Determination of molar masses via a measurement of the freezing-point depression (cryoscopy) (Item No.: P3022000)

Curricular Relevance

Area of Expertise: Chemistry
Education Level: University
Topic: General Chemistry
Subtopic: Molar Mass
Experiment: Determination of molar masses via a measurement of the freezing-point depression (cryoscopy)

Difficulty	Preparation Time	Execution Time	Recommended Group Size
Intermediate	10 Minutes	10 Minutes	2 Students

Additional Requirements:

- Precision balance, 620 g / 0.001 g
- Ice
- Vice

Experiment Variations:

Keywords:

cryoscopic constant, freezing point depression, molar mass

Overview

Short description

Principle

In order to train and demonstrate the determination of molar masses by way of a measurement of the freezing-point depression, urea or hydroquinone are used as test substances. The cryoscopic constant of water is determined from the freezing point depression.

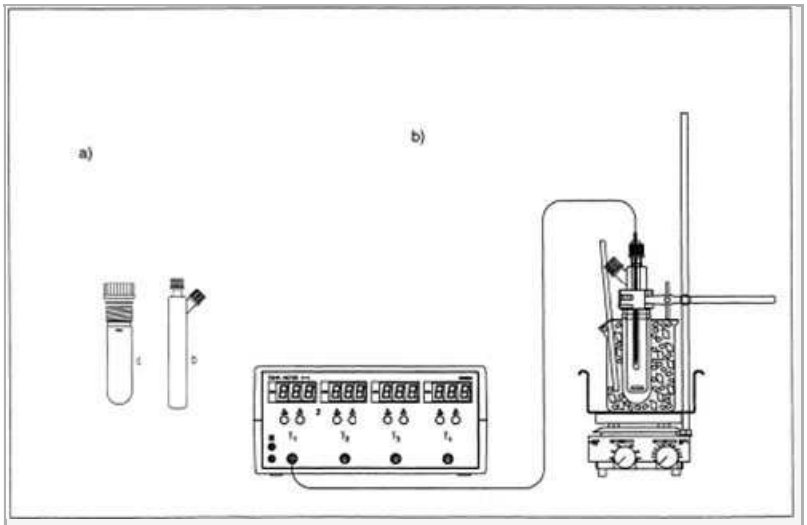

Fig. 1

Safety instructions

Ethanol
H225: Highly flammable liquid and vapour.
P210: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.

Equipment

Position No.	Material	Order No.	Quantity
1	Right angle boss-head clamp	37697-00	1
2	Universal clamp	37715-00	1
3	Supp.rod stainl.st.,50cm,M10-thr.	02022-20	1
4	Magnetic stirrer with heater MR Hei-Standard	35751-93	1
5	Apparatus for freezing point depression	36821-00	1
6	Temperature meter digital, 4-2	13617-93	1
7	Temperature probe, immersion type, Pt100	11759-01	1
8	Protective sleeves f.temp.probe,2	11762-05	1
9	Gasket for GL25, 12mm hole, 10pcs	41243-03	1
10	Magnetic stirring bar 15 mm, cylindrical	46299-01	1
11	Magnetic stirring bar 30 mm, cylindrical	46299-02	1
12	Magnetic stirring bar, 50 mm, cylindrical	46299-03	1
13	Dish, plastic, 150x150x65 mm	33928-00	1
14	Beaker, low, BORO 3.3, 1000 ml	46057-00	1
15	Volumetric pipette, 50 ml	36581-00	1
16	Pipettor	36592-00	1
17	Glass rod,boro 3.3,l=300mm, d=7mm	40485-05	1
18	Pellet press for calorimeter	04403-04	1
19	Mortar with pestle, 150 ml, porcelain	32604-00	1
20	Microspoon, steel	33393-00	1
21	Wash bottle, plastic, 500 ml	33931-00	1
22	Weighing dishes, square shape, 84 x 84 x 24 mm, 25 pcs.	45019-25	1
23	Pasteur pipettes, 250 pcs	36590-00	1
24	Rubber caps, 10 pcs	39275-03	1
25	Desiccator, vacuum, diam. 150 mm	34126-00	1
26	Porcelain plate f.desiccator150mm	32474-00	1
27	Watch glass, dia.80mm	34572-00	1
28	Denaturated alcohol (spirit for burning), 1000 ml	31150-70	1
29	Urea, 250 g	30086-25	1
30	Hydroquinone 250 g	30089-25	1
31	Sodium chloride, 500 g	30155-50	1
32	Water, distilled 5 l	31246-81	1

