BS-196

Experiment 1: Calibration of Volumetric Glassware

In this experiment, four types of glassware are used by an analytical chemist: volumetric flask, volumetric pipette, a measuring pipette and a measuring cylinder will be calibrated to avoid introducing systematic errors into measurements. To reduce the random errors when using these instruments, their proper use must be thoroughly understood. The quality of the measurements obtained from these tools depends heavily on the care taken in calibrating and in using each instrument.

Tips for Correct Use of Volumetric Glassware Pipettes

1. The pipette is used to transfer a volume of solution from one container to another. Most volumetric pipettes are calibrated To-Deliver (TD) with a certain amount of the liquid remaining in the tip and as a film along the inner barrel after delivery of the liquid. The liquid in the tip should not be blown out.
2. To fill the pipette, insert it vertically in the liquid, with the tip near the bottom of the container.
3. Apply suction to draw the liquid above the graduation mark. Quickly place a forefinger over the end of the stem. Withdraw the pipette from the liquid and use dry paper to wipe off the stem.
4. Now place the tip of the pipette in the container from which the liquid has been withdrawn and drain the excess liquid such that the meniscus is at the graduation mark.
5. Move the pipette to the receiving container and allow the liquid to flow out (avoid splashing) of the pipette freely.
6. When most of the liquid has drained from the pipette, touch the tip to the wall of the container until the flow stops and for an additional count of ten.

A General Calibration Procedure for Glassware

1. As was noted above, volumetric glassware is calibrated by measuring the mass of water that is contained in or delivered by the device.
2. This mass data is then converted to volume data using the tabulated density of water (See table below) at the temperature of calibration.
3. Finally, this volume data is corrected to the standard temperature of 25 °C room temperature. In this experiment a volumetric flask, a measuring pipette and a volumetric pipette will be calibrated using water. In each case, the measured mass of the calibrating water will be standardized to 20 °C or the room temperature.

The density of water at different temperatures is given below.

Temperature (°C)	Density (g/mL)
15	0.999 102 6
16	0.998 946 0
17	0.998 777 9
18	0.998 598 6
19	0.998 408 2
20	0.998 207 1
21	0.997 995 5
22	0.997 773 5
23	0.997 541 5
24	0.997 299 5
25	0.997 047 9
26	0.996 786 7
27	0.996 516 2
28	0.996 236 5
29	0.995 947 8
30	0.995 650 2

1. All the measurements needed to be taken thrice for the pipette, volumetric flask, and burette and measure the weight of the liquid.
2. Note the difference between the actual weight and the calculated one and estimate the error up to 3 decimal places.
3. Each group will be given a test tube of unknown volume, which they must determine with error bar.

All the results should be given in the tabulated form.

Prepare Salt Solution for the following:

1. Prepare **100mM** of **100ml** NaCl Solution in a volumetric flask.
   Calculation:

2. Prepare **10%** of **100ml** NaCl Solution in a volumetric flask.
   Calculation:

3. Prepare **200ppm** of **100ml** NaCl Solution in a volumetric flask.
   Calculation:

Experiment 1.2: Synthesis of a commercial polymer, Nylon 6,6

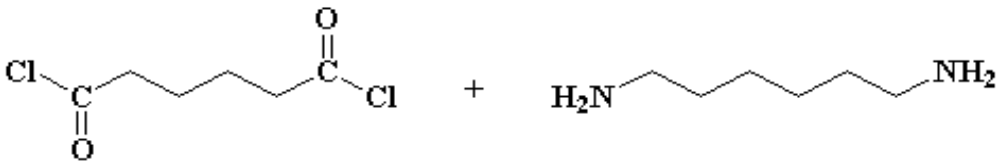
Introduction

The two most important kinds of nylon are nylon 6,6 and nylon 6. These two nylons have almost identical properties. Both were invented in the late 1930s. Nylon 6,6 was discovered first. It was invented in the United States by Wallace Carothers who was working for DuPont. Nylon 6,6 is a polyamide based polymeric material. It is commonly used in textile and plastic industries. Nylon 6,6 is normally produced by polycondensation of hexamethylenediamine and adipoyl chloride or adipic acid. It is a step-growth polymerization that produces high molecular weight Nylon 6,6.

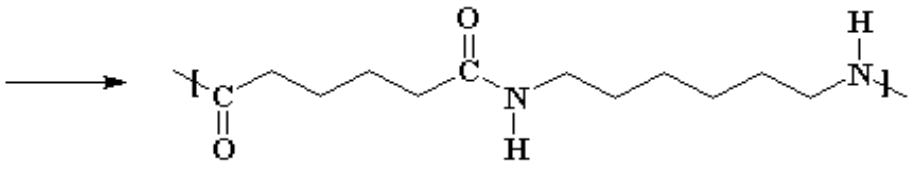
Materials: Adipoyl chloride, Hexamethylenediamine**,** NaOH (0.5M)**,** Cyclohexane

Experimental Procedure:

1. Dissolve adipoyl chloride in Cyclohexane (1% v/v).
2. Add hexamethylenediamine to the 0.5M NaOH solution (1% v/v) (for polymerization under basic condition).
3. Add hexamethylenediamine to water (1% v/v) (for polymerization under neutral condition).
4. Add 5 ml of hexamethylenediamine solutions to a watch glass.
5. Add 2ml of Adipoyl Chloride solution to the center of the solution surface obtained in step 3 and step 4.
6. Let the polymerization reaction occur for few seconds, forming Nylon 6,6 and releasing hydrogen chloride (HCl) as a byproduct.
7. Lift the polymer formed with forceps from the solvent and wrap it on a dropper.
8. Wash the resulting polymer with water, dry it first with some filter paper by pressing hard, and then with a heat gun (gentle heating).
9. Drain the excess solution and dry the product using filter paper while holding with a and collect in an Eppendorf tube
10. Take the weight of the resulting polymer
11. First, measure the FTIR spectra of the pure starting materials, i.e., Adipoyl chloride and Hexamethylenediamine
12. Measure FTIR spectra of the resulting polymers and compare with the IR spectra of starting materials.



adipoyl chloride **hexamethylene diamine**



nylon 6,6

Scheme 1. Synthesis of Nylon 6,6 from adipoyl chloride and hexamethylene diamine.