1 SCOPE

1.1 This Standard prescribes the requirements and methods of sampling and test for Lipstick / identified as lipstick by the regulatory body.

1.2 Lipgloss or lip rouge is not covered by this standard.

2 REFERENCES

NS 145:2044	Random sampling method
NS 17:2037	Rules for Rounding off numerical Values
NS 74: 2041	Distilled Water
ISO /TR 17276	Cosmetics - Analytical approach for screening and quantification methods for heavy metals in cosmetics
NS: XXXX1	Cosmetics- Classification of Cosmetic raw materials and adjuncts
NS: XXXX2	Method of test for Microbiological examination of Cosmetics and cosmetics raw materials.

3 REQUIREMENTS

3.1 Description

The lipstick shall be firm but not brittle in texture. It shall have an attractive appearance, pleasant taste and feel on the lips and shall be reasonably free from sweating, bloom and rancidity.

3.2 Ingredients

3.2.1 *Dyes, colours and pigments*

The dyes, colours and pigments used in the manufacture of lipstick shall comply with NS XXXX1 Annnex A and shall be of food grade**.**

3.2.2 *Other ingredients*

Ingredients other than dyes, colours and pigments shall comply with the provisions of NS XXXX1 (Annex C,D and E) and shall be of food grade**.** Prohibited Substances as listed in Annex B of NS:XXXX1 shall not be used .

3.3 The lipstick shall also comply with the requirements given in Table **1** when tested in accordance with the relevant methods given in Column (4) of the table.

TABLE 1 – Requirements for lipstick

SI No. (1)	Characteristic (2)	Requirement (3)	Method of test (4)
i)	Softening point, °C, min	55	Annex B
ii)	Peroxide number, max	10	Annex C
iii)	Breaking load value, min	200	Annex D
iv)	Particle size of undispersed pigments, microns, max	40	Annex E
v)	Pay Off test	To pass the test	Annex F
vi)	Microbiological examination	Not more than 100 microorganisms per g	Annex G/ NS XXXX2
vii)	Lead (as Pb), mg/kg, max	10.0	ISO/TR17276
	Arsenic (as As), mg/kg, max	3.0	ISO/TR17276
	Mercury (as Hg), mg/kg, max	1.0	Annex H
	Cadmium (as Cd), mg/kg, max	3.0	ISO/TR17276

For the purpose of deciding whether a particular requirement of this specification is complied with, the final value, observed or calculated, expressing the results of a test or an analysis shall be rounded off in accordance with NS 17.

4 PACKING AND MARKING

4.1 Packing
Each lipstick shall be packed in a metallic, plastic or any other suitable container, which shall not provide deleterious effect on the product from light, humidity and temperatures.

4.2 Marking
Each container shall bear a label marked with the following information and have a label marked or printed legibly and indelibly with following:
a) Name of the material
b) Manufacturers name, address and its recognized trade mark, if any
c) In the case of imported products, Name and address of the importer/distributor/marketer in Nepal
d) Maximum retail price
e) Brand name
f) Colour
g) mass, in g or kg;
h) Batch or code or lot identification number;
i) Date of manufacture;
j) Best before / shelf life;
k) Instruction, for use where necessary; and
l) Special precautions to be observed in use, if required;
m) Specific warning statement necessary or appropriate to prevent health hazard, if any;

4.3 The following information shall be legibly and indelibly marked on each package:
a) Name of the material
b) Manufacturers name, address and its recognized trade mark, if any
c) In the case of imported products, Name and address of the importer/distributor/marketer in Nepal
d) Brand name, if any; and
e) Number of containers.

5 SAMPLING
The method of drawing representative samples of the product for ascertaining conforming to the requirements of this specification shall be prescribed in Annex **A**.

6 METHODS OF TEST
Tests shall be carried out as per the methods given in Column (4) of Table 1 of this specification. Unless otherwise specified all reagents used shall be of recognized analytical grade and wherever water is mentioned distilled water (conforming NS :74) or de-ionized water shall be used.

