# Application Note

082/2012

Melting Point M-565

## Slip Melting Point Determination of Lipstick

SHORT NOTE

# Slip Melting Point Determination of Lipstick

*Measuring the slip melting point (SMP) is important to the quality control in the cosmetic- and food industries. Here, we demonstrate the feasibility to employ the M-565 for SMP measurements of a lipstick sample. We report a SMP of 74.7±0.9 °C for the evaluated lipstick sample when following the USP 741 class II procedure. Obtained results confirm that the M-565 is a reliable tool to record the SMP for waxy or fatty samples as cosmetics and food components. With the M-565 the melting process can be recorded and analysed without modification of the instrument.*

## Introduction

The slip melting point (SMP) is defined as the temperature at which a sample rises in an open capillary upon heating under defined conditions (Figure 1).

In the quality control of cosmetics the SMP plays a prominent role. Furthermore, this method can be employed to control the quality of fats for edibles as cacao butter or palm oil [1].

Here, as an example, it is shown how the SMP of lipstick can be determined using the M-565.

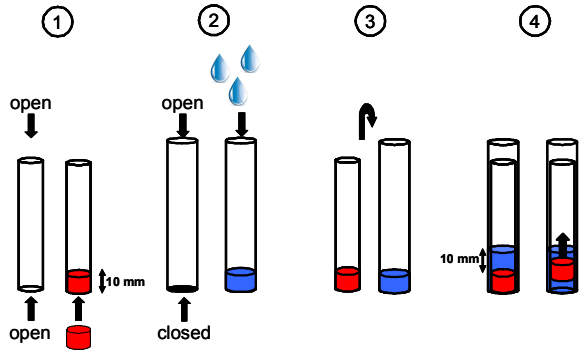


**Figure 1.** Illustration of the sample preparation (steps 1-3) and the rising sample (step 4).

## Experimental

For SMP measurements the procedure according to the US Pharmacopeia 741 for class II was followed.

About 1 g of the lipstick sample (Nivea Beauté, Cardinal 25 Colour Passion) was melted in a glass beaker using a heating plate. The melted liquid sample was filled into a capillary (open on both ends), by dipping the capillary into the solution. The height of the lipstick sample in the capillary was 9 to 10 mm. To harden the sample it was cooled in the refrigerator to 4 °C for 24 h.

The sample capillary was immersed in a boiling point tube containing water. To obtain reproducible results the upper edge of the sample has to be below the water surface at a defined distance (immersion depth). In the standard experiment, the immersion depth was 10 mm (Figure 1).

The lipstick sample was placed in the M-565 and a temperature gradient (0.5 °C/min in the standard experiment) was applied. The melting process was recorded using the Melting Point Monitor software. By programming a heating ramp, starting 5 °C below to about 5 °C above the expected melting point, the process can run without supervision. Recorded data were analysed after each run.

## Results

Three measurements from samples of the same lipstick revealed a SMP of 74.7±0.9 °C. The slip melting process is shown in Figure 2. Obtained standard deviation of less than 1.0 °C is well comparable to values found in literature for SMP measurements of fatty products [2].

When using an immersion depth of 30 mm instead of 10 mm a slip point of 72.5 °C was observed. Hence, the SMP is about 2 °C lower than when performing the experiment with an immersion depth of only 10 mm. These findings underline the importance to work according to a reproducible experimental procedure.

Another result of the deeper immersion depth is that the sample plug rises quicker and a longer distance in its capillary.

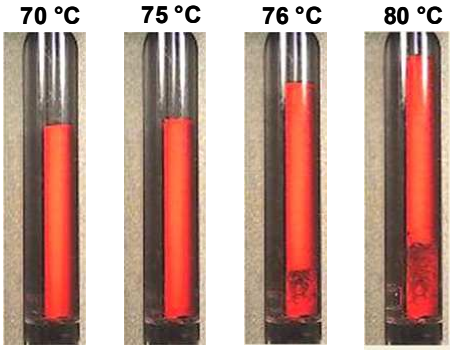


**Figure 2.** Lipstick sample, rising in its capillary by heating up. Measured slip melting point: 74.7±0.9 °C.

## Conclusion

It was successfully demonstrated that the slip melting point of lipstick can be determined using the M-565. Reproduction of the measurements was performed, yielding a standard deviation of less than 1.0 °C.

