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Experiment 14A: Isopentyl Acetate

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Experiment 14A: Isopentyl Acetate

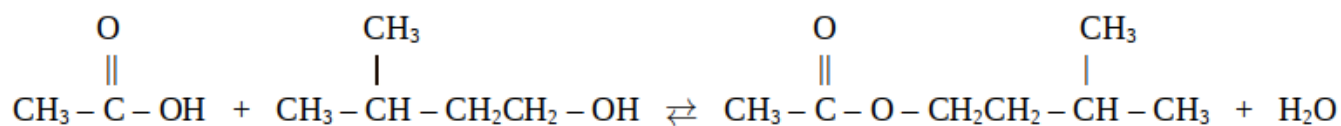
Ethan Sevedge

October 14th, 2020

Abstract:

In this experiment, the synthesis of isopentyl acetate via the Fischer esterification of acetic acid with isopentyl alcohol was studied.¹ Sulfuric acid acted as a catalyst for the reaction. A large excess of acetic acid and the addition of heat to the system shifted the reaction's equilibrium to the right, favoring the ester. The percent yield was 227%, most likely due to weighing errors. H1 NMR, C13 NMR, and C13 DEPT NMR spectra were taken to confirm the product. The peaks, integrations, and spin-spin splitting on the H1 NMR indicated that the molecule was isopentyl acetate. The peaks of the C13 and C13 DEPT NMR spectra also corresponded to the structure of isopentyl acetate, although the peak resulting from the carbonyl carbon was not clearly identifiable. In light of this data, as well as the product's strong banana-like odor, it was concluded that the product did in fact contain isopentyl acetate, though the purity of the compound could not be determined.

Chemical Reaction:



1.500 mL acetic acid

1.59 g acetic acid ----- produces 26.5 mmol. isopentyl acetate

0.781 g. isopentyl alcohol ----- produces 8.85 mmol. isopentyl acetate

↑
limiting reagent

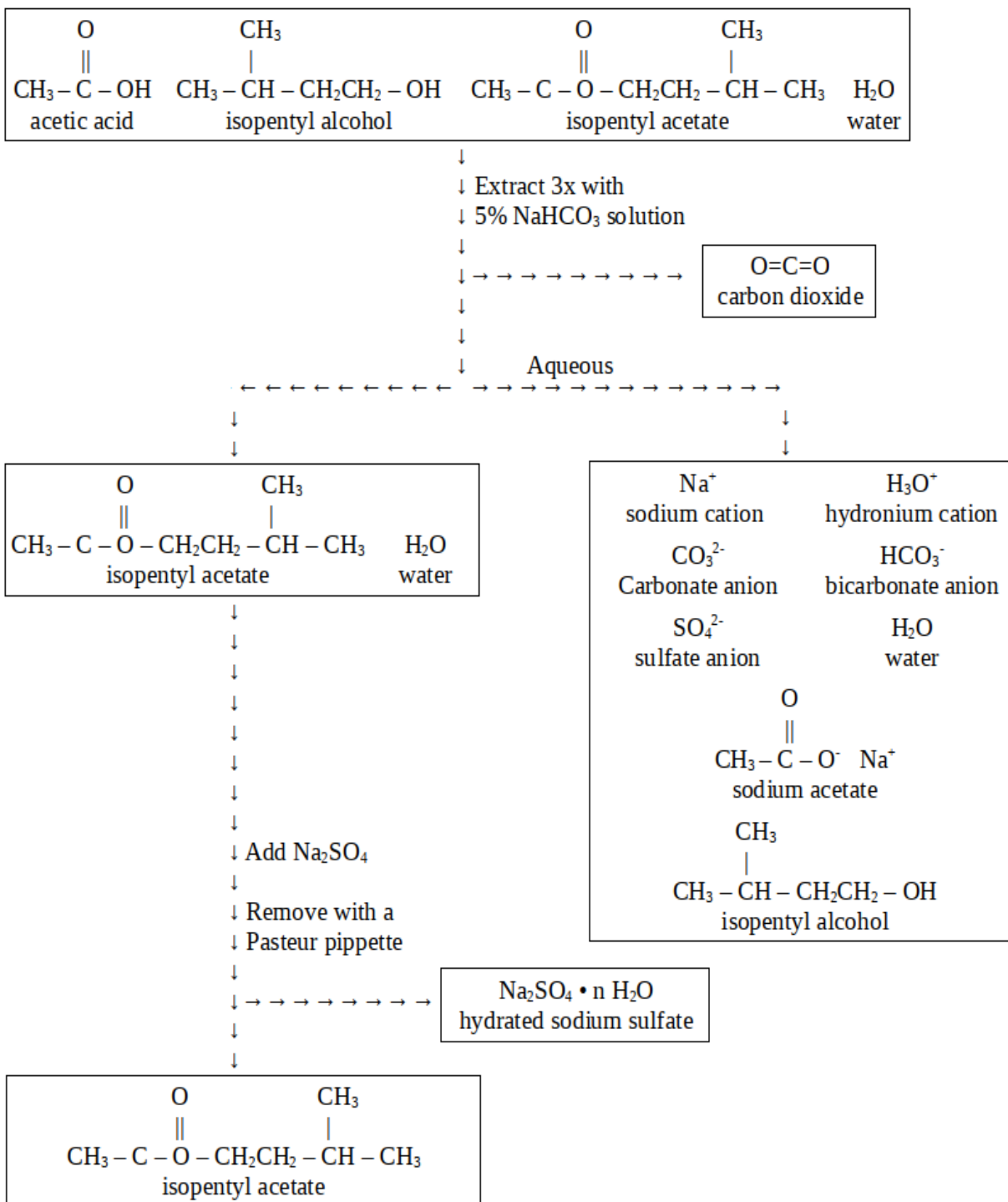
Theoretical Yield: 8.85 mmol. isopentyl acetate may be produced.

