Postlab Experiment 3: Diels-Alder Reaction

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I pledge my Honor that I have abided by the Stevens Honor System.

Purpose

The purpose of this experiment is to synthesize a 7-Oxanorbornene from furan and maleic anhydride by a Diels-Alder reaction.

Reagent Table

Name	**M.W.** (0.5 pts)	**Density** (0.5 pts)	**Amount (grams/mL)** (0.5 pts)	**Moles** (0.5 pts)	**Hazards/Precautions (MSDS data) and melting point or boiling point** (2 pts)	**Role of the reagent** (1 pts)*
Furan	68.07 g/mol	936 kg/m^3	3.3 mL	0.045 mol	Extremely flammable liquid and vapor. Harmful if swallowed. Causes skin irritation. Toxic if inhaled. Suspected of causing genetic defects. BP: 88.34°F (31.3°C) @760 mmHg, MP: -122.1°F (-85.6°C)	reactant
maleic anhydride	98.06 g/mol	1.48 g/cm^3	5 g	0.05 mol	Harmful if swallowed. Causes severe skin burns and eye damage. May cause an allergic skin reaction. Causes serious eye damage. May cause allergy or asthma symptoms or breathing difficulties if inhaled. Causes damage to organs (Respiratory system) through prolonged or repeated exposure if inhaled. BP: 395.6°F (202°C) @760 mmHg, MP: 127°F (52.8°C)	reactant
Tetrahydrofuran (THF)	72.11 g/mol	0. 889 g/mL	15 mL	0.185 mols	Flammable, toxic, hygroscopic. BP: 150.8°F (66°C) @760mmHg, MP: -163.1°F (-108.4°C)	Solvent
exo-7-oxabicyclo[2.2.1]hept-5-ene-2,3-dicarboxylicAnhydride	166.13 g/mol	1.4 g/cm^3			Causes skin and serious eye irritation MP: 118°C	Product

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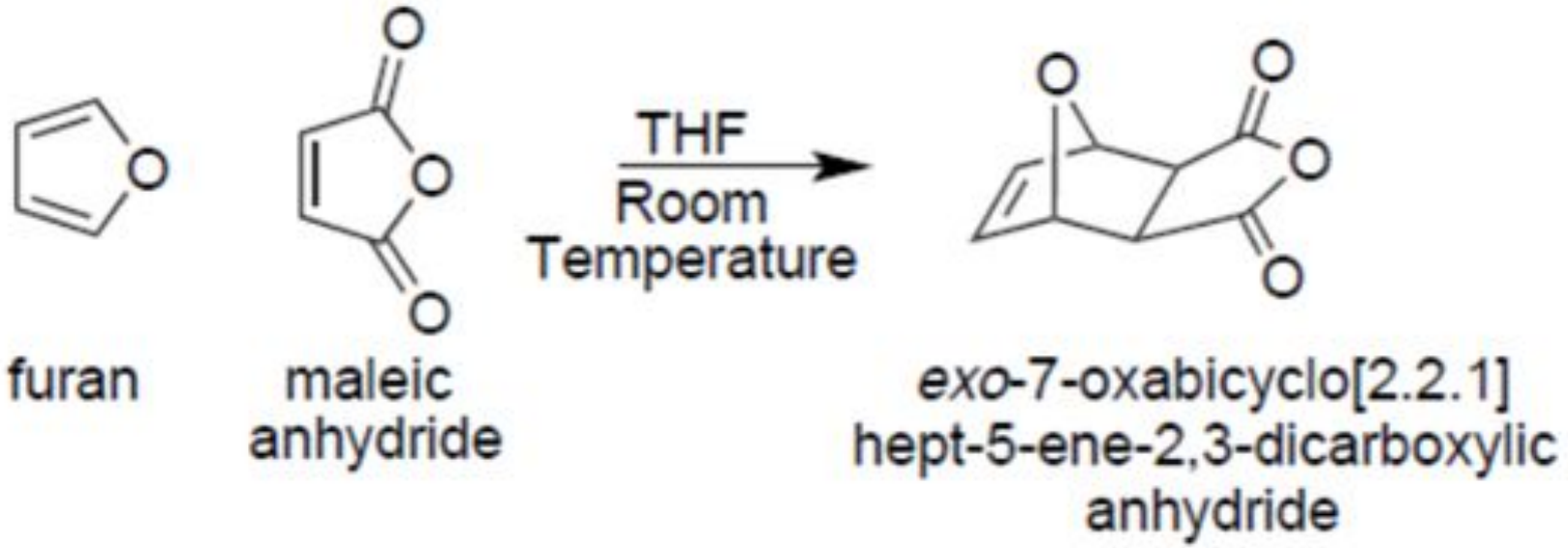
Reaction


THF
Room
Temperature
furan
maleic
anhydride
exo-7-oxabicyclo[2.2.1]
hept-5-ene-2,3-dicarboxylic
anhydride

Procedure

1.A 50-mL round-bottom flask was charged with maleic anhydride (5 g, 0.05 mol).

2.Tetrahydrofuran (THF) (15 mL) was added and the reaction flask was swirled until the maleic anhydride completely dissolved.

3.Furan (3.3 mL, 0.045 mol) was added and the flask was swirled for several minutes to completely mix the contents.

4.The flask was stoppered and allowed to stand until the following laboratory period (one week).

5.The crystals were collected by suction filtration and washed with cold THF.

6.Determine the melting point.

Stepwise Procedure

1.A 50-mL round-bottom flask was charged with maleic anhydride (5 g, 0.05 mol).

2.Tetrahydrofuran (THF) (15 mL) was added and the reaction flask was swirled until the maleic anhydride completely dissolved.

