

Isopentyl Acetate Synthesis

Name: Lindsey Fester

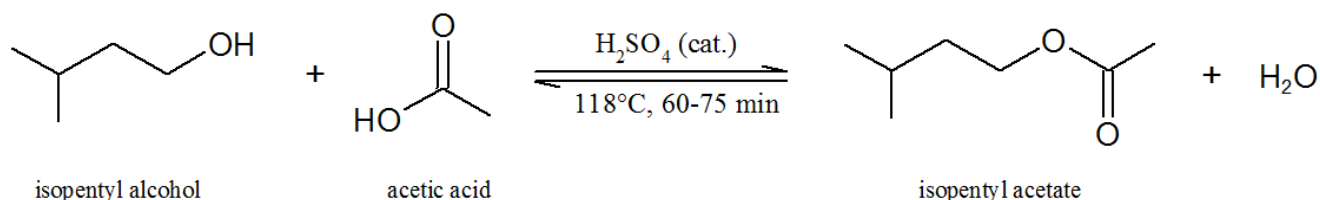
Lab: F 8:00 a.m. – 410 CE

Teaching Assistant: Qinggeng Zhuang (Albert)

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Purpose: To synthesize isopentyl acetate via an acid-catalyzed Fischer esterification between alcohol and acetic acid.

Reaction Equation:



Procedure:¹

A solution of isopentyl alcohol (8.09g, 91.8mmol, 10.0mL), acetic acid (13.7g, 229 mmol, 13.1mL), and sulfuric acid (1.7g, 17mmol, 1.0mL) was heated under reflux for 60min with stirring. The reaction mixture was cooled to room temperature and aqueous sodium bicarbonate (3×10mL, 5%) was added slowly to extract the acids. The organic phase was collected and dehydrated by anhydrous sodium sulfate. The crude product was distilled to obtain isopentyl acetate (7.95g, 61.1mmol, 67%) as a clear liquid, bp 127-132°C, (lit bp² 142°C); IR (thin film, NaCl) 3464, 2961, 2874, 1743cm⁻¹.

Discussion:

Using the IR data on page 3 as a reference for the purity and identity of the isolated compound:

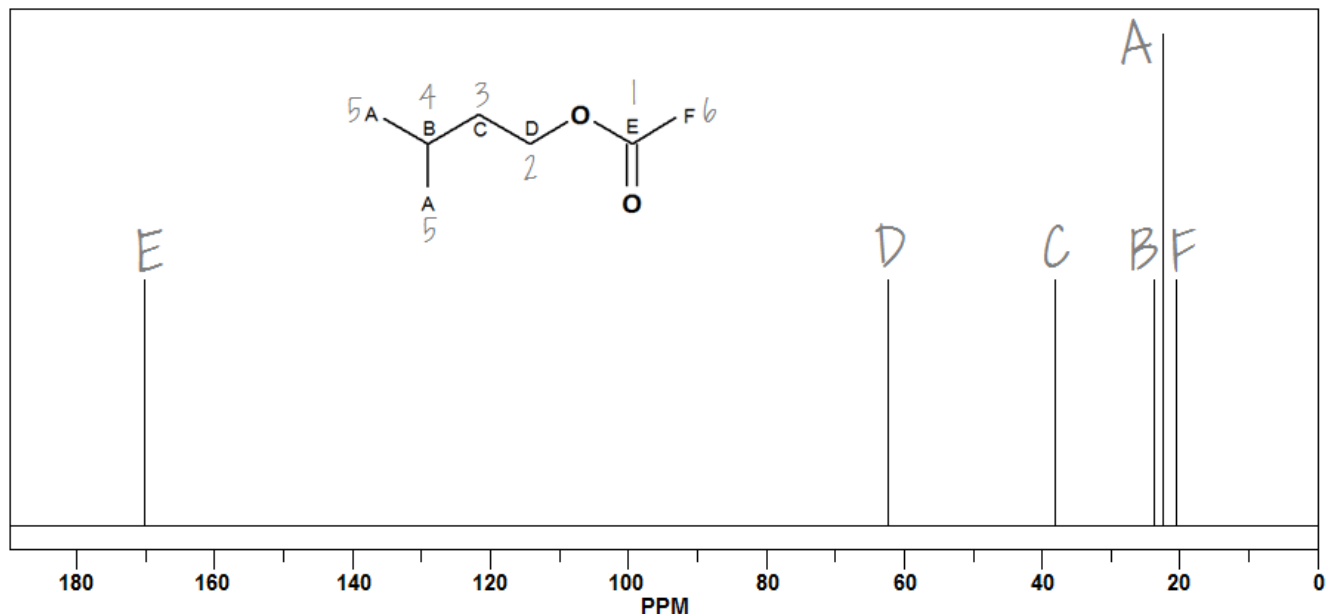
Peaks: 3464.06cm⁻¹ → overtone from carbonyl

$\left. \begin{array}{l} 2961.15\text{cm}^{-1} \\ 2874.09\text{cm}^{-1} \end{array} \right\} \rightarrow \text{C-H stretch of alkyl}$

