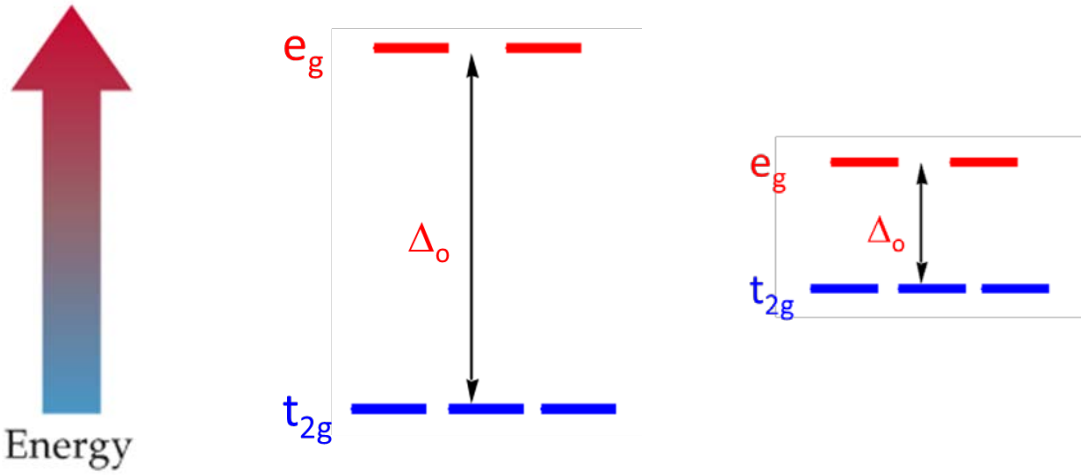
Preparation of $[Ni(NH_3)_6]Cl_2$ and its Analysis by Complexometric Titration

An arrangement of ligands according to their increasing ability to split the d-orbitals is termed as the spectrochemical series. This splitting is quantified using the crystal field splitting parameter (Δ). The parameter is determined experimentally.

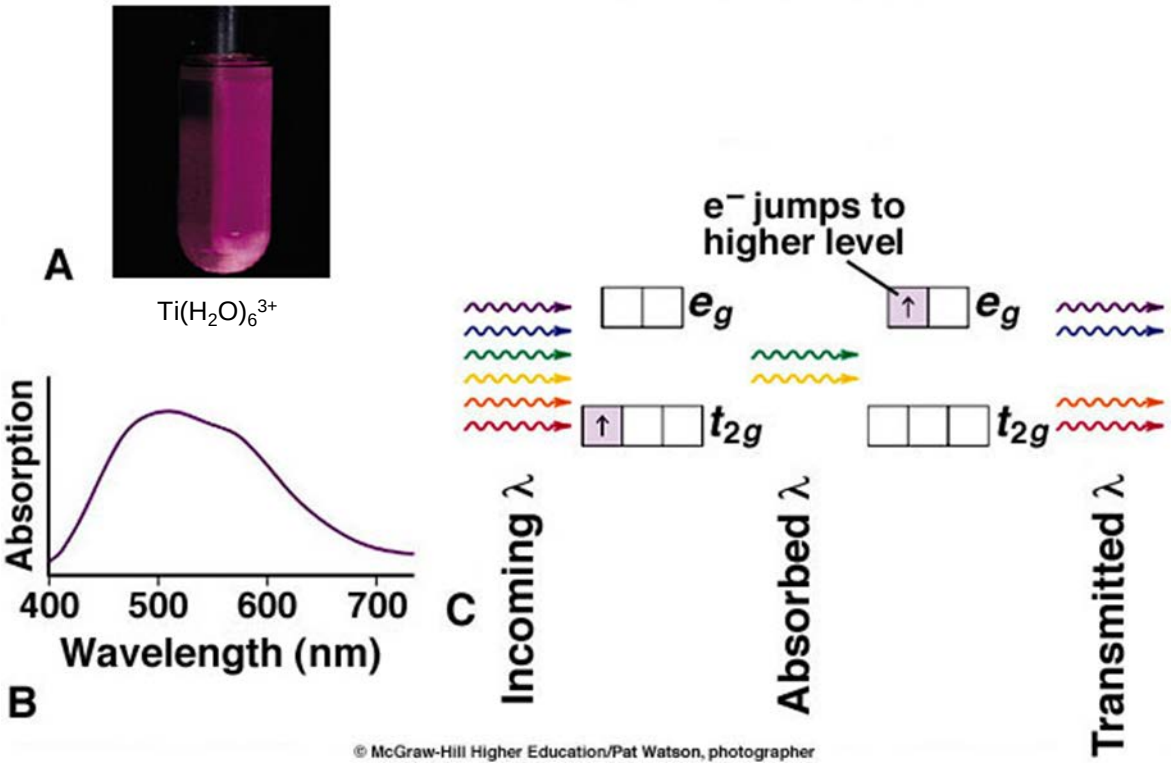
Weak Field $I^- < Br^- < S^{2-} < SCN^- < Cl^- < NO_3^- < F^- < C_2O_4^{2-} < H_2O < NCS^- < CH_3CN < NH_3 < en < bipy < phen < NO_2^- < PPh_3 < CN^- < CO$ **Strong Field**

