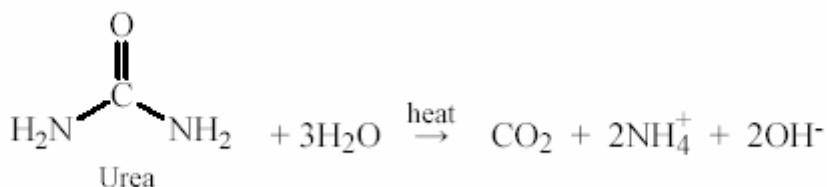


Laboratory Experiment 2.

Gravimetric Determination of Calcium as $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$.

Calcium ion can be analyzed by precipitation with oxalate in basic solution to form $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$.

The precipitate is soluble in acidic solution because the oxalate anion is a weak base. Large, easily filtered, relatively pure crystals of product will be obtained if the precipitation is carried out slowly. This can be done by dissolving Ca^{2+} and $\text{C}_2\text{O}_4^{2-}$ in acidic solution and gradually raising the pH by thermal decomposition of urea (Reaction 27-2 in the textbook).



REAGENTS

Ammonium oxalate solution: Make 1 L of solution containing 40 g of $(\text{NH}_4)_2\text{C}_2\text{O}_4$ plus 25 mL of 12 M HCl. Each student will need ~50 mL of this solution.

PROCEDURE

1. Dry a medium-porosity, sintered-glass funnel (Gooch crucible) for 1–2 h at 105° C; cool it in a desiccator for 30 min and weigh it. Repeat the procedure with 30-min heating periods until successive weighings agree to within 0.3 mg. Use a paper towel or tongs, not your fingers, to handle the funnels.
2. Weigh 1/3 gram sample into 200 mL beaker. Wash down with 5 mL water; add slowly, drop by drop, 20 mL of 5% hydrochloric acid. After the end of the intense reaction, heat gently and boil for 5-10 min. **Filter off silica into 200 mL beaker through #2 paper, wash 5 times with hot water (this is necessary only if there is a visible non-soluble residue)!** Dilute filtrate to 100 mL. Add 5 drops of methyl red indicator solution (Table 12-4 in the textbook) to each beaker. This indicator is red below pH 4.8 and yellow above pH 6.0. ***You can stop here and continue on Thursday!***
3. Add slowly ~20 mL of ammonium oxalate solution to the beaker while stirring with a glass rod. Remove the rod and rinse it into the beaker. Add ~12 g of solid urea to each sample, cover it with a watchglass, and boil gently for ~30 min until the indicator turns yellow.
4. Filter a hot solution through a weighed funnel, using suction (Figure 2-15 in the textbook). Add ~3 mL of ice-cold 1% ammonium oxalate solution to the beaker, and use a rubber policeman to

help transfer the remaining solid to the funnel. Repeat this procedure with small portions of this solution until all of the precipitate has been transferred. Finally, use two 10-mL portions of ice-cold water to rinse each beaker, and pour the washings over the precipitate.

5. Dry the precipitate with aspirator suction for 1 min.

Stop, you can leave it till the Thursday class.

6. Then dry it in an oven at 105° C for 1-2 h.

7. Bring a filter to constant mass. The product is somewhat hygroscopic, so only one filter at a time should be removed from the desiccator, and weighings should be done rapidly. The water of crystallization is not lost.

8. Calculate the weight percent of Ca in the unknown solid.

9. Using data from other students's reports for the same unknown, calculate the average, the standard deviation, and relative standard deviation ($s / x = \text{standard deviation/average}$).