colour matching required using this method was a great deal less than that using stock concentrate. The reason for this is almost certainly due to the reluctance of manufacturing staff to stir the drums of concentrate prior to weighing, and hence taking 'thin' material from the top.

The method worked extremely well for pigment concentration of 2% or more, i.e. the batch needed no colour matching. However, a lipgloss stick containing 0.02% pigment did require 20% extra colour, though in all fairness the time involved (in what was a difficult exercise) was greatly reduced compared to the concentrate method.

Advantages of each method of manufacture

Method I

- (1) Able to compensate for strength differences of incoming pigments; thus, once concentrate is manufactured there should be no further problem.
- (2) Fast clean down time of steam pan.
- (3) Optimum colour development and associated cost saving as a result of using best pigment to binder grinding ratio.
- (4) Operator required needs less skill to operate this method than when using the closed mixer.

Method II

- (1) Number of stages in manufacture reduced.
- (2) Quality control involvement reduced, hence reduced labour cost.
- (3) Storage area requirement reduced.
- (4) Number of formulae handled by manufacturing reduced.

- (5) Time saving on formula manufacture.
- (6) Bulk aeration is reduced.
- (7) Reduction in the amount of equipment used, and also cleaning down.

Disadvantages of each method of manufacture

Method I

- (1) Tedious to make up to twenty concentrates; time consuming.
- (2) Expensive requirement of colloid mills or stones for each colour.
- (3) Extra requirement that concentrates require stirring at least once a day to maintain dispersion.
- (4) Concentration gradient throughout drum because not stirred twice a day!
- (5) Bulk can become aerated.
- (6) Extra storage area required for concentrates.

Method II

- (1) Need to handle neat pigment/dyestuff which is unpleasant.
- (2) Need to know strength differences of incoming colour and alter each formula according to batch or lot used.
- (3) Clean down time is lengthy of mixer compared to steam pan.
- (4) Colour matching is difficult because additions are small.
- (5) As a result of (4) may need tinted wax bases.
- (6) Mixers are costly compared to the added cost of steam pan and colloid mill.
- (7) No facility to optimise colour development, i.e. pigment to binder grinding ratio.
- (8) Harder to pinpoint source of trouble in colourmatching, i.e. colour strength of pigment/low or pigment weighing inaccurate.

Comparison of times between methods of manufacture

Breakdowns of the times taken for lipstick manufacture by the concentrate method and the direct method are shown in Tables I and II.

Table I. Breakdown of lipstick manufacture time using concentrate method

Operation	Time (min)
Time to dispense materials	20
Load steam pan	2
Melt	25
Load colour and castor oil	5
Equilibrate temp	5
Add pearl and mix	5
Approval Q.C. assuming no colour match	10
Tray off	10
Clean down	10
Manufacture time excluding concentrate	92
Calculation of concentrate preparation time	
3 h to prepare and clean down 100 kg	
Assume 16 stock concentrates	
equivalent to 48 h to prepare 1600 kg of material	
On average 25 kg of concentrate/batch (100 kg)	
Thus enough for 60 batches. Time/batch 48/60 h =	48 min
True time of batch preparation	140 min

The results shown below are averaged over several shades of differing moulding quality.

I. Split moulding

	No. of operators	= 1 (using three moulds)
	Target production	= 340 doz/day
Downtime:	Breakdown	= Nil
	Rejects	= 85 doz (25%) handling, pinholes etc. etc.
	Changeover (i)	= 15 min/shade
	(ii)	= 10 min/product (to obtain new moulds)
	Actual production of good bullets	= 255 doz/day (one shade)
	Bullets are produced to bulk, i.e. no assembly into components	
	Labour cost/dozen	= £0.047
	Higher labour rate as per manual job evaluation and part assembly.	

II. Rotomould[©]

	No. of operators	= 2 (loading cases)
	Target production	= 900 doz/day
Downtime:	Breakdown	= 72 doz (8%)
	Rejects	= 90 doz (10%) mainly pinholing
	Changeover (i)	= 30 min/shade
	(ii)	= 2 h+/product
	Actual production of good bullets	= 738 doz/day (one shade)
	Cases can be reclaimed when rejects are produced, but require cleaning if used on another shade.	
	Labour cost/dozen	= £0.030 (incl. part assembly).

III. Ejectoret[©]

	No. of operators	= 2 (machine minders only)
	Target production	= 1300 doz/day
Downtime:	Breakdown	= 65 doz (5%)
	Rejects	= 4 doz (0.3%) low fills, damaged cases.
	Changeover (i)	= 3 min/shade (to purge pump)
	(ii)	= 5 h/product
	Actual production of good bullets	= 1231 doz/day (one shade)
	Rejects are a total loss, cases cannot be recycled.	
	Labour cost/dozen	= £0.018 (incl. part assembly).

Thus it can be seen that the labour cost is inversely proportional to the production. Split moulding requires experienced moulders to reach target production, which in turn requires higher hourly rates for the skill required, even so, the rate of production cannot match mechanised methods.

The mechanised methods require machine minders and components loaders only thereby reducing the labour cost standard, yet maintaining superior production figures, and low reject rates (II).

Split moulding

The following description refers to a single mould operation, in fact each station would operate a three mould rotation and in the case of a soft mass sometimes four moulds (Fig. 3).

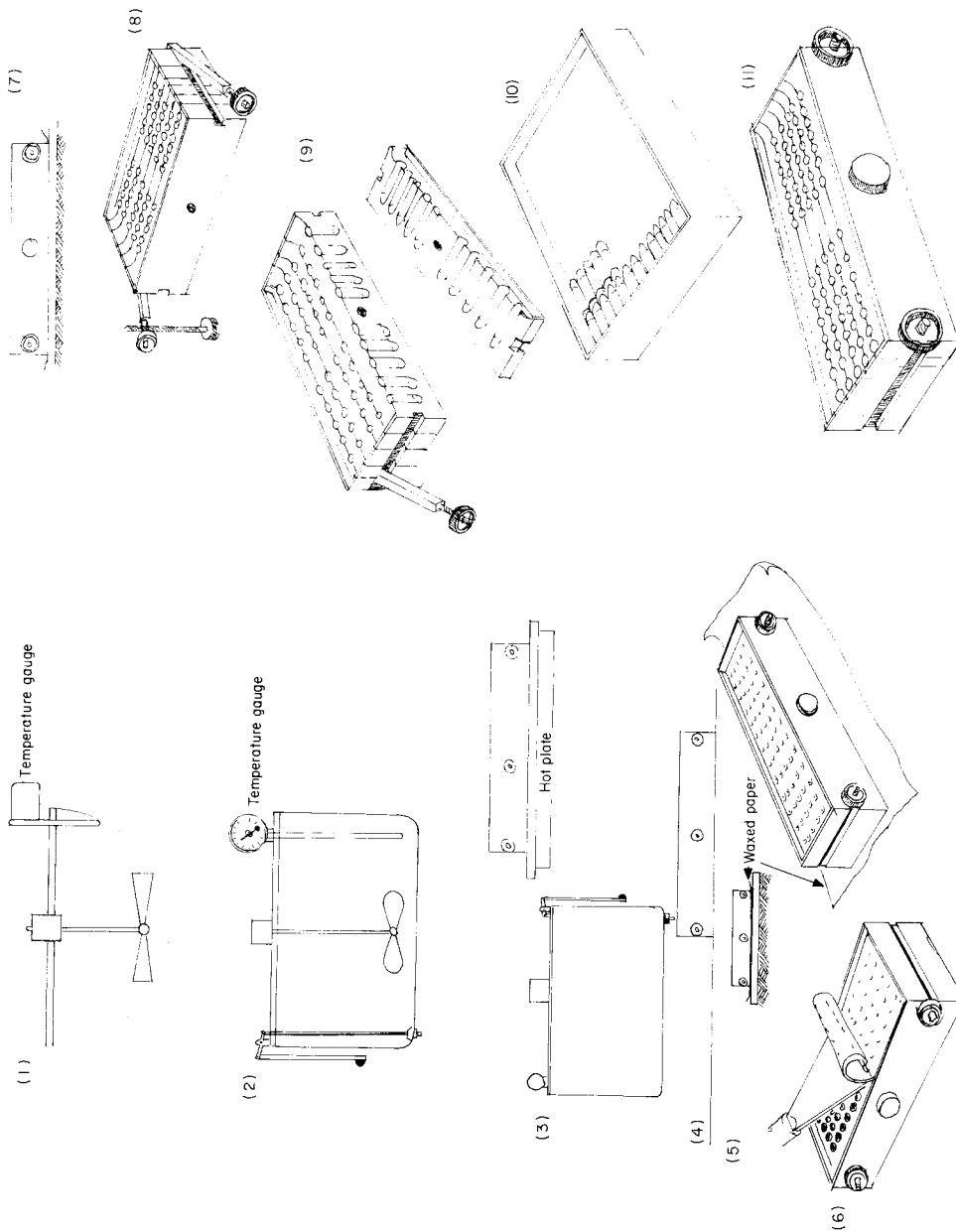


Figure 3. Split moulding of lipsticks. (1) Premelt lipstick in steam pan. (2) Fill dispenser. (3) Warm pre-oiled mould on hotplate. (4) Fill mould. (5) Allow mould to chill on cold plate. (6) Once surface has dimpled, remove top with scraper. (7) Replace mould on cold plate for final chilling. (8) Open mould. (9) Strip out bullets. (10) Examine sticks, reject poor moulds and pack acceptable sticks into tins. (11) Reassemble mould, oil with release agent. Return to (3) and recommence process.

