ISOPROPYL NITRITE (IPN) SYNTHESIS

DAY 1 – PREPARATION

APPARATUS

1 x 500mL graduated cylinder
1 x 100mL graduated cylinder
2 x 50mL graduated cylinder
1 x 1L beaker
2 x 125mL Erlenmeyer flask (one with stopper)
2 x glass stirring rods

CHEMICALS

distilled water
sodium nitrite, 97%
2-propanol
sulfuric acid, concentrated
sodium bicarbonate
sodium chloride

PROCEDURE

Solution #1: In the 1L beaker, dissolve 95g of sodium nitrite in 375mL of distilled water. Cover the beaker completely with Parafilm.

Solution #2: In the 125mL Erlenmeyer flask, combine 18mL of distilled water and 83mL of 2-propanol. Measure 34mL of sulfuric acid into a graduated cylinder and add to the alcohol solution by slowly pouring the acid down a glass stirring rod and into the flask. Add only a few milliliters at a time, keeping boiling to a minimum. Stopper the flask and seal it Parafilm.

Place both solutions in a freezer, and freeze overnight.

Wash: In the other 125mL Erlenmeyer flask, dissolve 8g of sodium chloride and 0.8g of sodium bicarbonate in 40mL of distilled water. Seal the flask with Parafilm and refrigerate overnight.

DAY 2 – SYNTHESIS

APPARATUS

1 x retort stand, tall/wide-bottomed
1 x retort stand, short
1 x small ring clamp
1 x plastic basin
2 x 50mL beaker
1 x electric mixer
2 x small funnel
1 x 125mL separatory funnel (with glass fittings)
1 x 500mL separatory funnel (with stopper)
1 x 500mL Erlenmeyer flask
1 x 125mL Erlenmeyer flask
1 x filter paper, 4”
1 x 50mL volumetric flask (with stopper)
1 x glass stirring rod

CHEMICALS

ice
table salt
sodium sulfate, anhydrous

PROCEDURE

1. Place the 500mL separatory funnel, 125mL Erlenmeyer flask, and 50mL volumetric flask in the refrigerator to cool while synthesizing IPN.

2. In the fumehood, set up the larger retort stand at the back, and place the plastic basin at the base. To the side of the basin, set up the short retort stand and attach the ring clamp.

3. Prepare an ice-salt bath in the basin, using a generous amount of table salt over each layer of ice. Place the beaker with Solution #1 in the ice bath (do this on top of about two layers of ice and salt, then continue layering around the beaker).

4. Securely clamp the electric mixer to the large retort stand, so that it is in the beaker and about halfway into the solution. Attach the glass fittings to the spout of the 125mL separatory funnel and secure with Parafilm. Place the separatory funnel in the ring clamp and lower it into the solution so that the opening is below the fluid line.

5. Turn on the electric mixer, and set between speeds 1 and 2. Using a small funnel, pour Solution #2 into the 125mL separatory funnel, and open the stopcock almost completely. The solution should be dripping out at a constant rate over a 10-15 minute period.
   As Solution #2 is being added, a milky, yellow slurry will begin to form and a reddish-brown gas (NO_2) will be observed, indicating that the product is decomposing. This is normal due to the quick addition, and thus quick rise in temperature.

6. As Solution #2 finishes draining from the separatory funnel, remove from the refrigerator the wash prepared on Day 1 as well as the glassware from Step 1 and place everything in the fumehood. Set the 500mL separatory, stopcock closed, in the 500mL Erlenmeyer flask.

7. When the addition of Solution #2 is complete, remove the 125mL separatory funnel. As someone manually stirs the solution, remove the electric mixer.

8. Pour the solution into the 500mL separatory funnel, and allow most of the greenish-yellow layer (IPN) to come to the top. Drain most of the bottom into the Erlenmeyer flask.

9. Wash 2-3 times with the solution prepared on Day 1. Gently swirl the separatory funnel, invert, and vent by opening the stopcock and allowing the solution to de-gas. After each wash, drain the bottom aqueous layer into the Erlenmeyer flask.

10. Collect the IPN in the cold 125mL Erlenmeyer flask and dry with anhydrous sodium sulfate. The amount of drying agent is dependent on the product yield, but generally, the solution is dry when there is excess sodium sulfate floating in the solution.

11. Using a small funnel lined with filter paper, filter the dried IPN into the cold 50mL volumetric flask. Stopper, seal with Parafilm, cover completely with aluminum foil, label (eg. IPN 0/25/06), and place in the refrigerator.

NOTES

→ From set up to IPN collection, the procedure takes approximately one hour, and clean up takes approximately 30-45 minutes. Thus, this can been executed up to 3 times in one day, if multiple batches of Solutions #1 and 2 are prepared on Day 1.
→ The typical yield for this method is 40-50mL of IPN.
→ The procedure for Day 2 should be completely carefully but quickly, to minimize the decomposing effects of light and heat.
→ The above method may be improved by experimenting with different factors such as:
  → height and speed of the electric mixer
  → period of time over which the solutions are combined
  → manual vs. electric stirring
  → brightness of the lab or surrounding area of the fumehood