Determination of molecular formula of a complex by spectrophotometry

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Abstract

Spectrophotometry is a very commonly used method in chemistry to study the progress of reactions, or the position of equilibrium. It is easy to make spectrophotometric measurements, and they are reasonably accurate (over the range that Beer's law is valid). In this experiment, I study the complex formed by salicylic acid and ferric ions, using Job's Method.

1 Introduction

The method I will be using here to determine the molecular formula of the ferric salicylate complex is *Job's method*, which is a method of continuous variation to find out the formula. Job's method is really effective only when a single complex is formed.

Job's method is dependent on the validity of the Beer's Law over the region of study. Beer's Law gives us the relation between absorbance (A) of a solution and intensity of the light (I):

$$A = \log I_0/I$$

where I_0 is the intensity of the incident light, and I is the intensity of the transmitted light. Another relation, from Beer's Law relating absorbance to concentration of the solution is:

$$A = \varepsilon c l$$

where ε is the molar extinction coefficient and l is the optical path length.

Armed with these formulae, we find the spectra of the solutions of the two species, recorded over a range of concentrations, each solution containing the same total reagent concentration. So, in my experiment [Salicylic acid] + [Fe^{3+}] = constant. By seeing the spectra, we pick a wavelength, at which there is good absorbance and the absorbance of each solution is noted.

As the concentration of one of the reactants, say salicylic acid goes up, so does the concentration of the product (which in my case, is purplish in colour), causing an absorbance rise. I would get a maxima at the point where the reactants have been mixed according to the ratio in their molecular formula. After that, the absorbance goes downhill again as Fe^{3+} present is insufficient to react with the salicylic acid, causing drop in complex concentration.

So if we plot the absorbance vs composition of solution, we will get the molecular formula by checking the maxima.

2 Experiment

2.1 What I needed

A Spectrophotmeter, Ferric Nitrate, Salicylic Acid, Glass beakers (100 mL), Round bottomed flasks (3).

2.2 Procedure

First, a solution of 0.0025M sulphuric acid was made. To do this I prepared standard 0.25M oxalic acid solution. NaOH was standardised against it and this standardised NaOH was used to standardise the sulphuric acid which after appropriate dilution was made to be 0.0025M strength.

A solution of 0.0025M Fe^{3+} was made by dissolving the appropriate amounts of ferric nitrate in 0.0025M sulphuric acid solution. Similarly a solution of salicylic acid was made.

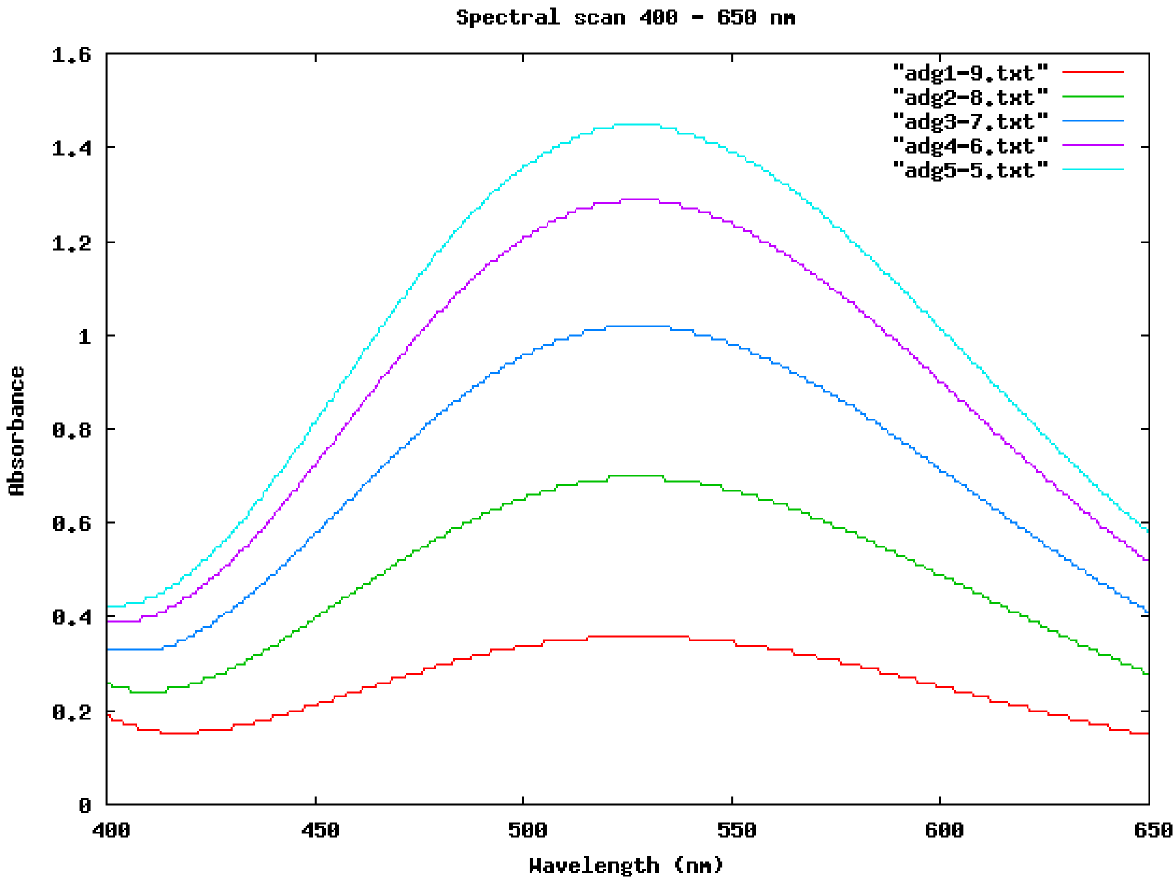
We check the spectra of the stock solutions of ferric nitrate and salicylic acid between 400nm to 650nm. The reference spectrum is 0.0025M sulphuric acid since it is the solvent.

