

TIKRIT UNIVERSITY COLLEGE OF PHARMACY



Inorganic Pharmaceutical Chemistry

Lab7

Back titration

Assay of aspirin

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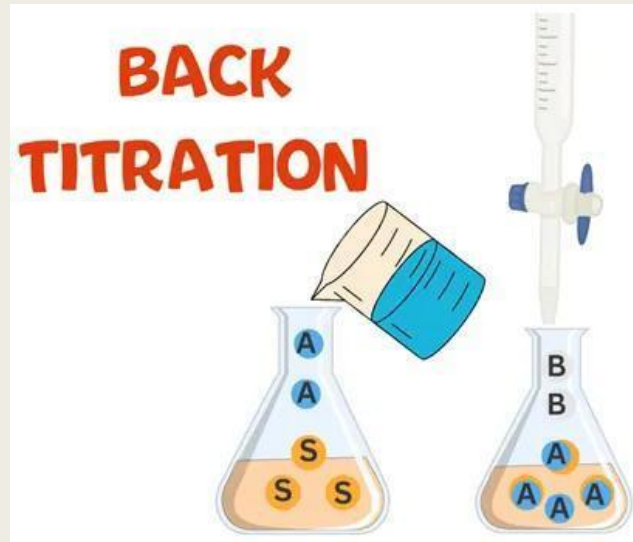
Back titration

- back titration is a titration method where the concentration of an analyte is determined by reacting it with a known amount of excess reagent. The remaining excess reagent is then titrated with another, second reagent. The second titration's result shows how much of the excess reagent was used in the first titration, thus allowing the original analyte's concentration to be calculated.

Back titration

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

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



Application of back titration

Back titration is usually carried out when the substance under estimation:

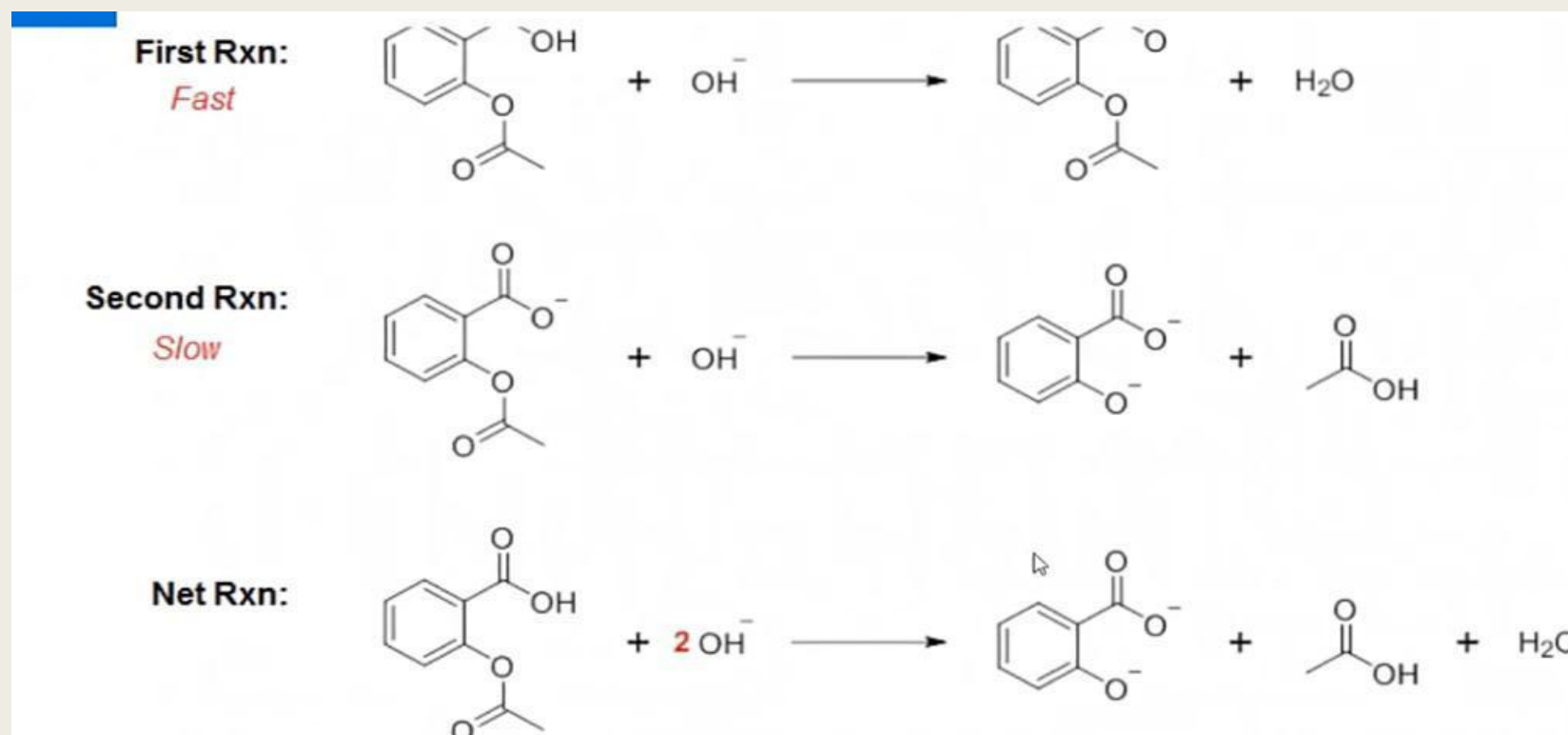
1. is insoluble in water (e. g, magnesium hydroxide and calcium carbonate; which require excess of the standard solution to be solubilized.
2. fails to give a sharp end point with the indicator used in direct titration.
3. is volatile (e. g, ammonia (some of which would be lost during the titration
reacts rapidly only in the presence of excess of the standard solution(e. G., aspirine and lactic acid(
4. decomposes when heated with the standard solution (e. G.,formaldehyde; heating is required during titration(

Assay of aspirin by back titration

- Aspirin, also known as acetylsalicylic acid (ASA), is a [nonsteroidal anti-inflammatory drug](#) (NSAID) used to reduce [pain](#), [fever](#), and/or [inflammation](#), and as an [antithrombotic](#). M.wt=180.17, is insoluble in water, soluble in ethanol.
- Aspirin is 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.

