Apparatus for freezing point depression

36821.00

Operating Instructions

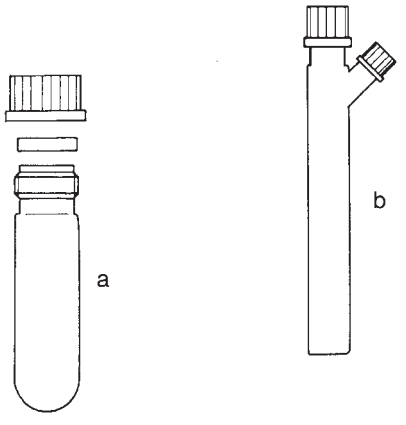


Fig. 1

1 DESCRIPTION OF THE APPARATUS

This apparatus serves to measure the lowering of freezing points. It consists of two cylindrical glass vessels, one of which fits in the other and is held in position in it by a GL 45 screw-top connector. The outer vessel (a in Fig.1) resembles a boiling tube and acts as a jacket around the inner vessel. This jacket can be used as it is, air-filled, but it is preferable to fill it with 35 to 40 ml of ethyl alcohol (or methylated spirits!). The jacket with filling medium (air or alcohol) ensures uniform heat transport from the inner vessel to the freezing mixture.

The solution of a substance or the solvent whose freezing point is to be determined is filled into the inner vessel (b). This vessel has a GL 25 screw-top connector for holding a Beckmann thermometer on the open end, and an upwardly inclined side-arm with GL 18 screw-cap for filling in the substance to be dissolved. The flat bottom of this vessel allows a small magnetic stirring bar (l = 15 mm) to rotate freely in it.

2 EXAMPLE OF EXPERIMENTAL PROCEDURE

The determination of molar masses by measuring the depression of freezing points (= cryoscopic method).

2.1 Experimental set-up

Place a dish or a bowl (e.g. a crystallizing dish or a plastic bowl) on a magnetic stirrer as shown in Fig. 2. Stand a 1000 ml glass beaker (short type) filled with freezing mixture in the dish (refer to the Table for possible freezing mixtures. A mixture of crushed ice and table salt is usually sufficient for teaching purposes). Insert the assembled apparatus for freezing point depression centrally in the mixture in the beaker, as shown in Fig. 2, and clamp it firmly in position.

Slip a magnetic stirring bar into the inner vessel. Adjust the Beckmann thermometer to the required range as described in the operating instructions supplied with it, then hold it in readiness in freezing mixture, whereby the use of a Dewar vessel is most expedient (Fig. 3). Also have a laboratory thermometer with stem (range -10°C to +110°C) readily available. When the exact density of the solvent which is to be used is not known, determine the exact mass of 50 ml of the solvent by weighing it (= m_L). The 50 ml must be delivered from a bulb (volumetric) pipette.

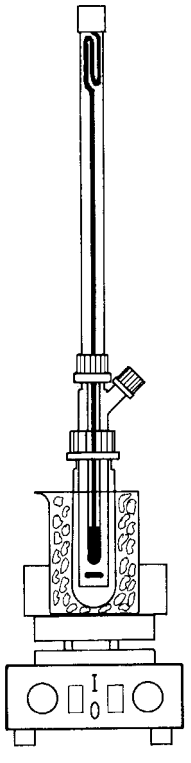
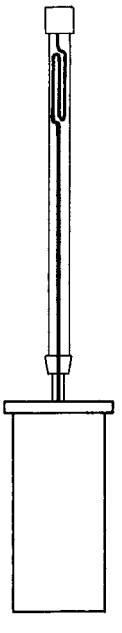


Fig. 2



Figb. 3

2.2 Determination of the relative freezing point of the solvent

Use a bulb pipette to transfer 50 ml of the solvent to be used (e.g. water) into the inner vessel. Switch on the magnetic stirrer and adjust it so that the stirring bar rotates briskly (Caution! Avoid inadvertently also switching on the magnetic stirrer heating!). Use the laboratory thermometer to follow the drop in temperature in the solvent. When this reaches about 1°C above the freezing point of the solvent, quickly transfer the Beckmann thermometer back into the apparatus (Fig. 2). As it is necessary to ensure that no contamination is brought into the solvent with the thermometer, wipe the thermometer quickly with a cloth when taking it out of the vessel it has been held in (Dewar vessel with freezing mixture). This complete procedure must be carried out very quickly, so that the mercury column does not rise up too far. It must under no circumstances connect up with the mercury in the reservoir. Should this happen, the thermometer setting is invalidated and the thermometer must be readjusted. Use a glass rod to move the freezing mixture around a little for uniform cooling of the apparatus.

Observation: The Beckmann thermometer shows that the temperature drops further, until suddenly the mercury column rises somewhat and so reaches a maximum.

Read off this maximum value as accurately as possible. It is the relative freezing point of the solvent (= t_1). The reading should be made to at least 1/100°C. The use of a magnifying glass enables this to be extended by estimation to thousands of a degree.

Hydroquinone or urea are suitable as test substances for practice and for demonstration of the determination of molar masses by measurement of the freezing point depression. The substance to be used must be well dried before use, however (desiccator!).

