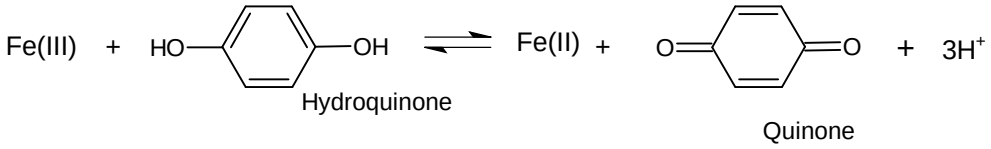
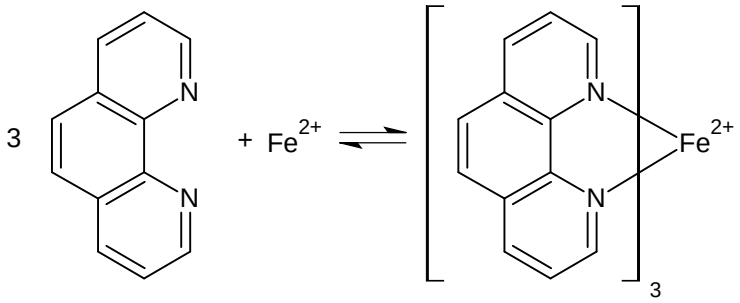
Spectrophotometric Determination of Iron in Vitamin Tablets

In this procedure, iron from a vitamin supplement tablet is dissolved in acid, reduced to Fe^{2+} with hydroquinone, and complexed with *o*-phenanthroline to form an intensely colored complex.



Hydroquinone

Quinone



o-phenanthroline

Reagents:

Hydroquinone: Freshly prepared solution containing 10g/L in water; stored in an amber bottle.

Trisodium citrate: 25g/L in water.

o-Phenanthroline: 2.5 g of *o-Phenanthroline* in 100mL of ethanol and diluted with 900ml of water; stored in an amber bottle.

Standard Fe Stock Solution: Dissolve ~0.3g (weighed accurately) of the reagent-grade $Fe(NH_4)_2(SO_4)_26H_2O$ (FW =392.13 g/mol) in water in a 1-L volumetric flask containing 1 mL of 98 wt% H_2SO_4. Calculate the concentration of Fe in the standard solution in mg Fe/mL.

Procedure:

1. Place on tablet of the iron-containing vitamin in a 100-mL beaker and boil gently (*in a fume hood)* with 25 mL of 4M HCl for 15min. Filter the solution directly into a 100-mL volumetric flask. Wash the beaker and filter several times with small portions of water to complete a quantitative transfer. Allow the solution to cool, dilute to the mark and mix well. Dilute 5.00mL of this solution to 100mL in a fresh volumetric flask.

2. Pipette 10.00mL of standard Fe stock solution into a beaker and measure the pH (with pH paper or a glass electrode). Add sodium citrate solution 1 drop at a time until a pH of ~3.5 is reached. Count the drops needed. (It will require about ~60 - ~70 drops.)

3. Pipette a fresh 10.00-mL aliquot of Fe standard into a 100-mL volumetric flask and add the same number of drops of citrate solution as required in Step 2. Add 2.00 mL of hydroquinone solution and 3.00mL of *o*-phenanthroline solution, dilute to the mark with water, and mix well. Use this first standard solution to generate the calibration plot with three other standards: as instructed below.

4. Prepare three more standard solutions as before from 7.00, 5.00, 2.00, and 1.00mL of Fe standard and prepare a blank containing no Fe. Use sodium citrate solution in proportion to volume of Fe solution. (if 10mL of Fe requires ~70 drops (may vary) of citrate solution, 5mL of Fe requires 35 drops of citrate solution)

5. Find out how many drops of citrate solution are needed to bring 10.00mL of the iron supplement tablet solution from Step 1 to pH 3.5

6. Transfer 10.00mL of solution from Step 1 to a 100-mL volumetric flask. Add the required amount of citrate solution determined in Step 5. Then add 2.00mL of hydroquinone solution and 3.0mL of *o*-phenanthroline solution; dilute to the mark and mix well. This is the prepared laboratory sample, "test solution".

7. Allow the solutions to stand for at least 10 min. Then measure the absorbance of each solution at the λ_{max}.and the absorbance of the "test solution" in triplicate (The color is stable, so all solutions may be prepared and all the absorbances measured at once.) Use a blank in the reference cuvette.

8. Prepare a blank.

9. Calculate the molarity of the colored ion each standard solution. (Remember that all the iron has been converted to the o-phenantrholine complex.)

10. Generate the UV spectra of the solutions made above from 350nm – 600nm. Measure the absorbances at the $\lambda_{max.}$ (unknown - in triplicate).

11. Generate the calibration curve and the least-squares parameters and associated uncertainties. Fit the data to the best fit straight line, leave out outliers. Calculate the molarity and the uncertainty in molarity of the "test solution" from step 6 using the "best fit line" information.

Estimate the Fe content in the ***tablet*** (and the uncertainty) in mg.

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Daniel C. Harris