بسم الله الرحمن الرحيم

AECENAR
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region
www.aecenar.com

MEGBI - Middle East Genetics and Biotechnology Institute
مركز أبحاث للجينات والتقنية البيولوجية
http://aecenar.com/institutes/megbi

GREEN CHEMISTRY

MEGBI REPORT 2024

Chemicals Production for Aspirin Production Plant

Editors:

Maryam El Rez, Dr. Samir Mourad

With the contributions of:

Muhamad Kalaoun

Abdullah Mourad

Nour Koulayb

Ahmad Jawhar

Ali Foul

Rawan Abdelmajid

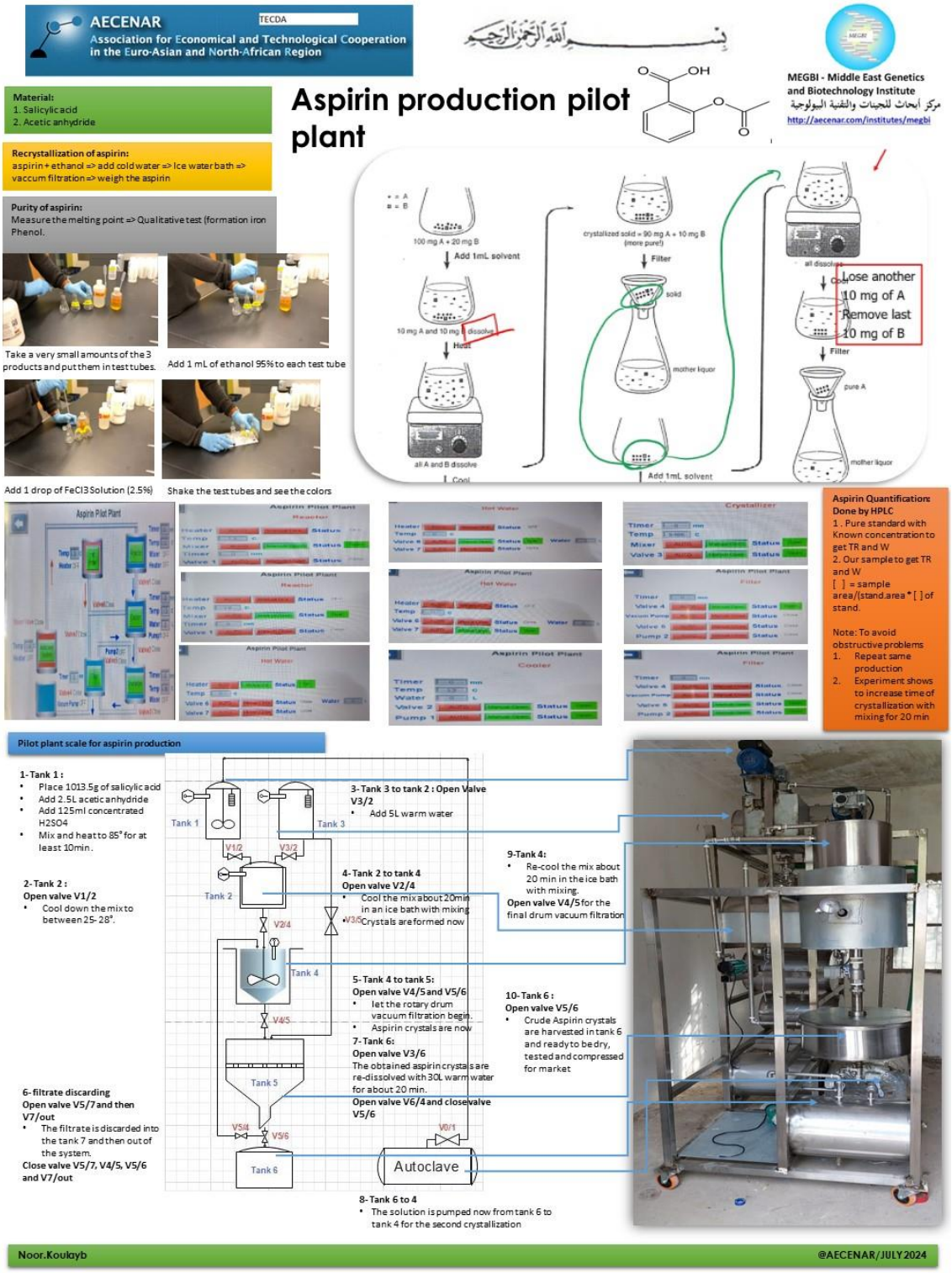
Last Update: 24.03.2025 15:17

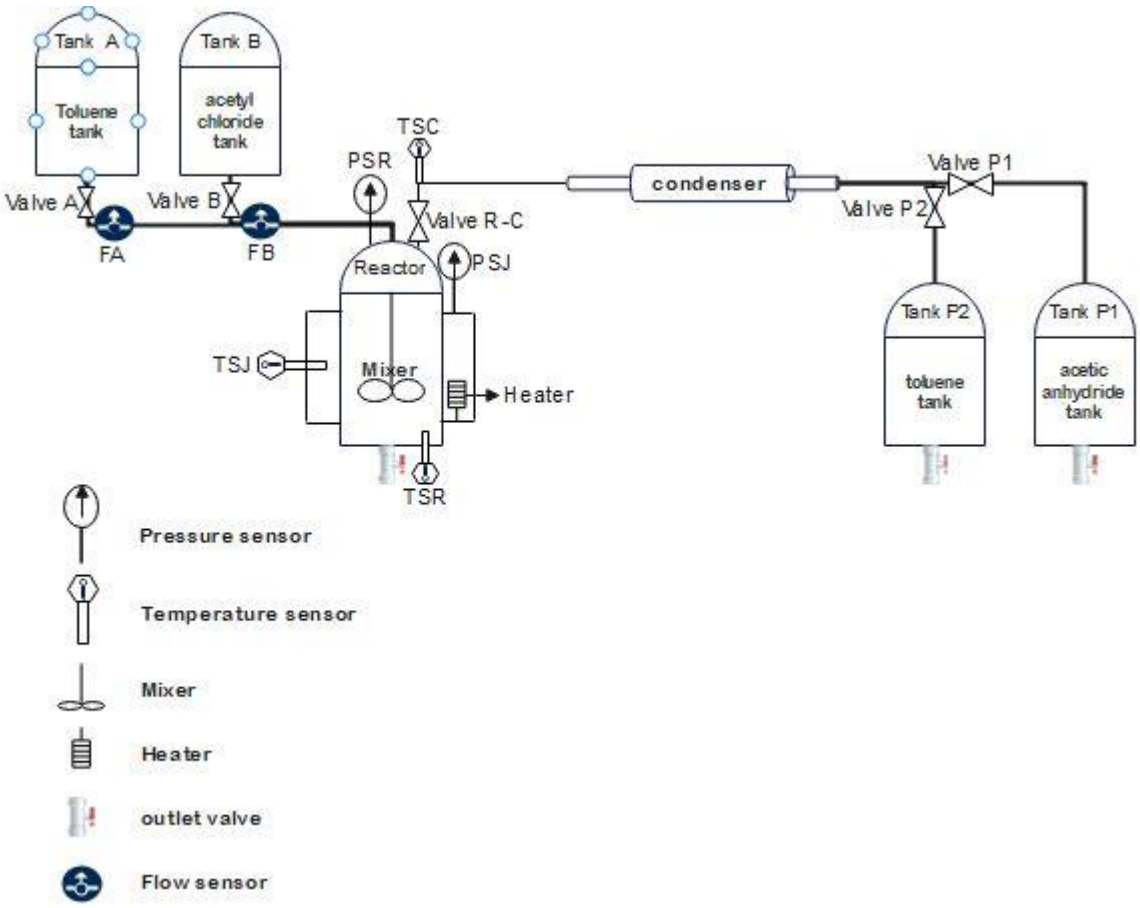
Table of Contents

1 Summary

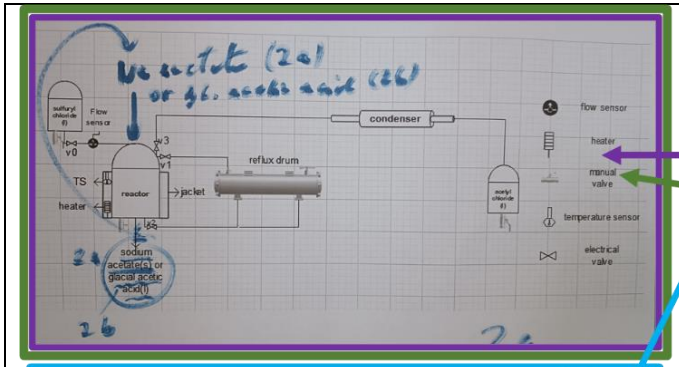