$$\frac{8.85 \text{ mmol.}}{1} * \frac{130.18 \text{ mg}}{1 \text{ mmol.}} * \frac{1 \text{ g}}{1000 \text{ mg}} = 1.15 \text{ g isopentyl acetate may be produced}$$

Table of Physical Constants:

Substance	MW (g/mol)	Density (g/mL)	MP (°C)	BP (°C)
acetic acid ^{2, 3}	60.1	1.06	16.635	117.9
isopentyl alcohol ^{2, 4}	88.2	0.813	-117.2	132.5
sulfuric acid ⁵	98.08	1.8302	10.31	337
isopentyl acetate ^{2, 6}	130.18	0.876	-78.5	142
water ⁷	18.02	0.998	0.00	100.00
sodium bicarbonate ⁸	84.007	2.20	~50 decomposes	decomposes
magnesium sulfate ⁹	120.37	2.66	1124 decomposes	decomposes

Separation Scheme:

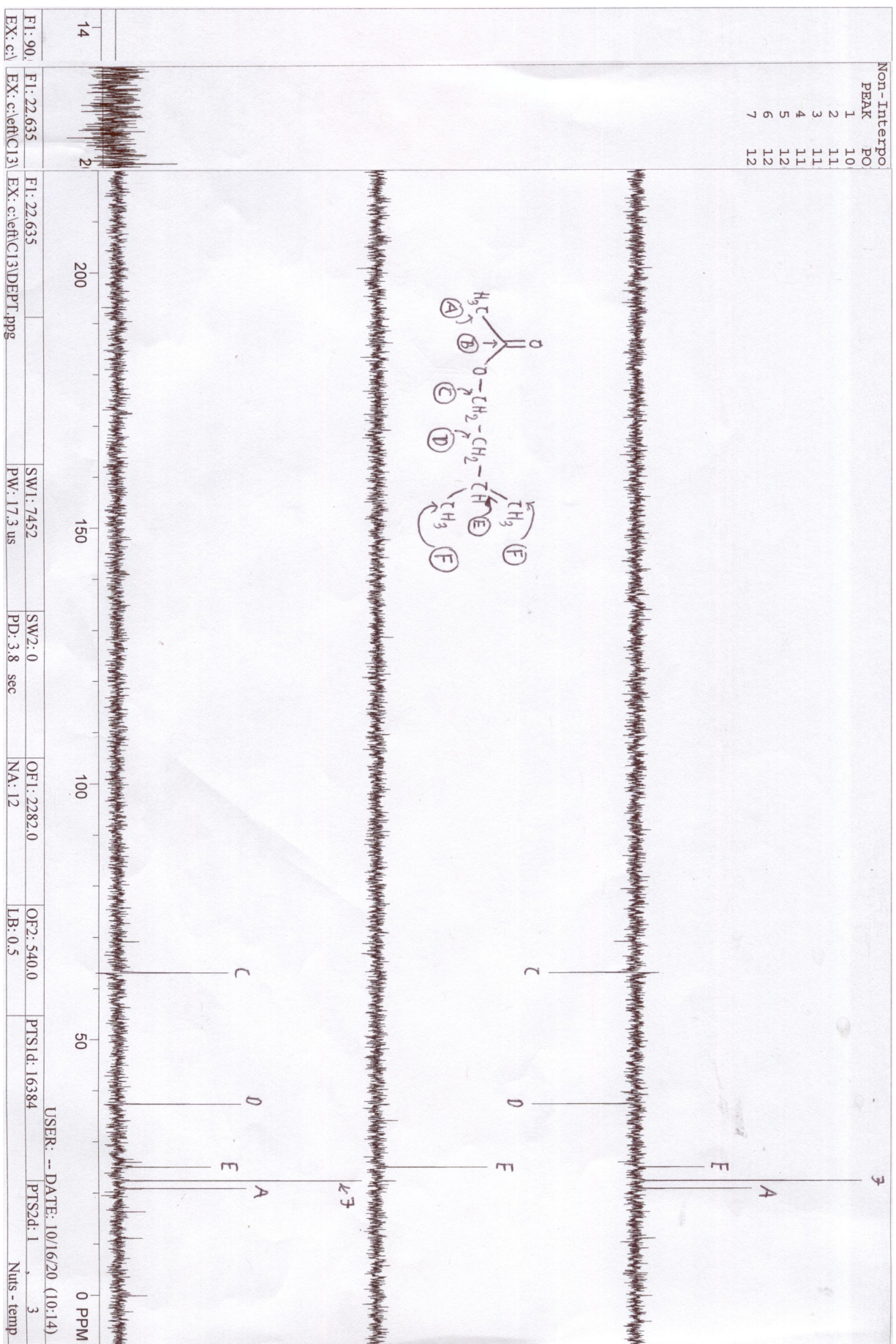


Procedure:

The procedure found in the lab manual was followed, except that the distillation and boiling point determination were skipped.

Data:

Mass of 5-mL conical vial	22.231 g
Mass of 5-mL conical vial with isopentyl alcohol	23.012 g
Mass of isopentyl alcohol	0.781 g
Volume of acetic acid	1.500 mL
Volume of 5% aqueous sodium bicarbonate	3.000 mL
Microspatulafuls of sodium sulfate	about 4
Loss	product was spilled three times
Amount of product observed to remain after spillage	approximately 0.3 mL
Mass of 3-mL conical vial	19.145 g
Mass of 3-mL conical vial with product	21.765 g
Mass of product	2.620 g
Percent yield	227%
Odor of product	reminiscent of bananas, with a bit of sharpness – i.e., a “chemically” smell



Conclusion:

This experiment focused on the synthesis of an ester by a Fischer esterification. A Fischer esterification is an acid-catalyzed reaction in which a carboxylic acid and an alcohol react to form an ester and water.