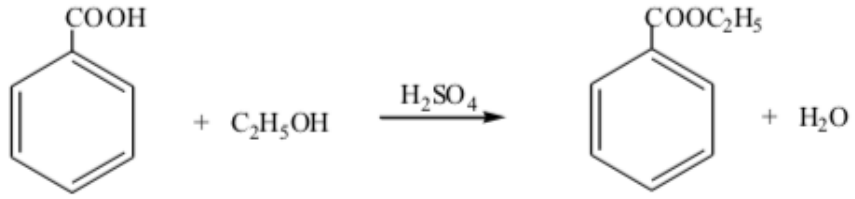
REPORT

Lab work: ETHYL BENZOATE

Student: Dike Moses Ekene

Data: 081836941, HM18 – 06M

The main reaction: Fischer Esterification



Benzoic acid + ethanol → ethyl benzoate + water (H_2SO_4 homogenous catalyst)

Reaction by-product: H_2O

Properties of the raw materials

Formula	Molecular weight (g/mol)	Boiling temperature (°C)	Melting temperature (°C)	Density (g/cm^3)	Notes
$C_7H_6O_2$	122.12	249.2	122	1.27	Benzoic Acid
C_2H_5OH	46.07	78.37	-114.1	0.789	Ethanol
H_2SO_4	98.079	337	10	1.84	Sulfuric Acid (Homogenous Catalyst)
$C_9H_{10}O_2$	150.17	212	-34	1.05	Ethyl Benzoate (Desired Product)
H_2O	18.015	100	0	0.997	Water By-product

The amount of the raw material used in the synthesis

Substance name	Amount of substance according to instructions			Amount of substance according to task			Conversion factor
	mole	g	ml	mole	g	ml	
Benzoic acid	0.1	12.2	9.6	0.208	25.4	20	1
Ethyl alcohol (95%)	0.6	27.61	35	0.39	18.15	23	1
Sulfuric acid	0.08	7.36	4	0.08	7.73	4.2	-
Diethyl ether	-	-	-	0.04	6.3	6	1
Calcium chloride	-	1.5	-	-	-	-	1

Theoretical yield:

Molarity condition	Benzoic Acid	Ethanol	Ethyl Benzoate
Before reaction	0.208 mol	0.394 mol	-
During reaction	0.208 mol	0.208 mol	0.208 mol
After reaction	-	0.186 mol	0.208 mol

Theoritical yield (g) = $n_{\text{ethyl benzoate}} \times MW_{\text{ethyl benzoate}}$ = 0.208 mol × 150.17 g/mol = 31.235 g

Safety:

1. When performing this synthesis, safety measures are observed when working with flammable and explosive substances, concentrated acids.

2. Care must be taken when extracting ethyl benzoate from the reaction mixture with ether, as the ether is flammable and forms explosive mixtures with air (1.2-51% by volume).

3. When working with ether, make sure that it does not contain impurities. Otherwise, during the distillation of the ether, explosions are possible. To detect peroxides, several millilitres of ether are shaken with an equal amount of a 2% solution of potassium iodide acidified with hydrochloric acid. In the presence of peroxides, the ethereal layer is coloured brown. Purified from peroxides by shaking with a solution of ferrous sulphate.

4. It is strictly forbidden to heat ether on open fire and tiles. Heating can be done only on a pre-heated water bath in a flask equipped with a water cooler. It should be remembered that pairs of ether are heavier than air and are spreading along the table, which can lead to fire and explosion if there is an open fire. At the strait of the ether, it is necessary to turn off electrical appliances, fill the air with sand and collect in an open container.

5. If you get a drop of sulfuric acid on the skin causing chemical burns, the affected area should be washed immediately with a copious stream of water, then with a solution of soda and again with water.

6. Benzoic acid irritates the skin, so if it gets on your hands rinse with plenty of warm water and soap.

8. The work should be carried out in a dressing gown and gloves. When working with concentrated sulfuric acid, wear protective glasses. **First aid:** After contact with skin, wash

immediately with a strong stream of water, followed by a 5% solution of soda. When irritating the mucous membrane of the respiratory tract - fresh air, inhalation of soda solution, warm milk with soda, peace, medical help.

Device image:

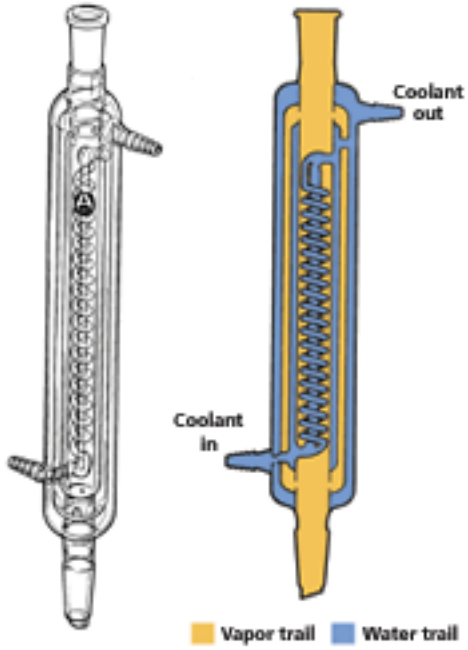


Figure 1 – Reverse condenser

Figure 2 – Laboratory electric heating mantle

Description of steps of synthesis:

Measure out 20 ml of benzoic acid, measure out 23 ml of 95 % ethanol using a measruing cylinder. Transfer the 20 ml benzioic acid to a round-bottom flask of 150 ml capacity, add the 23 ml 95 % ethanol to the round-bottom flask. Slowly add 4.2 ml sulfuric acid to the contents of the round-bottom flask, while shaking the flask gently. Add 3 pieces of boiling chips. Connect the round-bottom flask to a water-cooled reflux condenser and place on a distillation unit with an electric heater. Activate the heater and continue heating for 3 hours, after which, heating is discontinued. Ensure that the temperature does not exceed 112 ^{0}C.

