SOLUTION PREPARATION (5)

Objective

To prepare solutions from solid materials or by dilution from liquids or stock solutions.

Theoretical background

Solutions are homogeneous systems of two or more components in which one of the components is in some way distinguished or emphasized. This usually the (most) abundant component, which is called the solvent; the other(s) are the solute(s).

During the practice, aqueous solution of acids, bases or salts with given quantity and composition will be prepared in a volumetric flask. The density of the solution is also determined to check the composition of the prepared solution. The volume of the volumetric flask is determined by measuring the mass of water at a given temperature as

$$V_{flask} = V_{water} = \frac{m_{water}}{\rho_{water}} \quad , \tag{1}$$

where ρ_{water} is the density of the water at the given temperature.

Materials

volumetric flask of 100 cm^3 volume	*graduated cylinder of 10–20 cm^3 volume*
beaker of 100 cm^3 volume (2 pieces)	*lab spoon or metal spatula*
glass rod (2 pieces)	*wash bottle*

Experimental procedure

The first step is to determine the exact volume of the volumetric flask, which, if the flask is calibrated, is for practice.

Determination of the volume of the volumetric flask

- Fill a large beaker (400–600 cm^3 of volume) or a graduated cylinder (100–250 cm^3 of volume) with distilled water. Wait for 5–10 min and place a thermometer in the water. Wait until the thermometer takes the temperature of the water and then read the temperature and record it in the record sheet (**I.**).
- Take a clean and dry volumetric flask and measure its mass on a top loading balance. Record the mass in the record sheet (**II.**). Details on how to use the balance can be found in the Appendix.
- Fill the flask to its mark with distilled water, and measure the mass of the flask with water and record it in the record sheet (**III.**). The liquid level should be adjusted to the corresponding horizontal mark on the equipment. The liquid surface, the so-called meniscus, is approximately a spherical cone. With transparent liquids, such as water, the lower level of the meniscus is to be set to the mark. With nontransparent solution, such as potassium permanganate solution, the upper level could be adjusted to the mark. *Tips: When the meniscus is almost at the mark, carefully, drop by drop add the distilled water.*

t/°C	ρ/(g/cm^3)
20	0.9982
21	0.9980
22	0.9978
23	0.99755
24	0.9973
25	0.99705
26	0.9968
27	0.9965
28	0.9962
29	0.9959
30	0.9956

Table 1: Density of distilled water at different temperatures.

- Calculate the mass of water in the flask (**IV.**)
- From Table 1 find the density of water for the measured temperature. Use linear interpolation (details in the Appendix), if necessary, to obtain the exact density, when the temperature is not in the table.
- Calculate the volume of the volumetric flask from Eqn. (1) and record it in the record sheet (**V.**).

Solution preparation

Solution preparation from solids (A)

1. Record the formula of solute, its molar mass, the volume and the composition of the solution to be prepared in the record sheet.
2. Record the formula of the available solid chemical and its molar mass in the record sheet.
3. Calculate the mass of the solid compound needed to prepare the solution with the required amount and composition. Record the calculated mass in the record sheet.
4. Take a clean and dry beaker. Place it on an analytical balance. Tare it. Measure out the calculated amount of the solid using a clean and dry spatula. It is not a problem if the mass differs by 0.001 g or even a bit more but you have to record the measured mass (**VI.**) in the record sheet.
5. Add a small amount of distilled water (solvent) to the solid with the wash bottle and stir it with the stirring rod. We can speed up the dissolution by gently heating the solution. *Tips: Make sure that the amount of water is smaller than the amount of solution to be prepared and that the water will not be be spilled from the beaker. Wait until the warm solution cools down.*
6. Carefully transfer the solution into the volumetric flask. Wash the remaining of the solution from the beaker and its wall into the flask with small amounts of the solvent. Do not wash with too much water, the flask should not be washed to the mark. Close the flask with the stopper and while holding the stopper turn upright-down several times to mix the solution properly. Carefully, drop by drop fill the flask to the mark with a wash bottle. Finally mix the solution thoroughly by closing the flask with the stopper securely and inverting it several times.
7. Show your instructor the prepared, properly mixed solution of known volume.