1743.09cm⁻¹ → C=O stretch of carboxylic ester

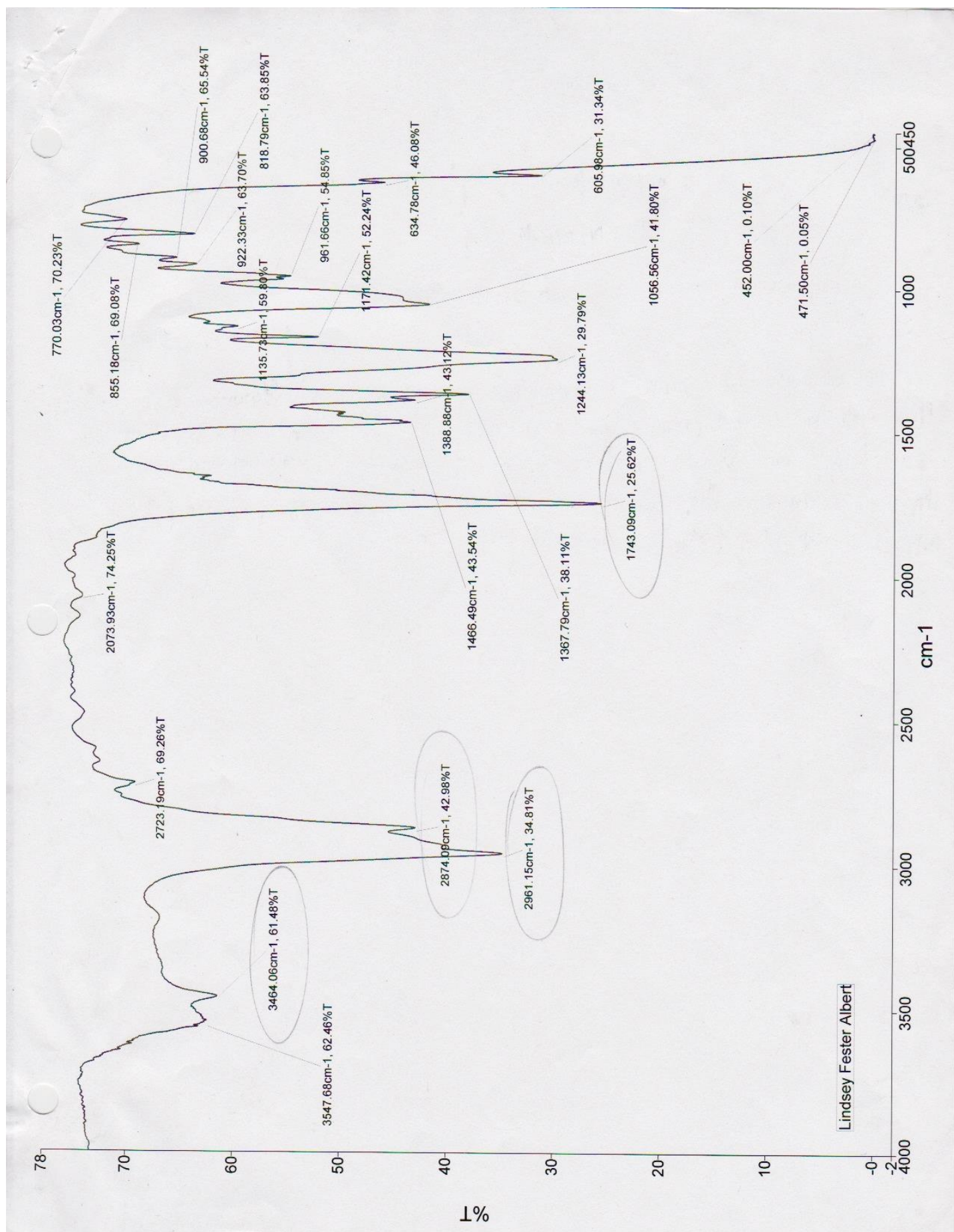
The IR data showed a peak at 1743.09cm⁻¹, confirming the presence of a carboxylic ester with an overtone peak from the carbonyl at 3464.06cm⁻¹. The two peaks at 2961.15cm⁻¹ and 2874.09cm⁻¹ represent the remaining alkyl chain, confirming isopentyl acetate as the identity of the product. The absence of any other distinct peaks represents the product's high purity.

Concept Question. Assign the peaks in the following ^{13}C NMR spectra to the lettered carbons indicated for isopentyl acetate.



References:

- (1) Callam, C. S.; Paul, N. M. Chapter 11—Electrophilic Aromatic Substitution—Nitration of Aromatics; Chapter 12—Bromination of Aromatics—Electrophilic Aromatic Substitution. *Chemistry 2550—Organic Chemistry Lab*; McGraw-Hill: New York; pp 87-94, 99-105.
- (2) Sigma-Aldrich Chemical Catalog Online. <http://www.sigmaaldrich.com/united-states.html> (accessed Feb 2013); search terms: veratrole, acetic acid, nitric acid, 4-nitroveratrole, water, acetanilide, potassium bromate, hydrobromic acid.



Lindsey Fester Albert