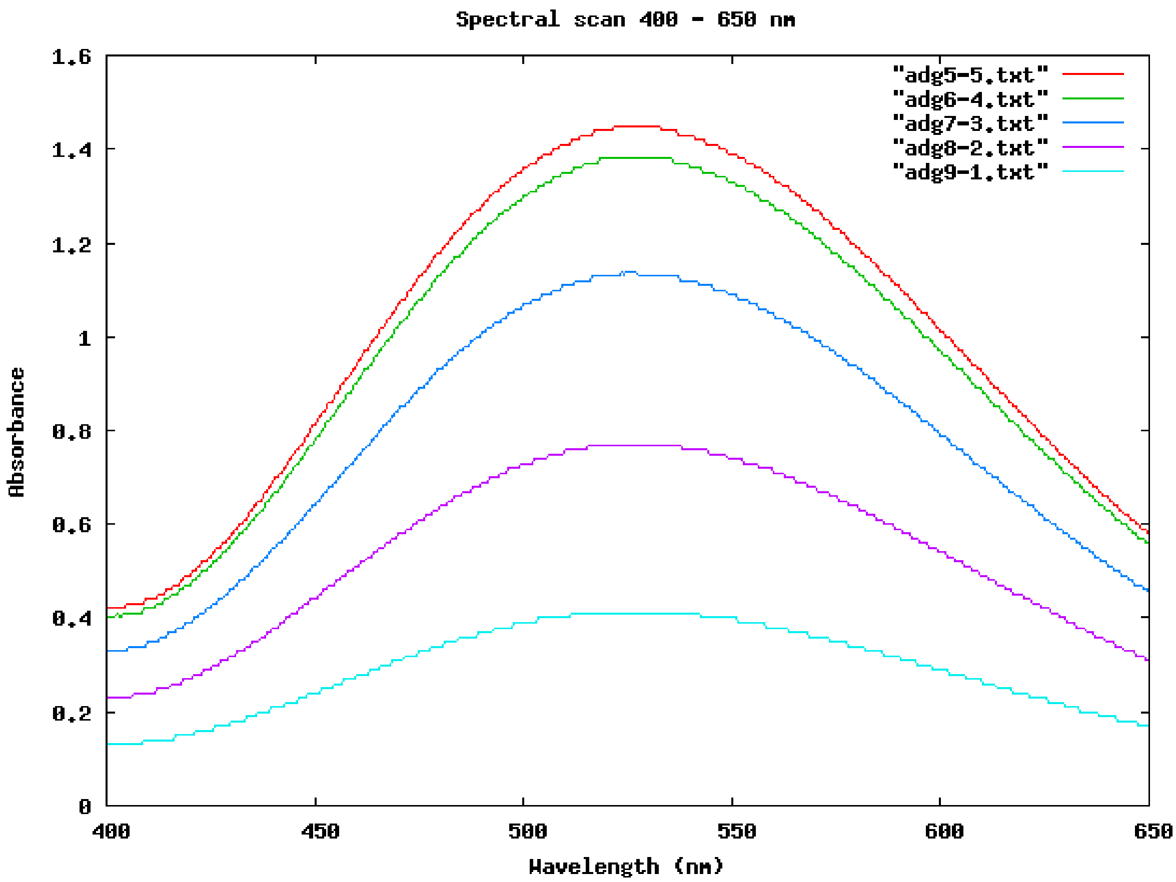
The spectra of 1:9, 2:8, 3:7, ..., 9:1 mixtures of Fe^{3+} and salicylic acid are recorded over same wavelength range. A particular wavelength is chosen for which there is strong absorbance, and the absorbances at this wavelength are plotted against the composition. The maxima gives me the molecular formula

2.3 Observations

The spectra of the stock solutions of Ferric nitrate and salicylic acid were recorded and no appreciable change was found from zero level.

