5.3. Polycondensation (1) – Formation of polyamide

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Printed: Apr 2, 2014 1:29:33 PM
interTESS (Version 13.12 B214, Export 2000)

Task

Task

How can polyamides be produced?

Produce a reaction of adipic acid by adding hexamethylene diamine.

Use the space below for your own notes.

Logged in as a teacher you will find a button below for additional information.

Additional information

Learning objectives

- Adipic acid and hexamethylene diamine react with each other to form a polymer.
- In this reaction, water is splitt off when the dimer is formed.
- The resulting dimer is capable of polymer formationdue due to the still present functional groups.
- This type of reaction is called step-growth polymerization (condensation polymerization).

Notes on set-up and procedure

The experimental process is more favourably when the AH-salt is finely pulverized in a mortar in advanced.

Make sure that the heating is carried out slowly.

Hazard and Precautionary statements

Copper (II) sulphate:

H301:	Toxic if swallowed.
H315:	Causes skin irritation.
H319:	Causes serious eye irritation.
H410:	Very toxic to aquatic life with long lasting effects.
P273:	Avoid release to the environment.
P302 + P352:	IF ON SKIN: Wash with plenty of soap and water.
P305 + P351 + P338:	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

Remarks on the students' experiments

The experiment can be stopped, when a sufficient quantity of condensate is formed. The yellow colouring of the condensate and the polymer is based on the partial decomposition of the AH salt.

Notes

This reaction is the prototype of polyamide formation, which counts to the classical condensation reactions. The originating nylon 66® (read: six, six) is one of the best known plastics.

Nylon was produced in 1935 for the first time, but the ability of condensation to polyamides is known since 1928.

Industrially, the AH salt is heated at 220 °C under oxygen exclusion and fibres are won from the molten mass.

The resulting thermoplastic is meltable and after heating it can be further processed.

Remarks on the method

This experiment can be used as an introduction to the condensation reactions that – despite extensive modifications of the starting materials – follow the reaction scheme formulated here. The problematic nature of the term "condensation" should be addressed here.

In a course of the SEC. II may be referred to the thermodynamic stability of the resulting in the condensation water. The unfavorable entropic chain formation, the entropy provided by the formation of water compared to.

In the last degrees of secondary school can be referred to the thermodynamic stability of water resulting from the condensation. The entropisch unfavorable chain formation can be compared with the increase of entropy because of water formation.

Waste disposal

- Put the produced plastic remains into the normal waste.
- Dispose of the copper sulphate solution in the heavy metals waste.

Material

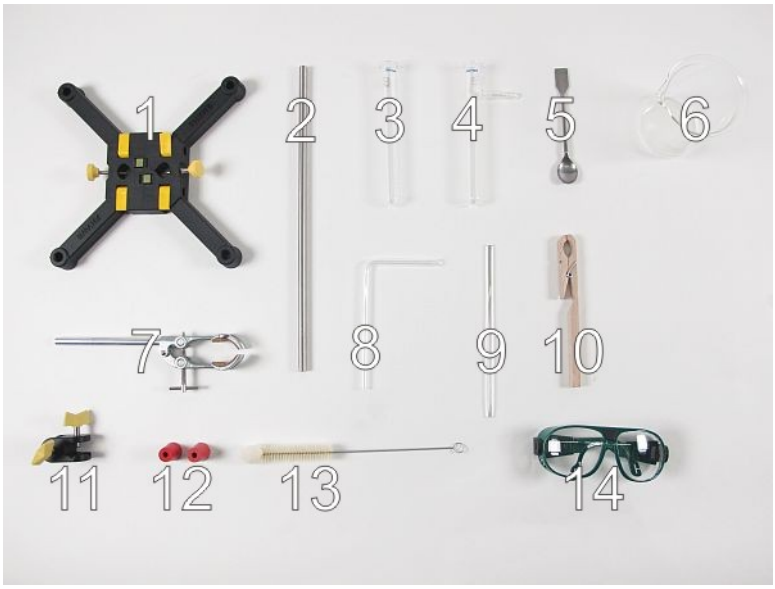
Material from "TESS Chemistry Set Polymer Chemistry" (Order No. 15305-88)

Position No.	Material	Order No.	Quantity
1	Support base, variable	02001-00	1
2	Support rod, stainless steel, *l* = 370 mm, *d* = 10 mm	02059-00	1
3	Test tube, *d* = 20 mm, *l* = 180 mm, DURAN, PN 19	36293-00	1
4	Test tube, *d* = 20 mm, *l* = 180 mm, side arm, DURAN, PN 19,	36330-00	1
5	Spoon, special steel, *l* = 150 mm	33398-00	1
6	Glass beaker DURAN, short, 400 ml	36014-00	1
7	Universal clamp, clamping range: 0...80 mm	37715-00	1
8	Glass tube, right angled, *l* = 85 mm + 60 mm, 10 pcs	36701-52	1
9	Glass rod, BORO 3.3, *l* = 200 mm, *d* = 6 mm	40485-04	1
10	Test tube holder, up to *d* = 22 mm	38823-00	1
11	Bosshead	02043-00	1
12	Rubber stopper, *d* = 17/22 mm, without hole	39255-00	2
13	Test tube brush with wool tip, *d* = 25 mm, *l* = 270 mm	38762-00	1
14	Protective glasses, clear glass	39316-00	1

Chemicals, Additional Material

Position No.	Material	Order No.	Quantity
	Bunsen burner DIN, natural gas	32165-05	1
	Safety gas tubing, DVGW, sold be meter	39281-10	1
	Copper(II)-sulphate, anhydr., 250 g	31495-25	
	AH-salt, 100 g	30910-10	
	Glycerol, 99 %, 250 ml	30084-25	
	Jam jar lid		
	Tap water		

Material required for the experiment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Set-up

Hazards

- When the AH-salt is heated there is a risk of bumping (boiling retardation). Carefully heating, wear protective glasses! Ventilate the room well!
- Copper (II) sulphate is harmful. Do not swallow.

