Nickel(II) 1,2-ethanediamine complexes

1. **The preparation of Tris(1,2-ethanediamine)nickel(II) chloride dihydrate**
   6 g (25 mmol) of nickel(II) chloride hexahydrate is dissolved in 20 ml of water. To the solution is gradually added 6,0 ml (5.4 g, 90 mmol) 1,2-ethanediamine while stirring the mixture, and (after filtration, if necessary) 20 ml of ethanol is added. After half an hour without stirring the mixture is cooled in ice-water. The red-violet crystals are separated by filtration and washed two times by 96% ethanol and dried in the air.

2. **The stepwise formation of Nickel(II) 1,2-ethanediamine complexes**
   Complexes of nickel(II) with amines are labile in aqueous solution, i.e. they participate in rapid equilibration reactions. The composition of a solution is therefore dependant on the concentration of amine ligand.

   a) Make up two stock solutions
      i) 1.00 M aqueous nickel(II) chloride solution
      ii) 1.00 M aqueous 1,2-ethanediamine solution
   b) Make four solutions in 25 ml measuring flasks:
      (1) 2.5 ml of 1 M nickel(II) chloride solution. Add up with water to the volume of the flask (the $Ni(H_2O)_6^{2+}$ ion is green)
      (2) 2.5 ml of 1 M nickel(II) chloride solution + 2.5 mmol 1,2-ethanediamine. Add up with water to the volume of the flask (the $Nien(H_2O)_4^{2+}$ ion is bluegreen)
      (3) 2.5 ml of 1 M nickel(II) chloride solution + 5 mmol 1,2-ethanediamine. Add up with water to the volume of the flask (the mixture of cis and trans-$Nien_2(H_2O)_2^{2+}$ ion is blue)
      (4) 2.5 ml of 1 M nickel(II) chloride solution + 7.7 mmol 1,2-ethanediamine. Add up with water to the volume of the flask (the $Nien_3^{2+}$ ion is red violet)
   c) Record an absorption spectrum of each of the 4 solutions (1200 -500 nm)

3. **The empirical formula of the red violet crystals**
   a) Weigh out approximately 0.3 g of the dry synthesis product and determine the mass better than 1 %. Transfer the sample to a 10 ml measuring flask and add enough of the stock solution ii) of aqueous 1,2-ethanediamine to ensure that the solution corresponds to the contents of solution (4) above. Add up with water to the volume of the flask.
   b) Record ithe absorption spectrum.
   c) Calculate the concentration of nickel(II) complex and determine the molar mass of the synthesis product.

JJ 13-11-2015

Report:

1. Yield of dry sample (in g and %)
2. $(\lambda,\varepsilon)_{max}$ of the four spectra
3. Confirm the average environment rule by predicting the position of the absorption maxima for the $Nien(H_2O)_4^{2+}$ and trans-$Nien_2(H_2O)_2^{2+}$ from spectra of solution 1 and 4
4. Use data from PC-exercise to confirm that each of the above four coloured solutions represent only one of the pure $[Nien_n(H_2O)_{6-2n}]^{2+}$ $(0<n<3)$
5. The analysis of the spectrum of the synthesis product, what is the number of water of crystallisation of the red-violet crystals (assuming that it is the pure tris(1,2-ethanediamnine)nickel(II) chloride.

JJ 13-11-2015