Attach one of the tubes to a thermometer graduated in 0.5 °C so that the substance is close to the bulb of the thermometer. Introduce the thermometer with the attached tube into a beaker so that the distance between the bottom of the beaker and the lower part of the bulb of the thermometer is 1 cm. Fill the beaker with water to a depth of 5 cm. Increase the temperature of the water gradually at a rate of 1 °C/min.

The temperature at which the substance begins to rise in the capillary tube is regarded as the melting point.

Repeat the operation with the other 4 capillary tubes and calculate the result as the mean of the 5 readings.

01/2008:20216

2.2.16. MELTING POINT - INSTANTANEOUS METHOD

The instantaneous melting point is calculated using the expression:

$$\frac{t_1 + t_2}{2}$$

in which t_1 is the first temperature and t_2 the second temperature read under the conditions stated below.

Apparatus. The apparatus consists of a metal block resistant to the substance to be examined, of good heat-conducting capacity, such as brass, with a carefully polished plane upper surface. The block is uniformly heated throughout its mass by means of a micro-adjustable gas heater or an electric heating device with fine adjustment. The block has a cylindrical cavity, wide enough to accomodate a thermometer, which should be maintained with the mercury column in the same position during the calibration of the apparatus and the determination of the melting point of the substance to be examined. The cylindrical cavity is parallel to the upper polished surface of the block and about 3 mm from it. The apparatus is calibrated using appropriate substances of known melting point.

Method. Heat the block at a suitably rapid rate to a temperature about 10 °C below the presumed melting temperature, then adjust the heating rate to about 1 °C/min. At regular intervals drop a few particles of powdered and, where appropriate, dried substance, prepared as for the capillary tube method, onto the block in the vicinity of the thermometer bulb, cleaning the surface after each test. Record the temperature t_1 at which the substance melts instantaneously for the first time in contact with the metal. Stop the heating. During cooling drop a few particles of the substance at regular intervals on the block, cleaning the surface after each test. Record the temperature t_2 at which the substance ceases to melt instantaneously when it comes in contact with the metal

Calibration of the apparatus. The apparatus may be calibrated using melting point reference substances such as those of the World Health Organisation or other appropriate substances.

01/2008:20217

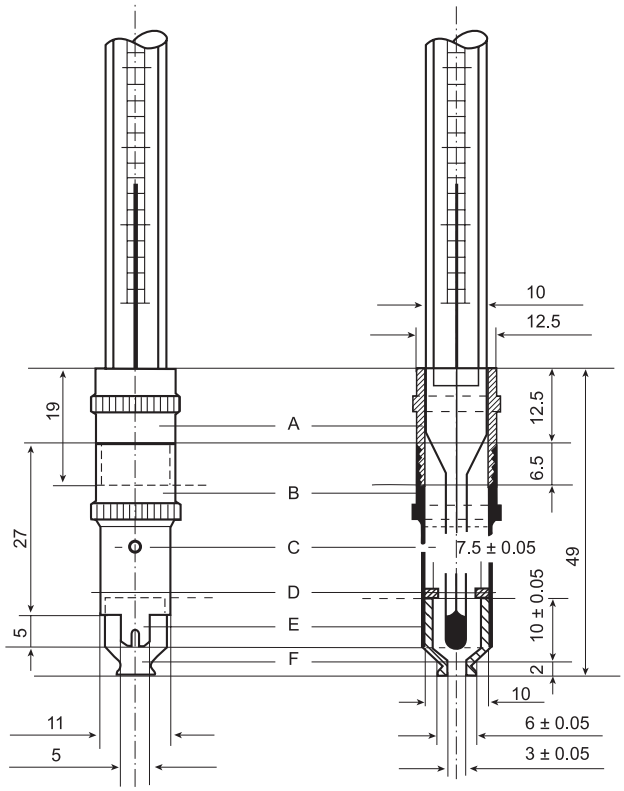
2.2.17. DROP POINT

The drop point is the temperature at which the first drop of the melting substance to be examined falls from a cup under defined conditions.

When a monograph does not specify the method to be used, method A is applied. Any change from method A to method B is validated.

METHOD A

Apparatus. The apparatus (see Figure 2.2.17.-1) consists of 2 metal sheaths (*A* and *B*) screwed together. Sheath *A* is fixed to a mercury thermometer. A metal cup is loosely fixed to the lower part of sheath *B* by means of 2 tightening bands. Fixed supports 2 mm long determine the exact position of the cup, and in addition are used to centre the thermometer. A hole pierced in the wall of sheath *B* is used to balance the pressure. The draining surface of the cup must be flat and the edges of the outflow orifice must be at right angles to it. The lower part of the mercury thermometer has the form and size shown in the figure; it covers a range from 0 °C to 110 °C and on its scale a distance of 1 mm represents a difference of 1 °C. The mercury reservoir of the thermometer has a diameter of 3.5 ± 0.2 mm and a height of 6.0 ± 0.3 mm. The apparatus is placed in the axis of a test-tube about 200 mm long and with an external diameter of about 40 mm. It is fixed to the test-tube by means of a laterally grooved stopper through which the thermometer passes. The opening of the cup is placed about 15 mm from the bottom of the test-tube. The whole device is immersed in a beaker with a capacity of about 1 litre, filled with water. The bottom of the test-tube is placed about 25 mm from the bottom of the beaker. The water level reaches the upper part of sheath *A*. A stirrer is used to ensure that the temperature of the water remains uniform.



