DICYCLOPENTADIENE CRACKING AND THE DIELS-ALDER REACTION

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Purpose: Use heat to crack, reverse diels-alder, dicyclopentadiene into cyclopentadiene. Form norbornene dicarboxylic anhydride using a Diels-Alder reaction between cyclopentadiene, a diene and maleic anhydride, a dienophile. Isolate the product via recrystallization and categorize it using IR and H NMR.

Reagent Table and Calculations(update with actual amounts):

reagents & products	**FW (g/mol)**	**Density g/mL at 25 °C**	**Bp °C**	**Mp °C**	**volume mL**	**mmol.**	**mass, mg**
dicyclopentadiene	132.20	0.98	170	NA	1.0	7.41	980
Cyclopentadiene (theoretical)	66.10	0.80	41	NA	NA	3.58 (14.8)	237 (980)
maleic anhydride	98.06	NA	NA	53	NA	3.94	388
the norbornene dicarboxylic anhydride	164.16	NA	NA	165-167	NA	0.833 (3.58)	145 (588)

Dicyclopentadiene: 1.0mL(0.98g/1mL)(1mg/$1x10^{-3}$g) = 980mg (1mmol/132.20mg) = 7.4mmol

Cyclopentadiene:

(7.41mmol dicyclopentadiene (2mmol cyclopentadiene/ 1mmoll dicyclopentadiene) = 14.8mmol (66.1mg/1mmol) = 784mg)

237mg(1mmol/66.1mg) = 3.58

Percent Yield: 3.58mmol/14.8mmol x 100 = 24.2%

Maleic acid:

3.58mmol cyclopentadiene (1.1mmol maleic acid/1mmol cyclopentadiene) = 3.94mmol(98.06mg/1mmol) = 388mg

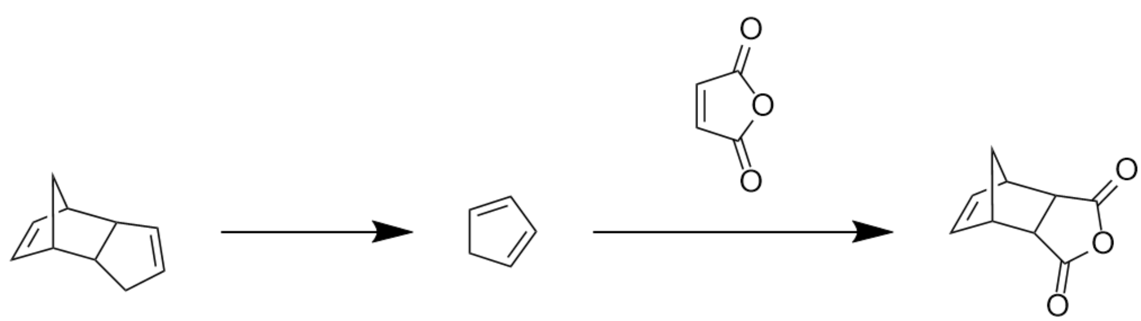
Norbornene Dicarboxylic Acid:

