



Apparatus for boiling point elevation

36820.00

Operating Instructions

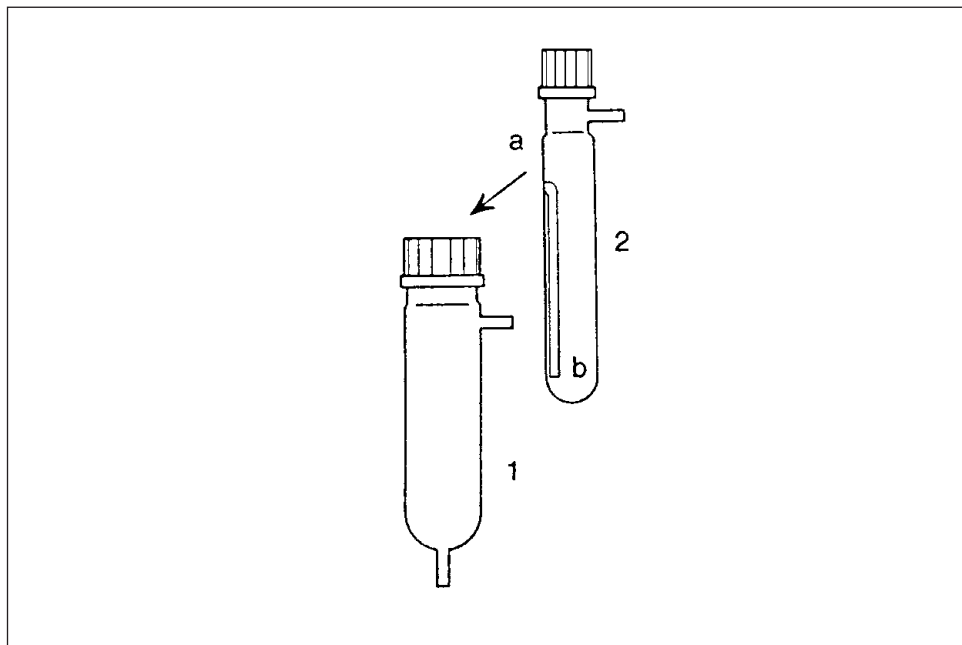


Fig. 1

1 DESCRIPTION OF THE APPARATUS

This apparatus serves to measure the rise of boiling points. It consists of two cylindrical glass vessels, one of which fits in the other. The outer vessel 1 consists of a glass tube with a GL 45 threaded neck, a connecting screw-cap and a GL 45/32 sealing ring. It has a side-arm just below the threaded neck and an extension tube from the rounded bottom (each $d = 8$ mm).

The inner vessel 2 is closed at the bottom. It has a GL 25 threaded neck with a connecting screw-cap and GL 25/12 sealing ring, and also an 8 mm side-arm just below the threaded neck. The small diameter glass tube in this inner vessel is fixed to an inlet opening in the glass wall at a and hangs down to just above the bottom of the vessel at b.

The inner vessel 2 must be so fitted in the outer vessel 1, that the inlet opening a is below the 45/32 mm sealing ring, which, together with the GL 45 screw-cap, ensures an airtight connection of the two vessels. The opening a must not be closed by the sealing ring!

2 EXAMPLE OF EXPERIMENTAL PROCEDURE

The determination of molar masses by measuring the elevation of boiling points (= ebullioscopic method).

2.1 Experimental set-up

Weigh the dry inner vessel to determine the exact mass of it and note this value ($= m_1$) before putting the inner vessel into the outer vessel and fixing them together. Connect the assembled apparatus 3 to a 250 ml round bottom flask which contains approx. 150 to 200 ml of the appropriate solvent and a few boiling stones. Seat the flask in a heating mantle.

