8. Preparation and titration of NaOH solution

Solid sodium hydroxide, NaOH, is hygroscopic compound and easily reacts with CO_2 in air forming Na_2CO_3. So, it is not a basic substance (i.e. substance that one can measure its amount by direct weighing). This means preparation of solution of this base with known concentration needs maintaining some rules, and exact concentration of the base can be measured by titration of a known amount of acid which is a basic substance.

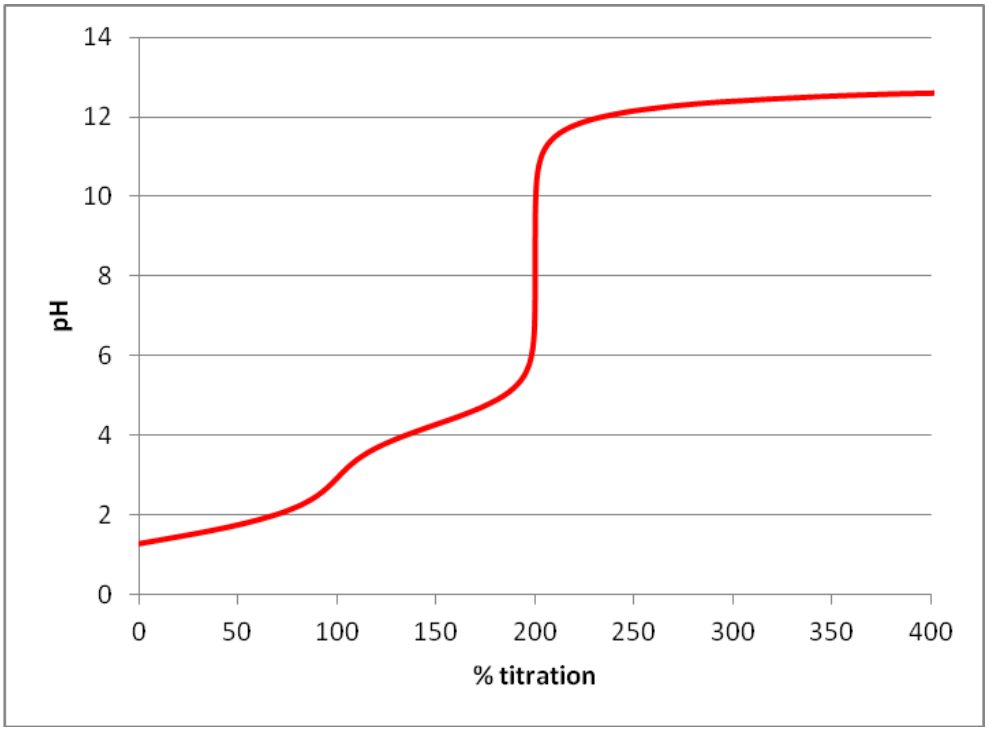
Preparation of ≈0.1 M NaOH solution

The solution to be prepared will have concentration sometimes rather different than the desired 0.1 mol/dm^3. This is not a problem, because the exact content of NaOH will be determined experimentally. Nevertheless, one should make efforts to have the solution free of dissolved sodium carbonate, the compound always present at the surface of granules of solid NaOH. Because of that, the best idea is preparing our titrant using concentrated, almost saturated NaOH solution. Such liquid can contain even ca. $50\%_{mass}$ of sodium hydroxide. Important is that in such highly concentrated solution the contaminating Na_2CO_3 has very low solubility and falls down the vessel as precipitate.

If one knows approximated mass concentration of NaOH and density of the solution, it is easy to calculate the volume in mL of it containing approximately 0.1 mole of the base. Such portion of the solution we will dilute to about 1 dm^3, exactly stir and measure exact concentration by titration.

Determination of the concentration of NaOH

This is the simplest version of direct volumetric titration. Pour your NaOH solution to 50 cm^3 burette. Weigh (on analytic balance) such the amount of the oxalic acid dihydrate, $(COOH)_2 \cdot 2H_2O$, which should react with ca. 20 cm^3 0.1 M NaOH. This portion of oxalic acid transfer quantitatively to a titration flask, dissolve in water, add few drops of Phenylphthalein and titrate to red color. The reason of use of this particular indicator is illustrated by the titration curve below. Note that the first inflection(formation of HOOC-COONa) is very weak and cannot be used for determination of the end-point of titration using the classical volumetric titration and indicators.



The titration curve of 0.1 M oxalic acid using 0.1 M NaOH. Acidity of$(COOH)_2$: pK_{a1}=1.27; pK_{a2}=4.28 (Wikipedia).

The procedure of titration should be repeated for three independent portions of oxalic acid minimum. The final result is an arithmetic average of these measurements.

Procedure

1. Prepare a bottle 1 L of capacity, wash it and check its closing. Make the label with your name and description: „"NaOH, ca. 0.1 M".
2. Calculate the volume of NaOH solution (44%$_{mass}$, density d=1.44 g/mL), which contains ca. 0.1 moles NaOH.
3. Take the calculated volume of the concentrated NaOH using calibrated pipette (Attention! The NaOH solution is harmful!).

Determination of the exact NaOH concentration

4. Calculate the exact mass of oxalic acid dihydrate $(COOH)_2 \cdot 2H_2O$ which reacts with ca. 20 mL 0.1 M NaOH.
5. Weigh oxalic acid (in weighing vessel, using analytical balance) as calculated at point 4.
6. Transfer this portion of acid quantitatively to an Erlenmayer flask and dissolve in it in ca. 150 mL of water.
7. Add few drops of Phenylphthalein and titrate using your NaOH until red color appears. Knowing the volume of base and the number of moles of acid calculate the exact concentration of NaOH.
8. Repeat points 5-7 two times more. Calculate the mean concentration of NaOH from these three titrations.

Report: should contain calculations of the concentrated NaOH used for preparation of titrant (point 2), as well as calculations of the mass of acid (point 4), and the results of titrations together with the final NaOH concentration in the solution calculated as the average.