Premelting

The contents of the tray delivered from the manufacturing area, holding approximately 20 kg, is cut out and placed in a steam pan held at 60°C. When the mass has melted it is kept at a constant temperature (60°C) and continually stirred.

The dispenser

The mass is held at $85 \pm 5^\circ\text{C}$, and kept stirring with a slow paddle stirrer.

The nozzle should not be too small for the size of mould being used, otherwise the mass will pour too slowly and excessive cooling will occur and result in poor mouldings, which could show signs of layering and/or cracking. On the other hand, the nozzle should not be too large, so that the molten mass is delivered as a flood, splashing and spluttering past the cavities causing air to be drawn into the final lipstick bullet, and causing pinholing (an effect which shows mostly after flaming).

The ideal size of nozzle, therefore, delivers mass at a reasonable pace, allowing it to flow first down one side of the mould cavity and then up the other, without premature setting of the mass, and without the production of turbulence. The ideal rate seems to be about one row of cavities/s.

Rate of dispenser stirring

This obviously depends on the shape of the paddle, but the top of the molten mass should show a gentle ripple. It is too fast if the surface is broken by vortexes, and too slow if the surface barely appears to be circulating.

A pearl lipstick should be stirred marginally faster than a plain shade, to prevent pearl fall out.

A dispenser should never be allowed to fall below half full, except for the completion of the bulk. This is because: the quality of the lipstick decreases, the incidence of titanium dioxide flotation increases, and air entrapment increases.

Oiling

Once the mould has been stripped of its bullets, the cavities should be rubbed down with a soft cloth to remove traces of lipstick which may have adhered. The mould is then re-assembled and clamped. A cottonwool bud is prepared by wrapping cottonwool around an orange stick, so that the bud just fits the cavity, the swab is then dipped into the chosen release agent and excess oil squeezed off. The swab or bud is re-oiled after each length of cavities (20 per line normally) and the next line oiled, etc.

If the operation has been performed successfully then only a trace of oil should be on the mould surface and there should be no droplets of oil at the cavity tip.

Moulding

The freshly oiled mould is placed onto the hot plate for about 5 min until it is warm to the touch, i.e. just above lukewarm, approx. 45°C. Rotatherms can be incorporated into the moulds, but generally the operators touch was found to be just as accurate!

Once the mould is at the correct prewarmed temperature, a few grams of mass is run out of the dispenser to clear the nozzle and the edge of the mould offered to the tip of it. The dispenser is run off slowly at first to clear the first few rows of cavities and the flow rate gradually increased to maximum.

Once the mould has been filled it is allowed to sit on the bench at ambient temperature for 3-5 min until such time that the top sets and dimples start to appear over the cavities.

The mould will feel cooler, just warm to the touch (about 40°C) and when the dimples are fairly pronounced, but full coring not evident, the surface mass is removed with a scraper and the mould placed on a cold surface. After a further few minutes the mould is placed on a piece of wax paper on the refrigerated surface. The mould is removed when it feels cold or chilled and the clamps are removed. The sticks are examined carefully and packed off into biscuit tins divided using wax paper between layers. The tins are refrigerated for 48 h prior to assembly and flaming.

Moulding trials

The following sticks were moulded under the specified conditions mentioned previously in this report:

- (i) Plain shade via direct method;
- (ii) plain shade via concentrate method;
- (iii) pearl shade via direct method;
- (iv) pearl shade via concentrate method;
- (v) glosser shade via direct method;
- (vi) glosser shade via concentrate method.

The direction of pouring was carefully noted, and sticks laid out in mould order. The areas of greatest interest are theoretically anticipated as the areas I, II and III in Fig. 4.

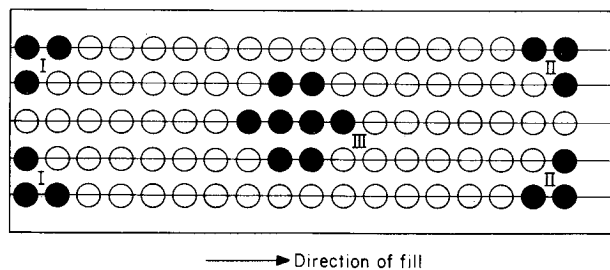


Figure 4. Lipstick mould showing direction of fill and areas of greatest interest.

Rotomould[®] method

General guidelines

The bulk is melted in a premelt area and the machine dispenser kept filled during production. The dispenser is kept at a temperature of between 75°C and 85°C depending on the set point of the bulk. The dispenser has twin nozzles which release bulk into a pair of cavities which pass beneath in a large moving ring containing such pairs of cavities (Fig. 5).

The rotating ring passes through three areas:

- (i) the prewarming chamber – prior to filling;
- (ii) the cooling chamber – immediately after filling;
- (iii) the refrigerated chamber – prior to ejection.

Immediately after the filling head are two top heaters to ensure that the bulk sets from the bottom of the cavity upwards, by keeping the top molten. This prevents contraction deformation in the stick.

Eventually the cavities align under a plate containing two sockets into which two empty cases are placed inverted. Compressed air is blasted through the base of the cavity, shooting

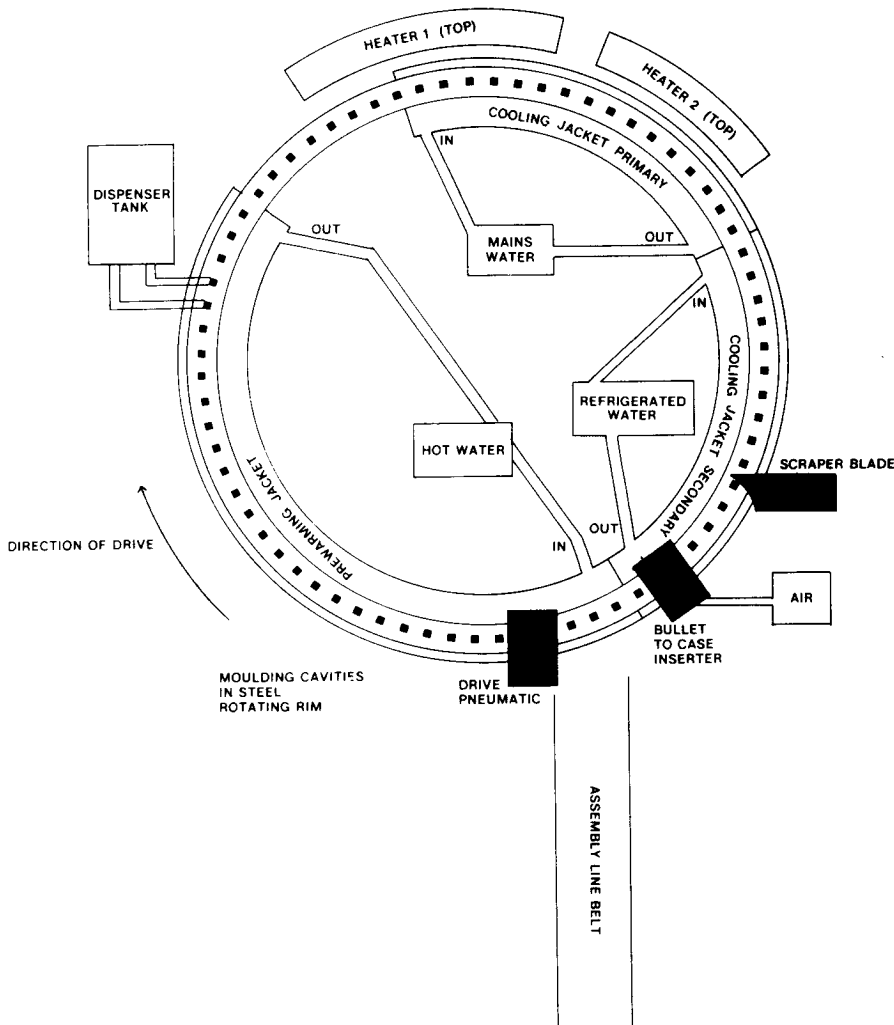


Figure 5. Rotamould[®] method of moulding.

the lipstick bullet into the case. The lipsticks are passed to a moving belt on which they are flamed and the mechanism wound down, caps assembled, etc.