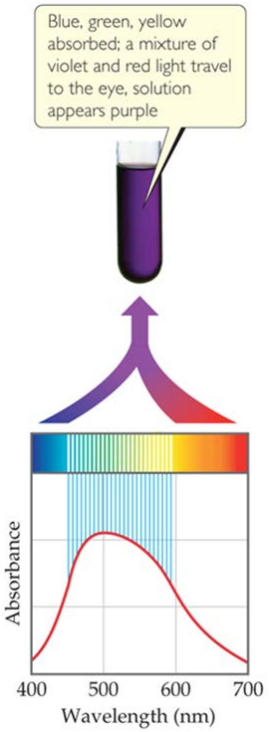
d orbital splitting for an octahedral geometry

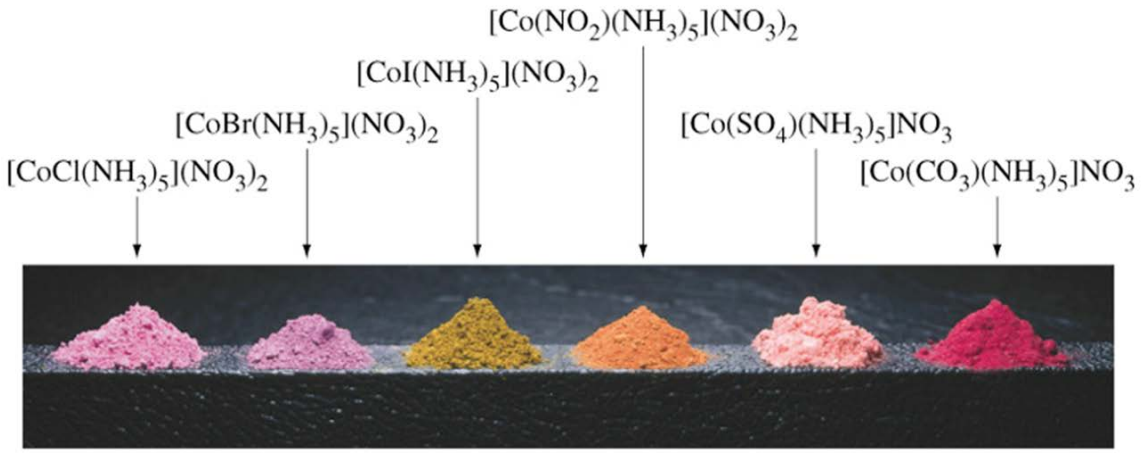


The splitting parameter Δ_o can be related to the colour of a complex

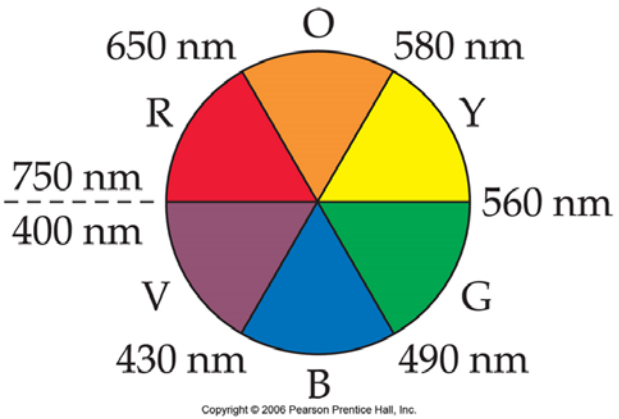
Δ is Relatable to Colour







Complementary Colour Wheel



Theoretical Background

In our experiment, we will synthesize the $[Ni(NH_3)_6]Cl_2$ and estimate the amount of Nickel

$$\underbrace{[Ni(H_2O)_6]^{2+}}_{\text{green}} + 6\ NH_3 \rightarrow \underbrace{[Ni(NH_3)_6]^{2+}}_{\text{blue}} + 6\ H_2O$$

Nickel hexamine complex is an octahedral molecule

$[Ni(NH_3)_6]Cl_2$

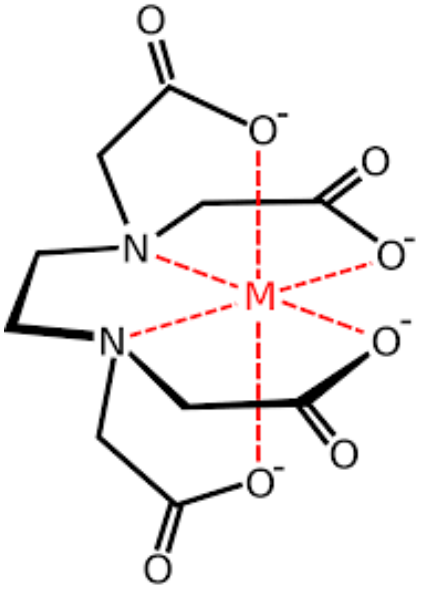
$[Ni(H_2O)_6]^{2+}$ $[Ni(NH_3)_6]^{2+}$

https://www.reddit.com/r/chemistry/comments/8d9c9s/hexaamminenickelii_chloride

Theoretical Background

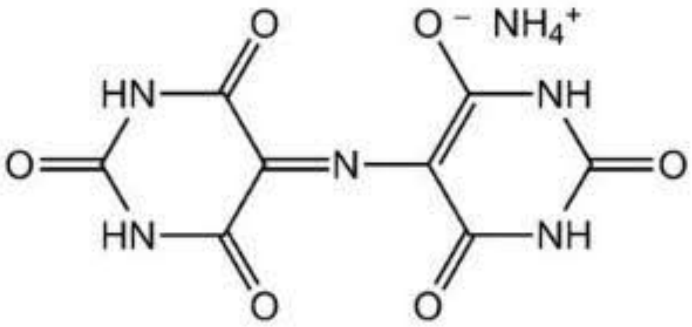
Estimation of Nickel is performed using a complexometric titration with EDTA

$Ni^{2+} + EDTA^{4-} \longrightarrow [Ni(EDTA)]^{2-}$



M = Ni^{2+}

Indicator used is Murexide



pH range : 10 – 11

Colour change for Ni: Yellow-green to violet

https://chem.hbcse.tifr.res.in/

Experimental Protocol

Preparation of Hexamminenickel (II) chloride

1. Take 10 mL solution of nickel chloride hexahydrate (contains 6g of $NiCl_2$) in a 250 mL beaker.
2. Take 12 mL solution of aqueous ammonia in a measuring cylinder.
3. Add the ammonia solution drop wise to the solution of nickel chloride with constant stirring till the colour of the solution has changed from pale green to intense violet.
4. Allow the solution to stand at room temperature for 5 minutes, cover with watch glass. Then cool it in an ice bath for about 15 minutes.
5. Filter the solution and wash the crystals with 3-5 mL ammonia solution.
6. Dry the crystals using filter paper.
7. Report the weight of the dried complex.

