Gravimetric Determination of Calcium Ion as Calcium Oxalate[1]

TSUD Department of Chemistry

Calcium ion forms insoluble calcium oxalate when reacted with oxalate ion ($C_2O_4^{2-}$) in basic solution:

$$Ca^{2+} + C_2O_4^{2-} + H_2O = CaC_2O_4 \cdot H_2O$$

If the calcium and oxalate ions are dissolved in mildly acidic solution, the oxalate is present as hydrogen oxalate ($HC_2O_4^{1-}$) and no precipitate forms. If the solution is made very slowly basic, large, clean, easy to filter crystals of $CaC_2O_4 \cdot H_2O$ precipitate. In this experiment, we will determine the calcium content of an aqueous solution by mixing the calcium solution with ammonium oxalate solution in acid and very slowly raise the pH by thermal decomposition of urea.[2]

Procedure

Dry three sintered glass funnels in the oven at 110 °C to constant mass.

Pipette replicate 25.00mL aliquots of the unknown calcium solution into 250- or 400 mL beakers. Dilute each sample with ~75 mL of 0.1 M HCl and add 5 drops of methyl red indicator. (Methyl red is red below pH ~5 and yellow above pH ~6). Add ~25 mL of 0.33 M $(NH_4)_2C_2O_4$ solution which is

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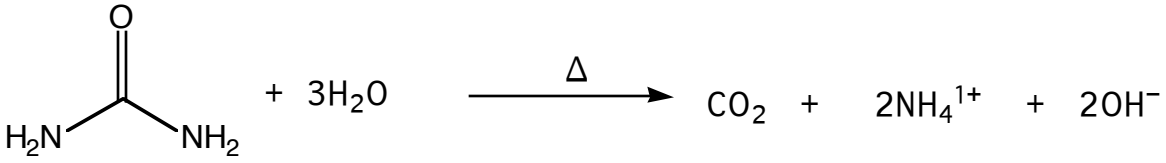
[1] adapted from D. C. Harris, Exploring Chemical Analysis, 2e, Freeman, 2001, pp 498-499.

[2]



0.33 M in HCl to each sample with stirring (glass rod). Add 15 g of solid urea to each sample, cover the beakers with a watch glass, and boil very gently for about 30 minutes until the solution turns yellow.

Filter the hot solutions through the glass funnels with suction. Use 3 mL portions of ice cold water to quantitatively transfer all of the solid to the funnel. A rubber policeman may prove helpful. Dry the funnels containing the sample in an oven at 105 ^{o}C for at least one hour and then transfer them to a desiccator to cool. Once the funnels are cool, remove them one at a time from the desiccator (the product is hygroscopic) and weigh.

Calculate the molarity of calcium ion in the unknown solution along with the average and standard deviation for the three samples. Prepare a complete laboratory report (in the proper format) for the experiment.