AUSTRALIAN SCHOOL SCIENCE INFORMATION SUPPORT FOR TEACHERS AND TECHNICIANS

Published on *ASSIST* (https://assist.asta.edu.au)

Making Nylon

Posted by Anonymous on Mon, 2015-02-16 15:35

Making Nylon: It has now been 14 years since I was in a high school classroom, but in those ‘good old days’ we could do an experiment to make nylon 6,6. I am writing some tasks at the moment but don’t want to include this in my tasks if OH&S regulations now means this can no longer be done by students. So, can you let me know if it is still an experiment that is acceptable. The experiment involves decanedioyl dichloride in cyclohexane being floated on an aqueous solution of 1,6-diaminohexane.

Voting:
No votes yet

Year Level:
Senior Secondary
Laboratory Technicians:
Laboratory Technicians

Showing 1-1 of 1 Responses

Making Nylon

Submitted by sat on 17 February 2015

Answer Reviewed 19 February 2023

The preparation of nylon is a classic reaction, which has been demonstrated to audiences since the late 1950s.[1,2] The reaction is a reliable one and, although the ingredients are hazardous, the product can be handled safely after washing it to remove any reagents.

Science ASSIST recommends that this reaction be carried out as a **demonstration only,** due to the corrosive and toxic nature of the reagents. The reaction should only be performed in a running fume cupboard and PPE (lab coat, gloves, safety glasses and closed shoes) should be worn throughout the procedure.

There may be circumstances where it is safe for senior students to carry out the reaction themselves, such as where the class is small and can be relied on to follow safe practices, and where there is sufficient fume cupboard space. The risks can be further minimised by providing the reagents as pre-prepared solutions, and by reducing the scale of the reaction (e.g., 5–10mL of each solution per group). Science ASSIST recommends using a nonhalogenated solvent, such as cyclohexane, to dissolve the adipoyl or sebacoyl chloride if the reaction is conducted as a student activity. A site-specific risk assessment will determine if it is safe to do this reaction as a class activity or only as a demonstration.

Safety and Handling Considerations

The reaction uses an aqueous solution of hexamethylenediamine (1,6-diaminohexane) and a solution of either adipoyl chloride (hexanedioyl chloride) or sebacoyl chloride (decanedioyl chloride) in an organic solvent. The reaction using adipoyl chloride gives nylon 6,6 as the product, while nylon 6,10 is the product from the reaction with sebacoyl chloride.

While the *J. Chem. Educ.* Article[1] from 1959 describing 'The nylon rope trick' recommended using a carbon tetrachloride solution of sebacoyl chloride, more recent procedures use less hazardous solvents such as dichloromethane,[3] hexane,[3] cyclohexane[4] or heptane[4,5] as the organic solvent.

Both adipoyl chloride and sebacoyl chloride are corrosive and can cause severe skin burns and eye damage. They hydrolyse on exposure to moisture in the air to produce hydrochloric acid and should therefore be protected from moisture during storage. Sebacoyl chloride is toxic in contact with skin. Bottles of these substances should be checked periodically, and if decomposition is observed, then the bottle should be disposed of via a licenced disposal company. It is not recommended to store solutions of these substances in organic solvent as this may hasten their decomposition.[1]

Hexamethylenediamine is corrosive and can cause severe skin burns, eye damage and respiratory irritation. It is readily biodegradable and poses a low bioaccumulation risk. Hexamethylenediamine is hygroscopic and should be protected from moisture during storage. The diamine is a solid at room temperature (m.p. ~40°C) but is generally melted when required for use. To melt the solid, the bottle is placed in a plastic bag in a warm water bath (temperature < 80°C to avoid the generation of irritating fumes). Hexamethylenediamine is stable in aqueous solution and can be stored thus.[1]

Dichloromethane is categorised as a Category 2 carcinogen (suspected of causing cancer). It can cause skin irritation and serious eye damage. Dichloromethane residues should be stored as halogenated organic waste and disposed of via a licenced disposal company. Science ASSIST has developed a List of Recommended Chemicals for schools, and dichloromethane has been accessed as being safe for use in Australian schools.

The nonhalogenated organic solvents hexane, heptane and cyclohexane are highly flammable, can cause skin irritation and are toxic to aquatic life. Waste hydrocarbon solutions should be stored as nonhalogenated waste and disposed of via a licenced disposal company.

The nylon product should be washed with 50% ethanol, followed by water to remove any residual salts or solvent.

Any unused solutions should be stirred together until no further polymerisation occurs. If an excess of the diamine has been used, then a dilute solution of adipoyl chloride (or sebacoyl chloride) should be added in small increments to the stirred mixture until no further polymerisation is observed. The nylon produced can be washed of any reagents and disposed of in the general rubbish. The layers can then be separated using a pipette (small volumes) or a separating funnel (larger volumes). The aqueous layer should be neutralised and washed down the sink and the organic layer stored as halogenated organic waste in the case of dichloromethane or otherwise as nonhalogenated organic waste.

Notes on variations of the procedure

If dichloromethane is used as the solvent for the diacyl chloride (adipoyl chloride or sebacoyl chloride), then the organic layer is the lower layer, and the nylon rope is drawn through the aqueous solution. Using a nonhalogenated solvent, with the diacyl chloride solution as the top layer, is reported to lead to a product with a 'sticky quality'.[1] However, the advantage of using a less toxic solvent would compensate for this drawback.

While some procedures use a 1:1 mixture of the diamine and diacyl chloride,[3, 4, 5] most of the procedures cited use the diamine in a 2–3-fold excess of the number of moles of the diacyl chloride. According to Bieber,[6] an excess of the diamine ensures that when the polymer has a terminating acyl chloride group, the reaction with the diamine will be fast, thus reducing the length of time the growing polymer is vulnerable to hydrolysis which would lead to chain termination. For the same reason, it is also recommended that care be taken to ensure that the diacyl chloride solution is well-mixed in order to avoid a high concentration of this reagent at the interface.[6] [Science ASSIST has not had an opportunity to test this particular variation, but using a hydroxide solution containing one equivalent of the diamine at twice the concentration of the diacyl chloride, if successful, would meet the above requirements and also minimise the amount of unreacted diamine at the end of the reaction.]

In the procedures cited, which include bases such as sodium carbonate[1, 5] or sodium hydroxide,[1, 3, 5] the base is used in a 2.5–4-fold excess of the number of moles of the diacyl chloride. An excess of base allows the hydrogen chloride by-product to be consumed quickly, with the diamine then free to participate in the reaction rather than act as a base.[6]

References

1 Morgan, P.W., & Kwolek, S.L. (1959) *J. Chem. Educ*., **36**, 182

2 Morgan, P.W.(1965). *J. Chem. Educ*., 42, 12

3 Elfick, J (Editor). (2022, July) *School Science Lessons, Topic 9*, 3.4.7 Prepare nylon polymer, School of Education. Retrieved from University of Queensland website: https://johnelfick.github.io/school-science-lessons/

4 Tranthim-Fryer, D. (2014, September) Personal communication. Senior Chemist & Research Officer, ChemCentre, Curtin University, Bentley W.A.

5 Helmenstine, A.M. (2020). *How to make nylon - Nylon Synthesis.* Retrieved from ThoughtCo. website: https://www.thoughtco.com/how-to-make-nylon-608926

6 Bieber, T.I., (1979). *J Chem. Educ*., **56**, 409