1.1 Aspirin Production Plant

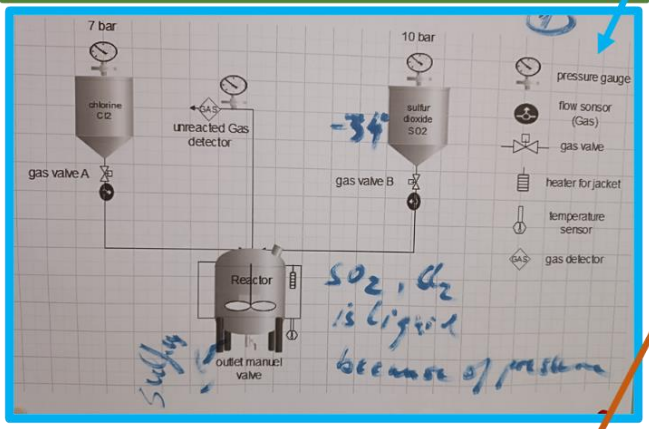
To produce Aspirin, the two raw materials salicylic acid and acetic anhydride are needed. In 2024 all pathways where worked out to produce acetic anhydride from basic, available materials. Also an acetic anhydride pilot plant was designed and the devices were realized.

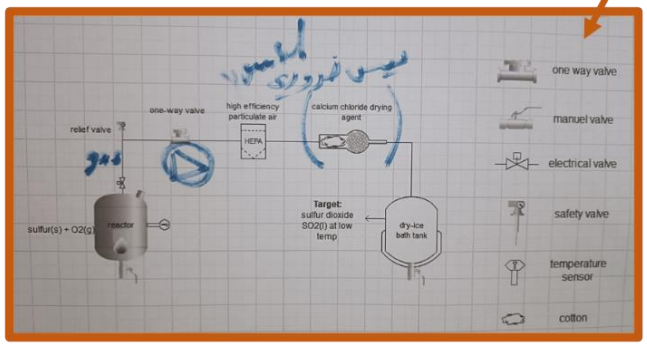


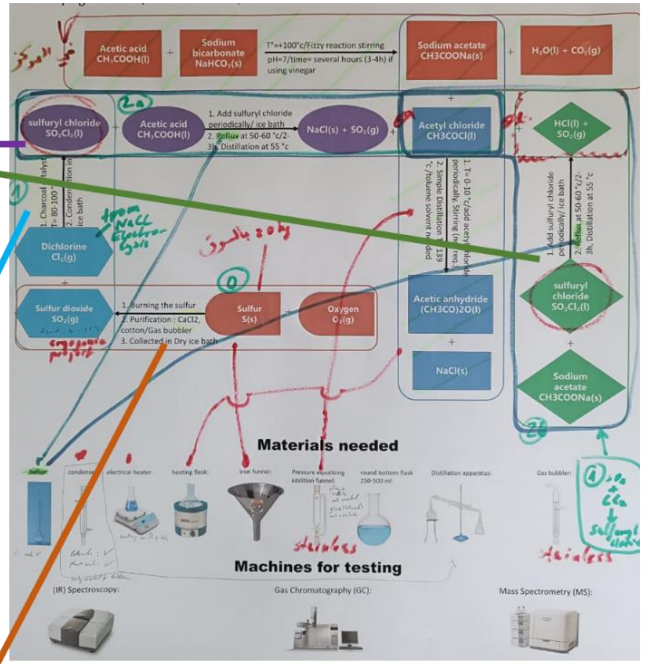

Tank A
Toluene tank
Tank B
acetyl chloride tank
Valve A
Valve B
FA
FB
TSC
PSR
Valve R-C
Reactor
PSJ
TSJ
Mixer
Heater
TSR
condenser
Valve P1
Valve P2
Tank P2
toluene tank
Tank P1
acetic anhydride tank
Pressure sensor
Temperature sensor
Mixer
Heater
outlet valve
Flow sensor

