Experiment 17: IR Determination of Pb binding to EDTA

SYNOPSIS The binding of Pb to EDTA will be studied using the vibrational changes in EDTA when Pb is added. An FTIR instrument is used.

READINGS 279-285 on EDTA and 319-325 on vibrational spectroscopy in Critical Reviews

Supplies

KBr Slides
Nujol Oil
Mortar and Pestle

Glassware

One 30 mL beaker

CAUTIONS AND PROBLEMS

This method is quite difficult because it relies on accurately bringing a solution chemistry down to a dry state. Lead EDTA has very different conformations and structures in the wet and dry states. Sample preparation must be performed with extreme care.

Solutions

These should be made up before this lab.

Combine 1.000 g (0.00342 moles) of Na-EDTA and 1.133 g (0.00342 moles) of $Pb(NO_3)_2$ in 10 mL of water and adjust the pH with 0.6 M NaOH to a pH 9-10 value under continuous stirring. Discontinue stirring as soon as there is no solid material left and dry the sample at 80 ^oC for more than 3 days.

Sample Preparation

Weigh out five 10 mg EDTA samples and to each of them add 0, 0.25, 0.5, 0.75, and 1 stoichiometric molar amounts of EDTA-Pb complex. Grind these separately to a fine paste on an Agate mortar about 15 minutes. Add 1 to 2 drops mineral oil and grind again. Use a rubber policeman to transfer the paste/oil to KBr plate . Press between two plates to create a thin film that is almost translucent.

Experiment

4. Take a spectrum of Nujol by itself. Export the data.

For our FT instrument the data will be exported into a text file. To get it into a form you can manipulate in excel you will need to import the data and delineate it into an Excel format.

A. Go to Data/Get External Data/Import Text File

B. In the Import Text File command box indicate that the data is delimited, and enter

C. On the next command box indicate that the data is delimited by commas and hit enter

D. You should be able to simply hit finish, finish and find your data imported into Excel in a readable format

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2. Obtain a spectrum of pure EDTA in Nujol. Compare the peaks that you observe to those listed in Table 27, p321 in Critical reviews.
3. Run each of the lead samples. Pay particular attention to the region between 1500 and 1700 cm^{-1}.
4. Overlay the different mole ratio spectrum.

REPORT: Report, in addition to materials, methods and results.

1. Why is the lead EDTA complex formed at pH 7? Give two separate reasons.
2. Why does the sample have to be ground to less than 2 Fm particle size?
5. Identify, with Table 27, page 321 in Critical Reviews, the major absorption bands of EDTA.
4. Can you observe the Pb-N band? Why or Why not? What would you need to do to see this band?
5. Prepare a table which quantitates the decrease in the C=O band and the increase in the C=O-Pb band. Is the decrease quantitative? What is the LOD?
6. Quantitate the effect of the increasing of co-added spectrums on the S/N of the spectrum. Plot the S/N as a function of square root of the co-added spectrum. Is the plot consistent with what you expect?
7. How does the k value for Pb-O compare to Pb-S?
8. Discuss the use of EDTA has a chelation therapy for lead. What are some of the pitfalls? How would **you** design a chelate to overcome those pitfalls?