

Synthesis of isopentene essay



Synthesis of Isopentyl Acetate (Banana Oil) Using A Fischer Esterification

Reaction Purpose: The purpose of this experiment is to synthesize Isopentyl Acetate using a Fischer esterification reaction.

Fischer esterification is an acid-catalyzed condensation of an alcohol and a carboxylic acid, yielding an ester and water. Isopentyl Acetate has the scent of banana oil, once synthesized it will be purified by distillation. Then the analysis of the sample using ^1H NMR and IR will determine purity of the product. **Reaction Scheme:** Mechanism: Fischer esterification is the acid catalyzed condensation of an alcohol and a carboxylic acid, where it is protonated at the carbonyl oxygen, then the nucleophilic attack of the alcohol. The proton is lost and then regained, followed by a decrease of water as electrons from the alcohol oxygen form the double bond.

Finally, the loss of a proton yields the ester. **Procedure:** The procedure is outlined in “–”, Seventh Edition, –, Thomson Custom Publishing, Mason Ohio, 2007, pp. 30. Calcium Chloride (CaCl_2) was used instead of Magnesium Sulfate (MgSO_4) as a drying agent. Boiling stones were used instead of the stir bar. **Reagent/Product Table:** Reagents/Product Table: *= measured values += calculated values Acetic Acid Isopentyl Alcohol Isopentyl Acetate Sulfuric Acid 1 Molecular Weight (g/mol) 60.

0.52488. 1.492130. 1.86498. 0.734 Grams (g) + 8.91 * 53.

852. 208 Moles + 0.14837 + 0.0567 + 0.

0.373 + 0.022 Volume (mL) * 8.5 + 6.15 + 5.54 * 1.2 Density (g/mL) 1.

04920. 08131. 4041. 84 1Boiling pt (? C)117. 9130142280 1Melting pt (? C)16. 6111-78.

53 Solubilitymiscible in H₂Osolublesolublesoluble 2HazardsCorrosive and flammable; do not contact with skin; or inhaleFlammable; causes coughingFlammable, avoid contact with skin & eyes; irritationcorrosive 2Appearancecolorless/clearcolorless/clearclearclear

1FormulaC₂H₄O₂C₅H₂O₂C₇H₁₄O₂H₂SO₄ 1Reference: <http://www.chemfinder.cambridgesoft.com> 2Reference: <http://www.hazard.com/msds> Observations: After adding isopentyl alcohol to acetic acid in a round-bottomed flask, the mixture was initially clear and colorless and then turned a tea-like brown tint.

Approximately ten minutes into the reflux period a condensation reflux ring was visible near the top of the round-bottom flask and then progressed up the approximately one-third of the condenser column length approximately twenty minutes into the reflux. Thirty minutes into the reflux, the mixture changed to a dark purple color while it bubbled and boiled. This color remained through the extractions but was left in the boiling flask during distillation. Reflux continued for a total of 60 minutes after the reflux ring formed and took approximately ten minutes to cool down to room temperature. Then the room temperature mixture was prepared for extraction with 10mL of water but discovered to be still very acidic after one wash.

To reach a neutral pH, it took three washes with Sodium Bicarbonate (NaHCO₃) to remove any excess acetic acid and catalytic sulfuric acid.

During distillation, the first drop was observed at 135 C and stopped at approximately 139 C when the distilling flask approached dryness. The final product was clear and colorless with a strong scent of bananas. The mass of the product was a 4.42g yield.

Results: Theoretical Yield Actual Yield Percent Yield Temp of 1st Drop Bpt
 Range Measured/Literature Appearance .42g 4.42g 59.56% 135 139/141 Clear
 and colorless Calculations Theoretical Yield: $5.0\text{g} \times (1\text{mol Isopentyl Alcohol}/88.1492\text{g of Isopentyl Alcohol}) \times (1\text{mol Isopentyl Acetate}/1\text{mol Isopentyl Alcohol}) \times (130.$

$864\text{g}/1\text{mol Isopentyl Acetate}) = 7.42\text{g}$ Mass of Beakers with Product: 26.53
 Mass of Beaker: 22.11 Actual Yield = $26.53 - 22.11 = 4.42\text{g}$

42g NMR Spectroscopy for Isopentyl Acetate: 200MHz, CDCl₃ Protons,
 H_x Chemical Shift (ppm) Splitting Pattern Integration Area (cm)² #H's
 Ha 0.91 Doublet 6 Hb 1.65 Multiplet 1 Hc 1.54 Quartet 2 Hd 4.09 Triplet 2 He 2.03 singlet 3

IR Spectrum for Isopentyl Acetate Bond Vibration Frequency (cm⁻¹) Intensity
 C=O stretch 1743.64 Strong C-O stretch 1243.82 Medium - Strong
 C-H stretch (or bend, etc.) for Alkanes 2871.70 & 2959.60 Medium C-H bend (-CH₃) 1367.48 Strong C-H bend (-CH₂) 1466.92 Strong

Conclusion/Discussion: The Fischer esterification of 5.0 grams of isopentyl alcohol using acetic acid yielded an amount of 4.42g of isopentyl acetate (59% yield).