Tasks

1. Determine the freezing point depression of water dissolving different amounts of urea.
2. Calculate the cryoscopic constant from the experimental results.

Set-up and procedure

Set-up

The apparatus consists of two cylindrical glass vessels that are placed with one inside the other and connected by way of a screw connection GL 45. The outer vessel that resembles a test tube (a in Fig. 1a) forms the jacket that surrounds the inner vessel (freezing vessel, b in Fig. 1a). The jacket can remain filled with air. However, it is better to fill it with approximately 35 to 40 ml of ethyl alcohol (it is possible to use methylated spirit). The outer vessel with the intermediate medium (air or ethyl alcohol) ensures the constant heat transfer from the inner vessel to the freezing mixture.

The inner vessel (b) holds the solution or solvent whose freezing point is to be determined. On its upper end, it has a screw cap for inserting a Beckmann thermometer and a glass tube connector with a screw connection GL 18 for filling in the substance to be dissolved. The bottom of the inner vessel is flat so that a small stirring bar (l = 15 mm) can rotate freely on the bottom.

As shown in Fig. 1b, a dish or tray (e.g. a crystallising dish, plastic tray, or similar) is placed on a magnetic stirrer. Place a 1000 ml beaker (short type) into this dish or tray. The beaker holds a freezing mixture (see the table for possible mixtures). For teaching purposes, a mixture of crushed ice and common salt is usually sufficient. Place the assembled apparatus into the middle of the beaker or freezing mixture and secure it on a support stand as shown in Fig. 1b. Then, place a stirring bar into the inner vessel and insert a protective sleeve for the temperature sensor into the upper screw connector. To do so, replace the gasket of the screw connector with the gasket with the 12 mm hole. Apply two to three drops of methylated spirit to the protective sleeve for better heat transfer and insert the temperature probe. Connect the temperature probe to the digital temperature meter. If the exact density of the solvent is not known, determine the precise mass of 50 ml of the solvent (= mS) by weighing. Use the graduated pipette for measuring 50 ml.

In addition, the experiment requires substance pellets with a thickness of approximately 5 to 7 mm. They can be produced with the aid of the pellet press. To do so, grind some sodium chloride finely in a mortar. Then, seal the hole in the bottom of the press cylinder with the small steel die, fill the hole with the finely ground substance, and insert the longer die from above into the hole. Clamp the press into a vice and press the powdered substance into a pellet that is pushed out of the cylinder by way of the longer die. Determine the mass of the pellet with an accuracy of 1 mg.

Measurement

Fill 50 ml of the intended solvent (e.g. water) with a graduated pipette into the inner vessel. Switch the magnetic stirrer on and adjust it so that the stirring bar rotates quickly (Attention! Do not switch the heater of the stirrer on as well by accident!). Check the falling temperature of the solvent by way of the temperature meter. The freezing mixture is gently moved in the beaker by the stirring bar in order to ensure the uniform cooling of the apparatus.