Connect short lengths of silicone tubing to the two side-arms. When water is used as solvent, simply allow these lengths of tubing to hang in a glass beaker, with their ends at about the middle of the beaker (do not allow them to

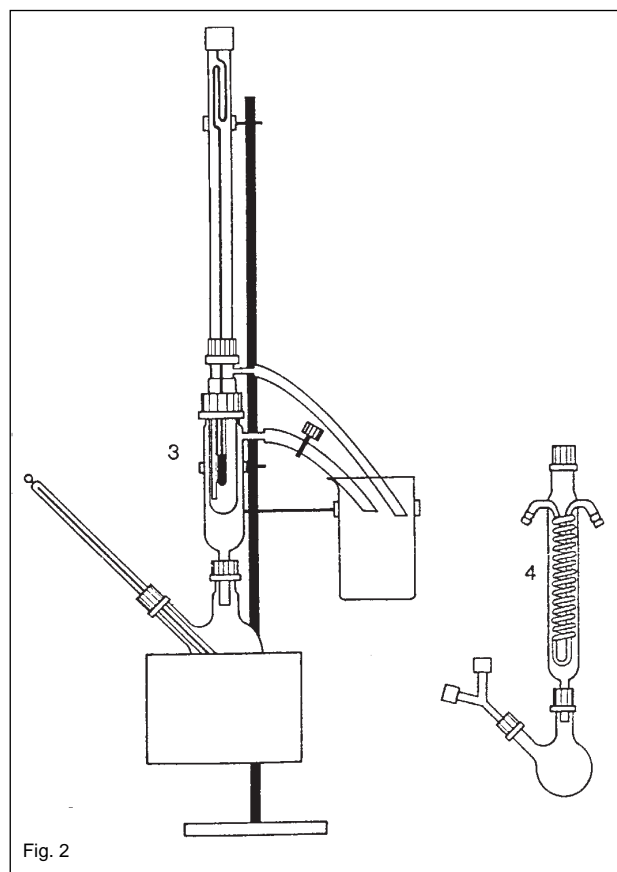


Fig. 2

reach to the bottom of the beaker!). When an organic solvent is to be used, connect the ends of these lengths of tubing to a Y-piece fitted to one neck of a two-necked 100 ml flask, which holds a reflux condenser (Dimroth cooler) in the other neck. Fit a pinchclip on the tubing attached to the outer vessel, but leave it open.

Pour approx. 40 ml of solvent into the inner vessel. Close this vessel at the top by fixing the Beckmann thermometer in position, after having adjusted the Beckmann thermometer to the solvent used (i.e. to the boiling point of the solvent and the anticipated elevated boiling point) according to the operating instructions provided with the thermometer. Remember that Beckmann thermometers are only suitable for measuring temperature differences of up to 5°C, and not for the measurement of absolute temperatures.

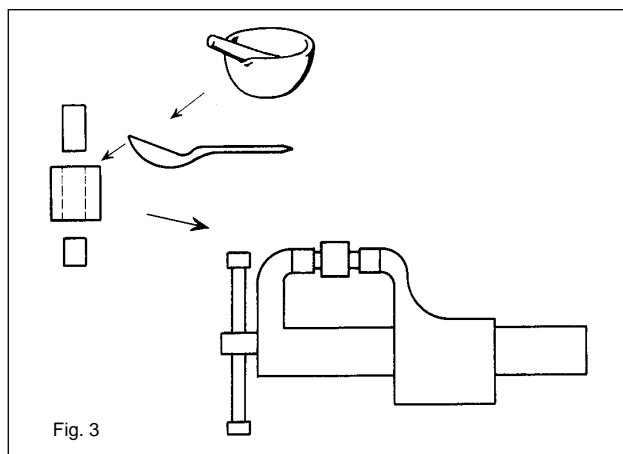


Fig. 3

2.2 Preparation of the test substance

The substance whose molar mass is to be determined must be brought to pellet form, which can be easily achieved with a simple pellet press (Fig. 3). Use the press as follows: Finely grind the substance in a mortar. Put the shorter steel rod in the steel cylinder. It closes the bottom end of the bore hole. Fill the bore hole with finely ground test substance, place the longer steel rod on the bore hole from above and press the substance a little together with it. Fit the assembled press in a vice, as shown in Fig. 2, and apply pressure on the press so that the substance is compacted to a solid pellet. After compaction, press the pellet out of the bore hole with the longer rod. One or two pellets, each of about 5 to 7 mm thickness, are required for a measurement. Determine their mass ($= m_s$) by weighing them to an accuracy of at least 1 mg.

Hydroquinone or urea are suitable as test substances for practice and for demonstration of the determination of molar masses by measurement of the boiling point elevation. The solid substances must naturally only be used in a dry condition. They should be dried for at least 24 hours in a desiccator.