Ejectoret[®] machine

General guidelines

The following parameters should be adjusted to satisfaction prior to operating the machine (12, 13) (Fig. 6).

Mass holding tank

The mass holding tank should be set at around 85°C, which is approximately 15°C above the setting point of the lipstick mass. The cooling and heating coils around the tank ensure that the set temperature can be maintained $\pm 0.5^\circ\text{C}$.

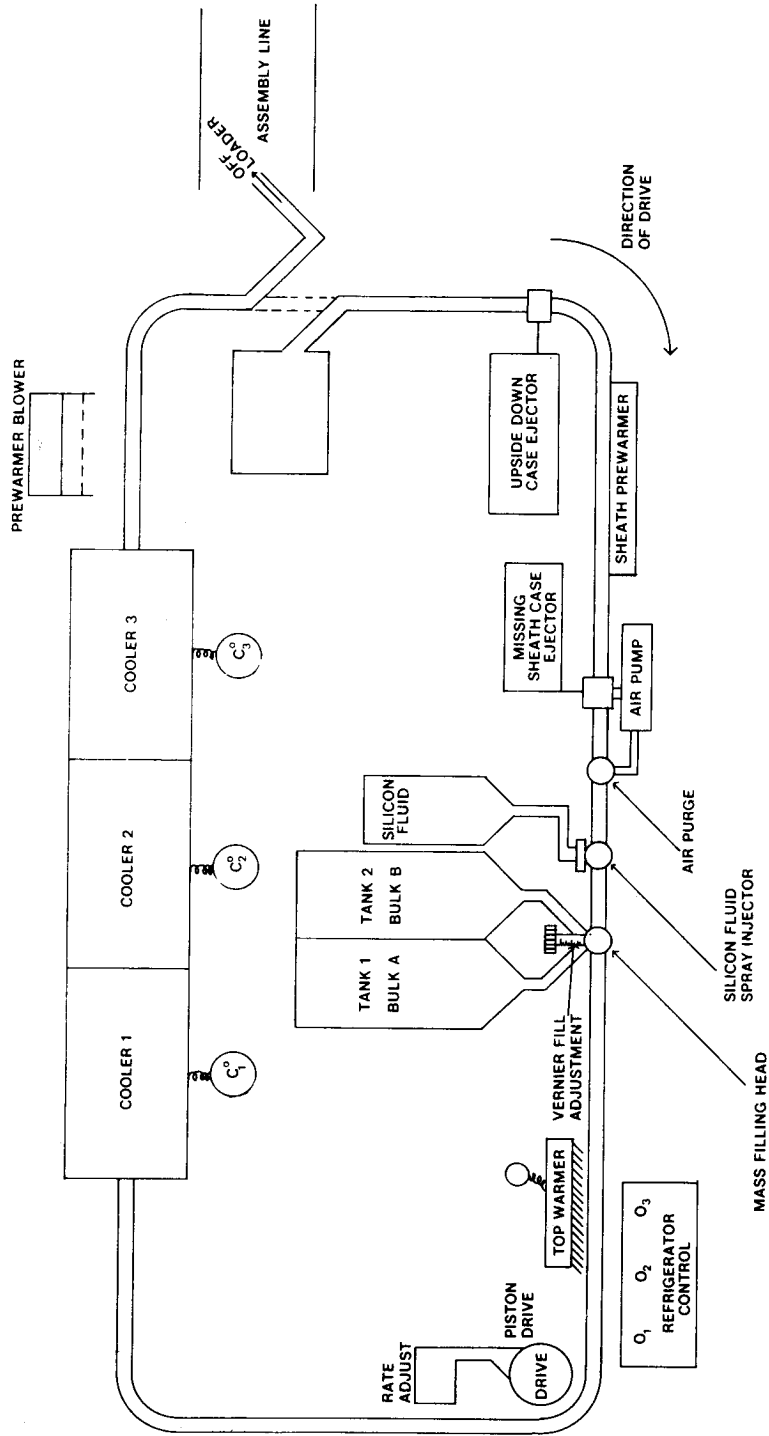


Figure 6. Layout of Ejectorette® Ipstick machine.

Silicon oil release agent spray

The machine should be allowed to run with six lipsticks through the silicon spray head and mass filling head, then allowed to cool for a few minutes until set. The sheaths should then be ejected and the bullets examined.

A normal stick exhibits a sheen, usually on one side extending over about 60% of the total visible surface of the bullet. There should be no visible droplets (like condensation) on this surface, but an even oil coating. A stick which is lacking in silicon oil is generally harder to desheath, has less gloss/surface area and may in extreme cases scuff on the removal of the sheath. Increase or decrease of the silicon oil as required may be facilitated by means of the silicon spray timer located on the control panel. In extreme cases the silicon oil delivery can be adjusted by means of a needle valve situated above the silicon spray nozzle.

A further six trial bullets should be run to check the adjustment. The oil on these sticks will migrate, once the sheaths have been removed, to cover the remainder of the stick which may originally have been dull. At 15–20 min from desheathing migration of the oil is well under way, and after 1 h the process should be complete. However, the ambient temperature could well be an influencing factor and these times could increase or decrease according to the temperature of the day.

The machine should never be run without silicon oil injection, as the reject rate on desheathing will be high, as will the lack of gloss finish.

The extractor fan over the spray should always be in place and running, to prevent possible build up of the silicon fluid in the lungs.

Adjustment of the mass fill

The adjustment of the fill weight can be carried out at the same time as the adjustment of silicon fluid spray. The machine has a calibrated vernier adjustment on the nozzle which presets the fill quite accurately.

Adjustment of the top warmer

The function of this stage is to prevent the top from setting before the middle of the stick has set, otherwise contraction dents can form at the centre of the stick instead of via the coring process at the top. This particularly applies to the softer lipstick formulae, such as lipgloss sticks. A suitable setting would be in the 200–210°C range.

Refrigeration compartments

The refrigeration compartments, three in all, decrease in temperature as the sticks approach the exit point of the machine. There is a fair degree of latitude on the settings, but a rough guide would be –10°C, –12°C and –17°C.

The machine rate

There can be no hard and fast rule for the rate which is dependent on the formula of the lipstick. However, an average rate would be in the region of 37 sticks/min with a high of 42 sticks/min.

PROBLEMS ASSOCIATED WITH LIPSTICK BULLETS

These are illustrated in Fig. 7. Table III compares faults in lipstick with type of moulding.

Table III. Comparison of faults with type of moulding

Moulding method	Aeration	Laddering	Cracking	Deformation	Cratering	Streaking	Sweating	Mushy failure	Low gloss
Split moulding	Often	Sometimes	Rarely	Sometimes	Sometimes	In high T_{10} , concentrate often	Depends on formula	Often	Always, requires flaming which can give further faults
Rotamoulding [©]	Often	Sometimes	Never	Sometimes	Sometimes	Rarely	Depends on formula	Sometimes	Always requires flaming, which gives further faults.
Ejectoret [©] moulding	Sometimes	Sometimes	Never	Sometimes	Never	Never	Depends on formula	Never	Initially but after 1 h never

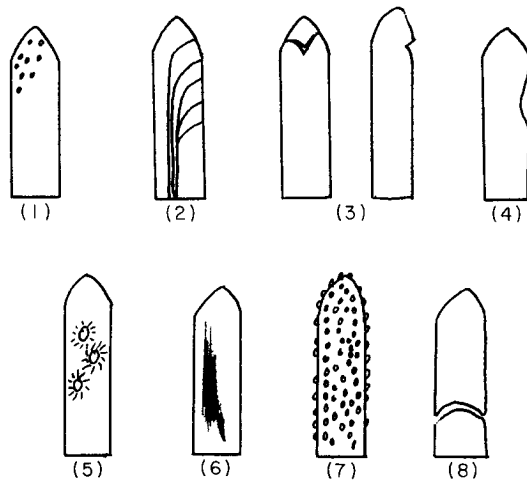


Figure 7. Problems associated with lipstick bullets. (1) Aeration, pinholing. (2) Laddering. (3) Cracked or chipped. (4) Deformation. (5) Cratering. (6) Streaking. (7) Sweating. (8) Mushy failure. An additional problem is low gloss which is not illustrated here.