Estimation of nickel(II) by EDTA

1. Take 80 mL of 0.05M EDTA solution in a 250/500 ml plastic beaker and fill it in a clean burette up to the mark.
2. Weigh accurately 1.15 g of $[Ni(NH_3)_6]Cl_2$ complex and transfer this to a 100 mL volumetric flask. Now add 50 mL of 1 N H_2SO_4 to dissolve it and makeup the solution to the mark with distilled water.
3. Pipette out 10 mL of the complex solution in a 250 mL conical flask and dilute it with 15 mL of distilled water.
4. Add 2-3 drops of murexide indicator and 5 mL NH_4Cl solution (0.5M) to the conical flask. Now add ammonia solution (7-10 drops) to maintain a pH 7 (light green color of the solution).
5. Titrate it with EDTA solution till the endpoint is near, add 3 ml of ammonia solution and continue the titration till the endpoint (bluish violet color appears).
6. Repeat the titration and get concordant values.
7. Calculate the amount of Ni present in the complex.

Estimation of nickel by spectrophotometery

1. Several solutions (of $NiCl_2$) of known concentration and one solution of unknown concentration will be provided to you.
2. Measure absorbance of all the solutions at 395 nm using a UV-Visible spectrophotometer.
3. Plot absorbance versus mg/mL of nickel. Determine the concentration of nickel present in the unknown solution in g/L.

Observations and Calculations

Given Concentration of EDTA solution = 0.05 M

S. No.	Volume of EDTA used from the burette
1	9.4 mL
2	9.4 mL
3	9.5 mL

1 mole Ni reacts with 1 mole of EDTA to form of the Ni-EDTA complex.

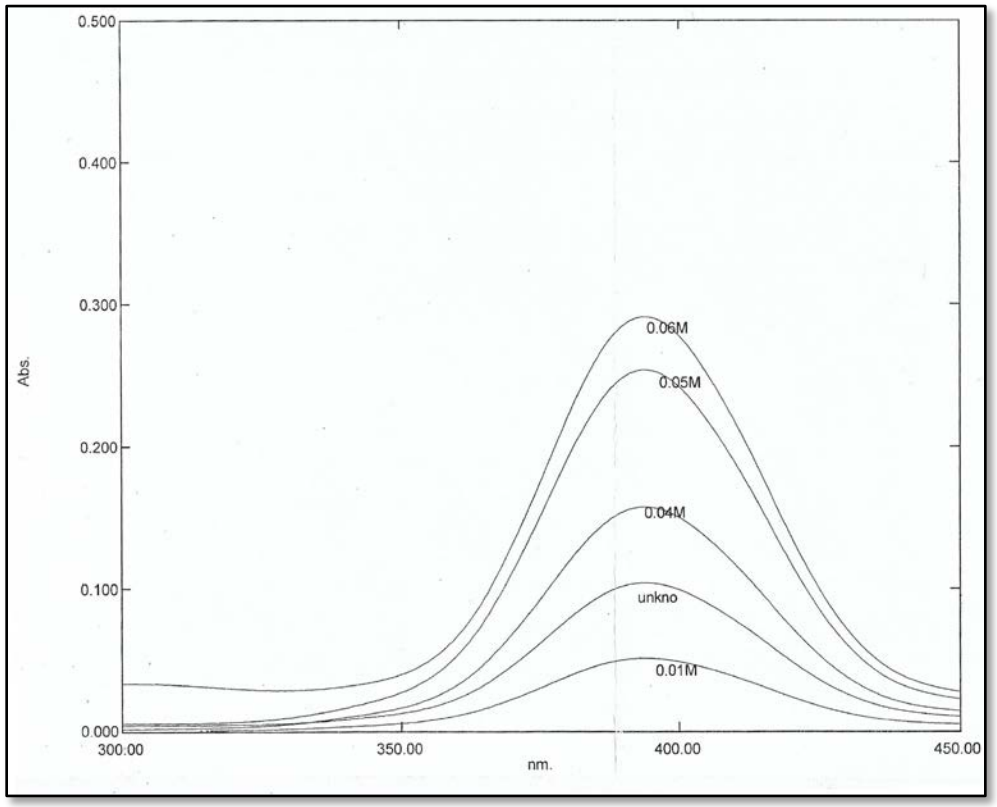
Moles of Ni = Moles of EDTA
(Molarity x Volume) of Ni = (Molarity x Volume) of EDTA