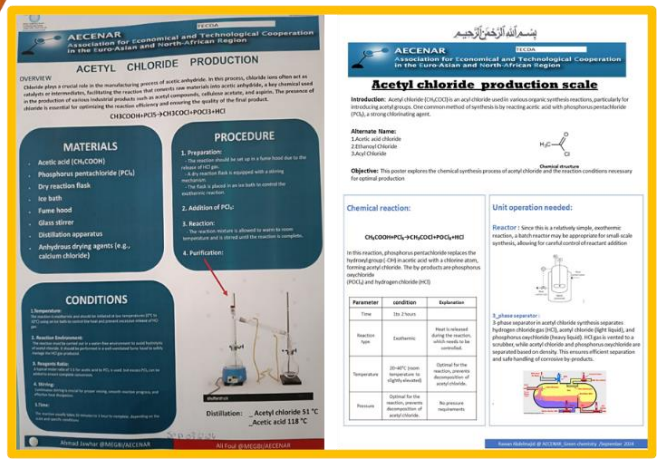
1.2 Overview of reactions to produce acetic anhydride









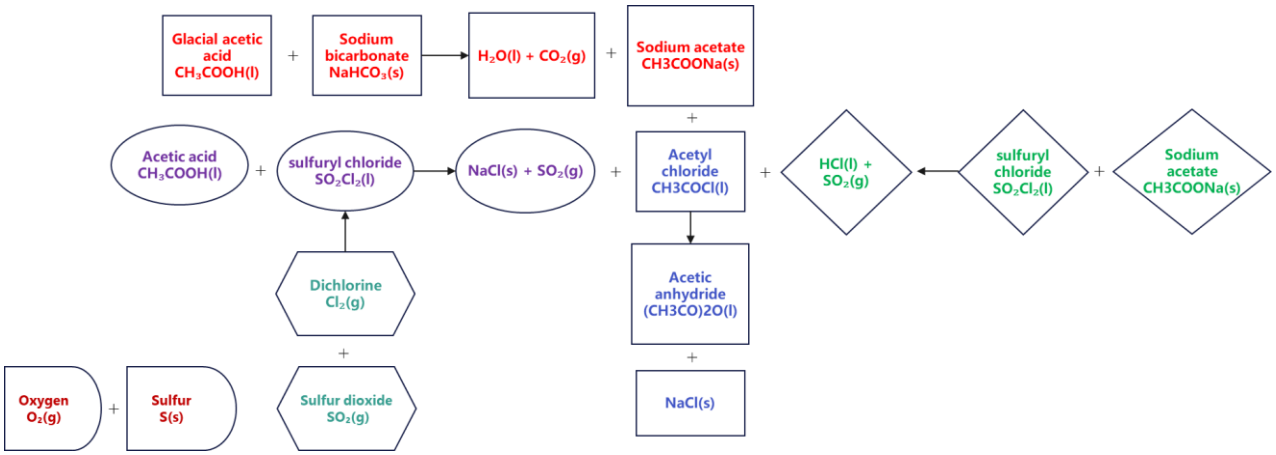


Overview reaction [pptx file:

Overview reactions.pptx

1.3 On aecenar.com site

1.4 Path to produce acetic anhydride (acetyl chloride with sulfur path)



General flow chart [pptx file]:	Drawing2.pptx

For aspirin we need as raw material acetic anhydride. So the goal of is to produce Acetic Anhydride [$(CH_3CO)_2O$] according to the following reaction:

Sodium Acetate + Acetyl Chloride → Acetic Anhydride + Sodium Chloride

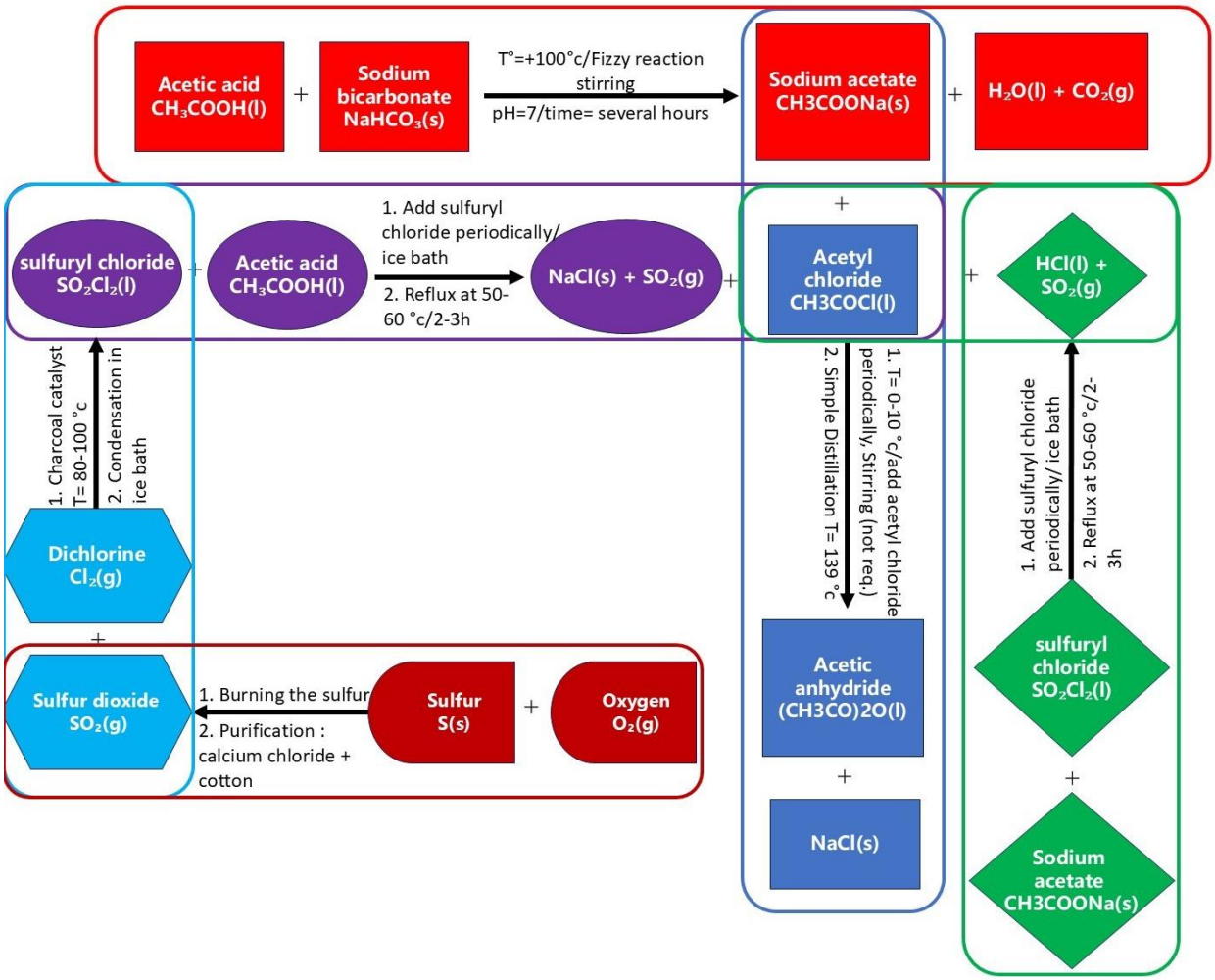
$CH_3COONa_{(s)} + CH_3COCl_{(l)} \rightarrow (CH_3CO)_2O_{(l)} + NaCl_{(s)}$