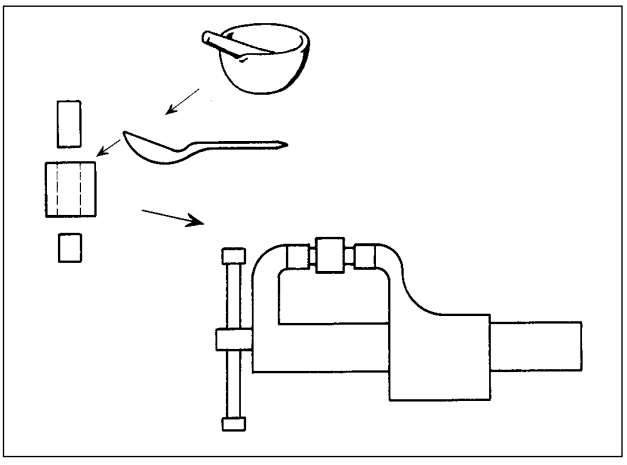
When other test substances are to be used, the appropriate solvent for each must be selected. The specific molal depression constants K for each solvent can be found in pertinent Tables.

An electronic instrument can be used to measure temperature in place of a Beckmann thermometer, provided it has the required measurement accuracy (1/100°C), e.g. the digital temperature measurement instrument 4-2, order no. 13617.93.

2.3 Determination of the relative freezing point of the solution of the test substance

After having determined the freezing point of the solvent, proceed as follows to determine the relative freezing point of the solution:
First, transfer the Beckmann thermometer back to the Dewar vessel so that it does not go out of adjustment.
Take the inner vessel out of the apparatus and warm it a little, so that the frozen solvent just re-liquefies. Dissolve an exactly weighed-out quantity of the test substance (between 0.5 and 1 g = m_S) in this solvent (50 ml, mass m_L). We recommend that you first compress the substance into 1 or 2 pellets, then weigh them and dissolve them. This pelleting avoids the adherence of particles of the substance to the walls of the inner vessel which occurs when the substance is added as a powder.

Fit the inner vessel back in the outer vessel and switch on the stirrer. Follow the drop in temperature with the laboratory thermometer as described above. As soon as the temperature is only about 1°C above the freezing point of the solvent, quickly transfer the Beckmann thermometer back into the apparatus.



Observation: The Beckmann thermometer first shows a continuation in the drop in temperature, but then the mercury thread suddenly rises up. The maximum reached by the mercury thread is the freezing point of the solution (= t_2).

2.4 Evaluation

Mass of solvent = m_L
Mass of dissolved substance = m_S
Relative freezing point, solvent = t_1
Relative freezing point, solution = t_2
Freezing point depression = $t_2 - t_1 = \Delta t$
Molal depression constant
(cryoscopic constant) = K
(the value of this constant for the solvent used must be found in pertinent Tables)
K has a value of 1.853°C/mol for water.

As the freezing point depression is proportional to the molal concentration, we have:

$$M: \frac{m_s \cdot 1000}{m_L} = K: \Delta_t$$

$$M = \frac{m_s \cdot 1000 \cdot K}{m_L \cdot \Delta_t} =$$

Example of results from a measurement:

m_L = 49.91 g (H_2O)
m_S = 0.59 g (hydroquinone)
Δ_t = 0.20°C (from 0.94 - 0.74)
K = 1.853 (for water)

$$M = \frac{0.59\ g \cdot 1000 \cdot 1.853}{49.91 \cdot 0.20} = 109.52\ g/mol$$

(hydroquinone = 110.1 g/mol)

Freezing mixtures

The numbers represent parts by weight	Decrease in temperature from (°C)	to
4 Water + 1 Potassium chloride	+ 10	– 12
1 Water + 1 ammonium nitrate	+ 10	– 15
1 Water + 1 sodium nitrate + 1 ammonium chloride	+ 8	– 24
3 Crushed ice + 1 sodium chloride	0	– 21
1.2 Crushed ice + 2 calcium chloride ($CaCl_2 \cdot 6H_2O$)	0	– 39
1.4 Crushed ice + 2 calcium chloride ($CaCl_2 \cdot 6H_2O$)	0	– 55
Methanol or ethanol + solid carbon dioxide	+ 15	– 77

2.5 Materials

Support rod, *l* = 500 mm, M10 thread		02022.02
Right angle clamp	(2x)	37697.00
Universal clamp	(2x)	37715.00
Magnetic stirrer, with hot plate		35714.93
Apparatus for freezing point depression		36821.00
Beckmann thermometer, 5-0 degrees		36821.10
Magnetic stirring bar, *l* = 15 mm		46299.01
Dish, plastic, 150 x 150 x 65 mm		33928.00
Glass beaker, short, 1000 ml		36017.00
Dewar vessel, 500ml		33006.00
Lab thermometer w. stem, -10...+110°C		38060.00
Volumetric pipette, 25 ml		36581.00
Glass rod, *d* = 8 mm, *l* = 300 mm		40485.06
Pellet press for calorimeter		04403.04
Mortar w. pestle, 150 ml, porcelain		32604.00
Spoon, special steel		33398.00
Balance		
Vice		
Raw alcohol for burning, 1000 ml		31150.70
Ice		
Table salt		
Cloth (e.g. paper cloth)		