Clearly, the found results confirm that the M-565 can be applied to perform SMP measurements, in addition to its conventional use for the determination of melting and boiling points. This makes the M-565 an economic and versatile instrument for the quality control of cosmetic products.

## References

[1] See for example: I. Karabulut et al, *Eur Food Res Technol*, 218, 3, 224-229, 2004.; P.S. Keng et al, *Ind Crop Prod.* ,Vol 29, no 1, p. 37, 2009.; SLMB 1024.1

[2] J.M. Deman et al, *JOACS*, 60, 1, 91, 1983.

# 1 Introduction

For cosmetic products, as for example lipsticks, we do not expect a precise temperature at which they change their state from solid to liquid. Contrary to pure substances, such complex molecular mixtures do not have a sharp melting point at which a transparent liquid is observed. However, it is important to know when the product changes its physical state. Especially, in warm countries, softening and melting of cosmetic products during transportation or storage is costly and should be absolutely avoided.

Furthermore, producers of cosmetics often use the slip melting point (SMP) to determine the melting properties and the quality of their raw material, intermediate- or final products. In addition to the cosmetic industry, the SMP is widely used to describe the quality of fats in the food sector [1, 2].

The SMP method is also known as the open capillary tube method or the rise melting point method. The SMP, an inherent material property, is determined to control the quality.

To measure the SMP, the sample is solidified in a capillary and heated in a water bath. At a certain temperature the sample rises in the capillary, this temperature is called the SMP temperature. Since many factors, as heating rate, sample amount and sample preparation may influence the reproducibility of the measured SMP [3] normative procedures were developed [4].

Here it is demonstrated how the SMP of a lipstick can be accurately determined using the M-565 and the Melting Point software following the US Pharmacopeia 741 Class II procedure.

# 2 Equipment

- Melting Point M-565
- PC with Melting Point-Monitor Software
- Analytical balance
- Heating plate equipped with contact thermometer
- Refrigerator set to 4°C
- Beaker 50 ml
- Syringe (2 ml) with needle

# 3 Chemicals and Materials

- Melting point capillaries, BUCHI, open on both ends (040808), Dimensions; Length: 80 mm, internal diameter: 1.10 mm, wall thickness: 0.225 mm
- Boiling point capillaries, BUCHI (019697)
- Distilled water

# 4 Samples

Lipstick Sample: Nivea Beauté, Cardinal 25 Colour Passion, Beiersdorf AG Hamburg Germany

# 5 Procedure

## 5.1 Working Principle

The steps for determining the SMP using the M-565 is shown in Figure 1. First, the sample has to be filled into a capillary that is open on both ends (Figure 1, step 1).

According to the procedure described by the USP 741 Class II, the height of the material in the capillary should be about 10 mm. Second, another capillary, the boiling point capillary has to be filled with a few drops of distilled water (Figure 1, step 2). The boiling point capillary is open on one end only and filling of water is best done using a syringe with a thin needle.

Third, the capillary with the sample has to be immersed into the boiling point capillary as shown in Figure 1 step 3. The upper edge of the sample has to be below the water surface. Depending on the procedure the sample level has to be below the water surface at least by 10-50 mm [4].

Then the sample is placed in the M-565, the position of the boiling point capillary is to be used. Upon heating, the sample material softens and expands. Also the water volume increases. When reaching the slip melting point temperature, the buoyant force exceeds the friction force between the sample and the glass wall and makes the sample to rise in its capillary (Figure 1 step 4).