A. upper metal sheath
B. lower metal sheath
C. pressure-balancing hole
D. fixed supports
E. tightening bands
F. metal sample cup

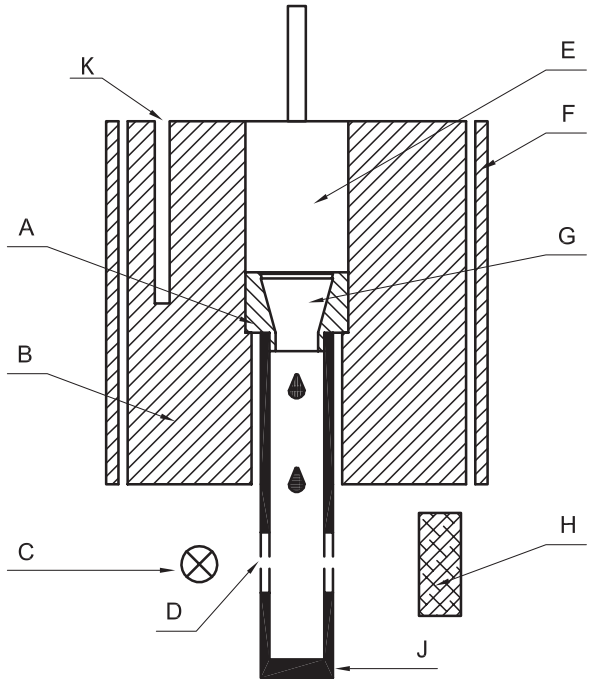
Figure 2.2.17.-1. – *Apparatus for the determination of drop point*

Dimensions in millimetres

Method. Prepare the substance to be examined according to the prescriptions of the monograph. Fill the cup to the brim with the substance to be examined. Remove the excess substance at the 2 ends of the cup with a spatula. When sheaths *A* and *B* have been assembled, press the cup into its housing in sheath *B* until it touches the supports. Remove with a spatula the substance pushed out by the thermometer. Place the apparatus in the water-bath as described above. Heat the water-bath and, when the temperature is at about 10 °C below the presumed drop point, adjust the heating rate to about 1 °C/min. Note the temperature at the fall of the first drop. Carry out at least 3 determinations, each time with a fresh sample of the substance. The difference between the readings must not exceed 3 °C. The mean of 3 readings is the drop point of the substance.

METHOD B - AUTOMATED METHOD

Apparatus. The apparatus (see Figure 2.2.17.-2) consists of a cartridge assembly comprising a cup holder into which the sample cup containing the sample is loosely fixed, and a collector sleeve with a horizontal light slit, which is fixed below the cup. This assembly is placed in a heating block. The block is a metal cylinder with a cylindrical hole along its vertical axis into which the cartridge assembly is placed. There is another, narrower cylindrical vertical hole in which a temperature sensor sits. This is positioned level with the sample cup. The heating block is surrounded by an electrical heating element. Below the heating block a lamp is mounted such that a beam of light shines through the light slit in the collector sleeve, and onto a photo-sensor mounted opposite. The heating block is capable of being maintained at a precise, pre-defined temperature by the heating element, and of being heated at a slow and steady, pre-defined rate after an initial isothermal period.



A. cup holder
B. heating block
C. light source
D. light slit
E. cartridge assembly
F. heating element
G. sample cup
H. photo-sensor
J. collector sleeve
K. temperature probe

Figure 2.2.17.-2. – *Example of automated drop point apparatus*

Method. Melt the substance to be examined and introduce it into the sample cup according to the prescriptions of the monograph, then proceed as follows or according to the manufacturer's instructions. Remove the excess substance at the 2 ends of the cup with a spatula. Condition the sample at the temperature and for the time prescribed in the monograph before making the measurement. Press the cup into the cup holder, and then press the collector sleeve onto the cup. Place the cartridge assembly in the heating block. Set the instrument to the initial isothermal conditions and rate for subsequent heating as described in the monograph of the substance to be examined. Start the temperature programme. When the first drop of molten sample falls through the hole at the bottom of the sample cup, interrupting the light beam, the signal from the photo-sensor causes the temperature of the heating block to be recorded automatically.

Calibration. Use the apparatus according to the manufacturer's instructions and carry out the prescribed calibrations and system performance tests at regular intervals, depending on the use of the apparatus and the substances to be examined. Benzoic acid and benzophenone are usually used as certified reference materials. Other materials may be used provided they show no polymorphism. Proceed as follows or according to the manufacturer's instructions. Prepare 3 sample cups for each of the 2 certified reference materials. Place the sample cups on a clean surface. Into each sample cup, introduce a small quantity of the sample and press it down with a rod (diameter about 4.5 mm). Check that the opening is completely filled. Fill the sample cup about half full and compact the sample with a rod (diameter about 9 mm). Fill the sample cup completely and compact, adding more sample and compacting again if necessary, until the sample cup is completely full.

Temperature programme for benzoic acid: start temperature = 118.0 °C; heating rate = 0.2 °C/min; end temperature = 126.0 °C. After inserting the cup at 118 °C, a waiting time of 30 s is set before heating starts.

Temperature programme for benzophenone: start temperature = 44.0 °C; heating rate = 0.2 °C/min; end temperature = 56.0 °C. After inserting the cup at 44 °C, a waiting time of 30 s is set before heating starts.

Check the 3 single results: the test is valid if the 3 results are within 0.3 °C of the mean value.

Calculate the corrected mean temperature $(T)_2$ using the following expression:

$$T_1 - F$$

T_1 = mean drop point temperature of 3 samples, in °C;

F = compensation for the difference in temperature between the sample and the point in the heating block where the temperature is measured; this will vary depending upon the design of the automatic drop point instrument and is provided by the manufacturer.

Taking into account the drop point (T_0) of the certified reference material, the accuracy of the temperature scale is satisfactory if $|T_2 - T_0|$ is not greater than 0.3 °C.