Laddering

This effect is shown in Fig. 8 and may be caused by any of the following: mould too cold; bulk not hot enough; filling rate too slow.

The first of the mass just fills the cavity and sets, the next mass comes over the top of the initial mass and sets. This process repeats until a series of layers are formed thus giving a laddered appearance.

The effect is particularly noticeable in pearl formulae where the platelets orientate themselves in different configurations at the various layer boundaries.

The solution to the problem is simple – increase the initial mould temperature and/or increase the bulk temperature and/or increase the dispenser nozzle aperture, until the stick is perfect.

Lipstick deformation

This problem is most noticeable in softer formulae (i.e. those with lower than standard set points, such as lipglossers). The effect is caused by adverse temperature gradients across the

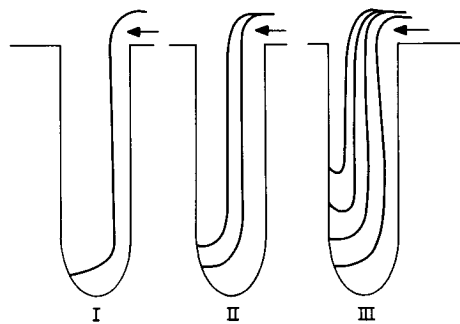


Figure 8. Formation of laddering.

stick during moulding. Contraction should always take place at the top of the mould as coring, the shrinkage at the surface of the mould just sufficient to facilitate stick release.

However, if the top of the mould sets prematurely, then coring cannot take place and the volume reduction must take place on the stick surface. In split moulding this normally occurs just above the tip, and in Ejectoret[©] moulding it appears as 'waisting' just above the rim of the godet.

The effect is prevented in Ejectoret[©] moulding by means of a top warmer which keeps the top molten until the tip has begun to set.

When split moulding the effect is normally caused by placing the mould too quickly onto the cold plate and/or prewarming the mould to too high a temperature.

Sweating

It would take another paper to explain the causes of sweating and would involve consideration of the structure of the materials used (whether linear or branch chained, functional groups, molecular length, etc), consideration of wax solubilities in relation to the oils present and finally a physical model of the lipstick structure.

Sweating is a problem directly related to formulation and not to the manufacture and handling of the bulk.

Blooming, bleeding and crystalline surface deposits

All of these problems relate to formulation, and the comments which applied to sweating also apply here.

The effect of flaming on a lipstick

The moulded stick when removed from its cavity will have a finer crystal structure on the outside than on the inside of the bullet.

This structure will be finest on the Ejectoret[©], slightly less on the Rotomould[©] and far less in a central stick from the split mould. Flaming refines the surface structure still further and reduces the fine crystal structure to hyperfine. This is achieved, obviously, by melting and cooling the surface wax rapidly, which produces so small a crystal size as to be almost amorphous or glass-like in structure, thus explaining the high gloss obtained.

Lipstick structure is not static in most cases, and rearrangements occur within the stick which can reduce this initial gloss, causing sweating, blooming, hazing, etc. The idea of a breathing lattice subject to changes in equilibrium must always be kept in mind when formulating any wax product.

Streaking

The reason for streaking is not fully understood. It is the pigment titanium dioxide that most usually exhibits this annoying phenomenon, but since the legal requirement to change over from carbon black to iron oxide black there have been isolated incidences of black flotation. The original theory for titanium dioxide flotation was that the surface coating was partially stripped off during milling to leave a non-oil compatible particle. However, with the incidence of iron oxide black flotation, which has no surface coating, this theory would seem unlikely.

The second theory is related to particle size, i.e. some particles were so small that they could not be wetted out, but would remain as a fine flotation or scum. The solution would seem to be that the titanium dioxide be milled in a surface active material such as an emulsifier — many of which were tried, all with varying degrees of failure.

The third and most likely theory is related to the surface tension of the lipstick base. Reformulation of the base, though a drastic step, does cure the problem. Modification of the base using montan wax derivatives can also cure the problem, but often produces dull sticks with structural problems. Certain silicon oils also eliminate streaking, but produce problems on flaming. The simplest solution is to change the moulding method, i.e. change from split moulding to Ejectoret[®] or Rotomoulding[®] on bulks with high titanium dioxide content. The rapid cooling does not permit sufficient time for the pigment to float to the surface whilst in the cavity.

Cratering

This effect is mostly found in split mouldings with rare outcrops using Rotomould[®]. It is a condition which shows up in flaming, when the stick develops 'dimples'.

The main cause was found to be trace amounts of silicon oils or machinery lubrication oil (from manufacturing mixers or the dispenser mixer). It is not possible to find an immediate remedy, though allowing sticks to 'rest' for 2 days prior to flaming can improve the condition.

Mushy failure

This puzzling phenomenon is more usually found in split moulding, and is not usually the result of any of the following: that the sticks are insufficiently cooled in the mould; that only the top half of a tray of bulk has been put in the dispenser, leaving the lipstick 'light' in waxes; incorrect weighing in manufacturing; sub-standard or out of specification raw material; heavy handed moulder; insufficient or excess release agent on mould. The sticks appear well set until pressure is applied to the tips to remove them, when the stick collapses. The central core of the stick lacks structure. The problem is not related to a particular formula or particular shade and arises maybe once a year for a few days then disappears. The same bulk seems to mould satisfactorily in Rotomould[®] or Ejectoret[®].

LIPSTICK STRUCTURE

It is necessary first to decide where the nearest anomalies exist. The thought of comparing a complex wax/oil blend to an ionic crystal would be illogical! There has been much discussion on wax crystallinity, but this is generally of single component behaviour and not helpful when considering a difficult mixture. Consideration of polymeric theory is helpful but lacks the depth to explain lipstick behaviour. Only when investigating the field of metallurgy does one find the closest similarity, particularly in respect to moulding and structure. Many factors are common between the two fields, such as effect of rate of cooling, contraction, break strength, crystal structure. Comparison of Ejectoret[®] and split moulded sticks to crude cast pig-iron ingots show startling similarity in structure (Fig. 9).

Manuel de Carames (14) makes the observation: 'despite the fact that they have similar melting points, one lipstick may have a hard outside layer with a soft inner core, while another is an extremely dry amorphous mass'.

Ejectoret[®] moulded stick structure

The mould in this technique is the plastic ejectable sheath contained in the case. Since the sheath is a single component surrounded on all sides by air, one may consider the mould to be exempt from the outside influence of thermal gradients. Also, since each stick is

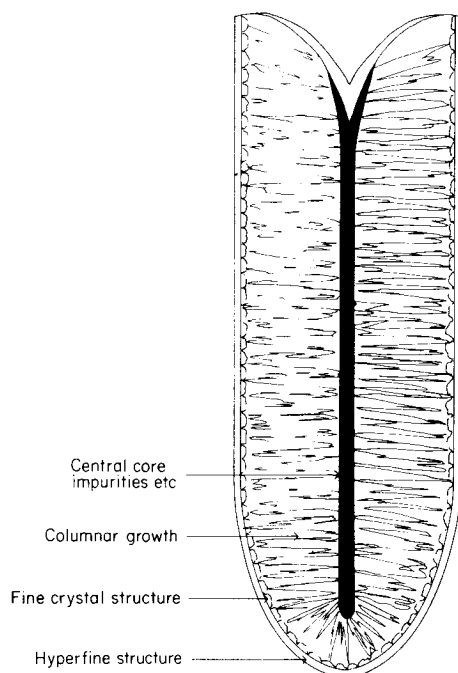


Figure 9. The pig iron model of lipstick structure.

individually moulded, there will be no influence or interaction of adjacent sticks. Thus the only thermal gradients that exist are purely in the mass (Fig. 10).

The mass entering the sheath is immediately cooled at the surface/mass interface and forms a fine grained lipstick skin from which the mass sets to form the final bullet. The skin is composed of small wax crystals or lattice mesh which become the nuclei for a fine needle shaped crystal to grow towards the centre. This needle growth is called 'columnar growth'.