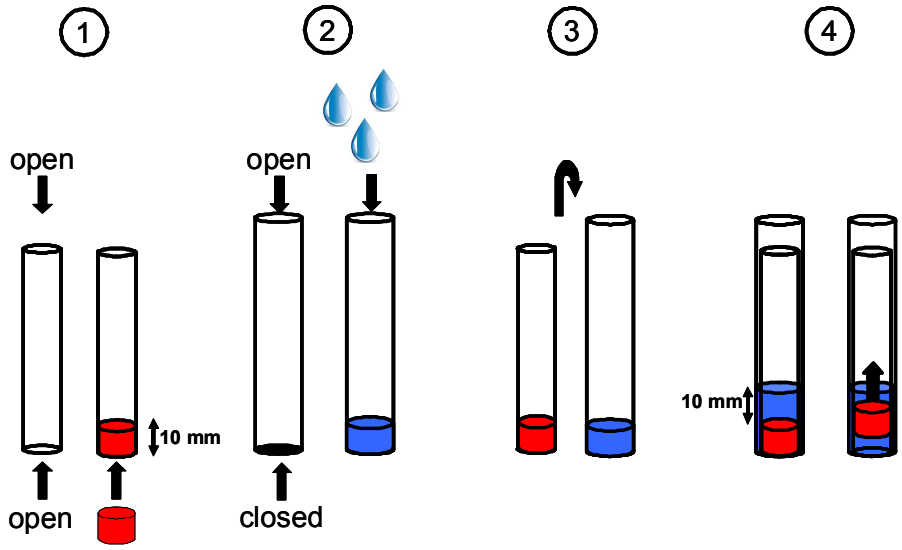


**Figure 1.** Illustration of the sample preparation (steps 1-3) and the rising sample (step 4).

## 5.2 Experimental

### 5.2.1 Sample Preparation

To determine the SMP the procedure to measure the melting point according to the US Pharmacopeia 741 for class II was followed.

About 1 g of Lipstick sample was carefully melted in a glass beaker using a heating plate. When completely melted the liquid sample was filled into a capillary that is open on both ends, by dipping the capillary into the solution. The height of the lipstick sample in the capillary was 9 to 10 mm. To harden the sample it was cooled in the refrigerator to 4 °C for 24 h.

The sample capillary was immersed in a boiling point tube containing water. When inserted the water level in the boiling point tube was 10 mm above the upper edge of the sample.

In Figure 2 left, the lipstick sample in the capillary and the boiling point tube are shown. On the right side in Figure 2, the sample capillary is inserted into the boiling capillary (without water for demonstration).

**Figure 2.** The lipstick sample outside (left) and inside (right) of the boiling point capillary.

### 5.2.2 Slip Melting Point Measurement

In a first experiment the SMP of a lipstick sample was measured. Therefore, the sample was placed into the boiling point position of the M-565. The sample was heated from 70 to 80 °C using a heating rate of 0.5 °C/min. To check the reproducibility, the SMP of three samples, prepared as described, were measured.

### 5.2.3 Influence of Immersion Depth

To obtain a reproducible result the upper edge of the sample has to be below the water surface at a defined distance (immersion depth). In the literature, different immersion depths are suggested. For example, according to the USP 741 Class II the immersion depth has to be 10 mm while according to the Eur. Pharm. the sample has to be immersed 30 mm below the water surface [4].

To study the influence of the immersion depth, SMP measurements with 10 and 30 mm immersion depths using otherwise identical conditions were performed.

### 5.2.4 Influence of Heating Ramp

Next the effect of the heating rate on the SMP was evaluated. In this Application Note the influence of the heating rate was studied using rates of 0.5 and 1.0 °C/min.

To ensure that the heat exposure time of the sample is similar for both heating rates, different starting temperatures were set. For the rate of 0.5 °C/min the starting temperature was 70 °C and for the heating rate of 1.0 °C/min it was set to 65 °C.

# 6 Results and Discussion

## 6.1 Slip Melting Point Measurement

In Figure 3 snapshots of a lipstick sample at different temperatures during a SMP measurement with the M-565 are shown. The red lipstick is surrounded by a capillary that is immersed in a water filled boiling point tube (see Figure 1). The water level is not visible in Figure 3 since it is out of the recorded picture. The melting progress was recorded using the Melting Point Monitor software.

At the starting temperature of 70 °C a compact plug of lipstick was present. Upon heating, the sample volume expanded and softened. Due to the softening the friction forces between the glass wall and the sample decreased. Suddenly, in this experiment at 75.1 °C, the softened lipstick plug rose in its capillary. A snapshot of the lipstick sample taken at 76 °C shows the rising lipstick sample. Finally, at 80 °C, heating was stopped.

Three measurements of samples prepared from the same lipstick revealed a SMP of 74.7±0.9 °C. Obtained standard deviation of less than 1 °C is well comparable to values found in literature for SMP measurements of fatty products [5].

