

Titration [2020-06-16]

Why would the pH value changes dramatically when reaching the equivalence point?

Let's assume there is 1dm³ of solution, and we don't increase the volume when titrating (for the ease of explanation), and that negligible amount of water is formed during the neutralization.

Consider **adding alkali into an acid** of (originally) pH 3.

	Number of moles of H ⁺	Number of moles of OH ⁻	pH value
Beginning	0.001	≈ 0	≈ 3
Add 0.0003 mol of OH ⁻	0.001	0.0003	
Shaked (so they reacted)	0.0007	≈ 0	≈ 3.15
Add 0.0003 mol of OH ⁻	0.0007	0.0003	
Shaked (so they reacted)	0.0004	≈ 0	≈ 3.40
Add 0.0003 mol of OH ⁻	0.0004	0.0003	
Shaked (so they reacted)	0.0001	≈ 0	≈ 4_{acidic}
Add 0.0003 mol of OH ⁻	0.0001	0.0003	
Shaked (so they reacted)	≈ 0	0.0002	≈ 10.3_{alkaline}
Add 0.0003 mol of OH ⁻	≈ 0	0.0005	≈ 10.7

可以看到，從某一刻開始，OH⁻ 突然反客為主，本來酸性的溶液變成鹼性
(這樣理解：酸多就全酸、鹼多就全鹼，都比較霸道——從酸變鹼有點像一刀切)

Experiment on *titration* (important for SBA as well as the coming S4 Sessional Examination):

(May refer to the worksheet *Topic IV Acids and Bases - Experiment 4.7.1 Finding the molarity of a given sodium hydroxide solution*, here I would complement something more, according to **Textbook 2 Chapter 19** and what is said by Mr.Ng in the demonstration of 2020-06-16)

- We are performing an experiment to determine the **molarity** (currently unknown) of a given dilute NaOH solution using a standard dilute HCl solution.
It is written on the worksheet: 'Standard dilute hydrochloric acid (~0.1M)'.
Notice that we may not need to have 0.1 M solution (can be 0.15 M, 0.12 M, 0.1 M, 0.08M). **As long as we clearly know the concentration very accurately** of the HCl solution **whatever we choose to use**, it's fine. You may not have 0.1 M solution, but you must **know the concentration (molarity)** of the HCl solution you're using.
- Refer to page 19-13 of the book, Figure 19.3, at the bottom of the page:
 - Whenever we read the reading of whatever solution by whatever *burette/pipette*:
 - Never read it too high or too low, we should instead read the **bottom of the meniscus with eyes at a proper level**.

- To read the level of a *burette*, use two fingers to hold the upper part of the *burette* but allow it to slowly restore to vertical by gravity on its own. Refer to the last point from above for how to read the solution level properly.
 - **Avoid** jumping high or climbing up the table to read: simply take it down.
- We have to wear *safety goggles* and *gloves* (to be safer, especially for strong & concentrated acid / alkali).
- We usually put the solution with unknown concentration in the conical flask at the bottom (with an accurate volume measured by a *pipette*), and then the standard solution in the *burette* (the *titrant*).
- We shall **first wash the apparatus**:
 - *Conical flask*:
 - We should **not** use a *beaker* instead, **nor draw a beaker** in the diagram of the experiment set up of *titration*.
 - The conical shape allows it to be swirled gently without spilling out the solution.
 - Use distilled water (*Plastic washbottle*) to rinse the inner wall of the *conical flask*.
 - We shall keep the **number of moles of NaOH** in the conical flask under control / within our expectation. Slightly more distilled water doesn't matter.
 - *Pipette*:
 - Use distilled water to wash for a few times then use NaOH solution to wash for another few times.
 - We may hold the *pipette* / *burette* horizontally and rotate it for several folds, then pour out the liquid inside from another end (we get the solution from an end, and there is another end), as long as the inner wall of the pipette is rinsed and cleaned, it's fine.
 - We shall keep the **concentration of NaOH** known (under control / within expectation).
 - After washing, we should support the *pipette* **and** *burette* with a *stand and clamp* above the table (instead of putting it somewhere on the table) to keep the apparatus clean.
 - *Burette*:
 - The cleaning procedure is almost the same as pipette, but we are delivering HCl solution instead of NaOH solution in this case.
- Then, we prepare the sodium hydroxide solution:
 - In this experiment, we shall measure exactly 25.0 cm³ of NaOH solution by *pipette* and transfer it to the *conical flask*.
 - Pour the NaOH solution out from the reagent bottle to a beaker.
 - **Don't directly** use *dropper* or *pipette* or whatever apparatus to get the solution in the *reagent bottle* in order not to contaminate the solution stored.

