

JADAVPUR UNIVERSITY**KOLKATA – 700 032****M. Sc. (CHEMISTRY) 2nd Semester Practical Examination-2018****Full Marks – 50****Time: 11.00 am – 5.00 pm**

- Q. 1. Determine amount of Ni present in Ni-steel in the given sample marked I-XX. 20**
- Q.2. Prepare tris(ethylenediamine)nickel(II) chloride dihydrate as per given procedure. 10**
- Q.3. Viva-Voce and Laboratory Note Book 10**
- Q.4. Sessional 10**

Q.1. Procedure: Transfer the given nickel steel to a 100 mL beaker, dissolve it in minimum volume of conc. HCl (~10 mL) and boil with successive addition of conc. HNO₃ (3 mL) to ensure complete oxidation of the iron to ferric state. Dilute it to somewhat, filter to remove any solid material and wash the filter paper with hot water. Dilute the filtrate to 250 mL in a 500 mL beaker. Add 5 g of tartaric acid and neutralize the solution with 1:1 aqueous ammonia solution. Then the solution was barely acidified (litmus) with dilute HCl, warm to 60-80°C. Add slight excess of a 1% ethanolic solution of dimethylglyoxime (HDMG) (15 mL), immediately followed by the addition of dilute ammonia solution dropwise until the liquid becomes slightly ammoniacal, stir well and allow to stand on the steam bath for 30 min. Allow the solution to stand at least for 1 h and cool to room temperature. Filter off the precipitate through a previously weighed G-4 crucible, test the filtrate with a little HDMG solution for complete precipitation and wash the precipitate with cold water until free from chloride. Dry the precipitate at 100-120°C and weigh as Ni(DMG)₂. Calculate the amount of nickel present in the steel.

Q.2. Procedure

In a 100 mL beaker, 6.0 g of nickel(II) chloride hexahydrate was dissolved in 10 ml of distilled water. The mixture was cooled in an ice bath/cold water and 10 mL of ethylenediamine was added slowly. 15 mL of 95% ethanol was cooled in ice bath/cold water and then slowly added to the solution. The mixture was allowed to settle for complete precipitation of the lavender product. The supernatant becomes nearly colorless. The product was filtered in a Buchner funnel, washed with 5 mL of cold 95% ethanol twice followed by 5 mL of acetone. The product was air dried.

Report yield.