Set-up

Set up the support system according to Fig. 1 - 4.

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Introduce carefully a stopper (with glycerine moisten) at each extrem of the right-angled tube (Fig. 5).

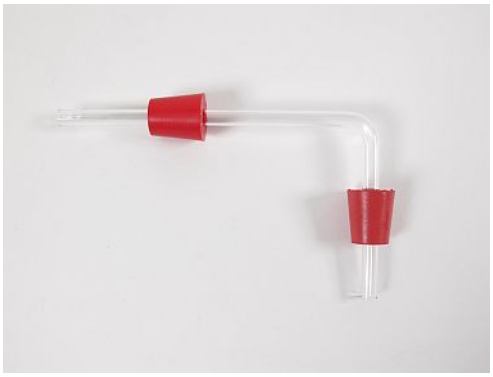


Fig. 5

Fill approx. 5 cm of the Duran-test tube with AH-Salz and fix it diagonally at the support rod.

Close it with the stopper with the hanging right-angled test tube, close with the second hanging stopper the test tube with side arm (Fig. 6 - 8).

Fig. 6

Fig. 7

Fig. 8

Place this test tube in the glass beaker, fill it almost till its edge with cold water (Fig. 9).

Fig. 9

Action

Procedure

Heat the AH-salt on low flame until it starts to melt (Fig. 10). Then also heat the upper part of the test tube until liquid accumulates in the second test tube (with side-arm) (Fig. 11).

Fig. 10

Fig. 11

Remove the stopper and pour the molten mass into the jam jar lid (Fig. 12). Try to pull threats from the molten mass by means of the glass rod.

Fig. 12

Add a spatula tip of anhydrous copper (II) sulphate into the liquid which has been accumulated in the test tube with side-arm (Fig. 13).

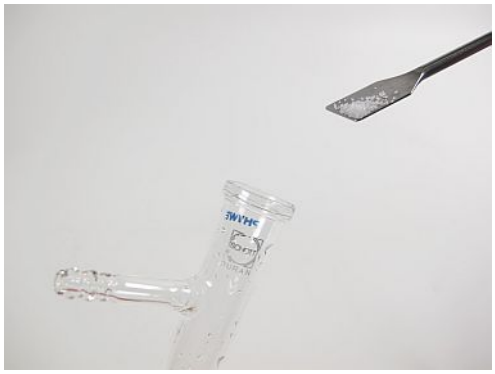


Fig. 13

After the solidification heat the resulting solid once more, and try again to pull threads from the melt.

Waste disposal

- Put the produced plastic remains in the normal waste container.
- Treat the copper sulphate solution as heavy metal waste.

Evaluation

Evaluation 1

Write down your observations in general form.

a) On heating

b) On re-heating

c) Addition of copper (II) sulphate (anhydrous)

a)

The AH-salt melts when it is heated and becomes more and more viscous, besides, it colours slightly yellowish. Watery droplets condense in the cold part of the test tube. After some time a watery liquid accumulates in the condenser. The poured melt, which is slightly coloured yellowish, solidified after some time. Before solidification threads can be pulled from the molten mass.

b)

The solidified reaction product melts when heated again, threads can also be pulled out from the melt. A formation of condensates cannot be longer observed.

c)

The anhydrous white copper (II) sulfate becomes blue.

Questions and exercises

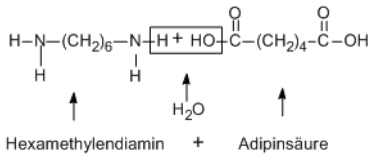
Question 1:

Draw the conclusions from your observations.

The melting of the AH-salt is not a simple change of the state of matter, since the melt becomes more and more viscous, besides it forms water, which can be recognized by the blue color of the copper (II)-sulfate. The result is a polymer which can be softened and reprocess by heating the AHsalt again.

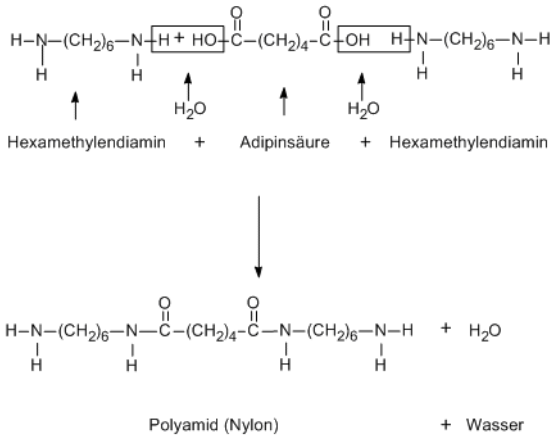
Question 2:

AH-Salz ist eine Mischung aus Adipinsäure und Hexamethylendiamin. Diese Edukte weisen folgende Struktur auf:



Versuche anhand der Strukturformeln und der Beobachtungen eine Aussage über den Verlauf der Reaktion zu formulieren.

Adipic acid and hexamethylenediamine react with each other, whereby water is split off from the carboxyl group and amino group.



Question 3:

Which reaction type has taken place? Try to state a general definition for this reaction type.

The molecules of the starting materials react while splitting off water molecules (condensation), the functional groups of both molecules contribute to this process.

The other two parts are bond to form a dimer, which continues carrying reactive functional groups because of the bifunctionality of the reactant molecules, and it is therefore capable of polymer formation. Thus, polycondensations are reactions of polymer formation in which (at least) bifunctional reactant molecules react to form a polymer under the splitting off of a thermodynamically stable molecule.