ANNEX A
COMPLIANCE OF A LOT
The sampling scheme given in this Annex A should be applied where compliance of a lot to the requirements of this standard is to be assessed based on statistical sampling and inspection. Where compliance with this standard is to be assessed based on manufacturer's control systems coupled with type testing and check tests or any other procedure, an appropriate scheme of sampling and inspection should be adopted.

A.1 LOT

In a single consignment, all the packages containing lipsticks of the same size (mass) and colour representing the same batch of manufacture shall constitute a lot.

A.2 SCALE OF SAMPLING

(i) Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of this specification.

(ii) The number of packages to be selected from a lot shall be in accordance with Table 2.

TABLE 2 - Scale of sampling

No. of Packages in the lot (1)	No. of Packages to be selected (2)
3 to 50 51 to 200 201 to 400 401 to 650 651 and above	3 4 5 6 7

(iii) The packages shall be selected at random. In order to ensure randomness of selection, random number tables as given in NS 145 shall be used.

A.3 PREPARATION OF SAMPLE

Two containers shall be drawn from each package selected as in A.2 . The containers so selected shall be divided into two equal sets, each set having containers from each package selected to draw containers. One set of containers shall constitute individual samples and the contents of the other set shall be mixed to form a composite sample.

A.4 Softening point, peroxide number, breaking load, particle size and pay off test shall be tested on each of the individual sample and tests for the remaining characteristics shall be carried out on the composite sample.

A.5 CRITERIA FOR CONFORMITY

A.5.1 *For individual sample*

The mean **(***x)* and Range (*R)* for the test results shall be calculated (range being the difference between the maximum and the minimum test results). The lot shall be declared to have satisfied the requirements for tests mentioned in **A.4** if the value of expression ($x - 0.6\ R$)
for each characteristic is equal to or greater than 99.

A.5.2 *For composite sample*

The test results on the composite sample shall meet the corresponding requirements specified in Table **1**.

A lot shall be declared as conforming to this specification if it satisfies the requirements for each of the characteristic listed in Table **1**. If the requirement for any of the characteristic is not met, the lot shall be declared to have not satisfied the requirements of the specification.

ANNEX B
DETERMINATION OF SOFTENING POINT

B.1 APPARATUS

B.1.1 *Flat bottom tube ,* 12 cm long and 2.5 cm in diameter.

B.2.1.2 *Thermometer , accurate to* 0.1 °C.

B.2 PROCEDURE

Place the lipstick with protruded salve in the flat bottom tube. Fix the thermometer through a cork in such a way that the tip of the thermometer just touches the lipstick salve. Insert this arrangement into a 1-litre beaker filled with water to a level one centimetre above the upper tip of the lipstick salve. Slowly heat the water while stirring so that temperature rises at a rate

not exceeding 2 °C per minute. When the temperature reaches about 45 °C, raise the temperature at the rate of 1 °C per minute. Constantly watch the lipstick salve. Record the temperature when the salve starts bending and losing its shape.

ANNEX C
DETERMINATION OF PEROXIDE NUMBER

C.1 GENERAL

This test when carried out on dark coloured lipsticks is likely to be vitiated because the end point in determination of peroxide number may not be very sharp. In such cases, it is expected, as a good manufacturing practice, manufacturer should check rancidity of lipstick raw materials, especially vegetable oils and other rancidity prone materials regularly in lipsticks base mixtures without colours, by peroxide number test.

C.2 REAGENTS

C.2.1 *Acetic acid*

C.2.2 *Chloroform*

C.2.3 *Potassium iodide solution, saturated.*

C.2.4 *Sodium thiosulphate solution,– approximately* 0.01 M.

C.2.5 *Starch solution,* freshly prepared.