Chemical principle

- ASA undergo to reaction with sodium hydroxide fast and slow reaction



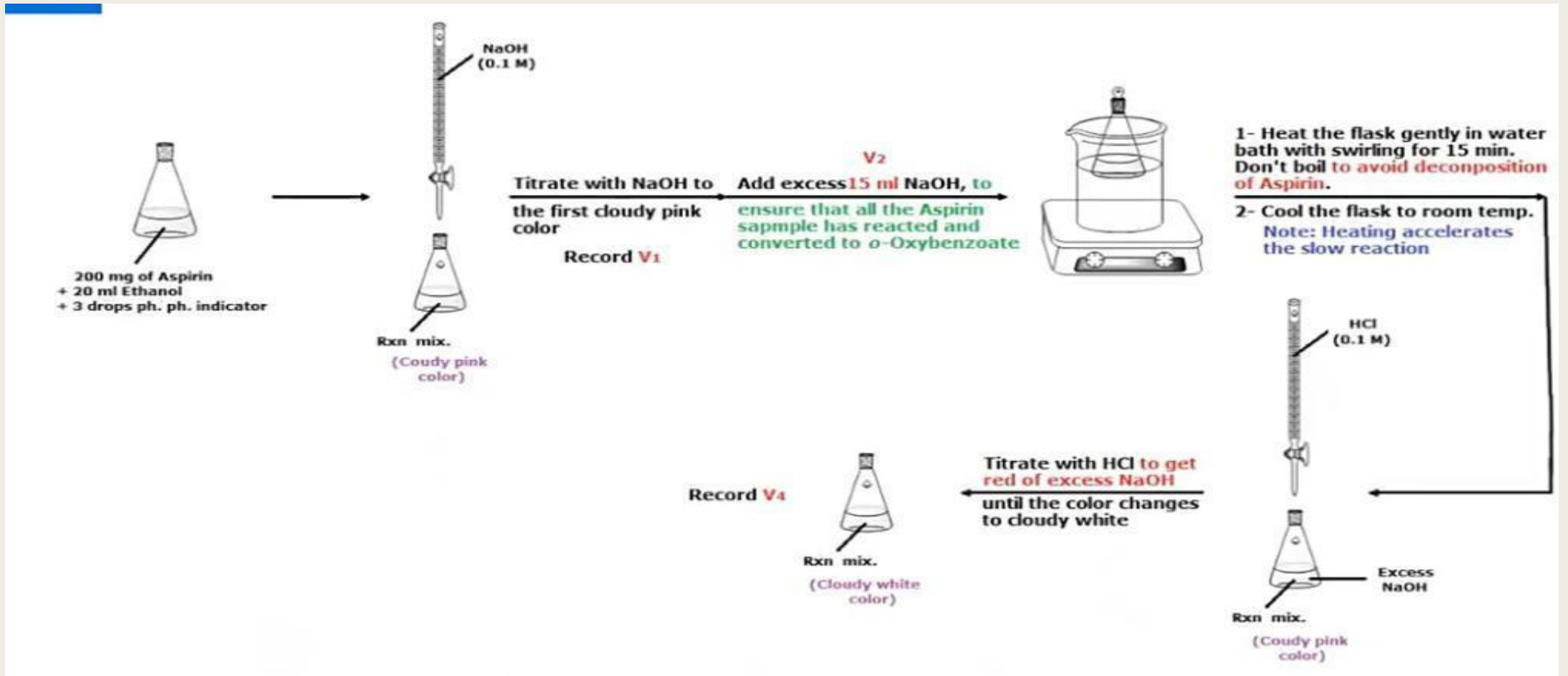
Chemical principle

- The first reaction is titration of weak acid , aspirin,with sodium hydroxide standard solution to produce sodium carboxylate .
- The second reaction is hydrolysis of ester group by adding excess amount of sodium hydroxide to ensure that all aspirin has reacted and converted to o-oxybezoate .
- The hydrolysis solution must be protected from air because CO₂ causes a color change of the indicator before the endpoint is reached.

Procedure

- Weight 0.2g of aspirin powder and dissolve in 20 ml ethanol %50
- Add 3 drop of ph.ph indicator
- Start titration With 0.1N sodium hydroxide until get faint pink color
- Record the volume V1
- Add V2 ,excess amount of NaOH 15ml.
- Heat solution in water bath for 15 min with swirling to accelerate the slow reaction.don't boil !
- Cool the flask at room temp .
- Titrate with HCL until get colourless ,. Record V4

Procedure



Calculation

- Volume of NaOH used in first titration V_1
- Volume of NaOH excess V_2
- Total volume of NaOH added in th flask $v_1+v_2=v_3$
- Volume of HCL titrated with excess NaOH= V_4
- Total volume of NaOH used in the reaction $V_5=v_3-v_4$
- Mole of NaOH used in the reaction = $M'NaOH*v_5$
- Mole of aspirin reacted = mole of NaOH*0.5
- Mass of aspirin used in reaction= mole of aspirin*M.wt
- %assay of aspirin= $m_2/m_1*100\%$