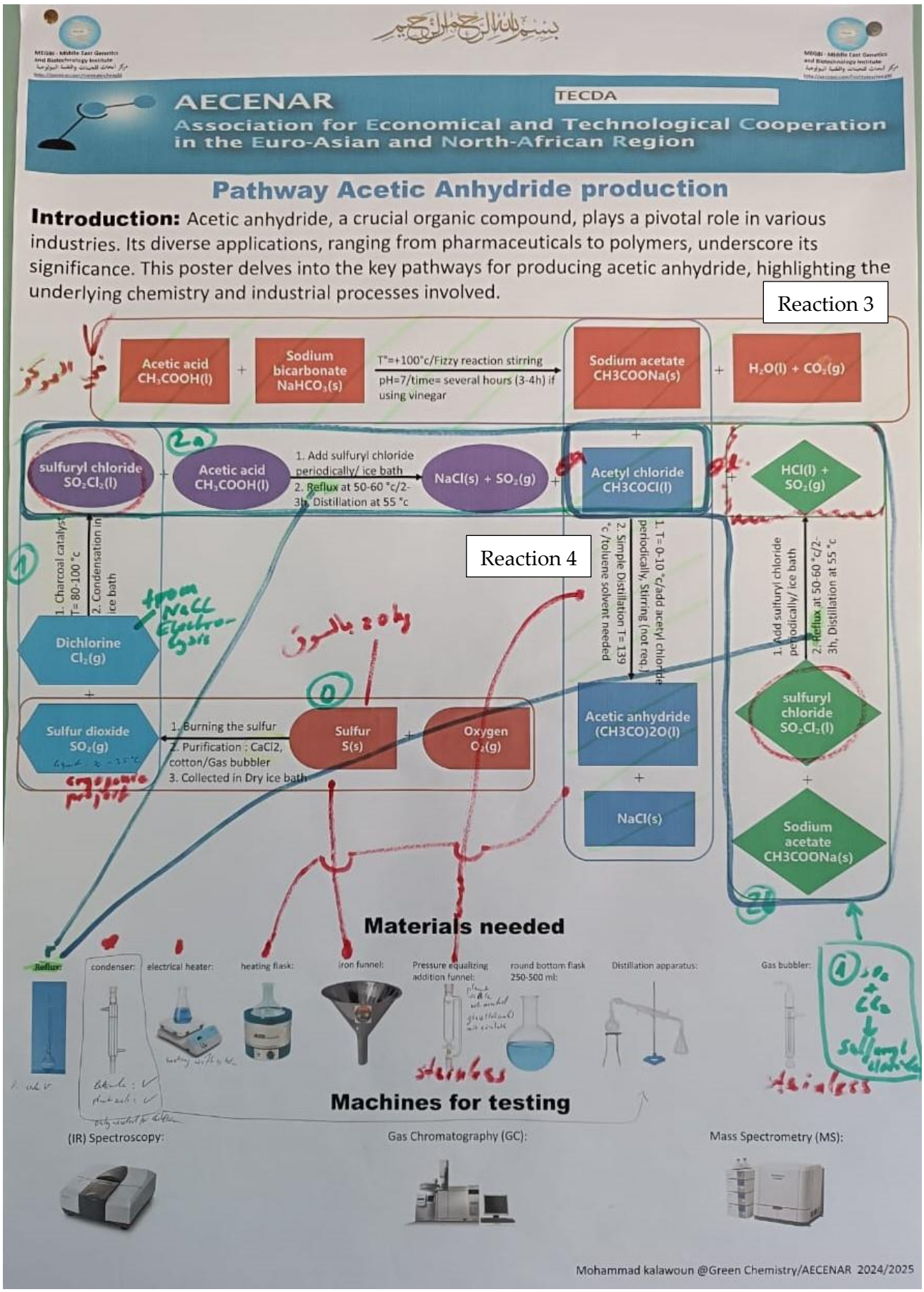
This reaction occurs under the following conditions:

1) T = 0-10℃, Add Acetyl Chloride periodically, Stirring (not req.)
2) Simple distillation at T = 139℃ / Toluene solvent needed

To achieve this reaction, Sodium Acetate and Acetyl Chloride must be obtained.

And therefore we need to produce Acetyl Chloride.