C.3 PROCEDURE

Weigh 5.0 ± 0.05 g of lipstick sample in a 250-ml conical flask and dissolve in 30 ml of acetic acid – chloroform mixture (3:2). Heat if necessary to dissolve the sample. Add 0.5 ml of freshly made saturated potassium iodide solution. Shake and after two minutes add 30 ml of distilled water and then titrate with 0.01 M sodium thiosulphate solution using starch as an indicator.

Peroxide number = Milli equivalents peroxide per 1 000 g sample

$$= \frac{A \times M \times 1000}{m}$$

A = volume, in ml, of sodium thiosulphate, and

m = mass, in g, of the sample

M = molality of sodium thiosulphate solution.

ANNEX D
DETERMINATION OF BREAKING LOAD

D.1 GENERAL

This test gives the value of maximum load a lipstick can withstand before it breaks.

D.2 APPARATUS

D.2.1 *Burette ,* 500 ml capacity.

D.2.2 *Screw chuck,* to hold the lipstick.

D.2.3 *Aluminium cup, o*f 6-cm diameter and 12-cm length with an arrangement of a hook to suspend it on lipstick salve.

D.3 PROCEDURE

Fix firmly the lipstick container with protruded salve of diameter ranging 11 mm to 13 mm, into a screw type of chuck so that the assembly is perfectly horizontal. Adjust the burette just above the lipstick salve. Make a marking at a distance of 15 mm from the base of the salve where lipstick salve sits in salve holder cap. Weigh the aluminium container along with the hook and suspend it on this 15 mm mark slowly. Release water from the burette into the aluminium container till the salve breaks. Burette reading added with the mass of the suspended container gives the breaking load of the lipstick.

ANNEX E
DETERMINATION OF PARTICLE SIZE OF UNDISPERSED PIGMENTS

E.1 APPARATUS

E.1.1 *Microscope*

E.1.2 *Glass slides*

E.2 PROCEDURE

Apply a small portion of the lipstick paste on glass slide. Press and spread it with the help of another glass slide. Separate both the glass slides. Observe one of the slides under microscope using a specially calibrated eye piece. Determine the particle size of the largest pigment particle.

ANNEX F
PAY-OFF TEST

F.1 General-This test gives the idea of mass release from the lipstick salve.

F.2 Apparatus

F.2.1 The apparatus (see Fig. 1) Consists of constant speed electric motor A of power 180 watt (0.25 hp approximately) attached to gear arrangement B, which pulls the strip of paper F (About 7cm wide) from a roller C on the another roller G fixed on platform D through supports H. A slot arrangement E having a cylindrical tube of 4 cm length and 1.7 cm diameter is also fixed on the platform.

