POLYMERS AND PLASTICS

TECHNIQUES REQUIRED : Reflux apparatus, extraction, vacuum filtration, yield calculation

EXPERIMENTAL PROCEDURE Work in pairs - **hand in individual reports**

A PETE DEPOLYMERISATION

- **1-Pentanol has an unpleasant vapour, make sure the reaction flask is cold before removing the condenser after the reflux is complete.**
- **Potassium hydroxide can cause skin burns. Avoid skin contact.**
- **Work in a fumehood whenever possible.**
- **Pay attention to the position of power cords near hot plates.**
- **Hot stirrers / metal blocks and hot glassware ! Avoid burns!**

We expect that you will provide your own PETE plastic from a clean, soft drink bottle. The bottle you use should be marked as "PETE" and / or with the logo shown to the right and NOT marked as "LDPE" or "HDPE". Ideally it should be precut into pieces about 0.5 cm square or smaller

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NOTE: In the equipment set up shown in reflux technique document, a heating mantle with a heating controller was shown being used to heat the round bottom flask. In your equipment set up, the heating mantle and heating controller will be replaced by an engineered (shaped) aluminium block sitting centered on the top of a stirrer hot plate. This is a more modern version of a heating mantle with some distinct benefits.

In the fumehood, set up a hot plate stirrer with a metal heating block (check the fit size for your 100 mL round bottomed flask). Make sure the power cords are well away from the heated surface. Don't turn the power on yet.

Put 3.0g of preweighed PETE from a clean, soft drink bottle cut into pieces 0.5cm square or smaller into a 100ml round bottomed flask containing 1-pentanol (25ml), solid KOH (2.65g) and a magnetic stirrer bar. Secure the round bottom flask in the metal heating block using a clamp to the fumehood racking (don't over tighten the clamp on the glassneck of the flask, but make sure it secures the flask in an upright position). Make sure that the flask is centered over the middle of the stirrer hotplate in the metal heating block. Attach a condenser equipped for reflux (see reflux technique document) with secured water hoses to the tap and the sink. Get your TA to check the set up before turning on the heat or the water.

Start the **gentle flow** of water to the condenser, start stirring and then heat the mixture to boiling (heat setting 200-250), monitor the heat process regularly and adjust the heat control as required. Once the

solution is boiling, continue the reflux for 1 hr. During the reflux monitor the stirring. If a thick white suspension forms that prevents stirring then add more solvent (pentanol). After 1 hr, stop heating and remove the heating mantle, but **don't remove** the reflux condenser and cool the reaction mixture to room temperature using an ice bath.

Caution: Only when the reaction flask is cold should you remove the condenser.

Add 25ml of cold water to the flask and dissolve the white solid that is present. Remove and weigh any unreacted plastic using a vacuum filtration, remove the stirrer bar and then transfer the filtrate to a separatory funnel. Remove the lower aqueous layer and put it to one-side (DO NOT THROW IT AWAY!). Extract the pentanol layer with water (25ml), remove the aqueous layer and combine the two aqueous extracts. **Slowly**, acidify the combined aqueous extracts with dilute HCl (add in 1 ml portions from a pipette), swirl the mixture and check the acidity using blue litmus paper after each ml (it needs to turn red). Collect the white crystals by vacuum filtration and while they are still under suction, wash them with a **small volume** of **cold** acetone (*i.e.* prechill the acetone by standing it in an ice bath) and allow the crystals to dry (keep the suction running to help draw air past the crystals and periodically stir / churn them up with a spatula). Once they are dry, weigh the product. Calculate the yield of product based on the mass of plastic that had reacted.

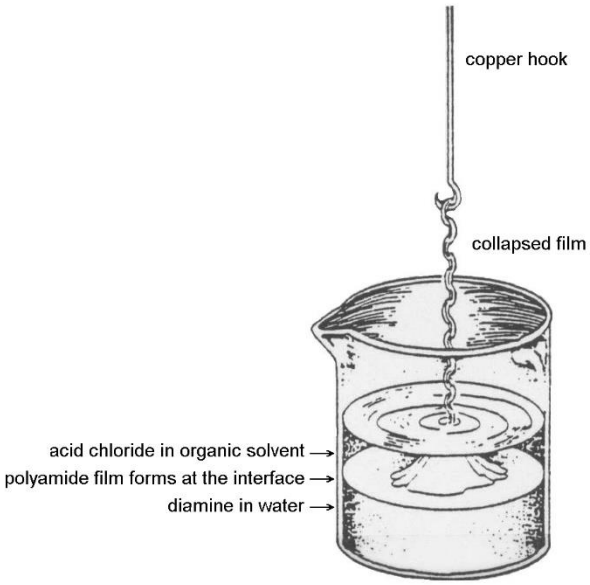
CLEAN UP

- Aqueous waste should be poured into the large white aqueous waste container.
- All other residues should be placed in the red drum for organic waste in the fumehood.
- Remember that you heated the aluminium heating block for an hour = it might be hot!

B. NYLON Your TA will set-up and demonstrate the following procedure.

- **1,6-hexanediamine is corrosive, causes burns and is harmful by inhalation and skin contact. Wear gloves and use in a fumehood.**
- **Adipoyl chloride is corrosive, causes burns, reacts violently with water and is a lachrymator. Wear gloves and use in a fumehood.**

Pour 10 mL of a 5% aqueous solution of hexamethylenediamine (1,6-hexanediamine) into a 50 mL beaker. Add 10 drops of 20% sodium hydroxide solution. Carefully add 10 mL of a 5% solution of adipoyl chloride in cyclohexane to the solution by pouring it down the wall of the slightly tilted beaker. Two layers will form (see figure), and there will be an immediate formation of a polymer film at the liquid-liquid interface. Using a copper-wire hook (a 6-in. piece of wire bent at one end), gently free the walls of the beaker from polymer strings. Then hook the mass at the center, and slowly raise the wire so that polyamide forms continuously, producing a rope that can be drawn out for many feet. The strand can be broken by pulling it faster. Rinse the rope several times with water and lay it on a paper towel to dry. With the piece of wire, vigorously stir the remainder of the two-phase system to form additional polymer. Decant the liquid and wash the polymer thoroughly with water. Allow the polymer to dry. Do not discard the nylon in the sink. Use a waste container.



REFERENCES

1. UofC “Organic Chemistry etext”, Ch 20 “Carboxylic Acid Derivatives. Nucleophilic Acyl Substitution” https://www.chem.ucalgary.ca/courses/351/Carey5th/Ch20/ch20-0.html
2. Kaufman, D. et. al. "New" Compounds from Old Plastics: Recycling PET Plastics via Depolymerisation , Journal of Chemical Education, 1525, 76 (1999)