5 g

Then

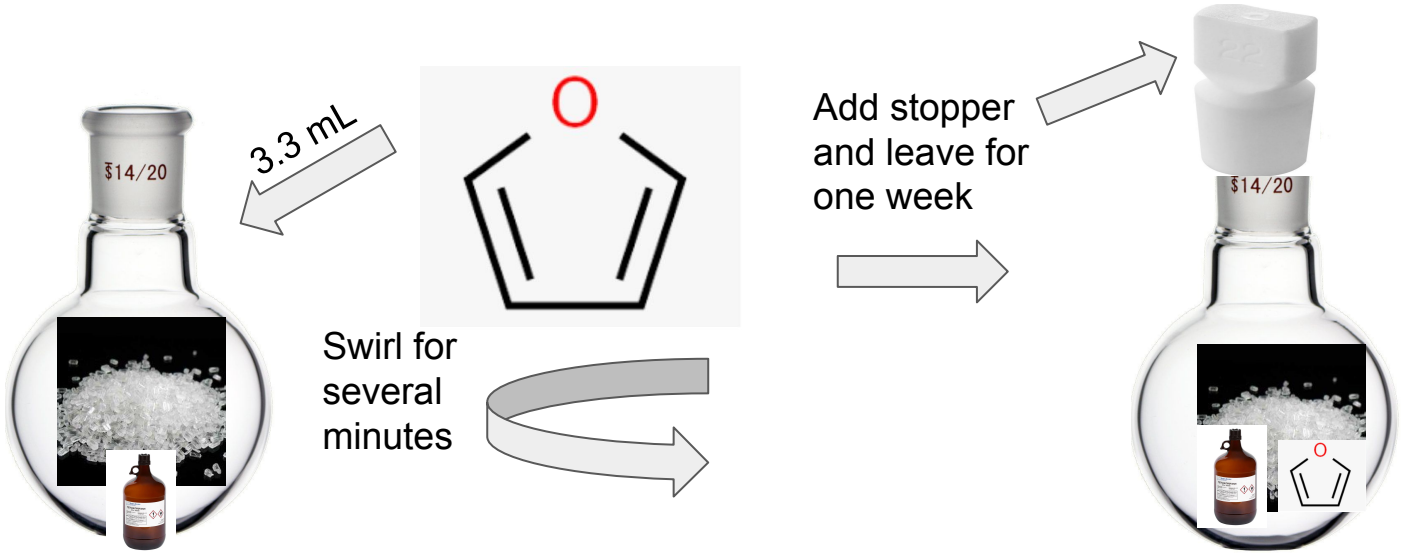
15 mL

Swirl!

Stepwise Procedure (cont.)

3.Furan (3.3 mL, 0.045 mol) was added and the flask was swirled for several minutes to completely mix the contents.

4.The flask was stoppered and allowed to stand until the following laboratory period (one week).

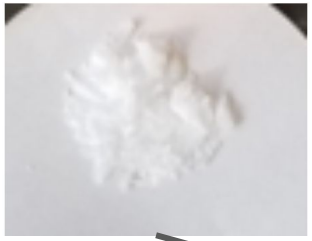


Stepwise Procedure (cont.)

5.The crystals were collected by suction filtration and washed with cold THF.

6.Determine the melting point.

Recover crystals



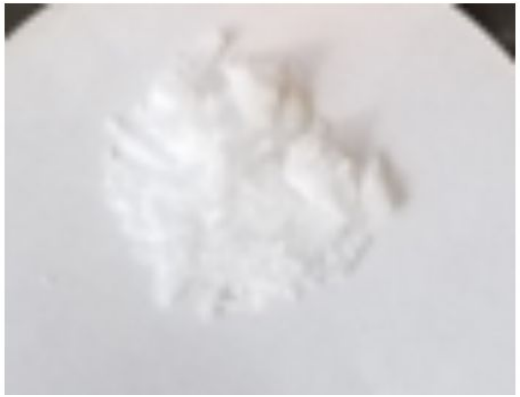
Perform melting point determination

Results

1) Weight of the product = 5.25 grams

   a) Percent yield = (5.25/7.54) * 100 = 69.6%

2) Melting point of the product = 115-117 degrees Celsius
3) Picture of the white crystallized product:



Conclusion

In this experiment, we accomplished synthesis of exo-7-oxabicyclo[2.2.1]-hept-5-ene-2,3-dicarboxylic anhydride through a Diels-Alder reaction between furan and maleic anhydride. Identity of the crystallized product was determined by melting point determination, where a measured melting point of 115-117℃ indicated the exo isomer. We learned about some of the precautions when approaching a Diels-Alder reaction, mostly involving correct choice of solvent to prevent side products. Also, we learned further about uses of melting point determination to identify the identity of a product. There were no issues, and no suggestions to the experiment, as it was performed remotely. The Diels-Alder reaction is a powerful tool in the pharmaceutical industry, as it is a cycloaddition reaction that proceeds under mild conditions, gives high yields, and does not require a metal catalyst.

Post Lab Questions

1. Based on your melting point suggest whether you prepared endo or exo product.
    a. The measured melting point of our product was 115-117℃. As the melting point of the exo isomer is 118-119℃, and the endo isomer is 80-81℃, we conclude that the synthesized product is the exo isomer, as its melting point is the most similar.
2. Why can’t we use acrylonitrile as solvent in place of THF for Diels-Alder reaction of furan with maleic anhydride?
    a. Acrylonitrile has a C=C double bond that can act as a dienophile in the Diels-Alder reaction, reacting with furan to perform cycloaddition in addition to the maleic anhydride. THF has no double bonds, thus this is not a concern with THF. Because of the formation of unwanted side products, acrylonitrile is not a suitable solvent for this reaction.