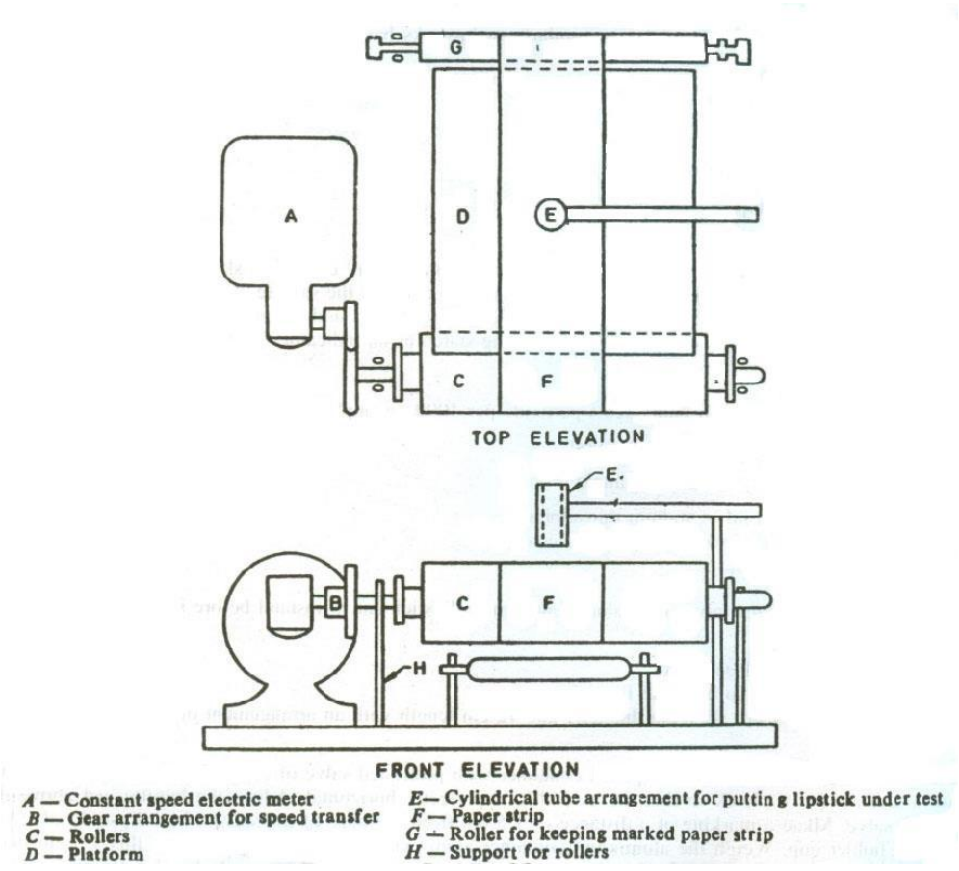

Fig. 1 Details of Pay-off Measuring Instrument

F.2.2 Constant Speed Motor - Of power 180 watt (0.25 hp approximately) attached to gear arrangement which pulls the strip of paper over a fixed platform.

F.2.3 Paper - 7 cm wide roll.

F.2**.4 Slot arrangement -** inner diameter 1.7 cm and length 4 cm (for inserting lipstick).

F.3 Procedure - Chop off the portion of lipstick salve one centimetre from the tip using a sharp blade. Rub remaining portion of the salve on a piece of paper and make the end portion perfectly flat. Run the constant speed motor and determine the time required for pulling out 100 cm of paper length. Weigh the

lipstick with chopped off tip on a balance accurately. Insert this lipstick in the slot arrangement so that the flattened salve portion rests on the surface of the paper strip (see Fig. 2). Place a total load of 50 g including mass of the lipstick on top of the lipstick. Start the constant speed motor and with the help of stopwatch allow 100cm length of paper to run. Reweigh the lipstick after the rub off and measure the length and width of the line drawn on the paper strip.

