Melting and boiling point
Laboratory Guide

Contents

1 Introduction

1.1 What is a melting point?

There are several material constants that can be used to describe a material, for example, its specific gravity, light refraction, adsorption capacity, or chromatographic behavior. The melting point is also one of these constants. Along with the boiling point and the solidification point, it is one of the important thermal characteristics that describe a material. The melting points of many pure materials can be measured with great accuracy.

Crystalline materials consist of extremely fine particles that form a certain regular 3-dimensional structure. These 3-dimensional arrangements are referred to as lattice structures or (crystalline) lattices. The particles within the lattice are held together by lattice forces.

Whenever this solid structure, the lattice, is heated, the particles in it begin to move more strongly, until finally the forces of attraction between them are no longer strong enough to maintain the crystalline structure. The lattice is destroyed and the solid material melts. At the melting point a material shifts from its ordered, solid state to an unordered, liquid state. The stronger the forces of attraction between the particles within the lattice, the greater the amount of energy that must be used to overcome them. The melting temperature of a crystalline solid is thus an indicator for the stability of its lattice. The higher the temperature, the more strongly the lattice structure in question holds together.

1.2 Why measure melting points?

There are various methods of chemical and physical analysis used to differentiate, identify, and classify materials. Measurement of the melting point is, among other things, one of these standard laboratory procedures. It is an experimental and easily performed method of physical analysis used to find out the identity, the purity, and the thermal stability of a material.

Identification
Pure materials have exactly defined melting points which can be obtained from reference tables. Thus, the identity of a material can be determined by measuring its melting point: One needs only to compare the melting point of the substance as determined in the test with the values in the technical literature. Of course, determination of the melting point alone is not yet enough for the clear identification of a substance. There may be several substances with the same melting point. In such cases, the shift in the mixed melting point (refer to Sect. 2.6.2) can provide an indication about the definitive identification of the material.

Purity
Even slight impurities in a material cause a lowering of its melting point or at least a widening of its melting range (the material melts within a range of temperatures and not at a precisely definable melting point).

This phenomenon is used to obtain indications about the purity of a material: The smaller the difference between the measured melting point of the substance and the melting point shown in the tables, the narrower its melting range and thus the purer the material.

Thermal stability
Many materials change at high temperatures, e.g., decomposition or discoloration. Measurement of the melting point is one method that can be used to determine how much the material can be heated without causing chemical changes. This value is useful as an indicator for the thermal stability of a substance and can be used to suggest a possible temperature for drying.

1.3 Who measures melting points?

Various groups of users use melting point determination in their daily work with chemical substances. Their priorities and their specific requirements for how the melting point is to be determined may differ. The needs of users are different, especially with regard to accuracy of measurement and the ability to observe how the substance behaves during the measurement process.

Synthesizing laboratories
A traditional research laboratory continually produces new types of chemical compounds. In order to find out how these new compounds behave, the research scientists observe the melting process closely.
Often, these researchers are dealing with different substances: They receive about 1 to 4 samples each week for investigation. Thus, having a high degree of automation for the measurement of melting points is only of secondary importance for them. Far more important is their ability to observe the sample comfortably during the melting process.

Analytical laboratories
Analytical laboratories perform the tests done on the receipt of goods (raw products) and when examining the end products of a production process. Their main interest here is checking the purity of the products. Their measurements must be as precise as possible. These laboratories analyze from 10 to 50 samples each week, whereby determination of the melting point is part of their daily routine. Because they are examining the same materials over and over again, a high degree of automation is an important advantage for them. Normally, these researchers do not need to observe the samples while measuring the melting point. Frequently, the substances they have to investigate are discolored or decomposing so that no automated determination of the melting point is possible. In this case, visual melting point determination may be considered.

Pharmacies
A mistaken identification of medication in pharmacies can become a danger for the health of patients. It thus becomes extremely important to verify the identity of the medications and agents. Measurements of melting points done in pharmacies therefore require both maximum accuracy and an opportunity to observe the melting process when necessary.

1.4 The boiling point

All elements and many inorganic and organic compounds have characteristic boiling points, which can be obtained from reference tables. Mixed liquids do not have a precisely defined boiling point. Instead, they boil over a fairly wide range of temperatures within clearly defined boiling limits. Accordingly, observation of their boiling behavior is an easily measurable experimental criterion for determining their purity. Whenever the boiling temperature changes during the boiling process, the material you are investigating cannot be a single pure material. However, it must be noted that impurities basically have less effect on the boiling point than on the melting point. For that reason, the boiling point is less as informative criterion for the purity or for the description of materials as the melting point.

2 Theoretical basis for the measurement of boiling points and melting points

2.1 Physical states

The «physical state» of a material is the state in which the material exists under the given external conditions (pressure/temperature). A distinction is made here between «solids», «liquids», and «gases».

Solids	Liquids	Gases
Defined volume	Defined volume	No defined volume
Defined shape	No defined shape	No defined shape
Particles have a defined position	Particle can shift positions with respect to one another	Particles can move freely; there is no longer any reciprocal attraction present

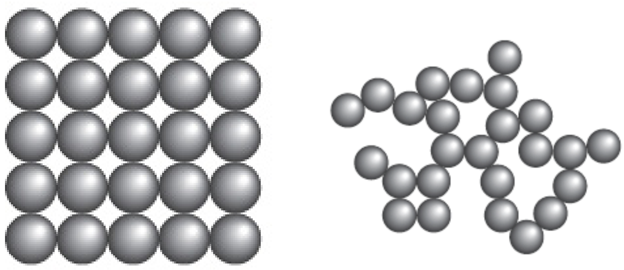

Table 1: Comparison of properties of the physical states «solid», «liquid», and «gaseous». The kinetic energy of the smallest particles, the compressibility of the material, and diffusion within the material increases as you go from left (solids) to right (gases).