¹ In this instance, concentrated sulfuric acid was used to catalyze the reaction of acetic acid with isopentyl alcohol, forming water and isopentyl acetate, an ester with the scent of bananas.¹⁰ The equilibrium for this reaction favors the reactants, however. To overcome this, excess acetic acid and heat were used to shift the equilibrium of the reaction to the right, in accordance with Le Chatelier's principle, in order to produce a more significant amount of products.¹⁰ After the reaction, a 5% aqueous sodium bicarbonate solution was added to the product to remove most of the acid and water, and was then discarded. The product was subsequently dried over sodium sulfate to remove the water which remained. H1 NMR, C13 NMR, and C13 DEPT NMR spectra were taken of the resulting substance to verify its identity. An IR spectrum was also assigned in order to determine the purity of the substance, but it was not taken.

The experiment had a percent yield of 227%. Seeing that the product was spilled three times, and only about 0.3 mL of product was observed to remain after said spills, it was concluded that the over-high percent yield was due to a simple weighing error. If the mass of exactly 0.3 mL of isopentyl acetate (0.2628 g) is subtracted from the mass of the product (2.620 g), the result is 2.357 g, a reasonable mass for the cap of a conical vial. Thus it is probable that the tare of the conical vial was taken without a cap, and the mass of the conical vial and product was taken with the cap, accounting for the large percent yield. Calculated simply from the observed mass of the isopentyl acetate, the percent yield would be 22.8%, a much more reasonable figure considering the significant amount of spillage.

