TEST PRINCIPLE
Evaluation of the hardness of lipsticks by penetration with a 2 mm needle probe.

BACKGROUND
Waxes and oils are the major components of lipsticks providing product firmness and enabling an even spread of the product during application. The capability of waxes to produce a good luster, adequate product firmness, along with their moulding properties, makes them complimentary to the final texture of the product. Petroleum waxes, however, differ in hardness. Measuring the hardness (firmness) of a lipstick by varying the composition and wax content may be a useful tool in quality control in assessing whether a wax of interest will be suitable in producing the desired properties. The test can also be used to monitor hardness over a range of temperatures that the lipstick may be subjected to during transportation and storage.

The method for testing lipstick hardness is adopted from the ASTM Standard Method of test D1321-10. This is a penetration test that measures sample hardness. In the standard method, a penetrometer determines the distance of penetration when applying a specified force over a period of 5 seconds. The method described here is an alternative method to the standard and measures the force of penetration at a defined deformation distance of 5 mm. The CT3 Texture Analyzer can also indicate the presence of unwanted air bubbles or a grainy texture, seen as fluctuations in the force values during penetration.

METHOD
EQUIPMENT: CT3 with 4.5kg load cell
Round Base Table (TA-TR-KIT)
Adjustable Vice Grips (TA-AVG)
2 mm Needle Probe (TA39)
TexturePro CT Software

SETTINGS: Test Type: Compression
Pre-Test Speed: 1.0 mm/s
Test Speed: 1.0 mm/s
Post-Test Speed: 10.0 mm/s
Target Value: 5 mm
Trigger Force: 5 g

Note: It is recommended that the pre-test speed be the same as or less than the test speed for accurate trigger detection; for example, 1 mm/s test speed will require ≤ 1 mm/s pre-test speed.

PROCEDURE
1. Attach the 2 mm needle probe to the load cell.
2. Ensure that the probe is clean.
3. Place the round base table at the base of the instrument and loosely tighten the screws to enable some degree of mobility wind out the lipstick to its maximum length.
4. Slide the adjustable vice grips onto the base table.
5. Wind the lipstick to maximum length and secure the base of the lipstick to the vice grips.
6. Align the sample centrally under the needle probe.
7. Start the penetration test.
8. Between tests, wipe the needle carefully towards its point with a clean, dry cloth to ensure complete removal of all adhering wax.

When optimizing test settings, the hardest sample is best tested first in order to predict the maximum testing range for subsequent samples.

RESULTS

Figure I shows force vs time for the penetration test on two lipsticks of different formulation tested at 20°C.

Figure II shows force vs. distance for the penetration test on two lipsticks of different formulation.
OBSERVATIONS
When a trigger force of 5g has been achieved, the probe begins to penetrate the sample to a defined distance (in this case 5 mm). The maximum force value is then measured as the force required to penetrate the sample to the specified distance. The higher the force value, the harder the sample. Sample A is harder (firmer) than sample B. The area under the curve is a measure of the work done to achieve the defined penetration distance.

The table below shows the hardness and work done values for the two lipsticks:

<table>
<thead>
<tr>
<th>Product</th>
<th>Hardness (g)</th>
<th>Work Done (mJ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample A</td>
<td>181</td>
<td>6.92</td>
</tr>
<tr>
<td>Sample B</td>
<td>91</td>
<td>2.111</td>
</tr>
</tbody>
</table>