Melting Point

Determining the melting point of a compound is one way to test if the substance is pure and is often used to test samples made from organic synthesis (eg of aspirin or paracetomol).

Pure samples usually have sharp melting points, for example 149.5-150°C or 189-190°C; impure samples of the same compounds melt at lower temperatures and over a wider range, for example 145-148°C or 186-189°C. So if your sample has a melting point at the temperature you expect, it is probably what you think it is. If the melting point is quite sharp, then it is likely to be fairly pure.

The general method is to heat a sample indirectly by placing the prepared sample (usually packed in a glass capillary tube) in or on a heated medium and observing it, and the temperature, closely until melting is complete.

There are a few ways of doing this

Melting point apparatus

This is the most common piece of apparatus for determining melting point. The sample is loaded in a capillary tube and the temperature of the sample gradually raised by means of a heated internal metal block.

Older, or budget, apparatus (like the one in Fig 1) is almost entirely manual. You have to manually control the rate of heating.

More modern, advanced ones, (see Fig 2), allow you to program the rate of heating and allow it to heat rapidly to a set point and then go more slowly.

In both cases, though, you need to watch (carefully) through a magnifier and determine for yourself when melting starts and finished.

The one problem with the melting point apparatus described above is that of cost. It is possible to purchase manual ones from around £200, or less, but the more advanced ones start around £700 or more.

But these days, even £200 is quite a chunk of an impoverished budget. Fortunately, there are lower cost alternatives.

Melting Point Block

This is a very simple arrangement, costing about £25, and used with care can give reasonable results.

It consists of a block of solid aluminium with a hole drilled in at an angle for a thermometer. You place a thermometer in the diagonal hole and put a small pile of your solid in the middle of the block.

You then heat the block, gently, from the bottom. The heat is conducted up and will eventually melt the solid on the top.

When you see it starting to melt, record the temperature and record it again when all the sample has melted.

It can be difficult to avoid heating the sample too rapidly, especially if you are using a Bunsen burner, but with care it is possible to get decent results.

Fig 1: Old MP apparatus

Fig 2: A more modern device

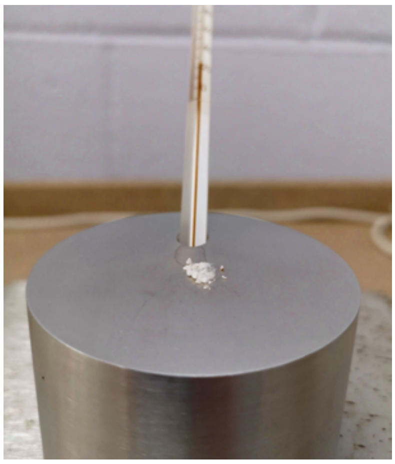

Fig 3: A melting-point block

Thiele Tube

Another cheap alternative that can, with care, still produce accurate results, is the Thiele tube. These cost only about £10 or so.

The Thiele tube is basically a boiling tube with a side loop (see diagram). The sample is placed in a capillary tube and is held next to the bulb of a thermometer by eg a band or wire and placed in the 'main', straight part of the tube.

The tube is filled to just above the loop with a liquid of a suitable boiling point, most commonly an oil and the side arm is heated. As the tube is heated, convection causes the liquid to circulate around the system distributing the heat.

Care needs to be taken to ensure that heating does not happen too fast. A mini burner or a spirit burner is easier to use than a Bunsen for this. It is worth noting that the temperature may continue to rise by 2 or 3 degrees after you stop heating.

It is possible, if even a Thiele tube is too expensive, to just use an ordinary boiling tube, again with a capillary tube fixed to a thermometer. But this is even harder to control to get a good result.

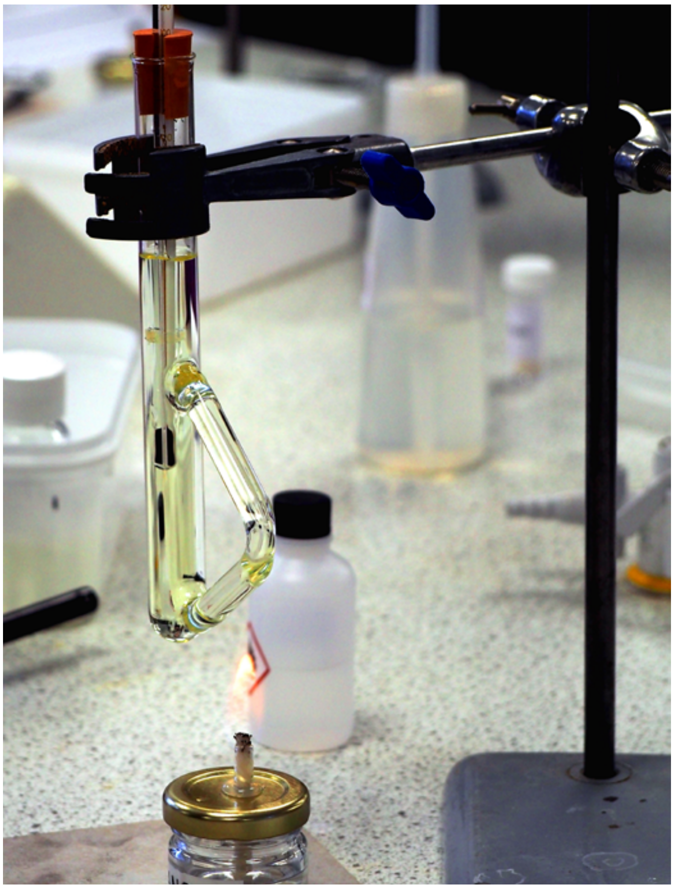

Fig 4: A Thiele tube in use

How to use a Thiele tube

1. Place some of your sample in a capillary tube.

2. Fix the capillary tube to a thermometer.

   We find the best option is to cut a small loop off a length of silicone tubing as a rubber band has a tendency to degrade and break in the oil.

3. Place the sample in your Thiele tube and start heating the side arm.

4. If you are testing the purity of your sample, you will have an idea of what the expected temperature is.

   If you are identifying an unknown though, it might be quicker to carry out a rapid melting point determination initially (by heating rapidly) to establish the approximate melting point before repeating it more carefully.

5. Once the temperature is getting close to your expected point, take care. It can often continue to rise 2 or 3 degrees after you stop heating. Try to increase the temperature as slowly as possible, by moving the burner or the tube nearer to or further from the flame.

6. When you see the first sign of melting in the tube, not the temperature.It is a good idea to use a magnifying glass (or some other device to magnify) so you get a clearer view of when the melting starts.

7. Keep heating, gently, until all the sample is melted. Record this temperature.

8. It is usually a good idea to carry out at least two further careful determinations until you obtain two consistent values.

Note that unlike boiling point, the melting point is relatively insensitive to pressure and no pressure correction needs to be made.

Conclusion

Determination of melting point is a useful analytical technique and although the apparatus can be expensive we hope this article has shown you that it is still possible to carry out a melting point analysis without breaking the bank.