Synthesis of Galactaric Acid (Experiment XIX-B)

Background Reading

Small Scale Syntheses, pp 453 and 455.
McMurry, J., Organic Chemistry, 8e 1008-9 or 7e 973-5 (Classifications of Sugars), 8e 1011-4 or 7e 981-3 (Configurations), 8e 1020-1 or 7e 992-4 (Oxidation).

Keywords

Aldose, Hexose, Anomer. Pyranose, Aldonic Acid, and Aldaric Acid

Compound, Reaction, and Yield Data

- Provide Fischer projections of reactant and product in your overall reaction.
- No reaction mechanism is necessary for your report.
- Note that the product decomposes at 230^{o}C.
  So, it has a decomposition range, not an actual melting range.
- For the yield calculations, the molarity of concentrated HNO_3 is 15.8 M,
  and 2 moles of HNO_3 are needed for each mole of galactose.
  Compare both reactants to determine which is limiting.

Procedure

1. Perform first three steps in a hood.
   Add 6.0 ml of DI water to a 30-ml beaker and clamp beaker to a stand.
   Place beaker in an ice-water bath using a larger beaker for the bath.
   Then, carefully add 6.0 ml of concentrated $HNO_{3(aq)}$ drop-wise and slowly.

   Caution – Concentrated HNO_3 solutions are corrosive and cause acid burns.
   Use gloves and avoid all contact with skin and eyes.

2. Add 3.0 g of galactose to a 50-ml Erlenmeyer flask.
   Then, slowly add the nitric acid solution to the flask while stirring.

3. Clamp the flask to a stand and heat gently over a steam bath using a metal pan.
   Continue heating until no more brown fumes are visible.

   Caution – Gaseous oxides of nitrogen (brown in color) are evolved by the reaction.
   These fumes are toxic and corrosive. The reaction is also exothermic.
   Perform this step in a hood.

4. Once reaction is complete, remove flask from heat and allow flask to cool.
   A white precipitate (galactaric acid) will form while cooling.
   After precipitation is complete, add crushed ice to the flask
   so that the final volume is between 30 and 40 ml.

5. Allow mixture to stand for 10 minutes. Place 30 ml of DI water in an ice-water bath.
   Weigh a piece of 55-mm filter paper, and then collect the product by vacuum filtration.

6. Wash product with three 10 ml portions of the cooled DI water.
   Place product on a watch glass and dry in oven at 100 ^{o}C for 10 minutes.

7. Weigh product. Determine decomposition point of product with a Mel-Temp.

Post-Lab Questions

1. Concentrated nitric acid is commonly used to oxidize organic compounds.
   What happens to nitric acid that causes other substances to oxidize?
   Show the balanced reduction half-cell reaction (gain of e^{-1}'s) between
   one NO_3^{-1} ion and two H_3O^{+1} ions that creates $NO_{2(g)}$ and H_2O.

2. How does the ice added in step 5 affect the oxidation reactions and the
   extent of crystallization? Explain in terms of reaction rates and solubility.

3. What effect do the water washes have in step 6?
   Explain in terms of the solubility of substances involved in the experiment.

4. What is the difference between an aldose and a ketose?
   Provide examples using Fischer projections using Section 25.1 of McMurry.

5. Galactose forms two cyclic hemiacetals called pyranose rings.
   They are diastereomers that are described as anomers of each other.
   The two molecules are similar to the glucopyranoses formed from glucose.
   Use Figures 25.3 and 25.4 of McMurry to draw and name the two structures.