However, the cooling rate is so rapid, that the initial fine structure laid down is of such a thickness that the area of columnar growth is very small. The overall structure is therefore fairly homogeneous, with the little columnar growth present running horizontally to the centre of the stick, but most of the structure being fine grained to such an extent as to be almost amorphous. Sticks that are frozen and sheared by a bending movement always break cleanly and at right angles to the applied force leaving a fairly flat surface, without concave dents or convex projections. None of the sticks examined showed any signs of a central core (which will be described shortly), and any patterning or markings were completely circular orientated.

Split moulded sticks

In this instance the *initial* picture is almost the same, with a fine grain structure lining the cavity, but considerably thinner. However, whereas the Ejectoret[®] mass fills from the centre of the cavity, the split mould mass flows from the side, and gives rise to an interesting (if not initially puzzling) phenomena of an oval rather than circular cross section orientation (Fig. 11).

As the mass initially runs down one side of the cavity it sets, but at the same time heats up the section of cavity in front of it. The mass rising on the opposite side of the cavity to

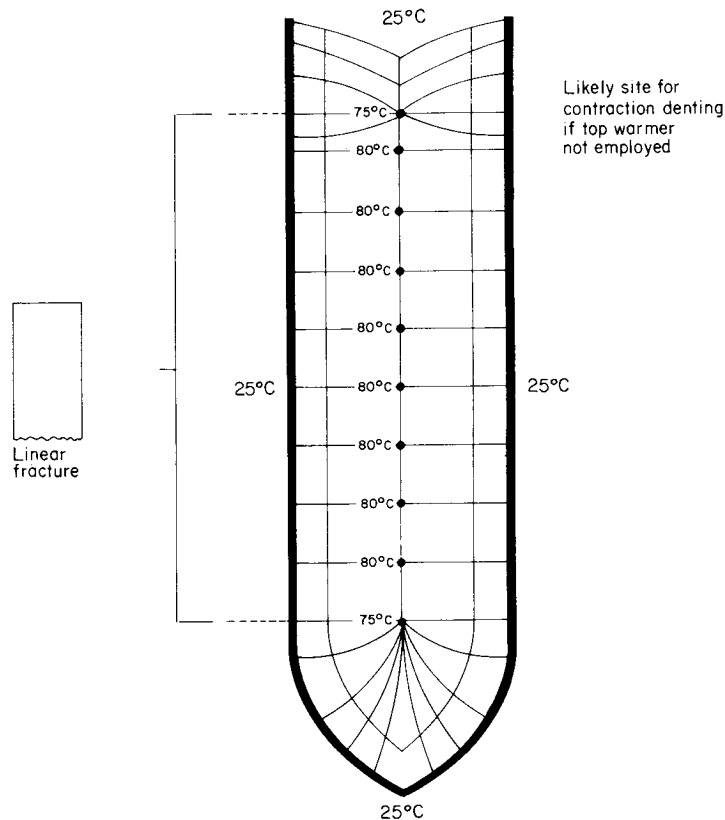


Figure 10. Ejectoret[®] moulding. Theoretical temperature gradients.

the fill also sets, forming the second half of the skin, but the section of cavity between front and back having been pre-warmed does not set, and therefore the molten mass in the centre of the cavity is oval in shape (Fig. 12).

Unlike the Ejectoret[®], the split mould cavity is surrounded by thermal gradients. The bottom of the mould stands on a cold plate, the top of the mould open to a relatively low ambient temperature, and the centre of the mould kept hot by adjacent cavities full of molten lipstick mass. Thus, whereas an Ejectoret[®] sheath containing 5 g of molten mass can set relatively unhindered in the space of 30 s, it takes 15–20 min for 500 g of molten mass and 4000 g of heated duralumin mould to cool and the sticks to set. It is this time delay together with the thermal gradients which give rise to such differing structure between the sticks from each method.

Figure 12 represents a thermal condition at a moment in time, and it can be seen that instead of the columnar growth being horizontal from skin to centre it in fact angles above and below the horizontal norm. This is because the thermal gradients produce a non-linear temperature scale within the cavity, and the columnar growth must proceed from a cool site to a warmer one. Sticks broken show both concave and convex structures depending on how the sticks set and where the fracture site occurred. In pearlised sticks particularly, the columnar growth and central oval core may be clearly seen, and on some occasions the fine structure of the skin may also be seen in the sharp edge at the site of fracture. The core itself is due to the slow cooling rate, where fractional crystallisation takes place as the mass

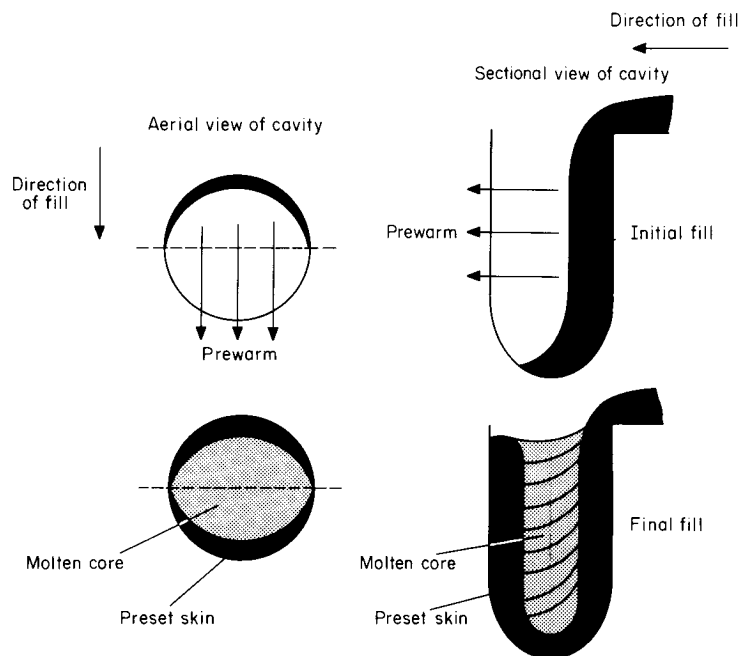


Figure 11. Split moulding. Formation of an oval core.

cools. Thus, the higher melting point waxes such as carnauba tend to set before the lower melting point waxes such as beeswax. In this way the centre of the stick has a higher concentration of oils and lower melting waxes than the skin. It is also suspected that a higher concentration of impurities would be found at the centre. Broken sticks have also shown pearl migration to centre. It is hoped that in the near future the analytical data will have been completed to verify conclusively this theory.

The shape of the central core is not expected to be linear, but to section a single lipstick into more than two pieces cannot be achieved at the present time. However, breaking sticks of the same moulding in different positions does show a variation (be it a small one) in core size (Fig. 13).

Preparation of samples for examination of crystal structure

The sticks are assembled into cases and stood in the freezer compartment for at least 24 h at -18°C to -20°C . After this time they are removed one at a time and snapped using thumb and forefinger of each hand, e.g. like snapping chocolate. At least 30% of the sticks either break off low in the godet or become smudged, making interpretation of the structure impossible. The introduction of a small cut into the middle of the stick to provide a site for brittle failure sometimes helped, but usually it breaks at the top of the godet.

EVALUATION OF LIPSTICKS USING PHYSICAL TESTING METHODS

Investigation of hardness (3, 16, 17)

The lipstick is held in a block and placed on the compression cell of an Instron tensiometer and tared to zero. The tensiometer is then tared for a full scale deflection of 100 g. The

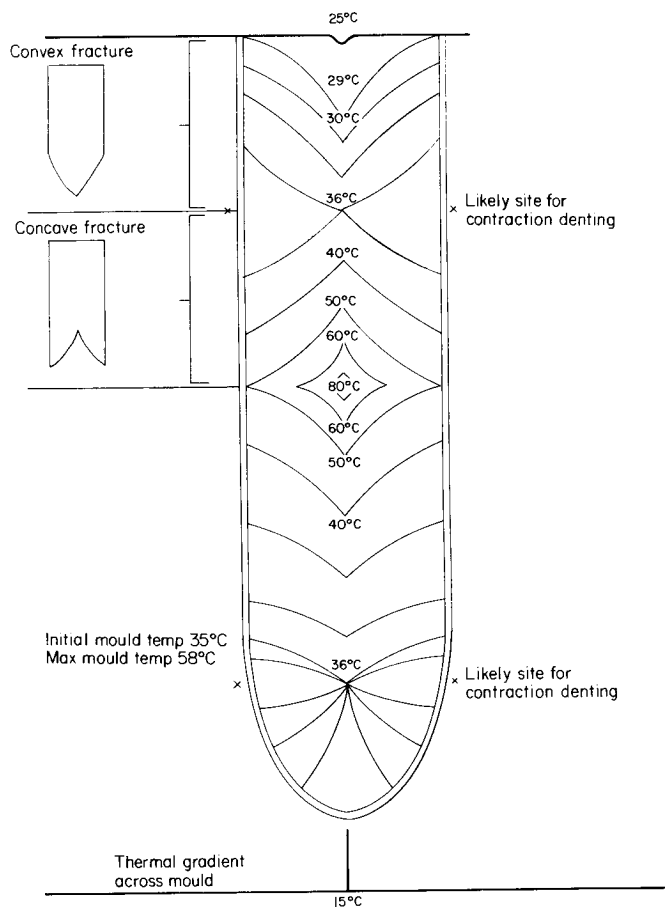


Figure 12. Split moulding. Theoretical temperature gradients.

hardness is determined by the maximum force required to penetrate the lipstick with a needle 0.52 mm in diameter. (Larger needles crack the stick, and smaller give small values for the penetration loading.)