Fischer esterification of isopentyl acetate is an equilibrium reaction, so it is expected that some of the product would hydrolyze back to acetic acid and

isopentyl alcohol. When the isopentyl acetate product reached room temperature after reflux the mixture had a mixture of both product and reactants. During the extraction process, the water will have dissolved most of the two reactants because both reactants are soluble, and they were removed through drainage. This removal of the two reactants would justify the ability for more of the product to be synthesized therefore this step resulted in a loss of the yield and prevented loss of yield in the reaction by removal of one of the products, water which prevents hydrolysis. But, if the water was not drained during this initial wash – the excess water in the mixture would hydrolyze products back to the reactants and shifting the equilibrium once more.

Another possibility of our low yields is because of the decanting process in which it cannot be determined if enough H₂O was absorbed into the CaCl₂ or if there was the possibility that we did not decant all we needed or some of it was potentially absorbed by Calcium Chloride. Human error could have played a large role in this experiment as transferring products could also result in a loss of yield, as well as improper extraction, separation funnels that had water residues, decanting instead of allowing the gravity filtration to completely finish. Yield increase could be achieved through more careful facilitation of techniques and knowing exactly how much drying agent to use. A suggestion for water removal could be to distill the reaction in the beginning and collect the water in a receiving flask since it has the lowest boiling point compared to all the other compounds. The collection of water should be stopped after the constant temperature of 100 C begins to rise, indicating all the possible water in the mixture has been collected.

This way, the remaining reactants in the mixture can only drive towards the right because any formation of water will be removed immediately without removing the reactants. The IR and ^1H NMR spectrums obtained in this experiment revealed some signs of impurities (the IR spectrum attached to back). All the peaks on the ^1H NMR spectra are identified with H's on isopentyl acetate compound. There were no other extra significant peaks in the NMR spectrum. The only extra peak observed had much too small of an integration to be considered part of the compound. However, they might show very minor impurities.

The peak at approximately 7.262 ppm, although small, may be due to the CHCl_3 from the deuterate chloroform solvent used in the NMR tube. There seems to be some extra but minor absorption peaks in the IR spectra. However, all significant peaks seem to identify with groups on our theoretical compound, isopentyl acetate. According to the IR spectrum, the C-H bend stretches at 1367.

8 cm^{-1} and 1466.92 cm^{-1} are both relatively strong in intensity. However, Table 18.2 in Mohrig suggests that these peaks should be much weaker in intensity. This, however, might be due to the result of different types of vibrations. Some C-H bonds may stretch while others might bend.

If there are more C-H bonds that stretch, the intensity will be lowered. With that into consideration and the values of the peaks matching, we can safely assume that these are C-H bend ($-\text{CH}_2$) and ($-\text{CH}_3$) bends even though they are more intense than expected. There are also very weak and inconsistent absorptions between 4000-3000 cm^{-1} that suggest that there had been some

hydrolyzing that had taken place and the IR might've absorbed traces of the -OH group of the reagents acetic acid or isopentyl alcohol. Since there were only slight traces and were weak and not broad at this region it was not enough to skew the spectrum. Otherwise, the IR and ^1H NMR spectrums agree with each other in terms of purity. The boiling point is on par with the IR and ^1H NMR spectrums.

The observed boiling point ranged between 135-139 C is moderately accurate when compared to the literature value of 141 C. The slight change in temperature and closeness of the actual to literature boiling point values indicate that the final product was only slightly impure. This may have resulted from the existence of the drying agent CaCl_2 solid in the distilling flask and might've been an error in decanting process. Pure compounds boil at the literature value due to the equal pressure inside the solution to the pressure of its surroundings, so the existence of these solids may have affected the pressure of the mixture and therefore lowered its boiling point.

In addition, effective drying is important before distillation. This is because water's boiling point (100 oC) is much lower than the boiling point of isopentyl acetate (140 oC), so it will condense into the receiving flask before the product, and contaminate its purity. Drying, however, was considered successful since no drops were observed at 100 OC, nor does the spectrum suggest the impurity. So overall, the obtained was accurate and relatively pure, and this may have been achieved through careful extraction and effective drying.