(3.58mmol(164.16mg/1mmol) = 588mg

145mg(1mmol/164.16mg) = 0.883mmol

Percent Yield = 0.883mmol/3.58mmol = 25%

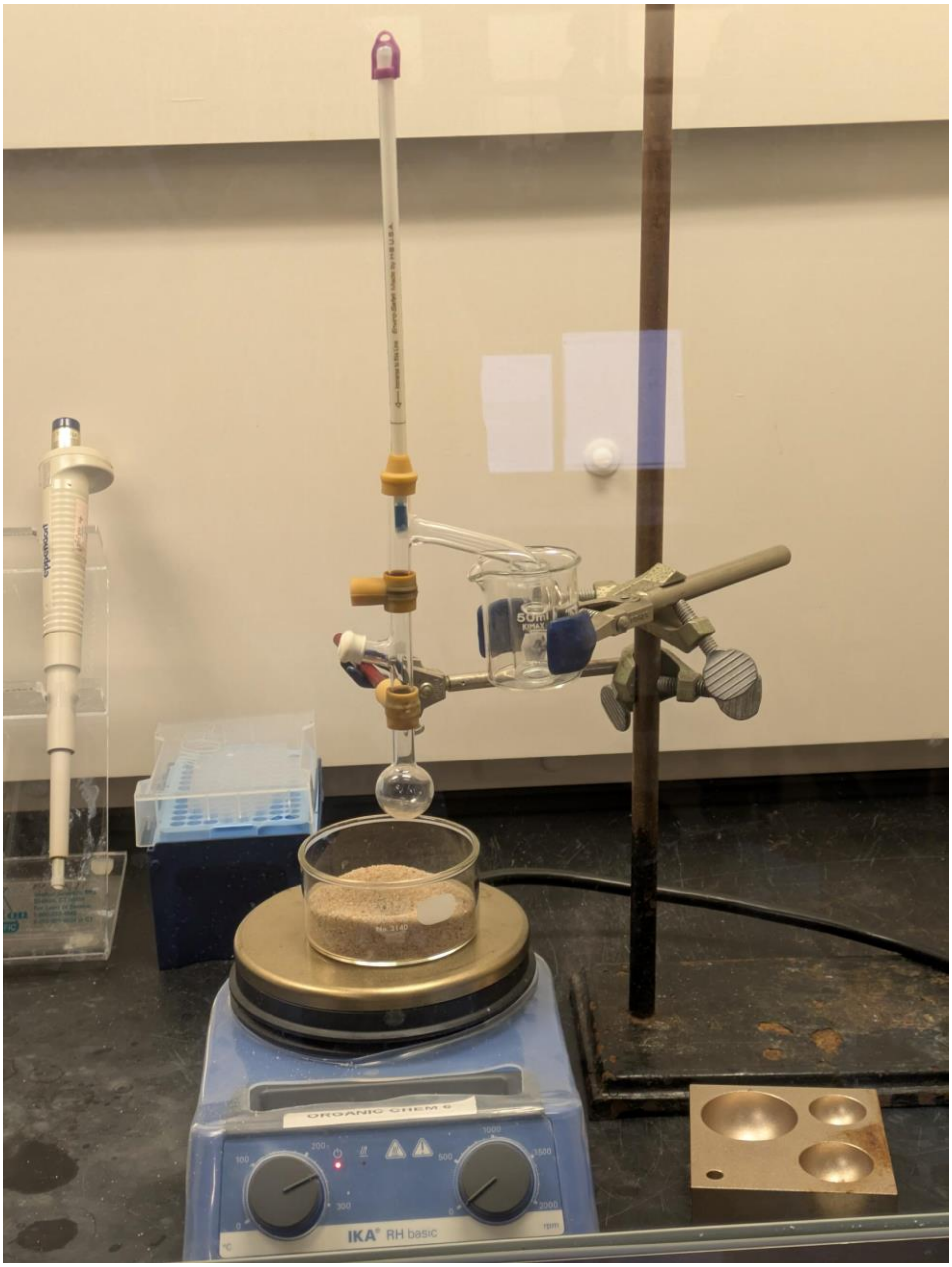
Net Reaction Equation: (Use Chemdraw)



References:

Kateley, L. J., *Guide for Organic Chemistry Laboratory*, Seventeenth edition, Lake Forest College, 2011

Apparatus:



Experimental: (be concise and use abbreviations h, min, and soln)

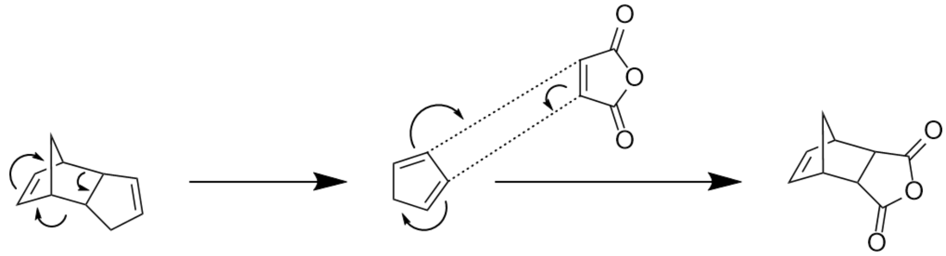
Bulb of 5mL round bottom flask was under half filled with mineral oil then attached back to apparatus. The round bottom flask was heated by the sand bath (set at max heat).