The Instron is set for a traverse of 0.2 cm/min^{-1} and run for 4.2 min (i.e. equivalent to a penetration of 0.84 cm). Samples are tested at least three times and an average result taken.

Investigation of break strength (3, 5, 18-21)

There have been a number of methods discussed in the literature regarding the evaluation of break strengths in lipsticks.

The method used in this paper is another to add to the list, and considers not only the physical strength of the stick, but also the interaction of the stick with the case, thereby giving a 'consumer-related' result. The lipstick is held at an angle of 45° and traversed at 0.05 cm/min^{-1} onto the compression cell plate. The break strength is the maximum recorded force to breakage.

Investigation of pay-off

There have been very few descriptions for a method to evaluate the pay-off of a lipstick, and

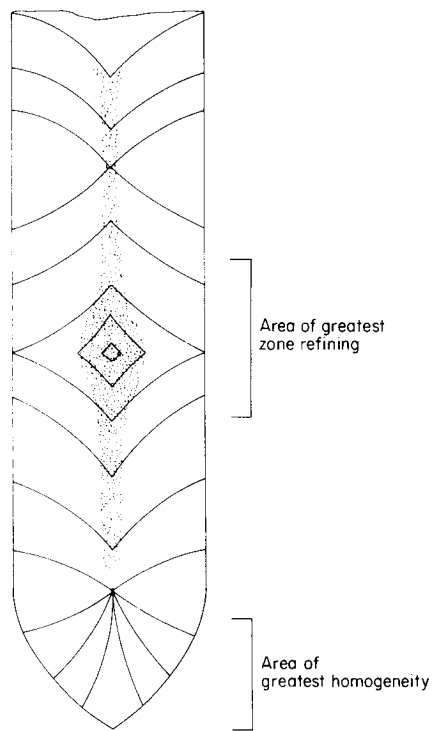


Figure 13. Split moulding. Anticipated shape of central core.

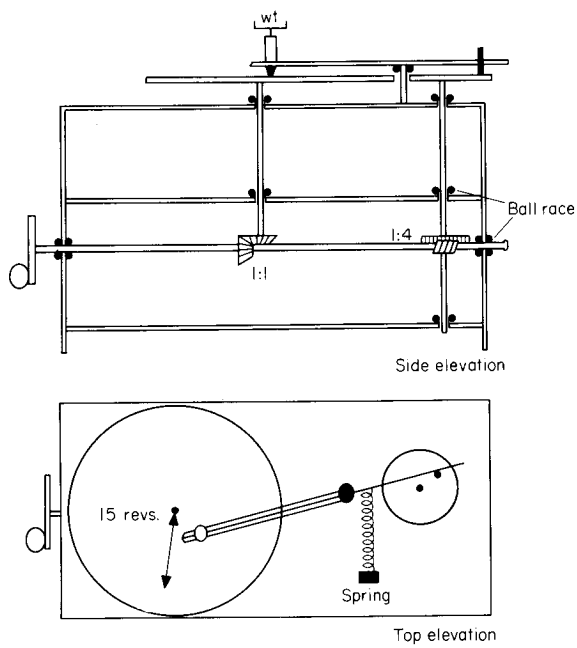


Figure 14. Construction of the pay-off testing equipment.

it was for these reasons that we constructed an apparatus suiting our own requirements (Fig. 14).

A filter paper is preweighed and pinned to the platter. The lipstick under test is removed from the 22°C preconditioning oven and the top cut off using a scalpel. This stick is lowered onto the outer rim of the paper and loaded with a 100 g weight, the handle is then turned at the rate of 1 turn/s⁻¹. As the platter rotates under the stick, so by a series of gears and cams the arm slowly arcs towards the centre. After 15 revolutions the paper is covered with lipstick and is removed for weighing, simple subtraction of the first weighing leaves the weight of applied lipstick.

RESULTS

Structure examination

The results are shown in Figs 15-17.

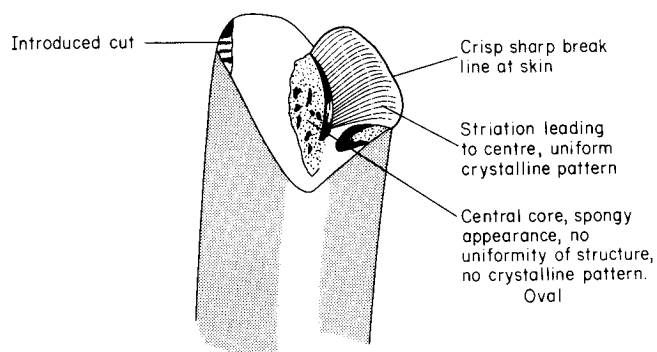


Figure 15. Fractured split moulded sample.

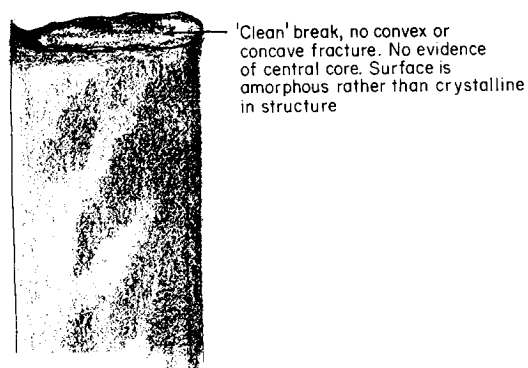


Figure 16. Ejectoret[®] moulded stick. Diagram of cross-section on breakage.

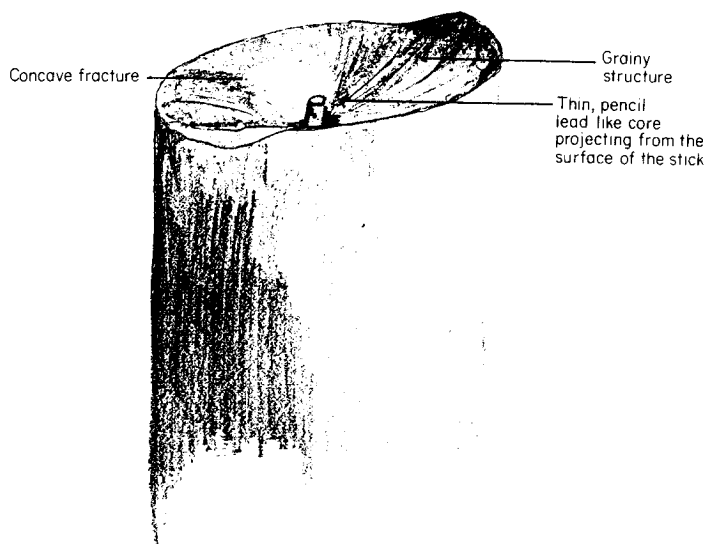


Figure 17. Rotamoulded[®] stick of fracture. The structure compares very closely to the split moulded structure, but the central core is very much smaller and more compact. The structure though similar tends to have a grainy rather than crystalline appearance, indicative of a trend to the amorphous state of Ejectoret[®]. The fractures are less convex than those found in split moulding, i.e. less deep, which again indicates an intermediate structure to split moulding and Ejectoret[®] moulding.

Physical tests

The results are listed in Tables IV and V and expressed graphically in Figs 18-21.

Manufacturing methods

Table IV shows that, when all results for lipstick type, lipstick moulding method and tests undergone, are averaged out, the difference between these mean results for the direct manufacturing method versus the concentrate method is statistically significant for plain and pearl split moulded samples when tested for penetration hardness and break strength.

