1. Determination of H_2O_2 Purity

Apparatus: Burette

: Erlenmeyer flask

Reagents: Hydrogen Peroxide (H_2O_2)

: Potassium Permanganate ($KMnO_4$)

: 25% Sulfuric Acid (H_2SO_4)

Procedure:

1. Weigh the sample such that there is no more than 0.06 g of H_2O_2 present.
2. Dilute this peroxide sample with 50 ml of distilled water and add 10 ml of 25% H_2SO_4. Take this solution into a conical flask.
3. Take 25 ml from the diluted solution.
4. Now titrate with 0.1N ($KMnO_4$) till pink color appears.

Calculation:

$$\%H_2O_2 \text{ Purity} = \frac{(Volume\ of\ KMnO4)\,X0.1X0.017}{Wt\ of\ sample}\ X\ 100$$

Note: 3.16 g in 1000 ml = 0.1N $KMnO_4$

$2KMnO_4 + 5H_2O_2 + 3H_2SO_4 = 2MnO_4 + K_2SO_4 + 5O_2 + 8H_2O$

2. Determination of Acetic Acid Purity

Apparatus: Burette

: Erlenmeyer flask

Reagents: 0.1 N NaOH [4g NaOH into 1000 ml distilled water]

: Phenolphthalein indicator

Procedure:

1. Weigh 5g of Acetic Acid to be tested
2. Dilute to 50 ml distilled water
3. Pipette 1 ml from the diluted solution into a conical flask
4. Add 2 drops of phenolphthalein indicator
5. Titrate with 0.1N NaOH to a faint pink end point

$$\% CH_3COOH = \frac{(Volume\ of\ titre\ X\ Normality)\ NaOHXMW\ of\ CH3COOH}{Wt\ of\ sample\ X\ 1000}\ X\ 100$$

3. Determination of Soda Ash Purity

Apparatus: Burette

: Erlenmeyer flask

Reagents: 0.1 M HCl [0.1g of the powder into 1000 ml distilled water]

: Methyl Orange Indicator [pH = 3.2 – 4.4]

Procedure:

1. Dissolve 1g of Soda Ash in distilled water in a 100 ml measuring flask
2. Make the volume upto the mark
3. Filled the burette with 0.1M HCl solution with the help of a funnel
4. Note the initial reading of the burette
5. Pipette out 10 ml of soda ash solution and transfer in the titration flask
6. Add 1-2 drops of Methyl Orange as indicator
7. Add 0.1M HCl solution from the burette drop wise with constant shaking until color change from orange to red
8. Note the final reading of burette

Calculation:

% of Na_2CO_3 Purity = 5.3 X Burette reading

DETERMINATION OF CHEMICAL CONCENTRATIONS IN BLEACH LIQUOR

It is important; to keep a constant and accurate check on the various liquors in scouring and bleaching baths and saturators. In continuous processing this will enable operatives to make any necessary modifications to the controls of automatic liquor feeding devices. Analysis of bleach liquors normally involves titration of the peroxide and alkali, usually caustic. Once a system is established the titrations are quickly and easily conducted and can be done in real time next to the machine.

1. Determination of Peroxide Content of Bleach Liquors

This is most frequently determined by titration against standard potassium permanganate solution. The results are usually expressed as ml/l of the commercial strength peroxide being used (the most common strengths are 35% and 50%).

Procedure

Accurately measure with a pipette 2ml of the bleach liquor and add this to 100ml of 10% sulphuric acid contained in a conical flask.

The solution is then titrated with 0.1N Potassium Permanganate solution ($KMn0_4$) to a faint pink colour.

Concentration of peroxide in the bleach bath can then be calculated from the following:

Each 1.0ml of 0.1N $KMnO_4$ used = 2.14ml/l of 35% hydrogen peroxide,

or = 1.43ml/l of 50% hydrogen peroxide.

2. Determination of Alkaline Content of Bleach Baths

Caustic soda is the alkali normally employed in peroxide bleach baths.

Procedure

An aliquot (say 10ml) of the bleach liquor can be titrated with 0.1N sulphuric acid using a suitable indicator. Phenolphthalein is frequently used as it has a very clear end point from pink to colourless.

Typical procedure:-

i. Place 20-25ml of water in a conical flask.
ii. Pipette into this a 10**ml** sample of bleach liquor- (or the solution of caustic under test)
iii. Add a few drops of indicator, if phenolphthalein is used the colour will be pink.
iv. Titrate the solution with the 0.1N Sulphuric acid until the solution becomes colourless. This is the **end point**

Calculation of strength

Take the volume (in ml) of 0.1N sulphuric acid used x 0.4 = Caustic concentration in **g/l** of solid

i.e. End point * 0.4 = **g/l** of Caustic

Example: End point for a titration using 10ml of test liquor is 25 ml of 0.1N Sulphuric acid.

Caustic strength = 25 (end point) * 0.4 = 10 g/l of solid caustic.

If a smaller or larger amount than 10 ml of the test solution is taken then adjust the 0.4 factor proportionately. eg if 5 ml is pipetted then 0.4 becomes 0.8 (you are titrating half the amount so to get the same value you have to double the calculation factor)

Note it is best to get ampules of concentrated solution which are standardised and made to give 0.1N on dilution to a set volume.

However if these are not available it can be done as follows.

Molecular weight of sulphuric acid (H_2SO_4) is :- 98.08 to make a 0.1N sulphuric acid you need 0.05Moles of sulphuric acid which equals 4.904 g of sulphuric acid (100%) however as most concentrated acids are not 100% but rather 95% then adjustments need to be made.

if a smaller or larger amount than 10 ml of the test solution is taken then adjust the 0.4 factor proportionately. eg if 5 ml is used then 0.4 becomes 0.8 (you are titrating half the amount so to get the same value you have to double the calculation factor)

Equipment for titration of bleach liquors.

2 * Auto zeroing Burettes

2* 250 ml Conical Flasks

1* 1000ml (or larger) bottle of 10% Sulphuric acid

Vials to make:-

0.1N Hydrochloric acid solution

0.1 N Potassium Permanganate solution

Bromothymol Blue or Phenolphthalein indicator solution

2 ml, 5ml and 10 ml Pipettes

Equipment for Cold Pad Bleaching in the Laboratory

Pad mangle

1) Compressor to at least 4 bar. Compressed air piping and connectors
2) Water supply. Not essential but preferable to avoid the need for carrying water to the machine for cleaning.
3) Drain pipe work
4) 1.25-1.5 litre plastic jugs
5) Sealable plastic bags
6) Stainless steel tongues for washing
7) Plastic gloves for use during the padding operation

4. Determination of Glauber's Salt Purity

Test Procedure

1. Weigh a dried beaker, **X**
2. Take 10g Glauber Salt in that beaker and dissolve it in 50 ml distilled water
3. Filter this solution to separate the solidified matters
4. This solution is taken in a beaker and evaporates.
5. Finally the weight of the beaker with sediments is taken, **Y**

Purity (%) =(Y-X)/10 *100÷10

Note: Required standard for specific textile processing is as required