Name
CHE 173 Sec 20#
January 18, 2005

Experiment 30: Identification of a Conjugated Diene from Eucalyptus Oil

Purpose: The purpose of this experiment was to determine the structure of the unknown conjugated diene in eucalyptus oil by means of a melting point determination and IR spectroscopy.

Introduction: A conjugated diene consists of two pi bonds separated by a single sigma bond. In a Diels-Alder reaction, these dienes, when in the s-cis conformation, are able to react with an alkene to form a Diels-Alder adduct. The alkene, known as a dienophile, reacts with the diene in a single step to form this adduct. This type of reaction is stereospecific, and may yield either an endo adduct or an exo adduct. An endo adduct forms when an overlap in the pi electrons of each reactant occurs, allowing for greater stability. In this experiment, the unknown conjugated diene in eucalyptus oil will be used to form the Diels-Alder adduct. The Diels- Alder adduct can then be separated and a melting point determination can be used to determine the identity of the diene. Furthermore, by obtaining an infrared spectrum of the adduct, the complete structure should be able to be determined using the functional group data.

Materials and Methods: The materials and methods used during this experiment may be found in the laboratory manual (Lehman, p. 247-248). However, some changes to the procedure were made. When heating the reaction mixture under reflux in a boiling water bath, foil was placed around in the flask in order to maintain the temperature of the water

for a longer period of time. Also, in the purification section of the experiment, after recrystalization, the adduct was then collected by vacuum filtration, in order to help dry the product. Other than these, no other significant changes to the materials and methods were made.

Results: The amount of maleic anhydride needed during the experiment was calculated to be 2.34g (see Calculation 1). During the heating of the reaction mixture, the mixture seemed to change color from clear to yellow. When the crystals were washed with petroleum ether, this yellow color was removed and the crystals appeared white. Other than these, no other notable qualitative observations were found in the experiment. When drying the adduct, the mass of the sample was found to be 1.78g (see Calculation 2). The melting point range of the product was found to be 122.0-127.2°C in the first trial and 122.4-127.0°C in the second trial (see Table 1). Given the IR spectroscopy data, peaks appear to occur at 3058.39, 2972.79, 2949.01, 2858.65, 1836.20, and 1769.62 (see attached IR spectrum). Other than those stated, no other significant qualitative data was collected during the experiment.

Discussion: Given the average melting point range for the product as 122.2-127.3°C, the maleic anhydride adduct of the diene _-phellandrene appears to have the most similar melting point, at 126-127°C (Lehman p.246). Thus, it can be determined that the unknown diene is most likely _-phellandrene. In the IR spectrum, the peaks at 3058.39 and 2972.79 possibly correspond to sp^2 C-H bonds. The peaks at 2949.01 and 2858.65

correspond to sp^3 C-H bonds. The peaks at 1836.20 and 1769.62 correspond to the acid anhydride structural unit. This data may be seen summarized in Table 2. The acid anhydride structural unit represents the portion of the Diels-Alder adduct taken from maleic anhydride. The structure of _-phellandrene is given to be:

(Lehman, p.244)

This structure appears to be consistent with the IR spectrum data. However, certain characteristics that could have appear did not, such as C=C. A reason why this may have occurred is experimental error. The product may have included some type of impurity, which could also be an explanation for the slight variance in melting point temperatures. Also, the slide prepared for use in the IR spectroscopy could have been contaminated or not properly prepared. However, as the given data still supports the structure of the Diels-Alder adduct to include _-phellandrene and maleic anhydride, the structure should appear to be:

References / Literature Cited:

Lehman, John; *Multiscale Operational Organic Chemistry*; John Challice, Prentice Hall, New Jersey, 2002, Experiment 30: Identification of a Conjugated Diene from Eucalyptus Oil, p. 243-249.

Figures, Tables, Data, Spectra:

Calculation 1-

The calculations to find the amount of maleic anhydride needed for the experiment may be found below:

5.00g x 0.650187 = 3.2509g

3.2509g x 136.2g/mol diene = 318.825

318.825/ 136.2 g/ mol maleic anhydride = 2.34g

This calculation uses the molecular weight of each substance and the gas chromatography data, which gives 0.650187 as the peak related to the diene.

Calculation 2-

Mass of watchglass + sample – Mass of watchglass = Mass of sample
44.31g – 42.53g = 1.78g

Table 1. Melting point ranges for sample

Trial	Start of melting (°C)	End of melting (°C)
1	122.0	127.2
2	122.4	127.4
Avg	122.2	127.3

Table 2. Peaks from IR spectrum data and corresponding structures.

Peak	Corresponding structure
3058.39	sp^2 C-H
2972.79	sp^2 C-H
2949.01	sp^3 C-H
2858.65	sp^3 C-H
1836.20	Acid anhyrdide
1769.62	Acid anhydride