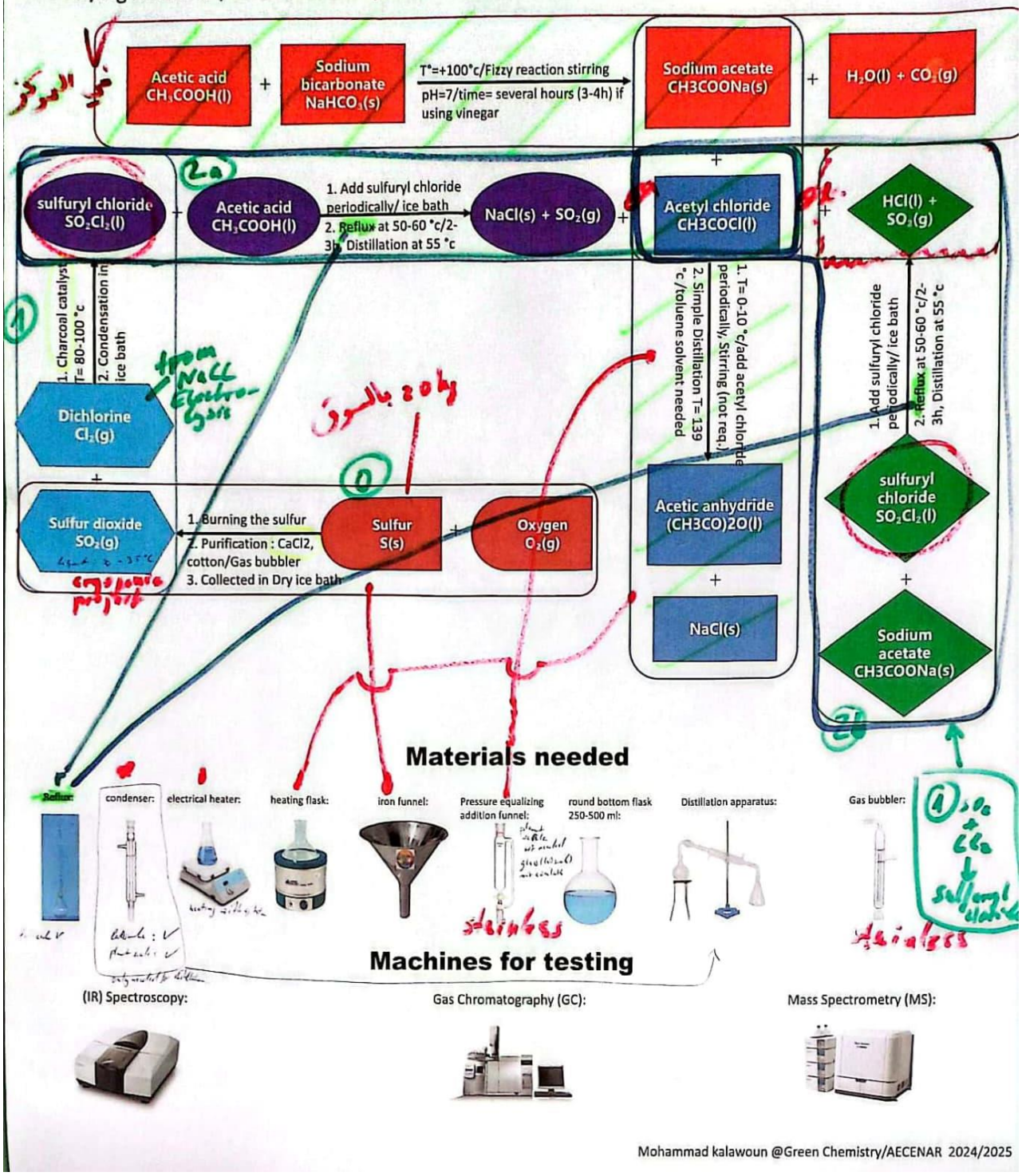

Acetic acid $CH_3COOH(l)$
Sodium bicarbonate $NaHCO_3(s)$
T°=+100°c/Fizzy reaction
stirring
pH=7/time= several hours
Sodium acetate CH3COONa(s)
$H_2O(l) + CO_2(g)$
sulfuryl chloride $SO_2Cl_2(l)$
Acetic acid $CH_3COOH(l)$
1. Add sulfuryl chloride periodically/ ice bath
2. Reflux at 50-60 °c/2-3h
$NaCl(s) + SO_2(g)$
Acetyl chloride CH3COCl(l)
$HCl(l) + SO_2(g)$
1. Charcoal catalyst T= 80-100 °c
2. Condensation in ice bath
Dichlorine $Cl_2(g)$
2. Simple Distillation T= 139 °c
1. T= 0-10 °c/add acetyl chloride periodically, Stirring (not req.)
1. Add sulfuryl chloride periodically/ ice bath
2. Reflux at 50-60 °c/2-3h
Sulfur dioxide $SO_2(g)$
1. Burning the sulfur
2. Purification : calcium chloride + cotton
Sulfur S(s)
Oxygen $O_2(g)$
Acetic anhydride $(CH3CO)2O(l)$
sulfuryl chloride $SO_2Cl_2(l)$
NaCl(s)
Sodium acetate CH3COONa(s)


بسم الله الرحمن الرحيم
AECENAR
TECDA
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region
Pathway Acetic Anhydride production
Introduction: Acetic anhydride, a crucial organic compound, plays a pivotal role in various industries. Its diverse applications, ranging from pharmaceuticals to polymers, underscore its significance. This poster delves into the key pathways for producing acetic anhydride, highlighting the underlying chemistry and industrial processes involved.
Reaction 3
Acetic acid CH3COOH(l)
Sodium bicarbonate NaHCO3(s)
T°=+100°c/Fizzy reaction stirring
pH=7/time= several hours (3-4h) if using vinegar
Sodium acetate CH3COONa(s)
H2O(l) + CO2(g)
sulfuryl chloride SO2Cl2(l)
Acetic acid CH3COOH(l)
1. Add sulfuryl chloride periodically/ ice bath
2. Reflux at 50-60 °c/2-3h, Distillation at 55 °c
NaCl(s) + SO2(g)
Acetyl chloride CH3COCl(l)
HCl(l) + SO2(g)
Reaction 4
1. Charcoal catalyst T= 80-100 °c
2. Condensation in ice bath
Dichlorine Cl2(g)
1. T= 0-10 °c/add acetyl chloride periodically, Stirring (not req.)
2. Simple Distillation T= 139 °c /toluene solvent needed
1. Add sulfuryl chloride periodically/ ice bath
2. Reflux at 50-60 °c/2-3h, Distillation at 55 °c
Sulfur dioxide SO2(g)
1. Burning the sulfur
2. Purification : CaCl2, cotton/Gas bubbler
3. Collected in Dry ice bath
Sulfur S(s)
Oxygen O2(g)
Acetic anhydride (CH3CO)2O(l)
NaCl(s)
sulfuryl chloride SO2Cl2(l)
Sodium acetate CH3COONa(s)
Materials needed
Reflux:
condenser:
electrical heater:
heating flask:
iron funnel:
Pressure equalizing addition funnel:
round bottom flask 250-500 ml:
Distillation apparatus:
Gas bubbler:
Machines for testing
(IR) Spectroscopy:
Gas Chromatography (GC):
Mass Spectrometry (MS):
Mohammad kalawoun @Green Chemistry/AECENAR 2024/2025