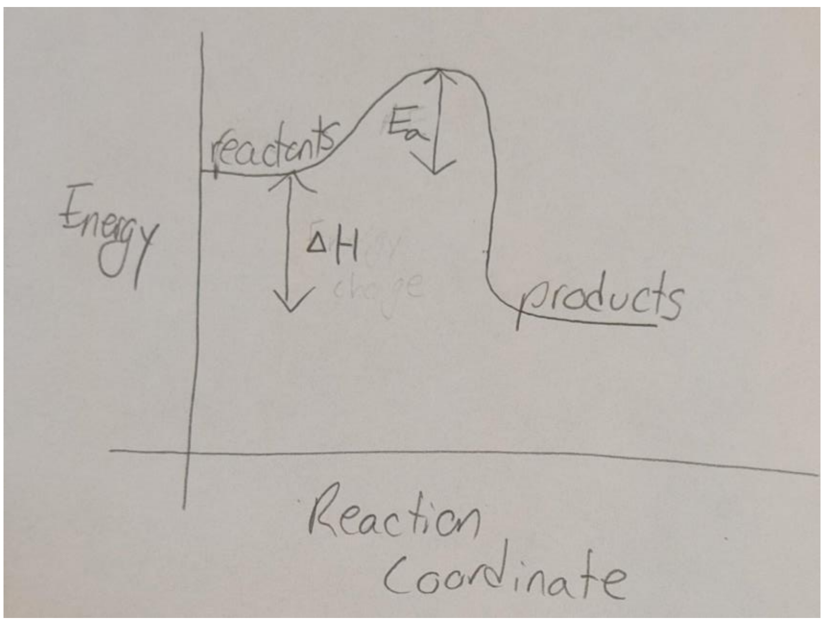
A syringe was used to slowly inject 1.0 mL of dicyclopentadiene through the septum of the side arm into the hot mineral oil. As the dicylopentadiene was added the soln bubbled. The cyclopentadiene escaped from the bubbles and began distillation at 28 C. It dripped from the Claisen head into a tared vial that rested in an ice bath until around 200mg of cyclopentadiene was collected. The vial was reweighted and the percentage yield of cyclopentadiene was calculated.

390 mg of maleic anhydride (1.1 equiv.) was added to an Erlenmeyer flask with a stir bar. 2 mL of ethyl acetate (0.5mL per mmol of maleic acid) was added. The cyclopentadiene was removed from the ice and pipetted in. The flask heated up indicating that the Diels-Alder reaction was exothermic.

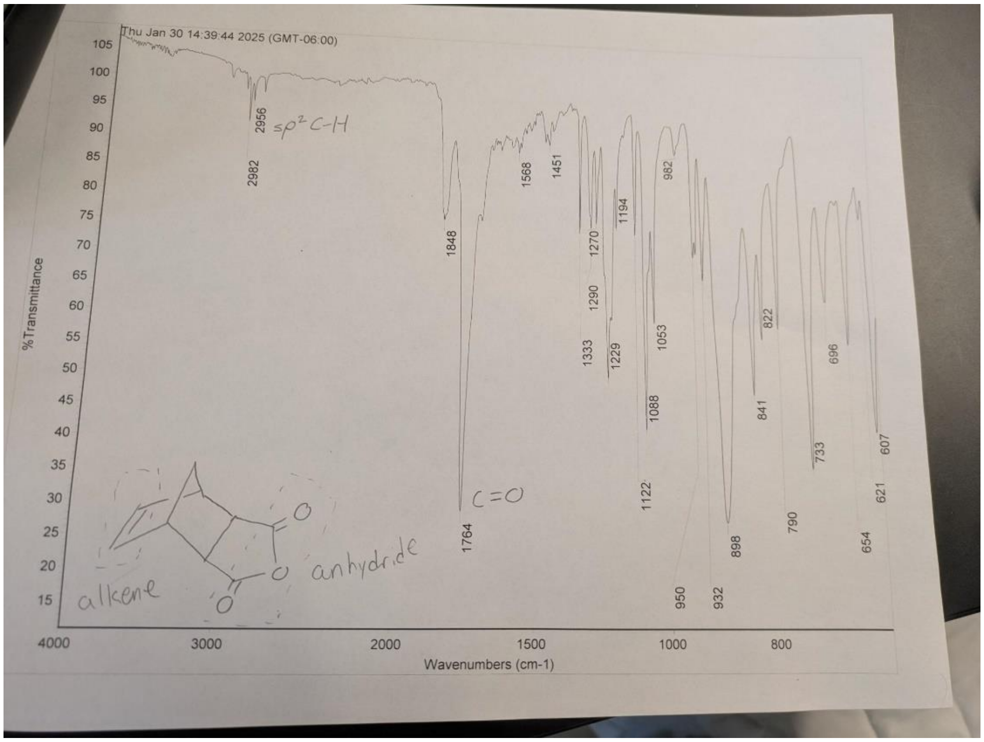
2.0 mL of hexane (equal to the mL of ethyl acetate) was added. Crystals of product began forming after vigorous scratching with a glass stir rod. Crystals were isolated with vacuum filtration.