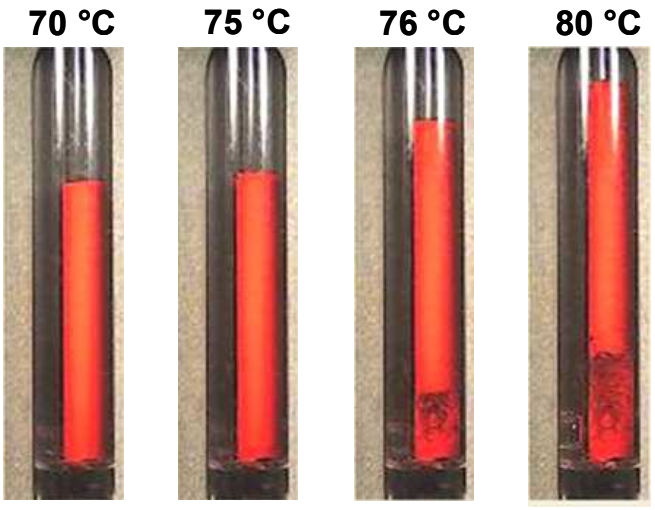


**Figure 3.** Lipstick sample, rising in its capillary by heating up. Measured slip melting point: 74.7±0.9 °C.

## 6.2 Influence of Immersion Depth

When using an immersion depth of 30 mm instead of 10 mm, using otherwise identical conditions, a slip point of 72.5 °C was measured. Hence, the SMP is about 2.0 °C lower than when performing the experiment with an immersion depth of only 10 mm.

Clearly, the immersion depth is an important factor when measuring the SMP. From the performed experiments it is expected that the deeper the immersion, i.e. the higher the water level above the upper edge of the sample, the SMP decreases. This is simply due to higher pressure of the water column acting on the sample.

Another result of the deeper immersion depth is that the sample plug rises quicker and over a longer distance in its capillary. When analysing the recorded data, it is easier to determine the SMP temperature in case of a fast rising sample plug.

Figure 4 illustrates the influence of the immersion depth on the rising distance. In Figure 4 left, the immersion depth is 10 mm on the right it is 30 mm.

**Figure 4.** Influence of the immersion depth. Left: the upper edge of the sample was 10 mm lower than the water level. Right: the upper edge of the sample was 30 mm below the water level. On the right the sample rose out of the recording area.

### 6.2.1 Influence of heating ramp

In order to evaluate the influence of the heating rate, three experiments using a heating rate of 1.0 °C/min were performed. Obtained average SMP was 74.3±0.8 °C.

The found slip point was slightly lower (0.4 °C) than for the measurement with a slower rate of 0.5 °C/min. Due to the very small temperature difference and the observed temperature distribution it is assumed that the influence of heating rates between 0.5 ° and 1.0 °C/min can be neglected.

Other parameter, as the tempering temperature and the sample height in the capillary are described in the literature. These factors have a greater influence on the SMP (up to 5 °C) than the studied heating rates [3].

### 6.2.2 Limitations

SMP determination with the M-565 is limited to samples with a SMP of at least 10 °C above the ambient temperature. Usually, this is about 35 °C.

## 7 Conclusion

It was successfully demonstrated that the slip melting point of lipstick can be determined using the M-565. Reproduction of the measurements was performed with a standard deviation of less 1.0°C .The slip point was found to be dependent on the immersion depth and independent on the heating rate, as long as the heating rate is between 0.5 and 1.0 °C.

Clearly, found results confirm that the M-565 can be applied to perform the SMP measurements, in addition to its conventional use for the determination of melting- and boiling points. No modifications of the instrument are necessary rendering the M-565 an economic and versatile instrument for the quality control in the cosmetic industry.

## 8 Acknowledgements

Marcos Y. Maekawa and Rafael C. Sartorelli from Pensalab, Sao Paulo, Brazil are kindly thanked for their important support.

# 9 References


[1] I. Karabulut et al, *Eur Food Res Technol*, 218, 3, 224-229, 2004.

[2] P.S. Keng et al, *Ind Crop Prod.* ,Vol 29, no 1, p. 37, 2009.

[3] K.G. Berger et al, *JOACS*, 59, 5, 244, 1982.

[4] see for example: USP 741 Class II; Pharm. Eur 7.0 , 2.2.15; ISO 6321:2002; JIS K 0064:1992; SLMB 1024.1.

[5] J.M. Deman et al, *JOACS*, 60, 1, 91, 1983.



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