Moulding methods

Table V* shows that the standard deviation in the results of the physical tests between different moulding methods for each lipstick type is proportionately as follows:

(1) Pearl lipsticks: penetration hardness and break strength results show the largest s.d. for the Ejectoret[®] method and the smallest for the split moulded method.

(2) Plain lipsticks: penetration hardness and break strength results do not follow the same pattern.

(3) Gloss lipsticks: again penetration and break strength results do not follow the same pattern.

Pay-off results are too small to register any significant differences in s.d.

Penetration hardness

Figure 18 shows that Ejectoret[®] lipsticks (plain and pearl) harden considerably between 1

*Plain and pearl split mould results expressed separately for direct and conc. methods following results in Table IV.

Table IV. Significance of difference of means between physical tests on different manufacturing methods (Student's 't'-test results)

Lipstick	Method	Test	't'-test	
Plain	Rotamould [®]	Break strength	t=1.80	P > 0.10 N.S
	Ejectoret [®]	Break strength	t=1.98	P < 0.10 N.S
	Split	Break strength	t=2.82	P < 0.01 V.sig
	Rotamould [®]	Penetration	t=0.61	P > 0.10 N.S
	Ejectoret [®]	Penetration	t=0.39	P > 0.10 N.S
	Split	Penetration	t=2.91	P < 0.01 V.sig
	Rotamould [®]	Pay-off*	t=4.04	P < 0.10 N.S
	Ejectoret [®]	Pay-off*	t=0.45	P > 0.10 N.S
	Split	Pay-off*	t=1.39	P > 0.10 N.S
Pearl	Rotamould [®]	Break strength	t=0.76	P > 0.10 N.S
	Ejectoret [®]	Break strength	t=0.30	P > 0.10 N.S
	Split	Break strength	t=6.74	P < 0.001 V.sig
	Rotamould [®]	Penetration	t=2.35	P < 0.05 N.S
	Ejectoret [®]	Penetration	t=0.22	P > 0.10 N.S
	Split	Penetration	t=3.83	P < 0.001 V.sig
	Rotamould [®]	Pay-off*	t=0.45	P > 0.10 N.S
	Ejectoret [®]	Pay-off*	t=0.45	P > 0.10 N.S
	Split	Pay-off*	t=0.72	P > 0.10 N.S
Gloss	Rotamould [®]	Break strength	t=1.12	P > 0.10 N.S
	Ejectoret [®]	Break strength	t=0.76	P > 0.10 N.S
	Split	Break strength	t=0.27	P > 0.10 N.S
	Rotamould [®]	Penetration	t=0.42	P > 0.10 N.S
	Ejectoret [®]	Penetration	t=0.86	P > 0.10 N.S
	Split	Penetration	t=2.01	P < 0.10 N.S
	Rotamould [®]	Pay-off*	t=1.35	P > 0.10 N.S
	Ejectoret [®]	Pay-off*	t=0.45	P > 0.10 N.S
	Split	Pay-off*	t=1.02	P > 0.10 N.S

*Small number of samples investigated.

and 4 weeks after moulding. Variation in split moulded and Rotamoulded[®] lipsticks is far less with time. Rotamoulded[®] lipsticks tend to be softer than Ejectoret[®] or split moulded lipsticks. Very little difference in hardness is shown, either with time or moulding method, for gloss lipsticks.

Break strength

Figure 19 shows generally that Ejectoret[®] lipsticks are stronger than Rotamoulded[®] and split moulded lipsticks. Rotamoulded[®] lipsticks being the weakest. Changes with time are not so marked (for Ejectoret[®] samples) as shown by the penetration hardness results.

Comparison of penetration and break strength results with time, lipstick type and moulding method shows a statistically very significant relationship between the two sets of results ($r = 0.6994$; $P < 0.01$).

Pay off

Figure 20 shows that for all moulding methods pay-off follows the trend

$$\text{gloss} > \text{plain} > \text{pearl}$$

No significant variation of pay-off with moulding type is apparent.

Comparison of penetration and pay-off results shows a statistically highly significant relationship between the two sets of results ($r = 0.8449$; $P < 0.001$).

Break strength/split mould position

Figure 21 shows that for plain and pearl lipsticks, samples moulded in Position I are stronger than those in Position II, and Position II lipsticks are stronger than Position III samples. The difference between I and II being larger than that between II and III.

Direct method pearl samples are stronger overall than concentrate pearl samples, but the converse occurs for plain samples. No similar patterns are shown for gloss samples.

Graphical representation of penetration results/split mould position does not follow the clear pattern shown in Fig. 21 for the break strength results (possibly due to the lower values obtained). However, a comparison of results of split mould position penetration versus split mould position break strength shows a statistically highly significant correlation ($r = 0.8298$; $P = < 0.001$).

Table V. Standard deviation of physical properties resulting from different moulding methods

Lipstick	Method	Test	Mean \pm s.d.	% S.d. of mean
Pearl	Rotamould [®]	Break strength	208.5 \pm 26.1	12.5
	Ejectoret [®]	Break strength	367.3 \pm 66.6	18.1
	Split	Break strength	320.3 \pm 19.4	6.1
			383.2 \pm 30.5	7.9
			46.6 \pm 3.9	8.4
	Rotamould [®]	Penetration	38.2 \pm 3.7	9.7
	Ejectoret [®]	Penetration	39.3 \pm 17.3	44.0
	Split	Penetration	41.2 \pm 3.8	9.3
	Rotamould [®]	Pay-off	0.21 \pm 0.01	4.7
	Ejectoret [®]	Pay-off	0.23 \pm 0.01	4.3
	Split	Pay-off	0.24 \pm 0.01	4.1
	Plain	Rotamould [®]	Break strength	171.6 \pm 31.3
Ejectoret [®]		Break strength	253.8 \pm 14.5	5.8
Split		Break strength	273.5 \pm 34.8	12.7
			241.3 \pm 27.2	11.2
			39.0 \pm 3.2	8.3
Rotamould [®]		Penetration	38.1 \pm 2.5	6.5
Ejectoret [®]		Penetration	35.2 \pm 11.4	32.5
Split		Penetration	43.3 \pm 4.8	10.9
Rotamould [®]		Pay-off	0.31 \pm 0.01	3.2
Ejectoret [®]		Pay-off	0.29 \pm 0.01	3.4
Split		Pay-off	0.31 \pm 0.01	3.2
Gloss		Rotamould [®]	Break strength	100.5 \pm 17.8
	Ejectoret [®]	Break strength	214.7 \pm 27.4	18.8
	Split	Break strength	194.6 \pm 21.7	11.1
	Rotamould [®]	Penetration	17.7 \pm 1.7	9.3
	Ejectoret [®]	Penetration	18.8 \pm 1.2	6.4
	Split	Penetration	17.6 \pm 2.4	13.7
	Rotamould [®]	Pay-off	0.39 \pm 0.01	2.5
	Ejectoret [®]	Pay-off	0.38 \pm 0.01	2.6
	Split	Pay-off	0.40 \pm 0.01	2.5

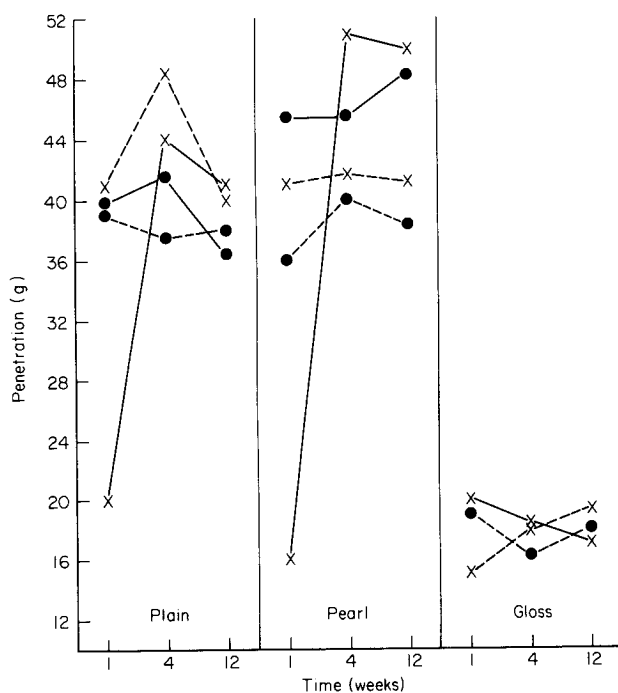


Figure 18. Penetration hardness results for different moulding methods of different lipstick formulae with time. (●- - ●) Rotamould[®] (mean standard + direct); ((X-X) Ejectoret[®] (mean standard + direct); (X- -X) split (mean standard); (●-●) split (mean direct).

