

SOLVAY VINYL CHLORIDE MONOMER

ANALYTICAL METHOD - ACETYLENE

This method has been replaced by a similar method being sent to my attention by Solvay.

Reagents:

Solution A: Weigh 20 grams anhydrous cupric sulfate, to this cautiously add 100 ml. NH_4OH with constant stirring. Make to 1 liter with distilled water.

Solution B: Weigh 70 grams of hydroxylamine hydrochloride. Dissolve in distilled water, make to 1 liter.

Note: Presence of iron in cuprous sulfate solution interferes with acetylene precipitation, filter to remove.

Hydrochloric Acid (Concentrated)
1:50 Ammonium Hydroxide
30% Hydrogen Peroxide
Acetic Acid
Potassium Iodide
0.10N Sodium Hyposulfate solution
Starch indicator

Apparatus:

250 ml. gas scrubbing bottle with fritted glass thimble and 25 ml. weighing bottle.

Gooch crucible
250 ml. beaker
50 ml. burette
Balance

Procedure:

Mix 60 ml. Solution A to 60 ml. of Solution B and add solution to gas scrubbing bottle. Allow all vinyl to evaporate. Filter off precipitate onto a Gooch crucible packed with HCl washed asbestos slurry. Rinse scrubbing bottle with 1:50 NH_4OH , filter through same crucible. Now wash acetylene precipitate free of all copper reagent with the ammonical water solution. Remove the precipitate from the Gooch to a 250 ml. beaker and dissolve in 75-100 ml. of concentrated hydrochloric acid. Boil to dissolve the precipitate. Cool and carefully add 5 ml. 30% H_2O_2 . Boil until excess H_2O_2 is removed and evaporate solution down to 25 ml. Neutralize with NH_4OH , and make acid with acetic acid adding an excess of 3 ml. Cool and add 2 grams KI. Titrate solution with 0.10N $\text{Na}_2\text{S}_2\text{O}_3$ using starch as an indicator.

$$\frac{\text{Net ml. of 0.10N Na}_2\text{S}_2\text{O}_3 \times 0.0013 \times 100}{\text{Weight of Sample}} = \% \text{ triple bond as acetylene}$$

CBY 1022843

RSV0030193