Experiment 4: Gravimetric Determination of Lead Chromate

SYNOPSIS   Lead is measured by precipitation with chromate, dried, and weighed.

READINGS: Pages 269-273 in Critical Reviews

SOLUTIONS

Concentrated NaOH
pH meter
0.1% Nitric Acid (Conc. HNO_3 = 76%, so about 1 mL conc./100 mL water)
0.10 M chromium nitrate, $Cr(NO_3)_3·9H_2O$ (4 g/100 mL)
0.12 M potassium bromate, $KBrO_3$ (2 g/100 mL)
Acetate buffer solution: 6 M in acetic acid, 0.6 M in sodium acetate.

GLASSWARE

2 erlenmeyer flasks
2 porous porcelain filter crucibles
  suction adapters

PROCEDURE

1.   To eache flask add 4 mL of 1000 ppm Pb. Bring to 20 mL vol.. To the fifth flask add 20 mL of one of your soil sample digests. To one of the first four (lead standard) flasks add 4 mL of 1000 ppm Zn.
2.   If necessary, neutralize 20mL solution with NaOH to pH 7 (use indicator paper) in a 100 mL beaker. Solution will be slightly cloudy. *What is the chemistry that makes it cloudy?*
3.   Adjust volume of sample to about 20 mL.
4.   Add 10 mL $Cr(NO_3)_3·9H_2O$ solution and 10 mL of $KBrO_3$. Solution will be clear blue.
5.   Heat but do not boil for 30 minutes.
6.   When solution is clear and yellow (= measure of extent of chromic oxidation), add 10 mL of buffer and heat 5 more minutes.
7.   Weigh your 6 filter crucibles.
8.   Cool the mixture and filter off the lead chromate on a sintered glass or porous porcelain filter crucible.
9.   Wash the precipitate with 2 or 3 small portions of 0.1% nitric acid.
10.   Dry at 120C for 30 minutes.
11.   Cool and weigh as $PbCrO_4$ (Pb/$PbCrO_4$ = 0.641108).

REPORT

1.   What is the chemistry of all of the reactions involved in forming lead dichromate? Write out

balanced reactions.

2. What is the chemistry that makes it cloudy?
3. What is the relationship between the solubility of $PbCrO_4$ and it's use as a yellow pigment for traffic paints?
4. What is the purpose of the added $KBrO_3$?
5. What is the relative standard deviation of your 3 ppm sample?
6. Does the addition of Zn to the standard affect your analysis?
7. What is the absolute error of your 3 ppm sample? What might cause the error?
8. How easy would it be to explain this method to a technician or a 5th grader?