Solution preparation by dilution from liquids (B)

1. Record the formula of solute, its molar mass, the volume and the composition of the solution to be prepared in the record sheet.
2. Record the composition and the solution density of the liquid available in the record sheet. You will find them on the glass holding the stock solution.
3. Calculate the volume of the liquid with given composition and density needed to prepare the solution in the required amount and composition. Record the calculated volume in the record sheet.
4. Find the measuring glass ware for the liquid. Graduated cylinder, pipet or buret can be used depending on the type of the liquid and on the required precision. use the latter two when high precision is required. Select always the glassware with sizes closest to the calculated volume. Special pipets should be used when handling concentrated acids.
5. Typically the liquid can be diluted in a beaker by adding distilled water about half the volume of the flask in small portions while stirring BUT NOT WHEN DILUTING CONCENTRATED ACIDS AND BASES or other liquids where the dissolution is highly exothermic and the density is much greater than that of water. In that case add about half the volume of the flask distilled water to the beaker. *Tips: Be sure that the total volume of the liquid and distilled water is still smaller than the volume of the flask.* Slowly transfer the measured stock solution into the beaker containing distilled water. Stir it with a

stirring rod. Wash the stirring rod into the beaker. If the solution becomes hot, wait until it cools down. Record the measured volume of the stock solution (**VI.**) in the record sheet.

6. Carefully transfer the solution into the volumetric flask. Wash the remaining of the solution from the beaker and its wall into the flask with small amounts of the solvent. Do not wash with too much water, the flask should not be washed to the mark. Close the flask with the stopper and while holding the stopper turn upright-down several times to mix the solution properly. Carefully, drop by drop fill the flask to the mark with a wash bottle. Finally mix the solution thoroughly by closing the flask with the stopper securely and inverting it several times.
7. Show your instructor the prepared, properly mixed solution of known volume.

Determination of the prepared solution composition

1. Calculate from the volume of the volumetric flask (**V.**) and the measured mass or volume of the solute (**VI.**) the solution composition (**VII.**). Write down the details of the calculations.
2. Write down the relevant H and P sentences in the record sheet.
3. Empty the solution from the volumetric flask where instructed. Clean the flask by washing it with detergent solution followed by rinsing with tap water couple of times. Finally rinse the flask by distilled water and return it to the technician.

Sample calculation

Solution preparation from solid material

The task is to prepare 100 ml NaCl solution with 5.70 $w\%$ composition from NaCl solid. The solid is available is NaCl (M_r = 58.44). Calculate the mass of NaCl needed.

1. Find the density of the solution to be prepared if it is not given. Use linear interpolation if needed.
   We have to calculate the density using linear interpolation from tables available in the lab based on data of $y_a = \rho_a = 1.0268$ g/cm^3 with $x_a = w\%_a = 4\%$ and $y_f = \rho_f = 1.0413$ g/cm^3 with $x_f = w\%_f = 6\%$. This yields for the density of the solution:
   $\rho_m = \rho_a + (w\%_m - w\%_a)\dfrac{\rho_f - \rho_a}{w\%_f - w\%_a} = 1.0391 \text{ g/cm}^3$
2. Calculate the mass of the solution from the density and volume of the solution as
   $m = \rho \cdot V = 1.0391 \text{ g/cm}^3 \cdot 100 \text{ cm}^3 = 103.91 \text{ g}$.
3. Calculate the mass of the solute as $m_2 = m \cdot w\%/100\% = 103.91 \text{ g} \cdot 0.0570 = 5.92$ g.

Solution preparation by dilution of stock solution

Our task is to prepare 100 ml hydrochloric acid with 5.00 $w\%$ composition using concentrated hydrochloric acid (M_r = 36.46). Calculate the volume of the concentrated HCl solution required to prepare the solution.

1. Find the composition and solution density of the concentrated (stock) solution on the glass holding the stock solution. For concentrated hydrochloric acid: 36 $w\%$ with density of 1.1789 g/cm^3.
2. Find the density of the solution to be prepared if it is not given. Use linear interpolation if needed.
   We find from a Table given by the instructor that the solution density is 1.0230 g/cm^3.
3. Calculate the mass of the solution from the density and volume of the solution as

$$m = \rho \cdot V = 1.0230 \text{ g/cm}^3 \cdot 100 \text{ cm}^3 = 102.30 \text{ g}.$$

4. Calculate the mass of the solute as $m_2 = m \cdot w\%/100\% = 102.30$ g·5/100 = 5.115 g.
5. Calculate the mass of the concentrated solution with that mass of solute:
$m = m_2/w = 5.115\ \text{g}/0.36 = 14.209\ \text{g}$
6. Calculate the volume of the concentrated hydrochloric acid required as

$$V_2 = m_2/\rho_2 = 14.209\ \text{g}/(1.1789\ \text{g/cm}^3) = 12.052\ \text{cm}^3 \quad \rightarrow \quad \mathrm{V}_2 = 12.1\ \text{cm}^3.$$

