# Assay of Aspirin by Indirect Acid- Base titration

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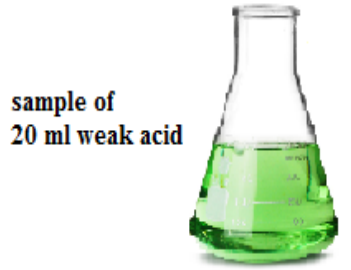
- Indirect titration is defined as the analytical technique that can be used to determine the weight of  an unknown (sample) by using an excess amount of a standard solution.
- Indirect titration (residual titration) is used

1. The sample is volatile
2. The sample is insoluble salt
3. Particular reaction is too slow
4. It is difficult to determine the end point

- Indirect titration ( residual titration) is a two stage analytical technique:

1. React the unknown ( sample) with an excess amount of the standard solution.
2. Determine the volume of the excess amount of the standard solution by using the direct titration with an another standard solution.

# Explain the base

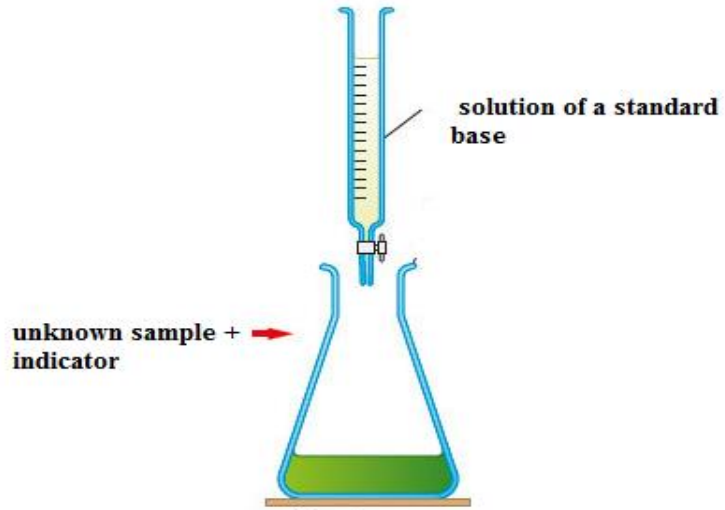



For example if you have:

1. A (20ml) sample of a syrup that is weak acid in a conical flask

2. Titrate this sample with a standard base (NaOH)

The 20 ml of the sample needs **40 ml of NaOH** to reach the end point, **then add an excess of the base 10 ml (the total volume of NaOH is 40+10 = 50 ml)**




**solution 2**

3 Now titrate the **solution 2** that contains an excess of NaOH with a standard HCl solution Why?

Because you will determine the excess (**residual)** volume of NaOH

HOW? Using the direct titration between the standard HCl and the excess NaOH

N V(HCl)= N V( excess NaOH)

V (excess NaOH) = NV(HCl)/ N (excess NaOH)

- Now you have determined the excess (residual) volume of NaOH suppose V (excess NaOH) = 21 ml.

