Experiment 6: Electrolytic Deposition of PbO_2 for Gravimetric Analysis

Synopsis Lead is measured by anodic deposition of PbO_2 to a Pt electrode and then gravimetrically weighed. This method can be modified for a quartz microbalance measurement.

READINGS Pages 269-273 in Critical Reviews

Materials and Supplies

6 V storage battery, regulated.
250 mL tall-form beakers covered with watch glasses.
Pt. Gauze electrodes (cathode (2 in diameter by 2.25 inces in height
Anode, 1 inch in diamter by 2.25 inches in height
stirrer
hot plate (gives more dense depostions)

Several samples of lead from 0.1 to 0.3 grams of lead total.

Instructions

1. To sample solution add 22.5 mL of concentrated nitric acid to give a solution 15% vol/vol of nitric on dilution to 150 ml
2. Add 0.3 g of copper by addition of solution of Cu nitrate.
3. Dilute sample to 150 ml with water
4. Bring beaker with sample solution beneath electrodes and cover at least 2/3 of electodes
5. Turn on hot plate and stirrer to temp of 95C
6. Pass 2 amp currents
7. Periodicially wash down walls to move lead from surface and to deposition
8 After 15minutes with no new depositon on newly submerged surface terminate
9. Leave current on, and lower beaker and rinse off electrodes from water with wash bottle
10. Remove anode, dip in alcohol then ether, and dry in a beaker in an oven at 220Cfor 1 hour. If > 0.3 grams anticipated dry for longer.
11. Cool and weight the electrode and calculate the amount of lead deposited

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

1. Calculate the theoretical gravimetric factor for this method.
2. How would a change in the current density affect your results.
3. How would the presence of Sn affect your results?

4. Why does chloride have to be absent in the method?
5. What is the point of the acidic solution?
6. What is the point of the hot plate?
7. What is the point of rinsing with alcohol and ether?
8. What was the percent recovery of the lead?
9. What constitutes a blank in this procedure? What are the sources of error embodied in the standard deviation of the blank?
10. What was the estimated time for turn around in samples?
11. Are there any problems with disposal of hazardous materials?
12. How easy would it be to instruct a technician on this method?
13. How easy would it be to construct a paper trail for this method?