Once the freezing point of the solvent is reached and remains constant, switch the temperature meter to the mode for measuring the change in temperature with a resolution of one-hundredth of one degree (button $\Delta T/K$; please refer to the operating instructions of the measuring instrument). Then, remove the inner vessel and heat it slightly until the frozen solvent has just about liquefied. Dissolve a precisely weighed substance quantity (approximately 0.5 to 1 g) (= ms) in this solvent (50 ml, mass mL). It is recommended to press the substance beforehand into 1 to 2 pellets (see above), to weight it, and to dissolve it. The advantage is that no substance can stick to the wall of the apparatus as it would be the case if the substance was added in its powder form. Add the substance pellets to the solvents via the lateral connector GL 18. Then, place the vessel with the solution back into the jacket and switch the stirrer on. Observe the falling temperature.

Robert-Bosch-Breite 10
D - 37079 Göttingen

Tel: +49 551 604 - 0
Fax: +49 551 604 - 107

info@phywe.de
www.phywe.com

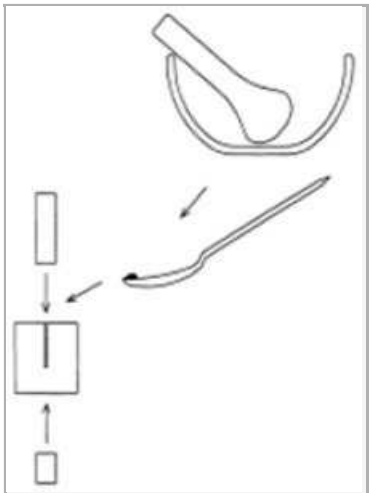

Fig. 2

Observation

First, the temperature drops. After some time, however, it remains constant, since the solution has solidified (frozen). The temperature meter indicates directly the difference with regard to the freezing point of the pure solvent (ΔT).

Theory and evaluation

Evaluation

The following values were measured:

Mass of the dissolved substance	m_s
Mass of the solvent	m_l
Freezing point of the solvent	T_1
Freezing point of the solution	T_2
Freezing-point depression	$\Delta T = T_2 - T_1$

The following equation for the calculation of molar masses of a dissolved substance based on the measurement of the freezing-point depression of the solvent is only valid for sufficiently diluted solutions. At higher concentration levels, the results deviate from the values that can be expected theoretically. The following applies:

$M = \frac{m_S}{m_L \cdot \Delta T} \cdot K$

M = molar mass; K = cryoscopic constant

The value for K can be found in the literature. In the case of water, K has the value 1.853 K•kg/mol.

Example of a measurement:

m_s (Urea)	= 0.62 g
m_l (water)	= 49.97 g
ΔT (freezing-point depression)	= 0.38 K
K (water)	= 1853 Kkg/mol

$M = \frac{0.62\ g}{49.97\ g \cdot 0.38\ g} \cdot 1.853\ K \cdot (\frac{kg}{mol}) = 60.50\ g/mol$

Theoretical value for Urea: M = 60.1 g/mol

The numbers indicate proportions of weight	**temperature decrease from (°C) to**	
4 water + 1 potassium chloride	10	- 12
1 water + 1 ammonia nitrate	10	- 15
1 water + 1 sodium nitrate + 1 ammonia chloride	8	- 24
3 ice, crushed + 1 sodium chloride	0	- 21
1,2 ice, crushed + 2 calcium chloride	0	- 39
1,4 ice, crushed + 2 calcium chloride	0	- 55
methyl alcohol or acetone + solid carbon dioxide	15	- 77

Notes

In order to train and demonstrate the determination of molar masses by way of a measurement of the freezing-point depression, urea or hydroquinone are suitable test substances. However, the substances must be well dry (desiccator). When using other substances, determine the most suitable solvent that can be used. The cryoscopic constant K that is specific for every individual solvent can be found in the literature.

Instead of a digital temperature meter, it is also possible to use a Beckmann thermometer for the temperature measurement. In this case, the experiment must be performed as described in the operating instructions of the "Apparatus for freezing-point depression".

Robert-Bosch-Breite 10
D - 37079 Göttingen
Tel: +49 551 604 - 0
Fax: +49 551 604 - 107
info@phywe.de
www.phywe.com

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Fax: +49 551 604 - 107

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