At the end of the heating, replace the refrigerator with a straight-water with distillation adaptor, the end of which is lowered into the receiver, and drive away the excess ethyl alcohol in a water bath. The residue in the reaction flask is cooled and poured into a glass with 60 ml of cold water. Add small portions of sodium carbonate until the medium becomes slightly alkaline.

The ethyl benzoate released in the form of oil is extracted with diethyl ether twice by 30 ml. the resulting ether extract is dried over anhydrous calcium chloride. Distilled off the ether on a water bath and ne-regnaut aniline, replacing water refrigerator air, collecting the fraction.

Properties of the obtained substances:

	Boiling point	Melting point	n^{D}_{20}	Yield	
				g	%
Literary	212	-34	0.208 mol	31.235	71.73
Actual	212	-34	0.042 mol	6.3	20.17

Answers to questions

1. What first aid should be given to the victim if splashes of sulfuric acid hit him on the skin or in the eye?

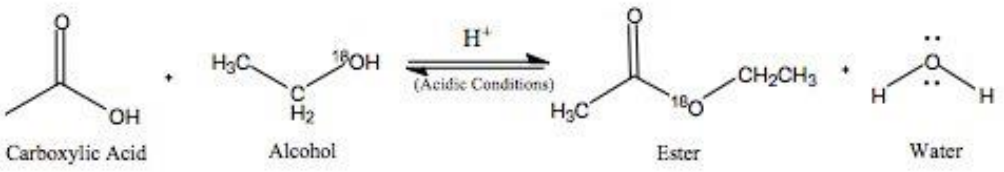
The most effective action to take is to try and flood the burn with water to disperse the chemical and stop the burning, it said. Bottled water is fine for this if it is the only thing to

hand. Try and douse the burn with water for at least 20 minutes, ensuring that no contaminated puddles are allowed to collect under the victim. While flooding the injury, try and gently remove any clothing with the substance on it and call an ambulance as soon as possible, while checking the casualty is still breathing and responsive.

If the substance has entered the victim's eyes, hold their eye under gently running cold water for at least 10 minutes, thoroughly irrigating the eyelid both inside and out, said the first aid experts. Do not allow the casualty to touch the injured eye, as they may have acid on their hands, and do not forcibly remove a contact lens. Make sure that contaminated water does not splash the uninjured eye,said the advice, adding someone helping should ask the casualty to hold a clean, non-fluffy pad over the injured eye, and arrange to send them to hospital.

2. What is the esterification reaction? Why is a drop of sulfuric acid added to the reaction mixture?

When a carboxylic acid is treated with an alcohol and an acid catalyst, an ester is formed (along with water). This reaction is called the Fischer esterification.



Reaction of carboxylic acid with alcohol to make an ester

The reaction is actually an equilibrium. The alcohol is generally used as solvent so is present in large excess. Many different acids can be used; it's common to see just "H+", although H2SO4 (sulfuric acid) and TsOH (tosic acid) are also often used.

Carboxylic acids and alcohols are often warmed together in the presence of a few drops of concentrated sulfuric acid to observe the smell of the esters formed. Because the reactions are slow and reversible, you don't get a lot of ester produced in this time.

3. Why when esterification of benzoic acid is used significant-six-fold-excess alcohol in relation to benzoic acid?

The ethanol serves as a reactant to form the ethyl ester. The alcohol is also generally used as solvent so is present in large excess.

4. What is the principle of extraction based on? Why ethylbenzoic EXT-reginout of water air?

Solvent extraction is a method used to separate compounds or metal complexes, based on their relative solubilities in two different immiscible liquids, usually polar and a non-polar solvent. There is a net transfer of one or more species from one liquid into another liquid phase. The transfer is driven by chemical potential, i.e. once the transfer is complete, the overall system of protons and electrons that make up the solutes and the solvents are in a more stable configuration (lower free energy). The solvent that is enriched in the solute(s) is called extract, while the feed solution that is depleted in solute(s) is called the raffinate.

5. With what refrigerator (air or water) distilled ether, ethyl alcohol? Why?

Water is used as cooling agent in the refrigerating process step of ethyl alcohol. Due to The overall heat transfer coefficient of water cooling refrigerator is typically 500-900 $W/m^2.K$, which is significantly higher than that of air cooling (typically around 100 $W/m^2.K$), water is preferably to be used to increase the efficiency of distillation for this process. On the other hand, using water as refrigerant offers high controllability the heating reflux to keep the condensation process of the solution stable.

6. Why in the distillation of ethyl ester of benzoic acid using air cooler?

To prevent the miscible components into water by using water as refrigerant. On the other hand, air cooler offers gentler cooling steps compare to water as the heat transfer coefficient is lower than water. However, the main consideration is to avoid any additional mixture of water into the solution mixtures.

7. What is the purpose in the ether extract ethyl benzoate add anhydrous calcium chloride? What are the dehumidifiers in addition to calcium chloride, you know? Given example

Anhydrous calcium chloride in this particular step works as drying agent over the solution as well as extractor of ether to separate from the desired product of ethyl benzoate. Uniquely, the behaviour of the anhydrous calcium chloride is that the drying agent behave as hygroscopic, which absorb and hold the water presence from the outer factors. Addition dehumidifier could be purposed to minimized the remaining humidity by means of desiccant which available in many options such as calcium oxide, calcium sulphate, silica gel, etc.