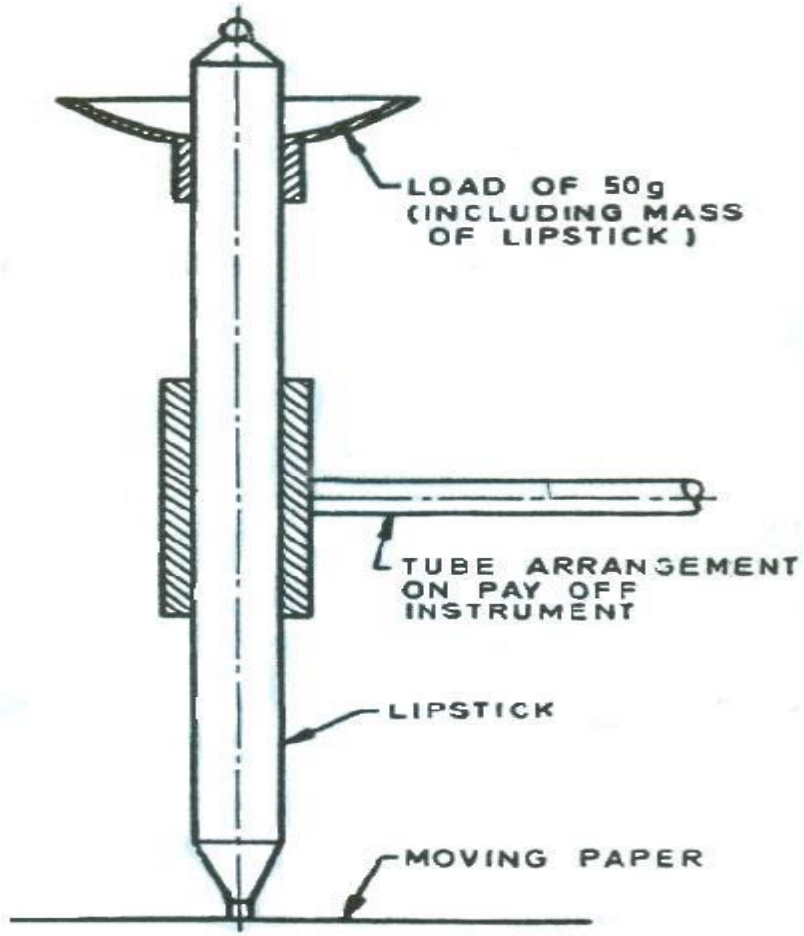

Fig. 2 Manner of keeping lipstick for test

4-7.3 Calculation

Pay-off, g/cm^2= $\frac{M1-M2}{l \times b}$

*M*1 = mass of the lipstick before the test,

*M*2 = mass of the lipstick after the test,

l = length in cm of the line drawn on paper strip, and

b = breadth in cm of the line drawn on paper strip

ANNEX G
MICROBIOLOGICAL EXAMINATION

G.1 OUTLINE OF THE METHOD

The test consists of plating a known mass of the sample on two selected culture media specifically suitable for the growth of bacteria and fungi and incubating them for a specified period to permit the development of visual colonies for counting.

G.2 APPARATUS

G.2.1 *Tubes,* of resistant glass, provided with closely fitting metal caps.

G.2.2 *Autoclaves,* of suitable size. They shall keep uniform temperature within the chamber up to and including the sterilizing temperature of 120 ℃. They shall be equipped with an accurate thermometer, located so as to register the minimum temperature within the sterilizing chamber, a pressure gauge and properly adjusted safety valves.

G.2.3 *Petri Dishes,* of 100-mm diameter and 15-mm depth. The bottom of the dishes shall be free from bubbles and scratches and shall be flat so that the medium is of uniform thickness throughout the plate.

G.2.4 *Colony Counter,* an approved counting aid, such as Quebec colony counter. If such a counter is not available, counting may be done with a lens giving a magnification of 1.5 diameter. In order to ensure uniformity of conditions during counting illumination equivalent to that provided by the Quebec colony counter shall be employed.

G.3 MEDIA

G.3.1 *Nutrient agar medium*

Dissolve 5 g of yeast extract (or meta extract), 5 g of sodium chloride and 10 g of peptone in 1 000 ml of distilled water contained in a 2-litre beaker by heating on a water bath. Add 25 g of powdered agar continue boiling until the agar is completely dissolved. Adjust the pH to 7.4 with sodium hydroxide solution using pH meter or comparator. Filter while hot through lint cloth placed in a funnel and dispense into tubes in 20 ml quantities. Filter only if necessary. Close the tubes with metal caps or cotton plugs and sterilize in an autoclave at 121 °C and 1.05 kgf/cm2 pressure for 20 minutes. After autoclaving, store the tubes in a refrigerator and use them within 3 weeks.

G.3.2 *Sabouraud agar medium*

Dissolve 10 g of peptone and 40g of glucose in 1000 ml of distilled water contained in a 2-litre conical flask by heating in water-bath. Add 25 g of powdered agar and continue boiling until the agar is completely dissolved. pH need not be adjusted (it automatically comes to 5.4). Filter while hot through lint cloth placed in a funnel and dispense into tubes in 20-ml quantities. Filter only if necessary. Close the tubes with metal caps or cotton plugs and sterilize in an autoclave at 121 °C and 1.05 kgf/cm2 pressure for 15 minutes. After autoclaving, store the tubes in a refrigerator and use them within 3 weeks.