When other test substances are to be used, the appropriate solvent for each must be selected. The specific molal elevation 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 Measurement procedure

Heat the solvent in the flask to boiling. The vapour evolved rises up into the outer vessel and heats up the inner vessel. Part of the vapour condenses, another part passes out into the beaker or flask with reflux condenser.

After some minutes, as soon as the mercury thread of the Beckmann thermometer has risen up into the region of the scale, lower the heating mantle for a few seconds, so that boiling briefly stops and the solvent which has condensed in the outer vessel flows back down into the flask. When this has occurred, lift the heating mantle up to the flask again and allow boiling to continue. When vapour is again evolved, close the pinchclip, so that vapour now flows into the narrow glass tube and out through the solvent in the inner vessel. The temperature of this solvent very quickly reaches the boiling point. Read this off to one hundredth of a degree. When the end of the mercury is exactly between two scale lines, the use of a magnifying glass allows an estimation to be made to thousandths of a degree. Note the (relative) temperature reading ($= t_1$).

Now lift up the thermometer and quickly throw the test substance pellets into the boiling solvent. Close the vessel again, bringing the thermometer back to its original position. Observation: As the substance dissolves, the boiling point temperature increases.

When the mercury thread has again reached a maximum, take the reading and note it ($= t_2$).

Re-open the pinchclip (Important! This is necessary to avoid solution being sucked from the inner vessel into the flask containing solvent when the heating is switched off!), switch the heating mantle off and lower it, so that the solvent in the flask quickly stops boiling.

The mass of the solvent in the inner vessel (m_L) must now be determined. Take the thermometer off and carefully remove the inner vessel (Caution! It is still very hot!). Weigh the inner vessel with the solution and note the mass ($= m_2$).

2.4 Evaluation

Measurements made:

Mass of empty vessel	$= m_1$
Mass of vessel with solution	$= m_2$
Mass of dissolved substance	$= m_s$
Boiling point, solvent	$= t_1$
Boiling point, solution	$= t_2$

Mass of solvent m_L	$= m_2 - m_1 - m_s$
Boiling point elevation Δt	$= t_2 - t_1$

As the boiling point elevation is proportional to the molal concentration ($=$ mol per 1000 g solvent), we have:

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

M = molar mass

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

K = the molal elevation constant (ebullioscopic constant, it has a specific value for each solvent).

The values can be found in pertinent Tables.

Example of results from a measurement:

m_S	=	0.675 g urea
Δ_t	=	0.128 °C (last place estimated)
m_L	=	45 g
K	=	0.515 (for the solvent water)

$$M = \frac{0.675 \text{ g} \cdot 1000 \cdot 0.515}{45.0 \text{ g} \cdot 0.128 \text{ °C}} = 60.35 \text{ g/mol}$$

(urea = 60.05 g)

To determine the molal elevation constants of solvents by use of known substances, K can be found from:

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

2.5 Materials

Retort stand, $h = 750$ mm	37694.00
Right angle clamp	(4x) 37697.00
Universal clamp	(2x) 37715.00
Universal clamp with joint	(2x) 37716.00
Apparatus for elevation of boiling point	36820.00
Beckmann thermometer, 0-5 degrees	36820.10
Round bottom flask, 250 ml, 1-neck, GL 25/12	35812.15
Jointing f. GL 25, 12 mm hole, 10 pcs	41242.03
Glass beaker, tall, 250 ml	36004.00
Flask, round, 2-neck, 100 ml, GL 25/12;GL 18/8	35842.15
Dimroth cooler, GL 25/12	35815.15
Y-piece connector, $d = 8$ mm	47518.03
Silicone hose, i.d. = 7 mm	39296.00
Rubber tubing, $d = 8$ mm	(2x) 39283.00
Mortar w. pestle, 150 ml, porcelain	32604.00
Pinchcock, width 15 mm	43631.15
Spoon, special steel	33398.00
Pellet press for calorimeter	04403.04
Heating mantle, 250 ml	47550.93
Holding rod for heating mantle	47557.01
Urea, 250 g	30086.25
Hydroquinone, 250 g	30089.25
Beads, 200 g	36937.20
Solvent according to requirements	
Balance + vice	