Total (V NaOH = **50ml**), V excess NaOH is **21ml**

The real Volume of NaOH that reacted with the unknown solution is

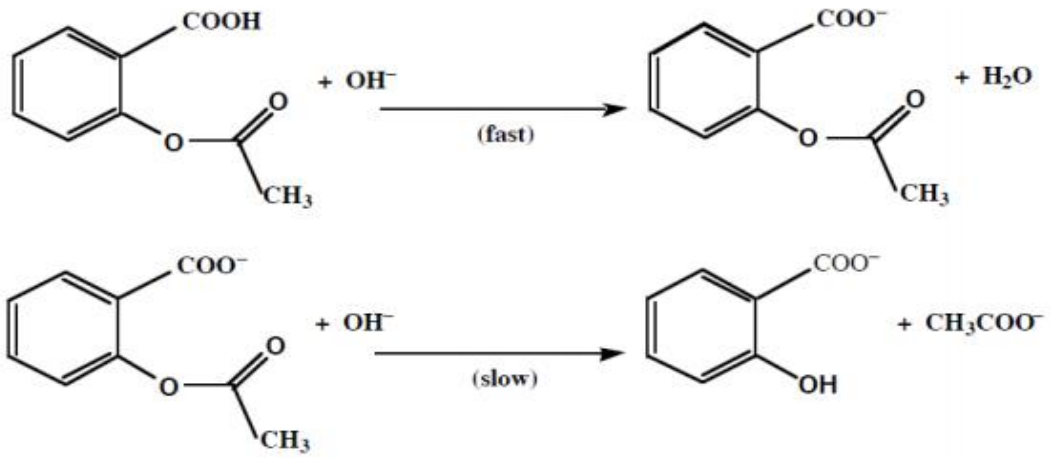
50 ml - 21ml= 29ml of NaOH reacted with the sample of the weak acid

The 29ml then can be used to calculate the weight of the unknown

# Indirect titration

## The aspirin

Is a weak acid that undergoes slow hydrolysis, therefore aspirin molecule reacts with two hydroxide ions. This problem is solved, by adding a known excess amount of the NaOH base to the sample solution and an HCl titration is carried out to determine the amount of unreacted base.



# Prepare the sample

1. Accurately record the weight of a group of three aspirin tablets so that you can determine an average tablet weight. Use a mortar and pestle to crush enough tablets to produce approximately ( 1 g) tablet powder.
2. Weigh approximately 300 mg aspirin powder, into labeled 250 mL Erlenmeyer flasks

3. To each flask, add 20 mL of ethanol (measure by graduated cylinder) and three drops of phenolphthalein indicator. Swirl gently to dissolve. Aspirin is not very soluble in water the ethanol helps the aspirin dissolve. Note that an aspirin tablet contains other compounds in addition to aspirin. Some of these are not very soluble. Your solution will be cloudy due to insoluble components of the tablet.)

# Indirect titration

4. Titrate the first aspirin sample with (0.1 N) NaOH to the first permanent cloudy pink color.

5. Heat gently the flask contents in a water bath. Avoid boiling, because the sample may decompose. While heating, swirl the flasks occasionally. After 15 minutes, remove samples from the water bath and cool for 5 minutes.

6. If the solution is colorless, add a few more drops of phenolphthalein. If it remains colorless, add 10 mL more of the base and reheat. (Don't forget to add this additional volume of base to the previously recorded total volume.)

7. The aspirin/NaOH acid-base reaction consumes one mole of hydroxide per mole of aspirin. The slow aspirin/NaOH hydrolysis reaction also consumes one mole of hydroxide per mole of aspirin, and so for a complete titration we will need to use a total of twice the amount of NaOH that you have already used, plus we will add some excess NaOH to ensure we really have reacted with all of the aspirin in the sample. (For example: if you used 26 mL of base in the previous step, the volume of base you would add now would be 26 + 10 = 36 ml. Thus, you would have added a total of 26 + 26 + 10 = 62 mL of base.)
Heating for Completion of Reaction

8. The only base remaining in each flask will be excess base that has not reacted with the aspirin. Using your burette with your ~0.1 NHCl solution, titrate the excess base in each flask with HCl until the pink color just disappears. The endpoint is best described as “cloudy white”

9. Record all the volumes of bases and acid in the data table.

# Data & Calculations

1. Wt of aspirin (practical) = (V NaOH reacted with sample) x N NaOH x Eq.wt  of aspirin
2. Recovery % = practical content / theoretical x 100

# Homework

1. Aspirin contains the carboxylate group that is considered as a problematic functional group, can you briefly ( 3 lines) discuss an alternative group to replace the carboxylate group? **Please, be careful, copy your answer from other students as the copy past answers will be marked zero.**
2. Draw to explain the indirect titration and its calculation (you can use power point) **draw to explain, copy past answers will be marked zero**

# Thank you