CONCLUSIONS

Manufacturing methods

The two methods show a statistically significant difference only for split moulded pearl and plain samples. Gloss samples contain such a small quantity of colour it is not surprising that no difference shows up. Pay-off results give very small numbers and less samples were tested for pay-off than for penetration and break strength during this investigation due to the unavailability of the pay-off equipment until a late date.

Where the difference resulting from the two methods is significant, a similar pattern is shown between break strength and penetration, i.e. in plain samples the direct method is softer and weaker than concentrate method whereas in pearl samples the concentrate method is softer and weaker than direct method.

The fact that the differences only show up in split moulded samples is probably due to the slower cooling rate allowing more movement of colour particles (hence no difference in low colour gloss samples). The inversion of plain and pearl results must therefore be caused by the presence and absence of pearl particles in addition to the colour particles.

Moulding methods

No real pattern emerges from these results to indicate whether or not one method is more variable than another. While we knew that split moulded lipsticks differed depending on

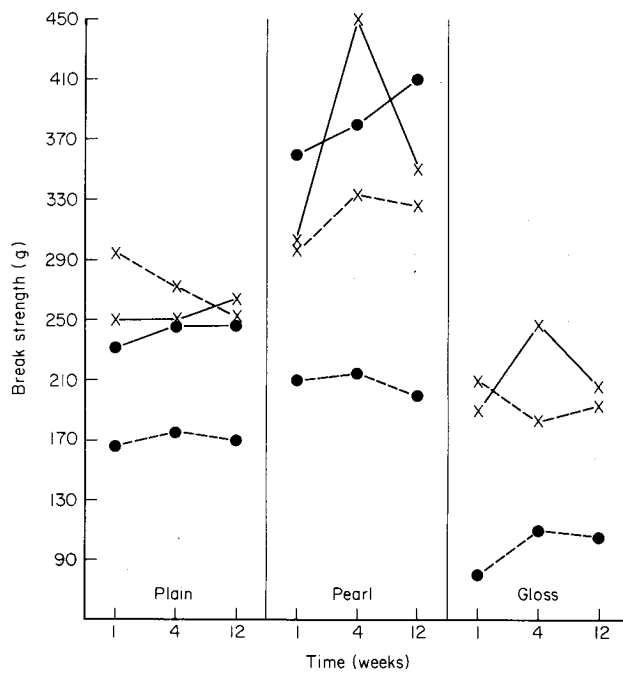


Figure 19. Break strength results for different moulding methods of different lipstick formulae with time. (●- -●) Rotamould® (mean standard + direct); (X-X) Ejectoret® (mean standard + direct); (X- -X) split (mean standard); (●-●) split mean direct.

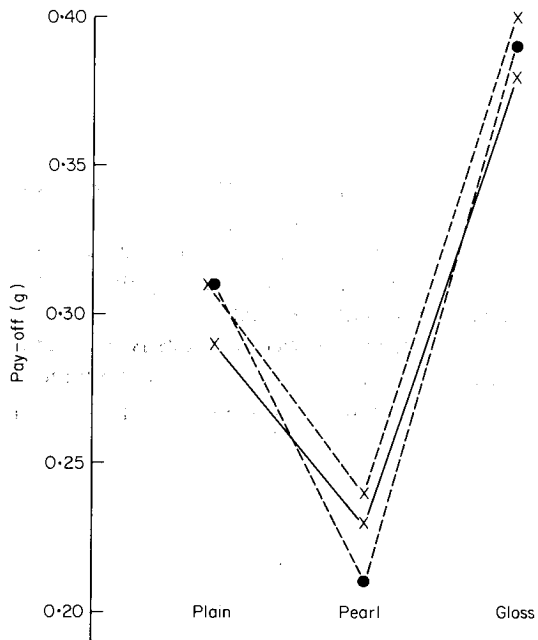


Figure 20. Variation of pay-off for different moulding methods of different lipstick formulations. (●- -●) Rotamould® (mean total); (X-X) Ejectoret® (mean total); (X- -X) split (mean total).

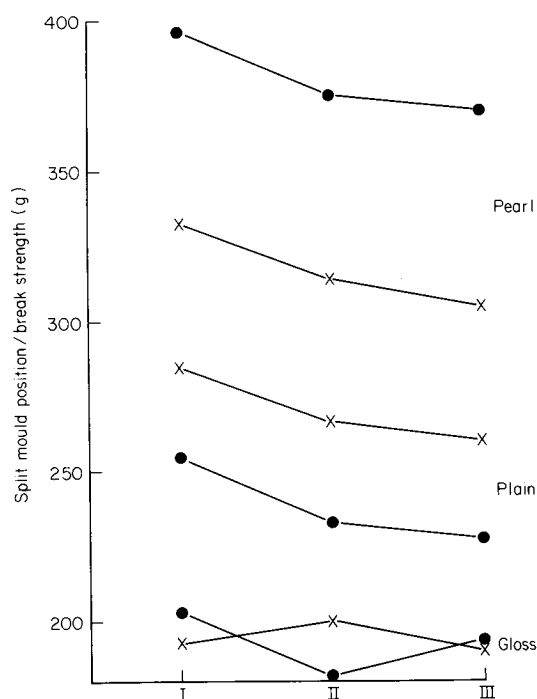


Figure 21. Variation of break strength with position in split mould for different lipstick formulae and different manufacturing method. (●-●) Direct; (X-X) standard.

their mould position, the overall variation in finished stick properties is generally less than that in Rotamoulded[®] and Ejectoret[®] moulded sticks. In these investigations only one batch of each manufactured bulk was used to test the moulding methods. In order to gain a realistic picture of which method is the most varied a large number of manufactured bulks should be tested, but, since each bulk would need to be moulded by each technique, this is a rather complicated task to perform in a commercial environment. However, all sticks examined were perfectly satisfactory from a users viewpoint.

Penetration hardness results

It seems that the very rapid cooling involved during Ejectoret[®] moulding gives an unstable soft structure which equilibrates itself during the first month, to give a much harder stick. Slower cooled sticks, rota mould and split mould form a more stable structure which does not appear to vary significantly with time.

Break strength results

As expected, Ejectoret[®] moulded lipsticks are stronger than other types due to the fact that they are actually moulded in the godet. Rotamoulded[®] lipsticks are mechanically shot into their godets and split moulded lipsticks are fitted by hand. It is interesting therefore to note that hand fitted (split moulded) samples were found to be more secure in their godets than those mechanically fitted by the Rotamould[®].

Break strength and penetration results were shown to correlate. Logically a harder stick

(unless extremely brittle) would be stronger, but firmness of fit in the godet is an additional factor. This factor shows itself for Ejectoret[®] lipsticks by inspection of Figs 18 and 19 which show Ejectoret[®] lipsticks to be extremely soft at one week and very much harder at 4 weeks. Break strength results show a much less marked difference between 1 and 4 weeks, indicating that the secureness of fit in the godet of Ejectoret[®] moulded samples does play a contribution to the break strength of the stick.

Pay-off

As expected, pay-off results are directly related to the softness/hardness of the stick. Differences between moulding methods and manufacturing methods did not show up. This may be because they do not affect the pay-off in any way, but it may also be due to the fact that we have not had sufficient time to obtain the greatest sensitivity out of the equipment, which is a prototype and only recently obtained.

Break strength/split mould position

The results for plain and pearl samples show that sticks moulded in Section I are stronger and harder than those in II, and sticks moulded in the middle of the mould (III) are softer still. This can be explained by the difference in cooling rates of the three positions: assuming that the lipstick bulk being poured is at a constant temperature, Position I samples will cool quickest due to the greatest heat differential between mould and bulk, as the mould is filled, its temperature will increase so that it will be warmer for Position III sticks, and hotter still for Position II sticks. However, Position II has external surfaces and will therefore cool quicker than Position III sticks which have no external (air cooled) edges. Position II sticks will therefore cool the slowest, be mushy in texture and therefore be the softest.

The physical test results and the examination of structure can not lead us to a firm conclusion about which lipstick manufacturing or moulding process is best. However, consideration of less scientific parameters leads to a definite conclusion!

The direct method of manufacture is more costly from the capital expenditure outlay, but less time intensive and requires fewer personnel to operate.

The Ejectoret[®] has a high capital outlay but has the highest output, the lowest labour rate and the lowest reject rate. It will handle softer bulks than Rotamould[®] or split mould with fewer of the quality control reject categories. Finally the number of stages involved in moulding is reduced considerably with no need to assemble or flame the final stick.

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