Appendix

Use of balances

1. Rules
   (a) Always check the maximum capacity of the balance. Never exceed it.
   (b) Never put any chemicals directly on the balances. Always use weighing paper, beaker, etc.
   (c) Never put hot containers on the balance, objects must be at room temperature.
   (d) Be sure that the container is dry outside when placing the container on the balance.
   (e) Always leave clean the pan and the balance after using it. Use brush to clean it.
   (f) Try to use the same balance during the same experiment.
2. Weighing on a top-loading balance
   (a) Turn on the balance and wait until the display stabilizes. Tare it if needed.
   (b) Place the object or container on the pan and wait until the display stabilizes. Record the mass if needed or tare it and carefully add the required material to the container. Wait until the display stabilizes and record the mass of the material.
   (c) Remove the object or the container from the balance and gently clean it with a brush provided if needed.
3. Weighing on an analytical balance when high-precision (0.1 mg) is needed
   (a) Close the doors of the balance if they are open.
   (b) Turn on the balance and wait until the display stabilizes. Tare it if needed.
   (c) open the balance door and place the object or container on the pan. Close the balance door and wait until the display stabilizes. Record the mass if needed or tare it and after opening the balance door carefully add the required material to the container. Close the balance door and wait until the display stabilizes and record the mass of the material.
   (d) After opening the balance door remove the object or the container from the balance and gently clean it with a brush provided if needed. Close the balance doors.

Linear interpolation

During an experimental work we often use tables that present certain physical properties, e.g., density, at different temperatures. It is also common that the data we need are not listed in the table. In such cases, we can also use the table and infer the unknown from the known data. The simplest method to use is the so-called linear interpolation during which we assume that between two adjacent points in the table there is a linear relationship. Assuming linear relationship between the physical property, e.g., density, as a function of the temperature in a narrow range, the density can be calculated between any two points, as it is shown in Fig. 1.

If we want to approximate y_m (e.g., density) for the measured quantity x_m (e.g., temperature), we select a quantity less than x_m, for example x_a, and a quantity greater than x_m, for example x_f, for which y_a and y_f

are known. We select x_a and x_f as close as possible to the measured quantity x_m to have the best assumption possible. Having similar triangles in Fig. 1 the figure, it can be written, that

$$\frac{y_m - y_a}{x_m - x_a} = \frac{y_f - y_a}{x_f - x_a} \quad , \tag{2}$$

which after rearrangements leads to

$$y_m \;=\; y_a \;+\; (x_m - x_a)\frac{y_f - y_a}{x_f - x_a} \; . \tag{3}$$

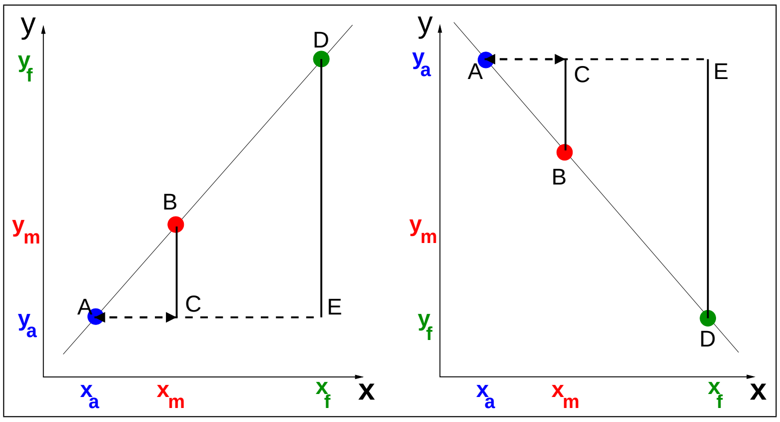

Figure 1: Schematic diagram of linear interpolation.

Example: Assuming linear relationship between the density and the temperature in a narrow range, the density can be calculated between any two points. We want to determine the density of water at $x_m = 20.3$ °C, therefore from Table 1 we have to select the closest temperatures around it, which are $x_a = 20$ °C and $x_f = 21$ °C with their corresponding densities of $y_a = \rho_a = 0.9982$ g/cm^3 and $y_f = \rho_f = 0.9980$ g/cm^3. The density of water at 20.3 °C substituting into Eqn. 3 is

$$y_m \;=\; \rho_m \;=\; 0.9982\text{g/cm}^3 + (20.3 - 20)^\circ\text{C}\frac{(0.9980 - 0.9982)\text{g/cm}^3}{(21 - 20)^\circ\text{C}} \;=\; 0.99814 \text{ g/cm}^3 \; .$$