The number of hydrogens in the H1 NMR and the way they were shown to interact with each other was in accordance with the structure of isopentyl acetate. Seeing that all of the groups of electronically equivalent hydrogens except for one were adjacent to at least one other group, it was

determined that all of the NMR signals except one would show spin-spin splitting. That group, the hydrogens on the acetate carbon, gave rise to a singlet at around 2.06 ppm which integrated to 115.83. By setting this value equivalent to 3 hydrogens, the number of hydrogens represented by the other signals were calculated. The triplet at around 4.14 ppm integrated to 2 hydrogens. Seeing that this signal was the farthest downfield, it was concluded that it was due to the hydrogens of the methylene carbon bonded directly to the ester's oxygen— the two methylene hydrogens bonded to the adjacent carbon account for the spin-spin splitting. The doublet at about 0.41 ppm integrated to 6 hydrogens, which must have been from the the two equivalent hydrogen groups on the isopropyl group. The single hydrogen on the central carbon caused this peak to split into two. The last group of peaks at around 1.53 ppm integrated to 3 hydrogens. There are no groups of three equivalent hydrogens in the structure of isopentyl acetate that were not already accounted for; however, the splitting of the group of peaks was not easily identifiable either. It would seem plausible, then, that the group of peaks contained overlapping signals from both the hydrogen on the central isopropyl carbon and the two hydrogens of the methylene group bonded to said carbon, which would account for both the integration and the irregular splitting pattern. Thus all of the hydrogens on the isopentyl acetate molecule were accounted for by peaks in the spectrum.

The C13 NMR and C13 DEPT NMR also gave compelling evidence that the product was isopentyl acetate, although the signal from the carbonyl carbon was not easily identifiable in the former spectrum. The DEPT 90 NMR showed that only one carbon in the molecule was tertiary, and it gave rise to a peak at 25.054 ppm. Seeing that the central carbon in the isopropyl group of isopentyl acetate was the molecule's only tertiary carbon, it must have been the one responsible for the peak. The DEPT 135 NMR showed that two carbons were secondary, with one peak at 63.056 ppm and another at 37.353 ppm. These corresponded to the two methylene carbons in the molecule. The former peak was thought to be caused by the carbon directly bonded to the ester oxygen, as it was shifted downfield in the manner of most peaks resulting from carbons bonded to electronegative atoms; and the latter was

concluded to be caused by the carbon bonded to the central isopropyl carbon by process of elimination. The two unidentified peaks that remained in the DEPT 135 NMR, therefore, had to have resulted from the molecule's primary carbons. Seeing that the signal at 22.382 ppm was much larger than the others, it was concluded that it resulted from the two equivalent carbon atoms of the isopropyl group, leaving the peak at 20.874 ppm to be from the acetate group's methyl carbon. This left only one carbon of the molecule's structure that hadn't been accounted for in the C13 NMR: that belonging to the carbonyl group. Carbonyl carbons, being bonded to no hydrogen atoms, give rise to relatively weak peaks in comparison to primary, secondary, or tertiary carbon signals in proton-decoupled C13 NMR spectra, due to the nuclear Overhauser effect.¹¹ Unfortunately, this meant that finding the carbonyl carbon's peak on the C13 NMR spectrum was not as straightforward as finding the others, because several low-intensity signals were present in the spectrum that rose just high enough to be considered of possible significance. Of these peaks, however, there was only one that fell in the 155-185 ppm range given in the textbook, located at around 179 ppm; and so this peak was interpreted as being from the carbonyl carbon. This accounted for all of the carbons in the isopentyl acetate molecule, though the last interpretation was not as conclusive as would be desired.

Overall, it was determined that the experiment was successful in that the desired compound was produced beyond a reasonable doubt. Because an IR spectrum was not taken, the purity of the substance could not be determined even qualitatively, and the lack of a reasonably accurate percent yield calls into question even the amount of substance produced. The quick observation that about 0.3 mL of product was left in the conical vial after spillage, while useful for theorizing why the percent yield is inaccurate, is not certain enough to present more than a very rough idea of what the percent yield should be. However, the H1 NMR, C13 NMR, and C13 DEPT NMR all reflected the structure of isopentyl acetate, with little room for inaccuracy. Furthermore, the scent of the product was most decidedly of bananas, which is consistent with descriptions of the scent of isopentyl acetate.¹⁰ Therefore

it is reasonable to conclude that at least some isopentyl acetate was formed; and thus the basic goal of the experiment was achieved.

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