Mechanism and Energy Diagram: (Use chemdraw and net reaction equation)

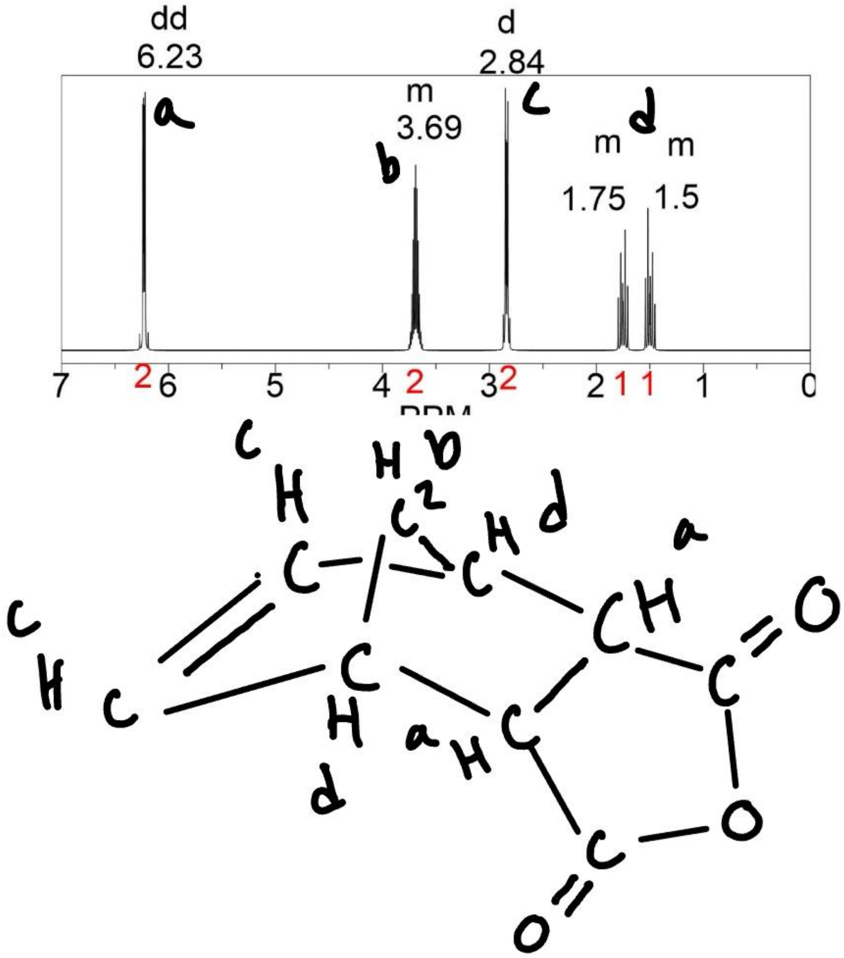




Spectra:



Thu Jan 30 14:39:44 2025 (GMT-06:00)
%Transmittance
Wavenumbers (cm-1)
2982
2956
sp² C-H
1848
1764
C=O
1568
1451
1333
1290
1270
1229
1194
1122
1088
1053
982
950
932
898
841
822
790
733
696
654
621
607
alkene
anhydride



Conclusion: (key findings, yield, and improvements)

“cracking” dicyclopentadiene takes a lot of energy because it is a reverse Diels-alder reaction and Diels alder reaction are exothermic so they form a more stable product then the reactants are independently. This means that the molecules want to stay as the products and not go in reverse.

The hotplates and sand baths did not reach temperatures high enough to break the dicyclopentadiene. So a big improvement would be using a more powerful hot plate. A small improvement would be to use tare the dram with its lid since the cyclopentadiene has such a strong smelling compound.

The percent yield was around 25% for both stages of this experiment meaning in total I only got about 6% of the overall percentage of initial dicyclopentadiene.