However on mixing the two stock solutions, we immediately get a nice purple colour, so we know that we will get measurable absorbance in the visible region. These are the spectrums I got:

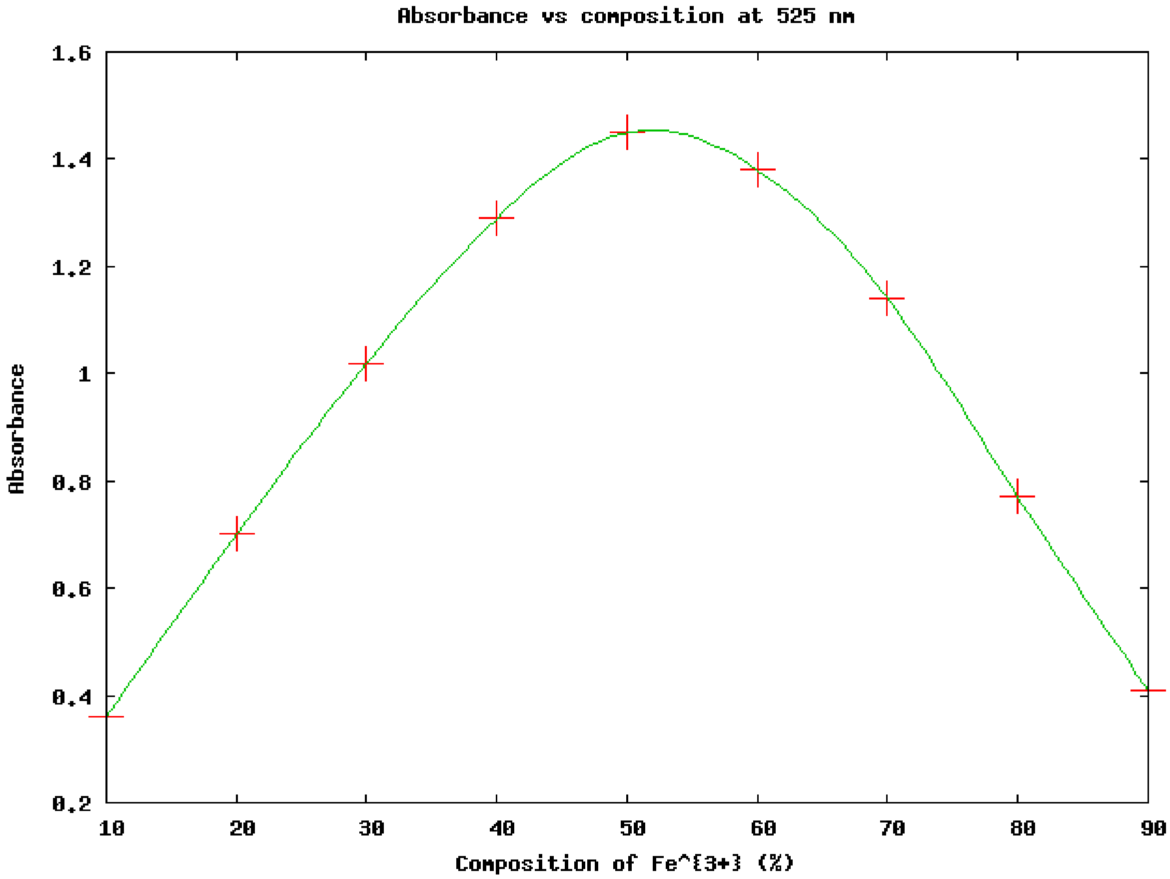




The first graph is for the concentrations 1:9 to 5:5, the second one is from 5:5 to 9:1; as expected they are similar.

As most of the peak absorbances occur around 525 nm, I take that wavelength and tabulate the corresponding absorbances:

Composition Fe^{3+}: Salicylic acid	**Absorbance**
1:9	0.36
2:8	0.70
3:7	1.02
4:6	1.29
5:5	1.45
6:4	1.38
7:3	1.14
8:2	0.77
9:1	0.41



By drawing the graph, we can clearly see that in the molecule of the complex, Fe^{3+} and Salicylic acid are in the ratio 1:1.

3 Conclusion

Thus from this experiment, I conclude that in the complex formed by ferric nitrate and salicylic acid, the reacting species are in the ratio 1:1. So we have determined the molecular formula of the complex using Job's method.

The precautions that we need to take while doing this experiment are that we need to ensure that the solutions are standardised properly, and that we are taking the spectral data over the range where Beer's Law is valid (otherwise absorbance will not have a linear relationship to the concentration).

4 Acknowledgements

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5 References

- Physical and Theoretical Chemistry Laboratory, University of Oxford
  http://ptcl.chem.ox.ac.uk/