G.4 STERILIZATION OF APPARATUS

G.4.1 *Tubes*

These shall be sterilized in the autoclave at 121 °C and 1.05 kgf/cm2 pressure for 20 minutes or in a hot air oven at 160 °C for one hour.

G.4.2 *Petri Dishes*

These shall be packed in drums and sterilized in the autoclave at 121 °C and 1.05 kgf/cm2 Pressure for 20 minutes or individually wrapped in kraft paper and sterilized in a hot air oven at 160 °C for one hour.

G.4.3 *Pipettes*

These shall be placed in pipettes cone (of copper, stainless steel, or aluminium) after plugging the broader and with cotton and sterilized in an autoclave at 121 °C and 1.05 kgf/ cm2 pressure for 20 minutes or in a hot air oven at 160 °C for one hour.

G.5 PROCEDURE

G.5.1 Melt sufficient number of nutrient agar tubes and sabouraud agar tubes in a waterbath and transfer while hot into a constant temperature water-bath maintained at 48 ± 2 °C.

G.5.2 Weigh and transfer aseptically four 0.5 g portions of the sample to four melted nutrient agar tubes, and four 0.5g portions to four sabouraud agar tubes. Shake the tubes to mix the contents thoroughly and pour into sterile petri dishes. Incubate the nutrient agar tubes at 31±0.5°C for 48h and the sabouraud agar tubes at 20°C to 25°C for 7 days.

G.5.3 Determine the average number of colonies per gram of the sample on nutrient agar tubes, as well as the average number of colonies per gram of sample on sabouraud agar tubes. The mean of the two average numbers shall be taken as the number of micro-organisms per gram of the samples.

Annex H
Determination of Mercury
by Atomic Absorption Spectrophotometric method
(Mercury vaporizer unit)

H - 1.0 Principle - Digested mercury react with reducing agent to form elemental mercury.
H - 1.1 Nitric acid -
H - 1.2 Distilled water
H- 1.3 Potassium permanganate solution
H- 1.4 Sulphuric acid
H - 1.5 Hydrochloric acid
H - 1.6 Reductant - 10% SnCl2 stabilized in 22 HCl mL
H - 2.0 Quality of Reagents
H - 2.1 Unless specified otherwise, pure chemicals and distilled waters shall be used in tests.
NOTE – 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.
H - 2.3 Apparatus/Equipments
H- 3.1 Volumetric flasks - 50 mL, 100 mL.
H - 3.2 Beaker - 400 mL
H - 3.3 Centrifuge machine
H - 3.4 Method of sample preparation- Weigh approximately about 2.0 g of the sample into a 400 mL beaker and then add 5-10 mL of distilled water. Digest with 2.5 mL nitric acid by heating on a water bath at 70-80°C about 1 hour and evaporate to dryness. Cool the sample to room temperature and mix a small amount of distilled water and centrifuge at 400-500 rpm for 10-15 minutes and filter the solution through a filter paper (No. 41). Transfer the filtrate into a volumetric flask and make up the volume up to the mark (50 mL or 100mL) with distilled water.
H - 4.0 Blank preparation - 2.5 mL concentrated nitric acid and volume up to mark (50 mL or 100 mL) with distilled water.
H - 4.1 Instrumental parameters / Conditions
Method name - Cold Vapor Atomic Absorption Spectroscopy (CVAAS).
H - 4.2 Spectrometer parameters
H- 4.3 Wavelength - 253.7 nm
H - 4.4 Background correction - D2
Calculation :

Mercury content (ppm) $= \frac{Absorbance \times Total\ Volume}{Weight\ of\ sample \times 1000}$