- Use a hand to hold the **upper part** of the *pipette* (use the **index finger** to block the upper end of the pipette) and make it stand on the beaker, touching the bottom of the beaker of NaOH solution just poured out.
 - **Never hold the bulb (middle part) of the pipette.** By thermal expansion, heat would be transferred from your hand to the *pipette*, so as to the solution inside, causing the volume of solution to be inaccurate (less than we expect).
- Use a *pipette filler* (plastic), compress (deform) it, then put it at the top of the pipette, **slowly** release it, and wait for the solution to be pumped up and the pipette to be filled.
 - You may hold the *pipette* slightly above the bottom of the beaker in order to fill the *pipette* faster, but **never** make it above the solution level, in order that **no air** would be pumped up when filling the *pipette*, causing spilling of solution.
- When the *pipette filler* returns to its original shape, take away the *pipette filler* temporary and simultaneously use the index finger of your hand to block the upper end of the *pipette* **immediately**, otherwise the solution held by the pipette would be drained out and you have to refill again.
- Repeat the process a few time (compress the pipette filler, fill the pipette, use the index finger to block the upper end while re-compressing the pipette filler using another hand, so on and on)
- Make sure there are no air bubbles inside the *pipette*, or else you shall wait until all air bubbles move up and escape the solution.
- We should fill the *pipette* to somewhere above the graduation mark (25cm^3).
- You should be holding the upper end of the pipette and blocking air into the pipette. Release just a tiny little bit (按得沒那麼緊、稍微鬆手) and then the solution would drain out a little bit, until the graduation mark (25cm^3) is reached. Usually you may just kind of slightly shake your index finger continuously and the solution would slowly drain accordingly, to 25cm^3 .
- After exactly 25cm^3 of NaOH solution is measured, transfer it to the *conical flask*. Release your index finger and then the solution would slowly drain on its own.
- There may be a tiny amount of solution left inside the *pipette*. Touch the inner wall of the *conical flask* using the *pipette* to transfer that drop of solution. In case any further solution remains inside the *pipette*, **do not** force it to get out: it is calculated (under consideration) during the design of *pipette*.
- We should then add **one drop** of acid-base indicator:
 - Refer to page 19-15 of the book, we shall use *methyl orange* or *phenolphthalein* in this experiment as NaOH and HCl solutions are strong alkali and strong acid respectively.
 - To increase the ease of detection of the *end point* (**sharp colour change** of the indicator), two drops of *methyl orange* would be even preferred.
 - Then we're ready with the *conical flask*.
- We should place a *white tile* below the *conical flask* to allow clear observation of the colour change of the indicator.

- After that, we should fill the *burette* with the hydrochloric acid (the solution in burette is called the **titrant**):
 - Always check if the valve of the *burette* is closed. Only when the *stopcock* is nearly vertical, the *burette* would let the liquid drain.
 - Place a *filter funnel* above the *burette* and pour the HCl solution into the *burette*.
 - We should better keep the *filter funnel* clean as well in SBA, but it may not be of high priority.
 - We should fill the *titrant* up to a certain (arbitrary) level and then **accurately** jot down the initial *burette* reading.
 - No air bubbles should be in the solution, or else you should wait until the air bubbles move up and escape the solution.
 - Make sure the **jet** (噴嘴) is filled completely.
 - We should let it try to drain a little bit once (let the solution pass through the *jet* once) in order to *fill the jet completely*.
 - But when rinsing the *burette* ('washing' it with HCl), or as the first trying trial has been performed, the *jet* would already be filled, so we shall not worry about this.
 - **Take away** the filter funnel after filling the *burette*.
 - This is to prevent any remaining HCl solution in the filter funnel from dripping down to the *burette* which reduces the accuracy of the volume of HCl solution in the *burette* measured.
- Then we should perform *titration*:
 - Add solution drop by drop (or after it comes close to the estimated *titre*) and **swirl** the conical flask (gently) meanwhile, until the solution **just** turns another colour sharply, indicating the *end point* of titration.
 - There may be a small region of the solution becoming red (in case that methyl orange is used) when a drop of HCl solution is just dripped. However, after we swirl it, if the colour changes back, it means that the solution is not acidic **overall** yet.
 - We should only take account of **persistent / permanent** colour change.
 - The sharp colour change indicates the *end point*. The titration should end now.
 - Take the final *burette* reading. It should be larger than the initial *burette* reading as the marking increases with decreasing height. Subtract initial *burette* reading from the final *burette* reading, and then you can get the **titre**, the volume of titrant used to react completely with the solution in the *conical flask*.
- The first time performing titration should be a trial, and is **never** counted when calculating.
 - We let the solution drain quickly and record the rough estimation of *titre* and give a suitable expectation for following trials of experiment.
 - In (paper) examination, if a data set is given, we should **never** include the first column of data, else marks may be deducted.
 - After the first trial, we know roughly how much solution should be added (know roughly the **titre**), **so that time can be saved** next time (as we could just allow the solution to drain quickly until it is close to (but smaller than) the estimated

titre, and only perform titration **drop by drop** very slowly and carefully after that).

■ Notice that one drop of solution is roughly 0.05cm^3 .

- With **titre** obtained, we shall (**excluding** the first trial) take three **consecutive** data with **similar** results, then calculate the average of them as the final value of **titre** to be used in calculations.