Molarity of Ni x 10 mL = 0.05 M x Volume of EDTA (Burette Reading)
Suppose Burette Reading is 9.4 mL
Molarity of Ni = (0.05 x 9.4)/10 = 0.047 M
Thus, the solution contains 0.047 moles/Liter of Nickel
Grams/Liter of Ni = 0.047 x 58.69 (Atomic Weight of Ni) = 2.76 g/L

Hence, in 100 mL of solution 0.276 g of Ni is present.
We started with 1.15 g of $[Ni(NH_3)_6]Cl_2$, thus 0.24 g of Ni is present per gram of $[Ni(NH_3)_6]Cl_2$

Observations and Calculations

Absorbance *vs* Concentration lot of given Nickel solutions

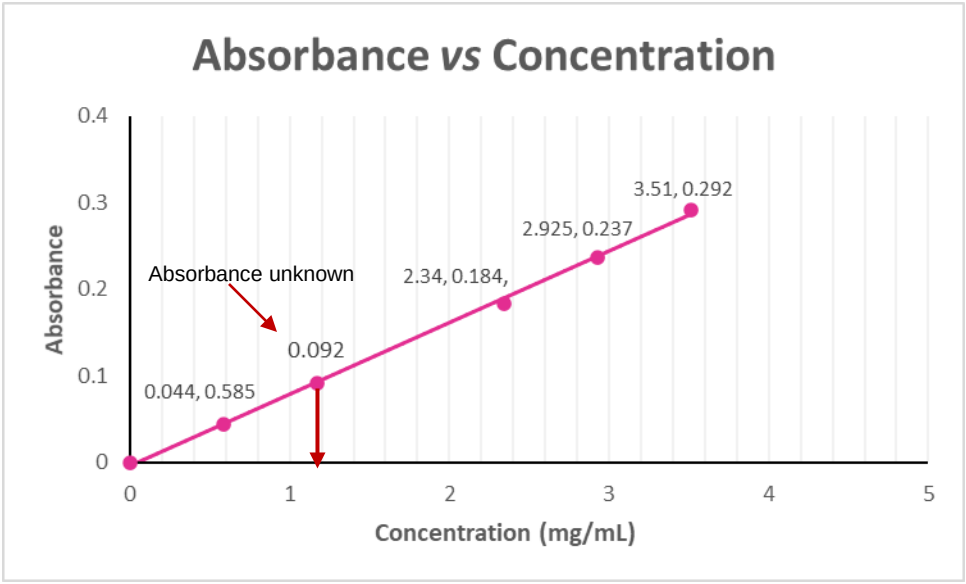


An overlay of absorbance plots for Nickel solutions with concentrations; 0.01M, 0.04M, 0.05M, 0.06M and an unknown solution
Absorbance measured at 395 nm

Observations and Calculations

UV-Visible Spectroscopy

S. No.	Concentration of Solution (M)	Concentration of Solution (mg/mL)	Absorption at 395 nm
1	0.01 M	0.585	0.044
2	0.04 M	2.34	0.143
3	0.05 M	2.925	0.237
4	Unknown	Unknown	0.092
5	0.06 M	3.51	0.292



The absorption of light is described by the Beer-Lambert law

$A = \varepsilon \, . \, c \, . \, l$

where A is the absorbance, ε is the molar absorption coefficient, c is the concentration and l is the path length

Using the absorbance vs concentration plot, concentration for the unknown solution can be inferred

Concentration for an absorbance of 0.092 corresponds to the value of 1.17 on the X axis

Concentration of unknown solution = 1.17 mg/mL = 0.02 M

Results

- $[Ni(NH_3)_6]Cl_2$ was synthesized from $Ni(H_2O)_6Cl_2$
- The obtained compound was then used to estimate the amount of Nickel present
- Amount of Nickel present : 0.24 g of Nickel present per gram of $[Ni(NH_3)_6]Cl_2$
- Concentration of a given solution was determined using solutions of known concentration with the help of UV-Visible spectroscopy
- Concentration of unknown solution = 0.02 M