

Experiment title. report sample

[Law](#), [Evidence](#)



Section _____

Stoichiometric Data (For any reactants that are part of the balanced equation, base on the amounts measured in lab)

Figure 3-sulfolene

Figure IR spectrum for cis-4-Cyclopentane-1,2-dicarboxylic acid anhydride

Figure from experiment in the text

NMR 1 from lab results endo norbornene-cis-5, 6 dicarboxylic anhydride

Figure 2 from lab results the exo norbornene-cis-5, 6 dicarboxylic anhydride (less stable)

Theoretical from http://www.ch.ic.ac.uk/local/organic/pericyclic/p1_sigma.html

Discussion

Figure 1. The diene (1,3-butadiene) plus the ethene (sulfolene) produce a cyclohexene (either the endo or exo isomer depending on reaction conditions).

Figure 2. Under acidic conditions to sulfuric acid the endo isomer plus water form the less stable exo isomer

Figure 3 Under conditions of low heat the endo isomer produces 2 cyclopentadiene

Figure 4. In a reverse reaction cyclopentadiene plus maleic anhydride produce the endo isomer (low heat)

The Diels-Alder Reaction is a strong reaction in organic synthetic chemistry.

The challenge in carrying out the laboratory is number one handling all the

reagents carefully and with professional laboratory precautions. This is because the reagent, dicyclopentadiene is seriously toxic and the reagent is also flammable. The compound must be kept under the hood at all time when possible and any containers holding the reagent need to be covered. The reaction involves an electron donating group on the diene and the electron withdrawing group on the dienophile. The greater challenge is to identify the endo-norbornene-cis-5, 6- dicarboxylic anhydride and exo-norbornene-cis-5, 6- dicarboxylic anhydride and successfully distinguish between the two stereoisomers. The endo product of a Diels Alder reaction is expected to be the first product that will form unless the reaction is carried out under reversible conditions. Under reversible conditions the exo product is more stable and it does not have the steric strain of the endo isomer so the exo product dominates.

Zaitsev's rule explains the exothermic reaction that takes place when alkenes are hydrogenated to form alkanes. It states that when the removal of HX (where X= OH) takes place to form an alkene then hydrogen is lost preferentially from the carbon atom of a molecule adjacent to a carbon bonded to X - in particular the one with the lesser number of hydrogens. This is important when considering stereochemistry and trying to predict a product for a synthesis reaction.

Figure 1 is the schematic of the Diels-Alder reaction for this lab. 1, 3 butadiene reacted with an ethane will form a cyclohexane. In other terminology a diene plus a dienophile reacts to form a cyclohexane. A maleic anhydride is a cyclopentadiene with 2 C= O and one C-O. The reaction of cyclopentadiene and maleic anhydride results in an endo isomer and an

exo isomer. The products are stereo isomers. Bruice (2008, p. 319-321) explains that The endo isomer is product is the kinetically stable product due to an overlap with the maleic anhydride's oxygen-carbon p-bonds and the newly formed p-bonds. The synthesis reaction will have an NMR with three peaks because there are three types of protons, (a) the protons on the carbon bridge, (b) the double bond protons, and (c) the protons where the two rings meet (maybe they will be detected and reported as two distinct peaks).

Nuclear Magnetic Resonance (NMR) is used to interpret a compound's structure by detecting the C and H nuclei. NMR works by putting nuclei in a static magnetic field and then exposing them to a second magnetic field. ((Homak, n. d.) Nuclei spin causes the signal that the instrumentation measures but spin only exist for an odd number of protons or nuclei. A chemical field is when a shift is noted in the measurements. H+ range from 0-12 ppm and C range from 0-220 ppm. A high shift is when the low ppm values are in evidence while the low shift is when the high ppm values are in evidence. It is the difference of the nucleus resonance frequency subtracted from a standard and then divided by the standard. (Hornak, n. d.) The lab resulted in a yield of approximately 12 percent endo-5 Norbornene-2, 3 dicarboxylic acid. The limiting reagent was malic anhydride(0. 0036 mole).

Appendix 1

Calculations

1) Show calculation for each starred (*) item in the report. write a complete calculation please

(Theoretical yield of the cis-5-norbomene-endo 2, 3 dicarboxylic anhydride =

$180 \text{ g} \times 1.06 \text{ mol} = 190.8 \text{ g}$ from the amount exp)

Maleic anhydride $0.3 \text{ g} \times \frac{1 \text{ mol}}{98.06 \text{ g}} = 0.00306 \text{ mole}$

Sulfolene $0.00425 \text{ mol} \times 118.16 \text{ g/mol} = 0.5 \text{ g}$

p-xylene $0.002441 \text{ moles} \times 106.16 \text{ g/mol} \times 0.87 \text{ g/ml} = 0.22 \text{ g}$

Endo-Norbornene-5, 6-cis-dicarboxylic acid $1.06 \text{ mol} \times 182.17 \text{ g/mol} = 190.8 \text{ g}$

$C_9H_{10}O_4$ (9) $(12.0107 \text{ g/mol}) = 108.0963 \text{ g/mol}$

$(10)(1.00794) = 10.0794$

$(4)(15.9994) = 63.9976$

Total = 182.17

$0.22 \text{ g} \times \frac{100}{182 \text{ g}} = 12.1\%$

Cyclopentadine $81 \text{ g/ml} \times 6 \text{ ml} = 486 \text{ g} \times \frac{1 \text{ mol}}{66.1 \text{ g}} = 7.4 \text{ mol}$

Appendix 2

Bruice, P. Y., (2008) Organic Chemistry 5th ed., Saddle River, NJ: Pearson Education, Inc.

Homak, J. (n. d). Basics of NMR. Ebook, Available from <http://www.cis.rutgers.edu/htbooks/nmr>

Mayo, D. W, Pike, R. M. and Forbes, D. C. (2010). Microscale Organic Laboratory: with Multistep and Multiscale Synthesis. New York, NY: John Wiley & Sons. pp. 261-267.

Material Safety Data sheet for cis-5-Norbornene-endo-2, 3-dicarboxylic acid, 99%, Fischer Scientific, ACC# 72550. Available from <https://fscimage.fishersci.com/msds/72550.htm>

LookChem Available from <http://www.lookchem.com/cas-385/3853-88-1>.

html

ChemBlink <http://www.chemblink.com/products/3813-5>