In general, a material may be present in any of these three physical states (Fig. 1).
When the external conditions change, materials can undergo a physical state. This process is referred to as «phase transition». A change in physical state always entails taking up or giving off a considerable amount of energy.

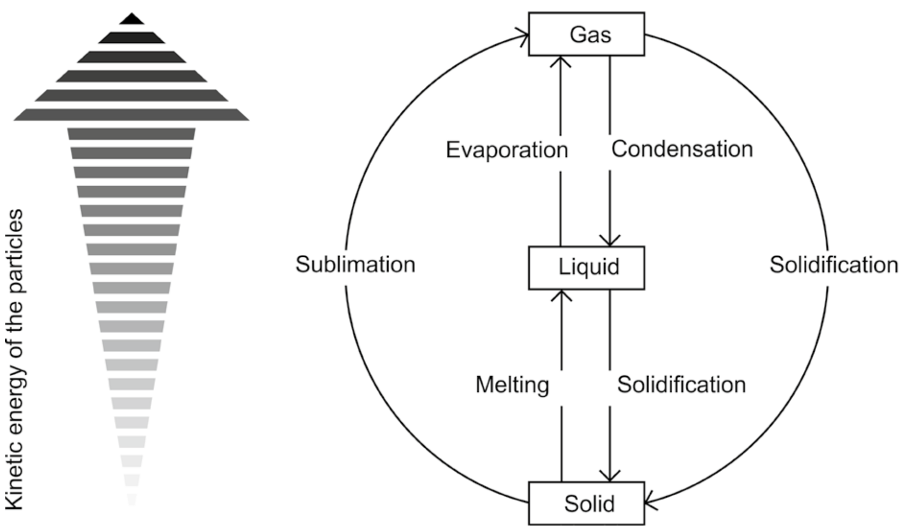

Figure 1: Changes in state: Transitions between the various physical states.

Most materials are crystalline when solid, i.e., in that state, their smallest particles (atoms, molecules, or ions) form an orderly, 3-dimensional arrangement – a crystalline lattice. The stability of the lattice depends not only on how strong the forces between two components of the grid are between themselves, but also on how uniformly these forces act in all directions in space. In addition to crystalline solids, there exist also amorphous solids, whose particles are in a random arrangement in the solid state. Glass, resin, and many synthetic plastics are examples of amorphous materials.

2.2 Phase transitions

The «phase» or physical state refers to material that is homogeneous in chemical composition and in a spatially constant physical condition. As already mentioned, materials may transform from one phase into another by means of phase transition. A phase transition always takes up or gives off energy (the heat of melting or evaporation, and/or the heat of condensation or solidification, cf. Sect. 2.3) and always occurs at the same pressure for any given temperature. The density of materials also changes during phase transitions. During the actual transition, the two physical states (phases) of the material always exist side-by-side, and the temperature remains constant until the change in phase has been completed. A temperature/energy diagram shows these relationships.

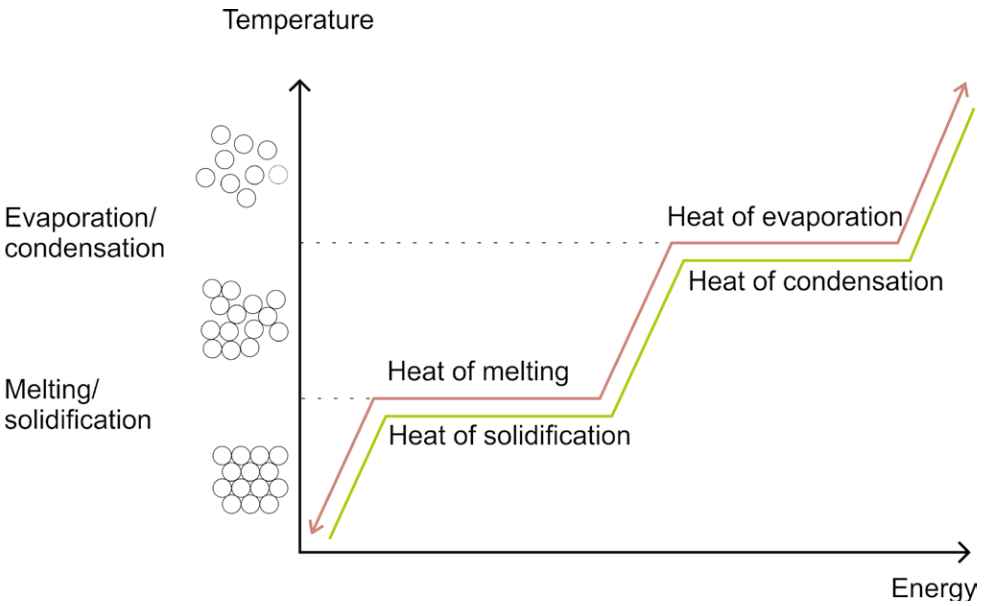

Figure 2: Temperature/energy diagram.