

Experiment 10: Direct Titration of Lead with EDTA: UV-Vis detection

Synopsis Direct titration of lead by EDTA is accomplished at a low pH using UV-Vis detection of the Pb-EDTA chelate.

READINGS Read pages 279-285 in Critical Reviews.

Reagents 0.01 M HClO₄
0.2 mg PbCl₂ in 100 mL 0.01 M HClO₄
5x10⁻⁴ M EDTA, standard. Dilute from 0.100 M EDTA
37.22 g of disodium EDTA in 1000 mL deionest water.
Or

Dry the acid for two hours at 130-150C. Cool. Weigh 29.210 g of acid EDTA, add to 600 mL water, add pellet by pellet NaOH, until the EDTA comes into solution. Dilute to 1L.

Instrument UV-Vis spectrometer.

Procedure

1. Scan the 5x10⁻⁴ M EDTA solution from 210 to 300 nm.
2. Scan the lead/HClO₄ solution from 210 to 300 nm.
3. Add 2 mL of EDTA to 100 mL of the Pb/HClO₄ solution
4. Scan this solution of lead/EDTA from 210-300 nm.
5. Based on the three scans choose a wavelength to monitor. You should find that 235 nm is an appropriate wavelength where Pb/EDTA maximizes and Pb/HClO₄ and pure EDTA minimizes.
6. At the wavelength selected in step 5 monitor the change in absorbance as mL of EDTA standard are titrated into 100 mL of Pb/HClO₄ solution. Be sure to add more than 3 mL in order to observe the endpoint.
7. Repeat step 6 two times in order to be able to obtain an rsd on the measurement.

REPORT In addition to material, methods and results, include:

1. What is the rsd of this method?
2. What will determine the minimum amount of lead that can be measured in this method?
3. What constitutes a blank in this procedure? What are the sources of error embodied in the standard deviation of the blank?
4. How does sample matrix affect your results?
6. What was the estimated time for turn around in samples?
7. Are there any problems with disposal of hazardous materials?
8. How easy would it be to instruct a technician on this method?
9. How easy would it be to construct a paper trail for this method?