AECENAR
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region
TECDA

Pathway Acetic Anhydride production

Introduction: Acetic anhydride, a crucial organic compound, plays a pivotal role in various industries. Its diverse applications, ranging from pharmaceuticals to polymers, underscore its significance. This poster delves into the key pathways for producing acetic anhydride, highlighting the underlying chemistry and industrial processes involved.



Mohammad kalawoun @Green Chemistry/AECENAR 2024/2025

Dichlorine can we get from NaCl electrolysis

1.4.1 Dichlorine (Cl_2) production

Dichlorine (Cl_2) can be obtained from the NaCl electrolysis

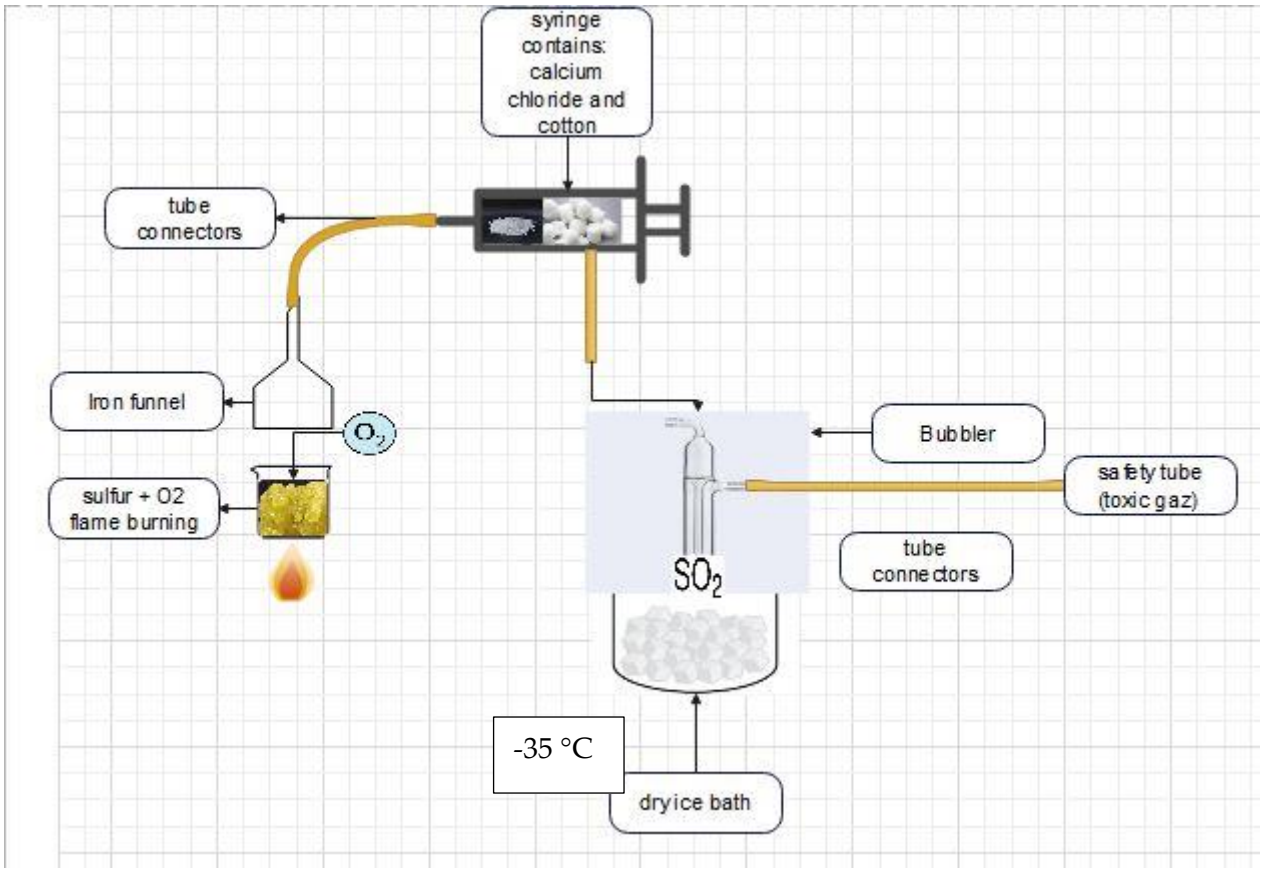
1.4.2 Sulfur dioxide production (reaction 0)

Sulfur Dioxide can produced by the following reaction:

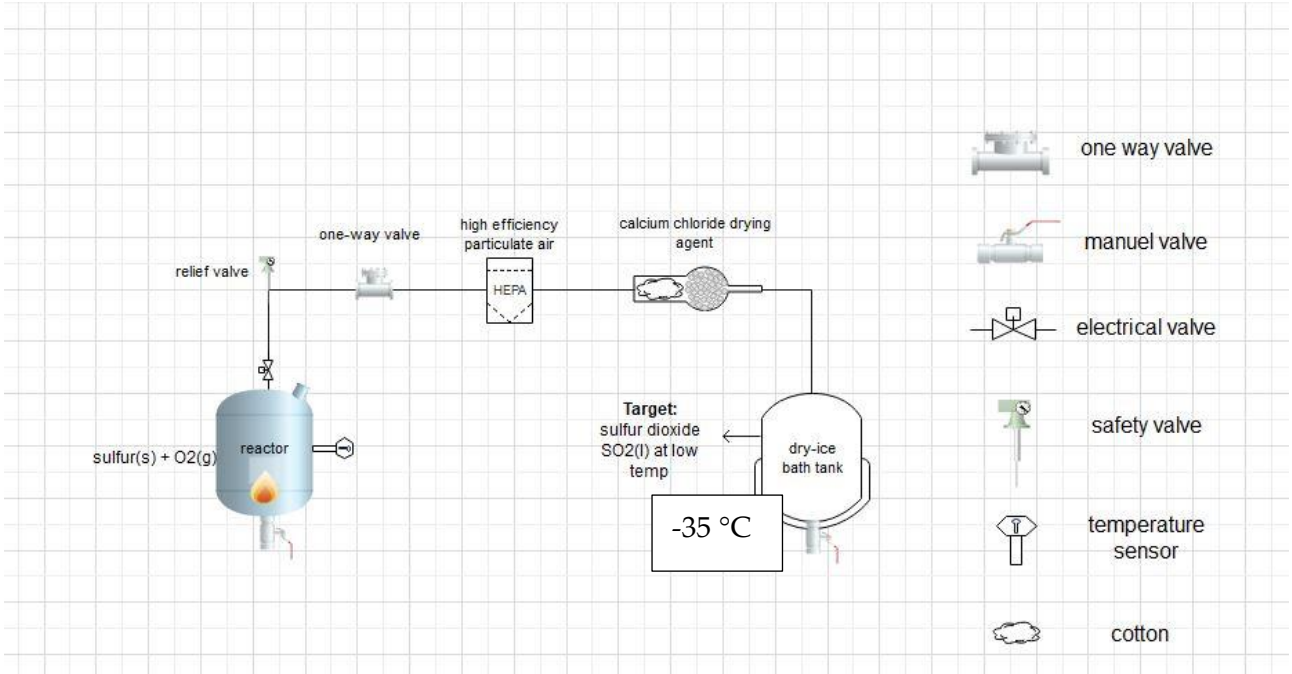
$$\text{Sulfur} + \text{Oxygen} \rightarrow \text{Sulfur Dioxide}$$

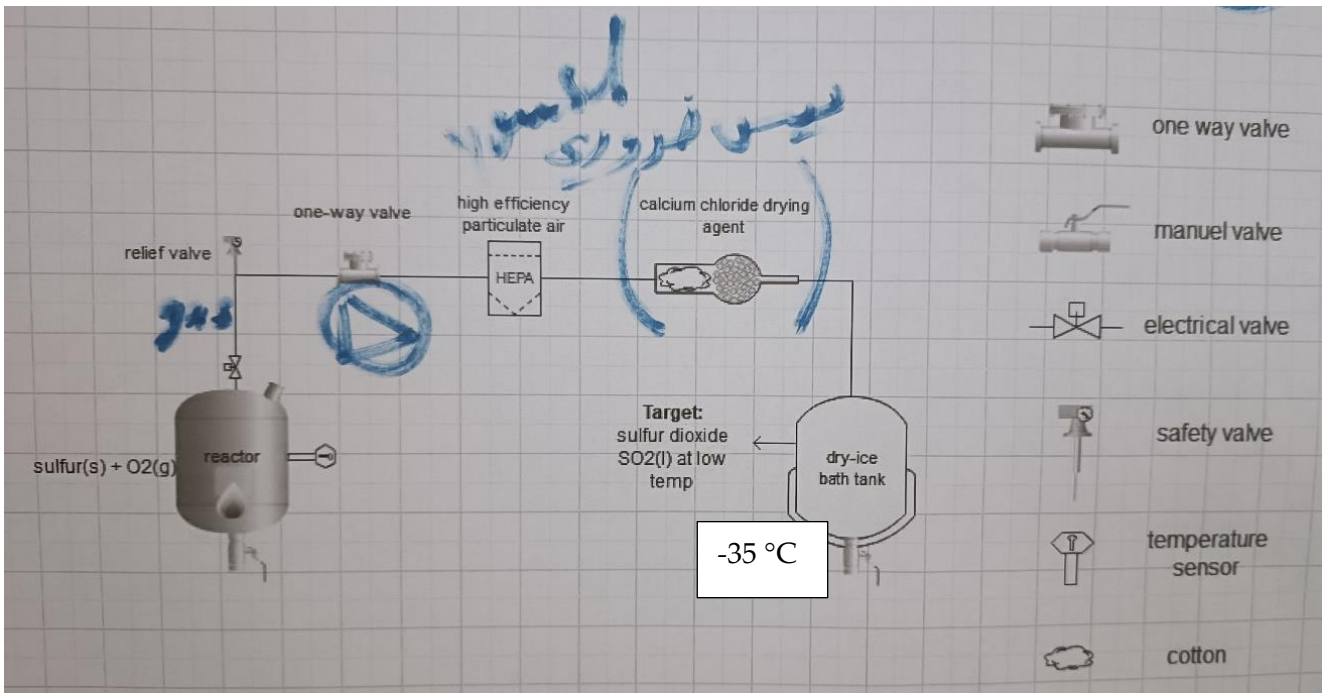
$$S_{(s)} + O_{2(g)} \rightarrow SO_{2(g)}$$

1.4.2.1 Lab Scale



1.4.2.2 Pilot Plant Scale





This reaction occurs under the following conditions:

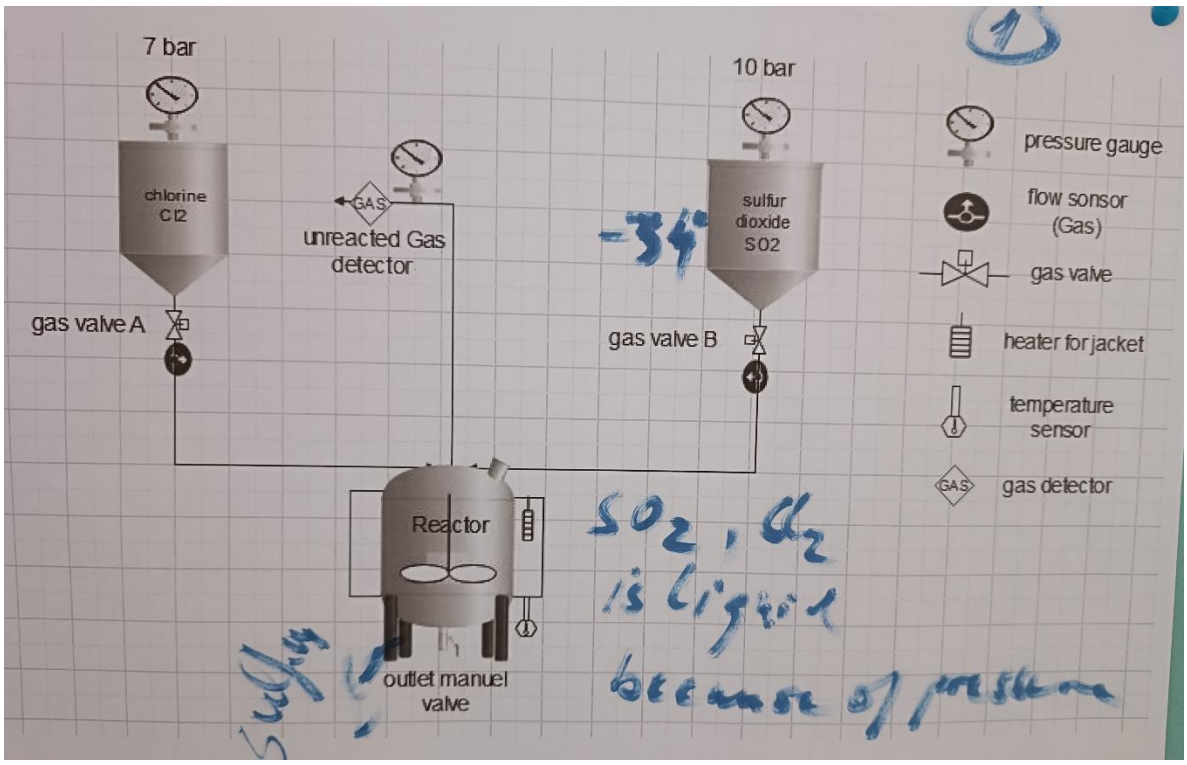
1) Burning the Sulfur
2) Purification with $CaCl_2$ cotton / Gas bubbler
3) Collected in Dry ice bath

Note: SO_2 liquefied at -35℃ [Cryogenic project]

1.4.3 Sulfuryl chloride ($SO_2Cl_{2(l)}$) production (reaction 1)

Sulfur dioxide + Dichlorine → Sulfuryl Chloride

$SO_{2(g)} + Cl_{2(g)} \rightarrow SO_2Cl_{2(l)}$



This reaction occurs under the following conditions:

1) Charcoal catalyst T = 80-100°C
2) Condensation in an ice bath

1.4.4 Acetyl chloride production (reaction 2a and 2b)

We can obtain Acetyl Chloride [CH_3COCl] can be obtained in the following two ways:

- **1st way (reaction 2a)**, Through the following reaction:

Sulfuryl Chloride + Acetic Acid → Sodium Chloride + Acetyl Chloride

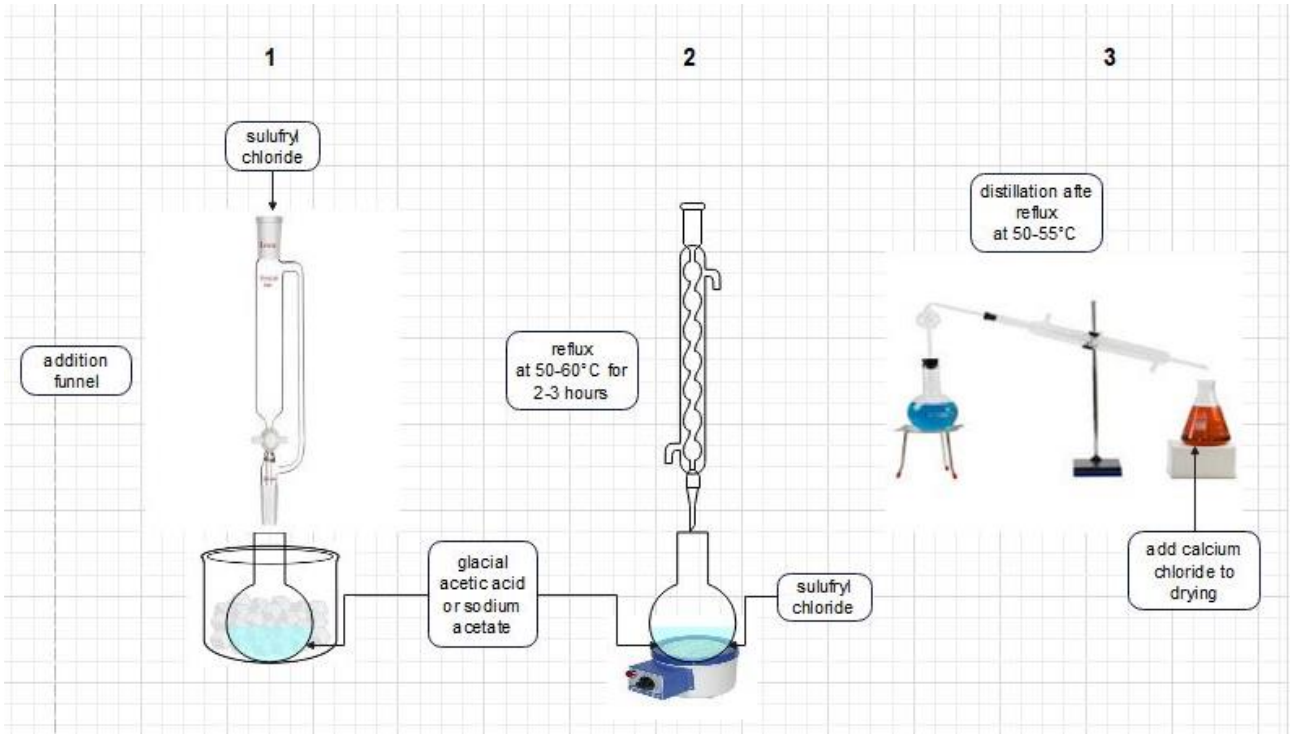
$$SO_2Cl_{2(l)} + CH_3COOH_{(l)} \rightarrow SO_{2(g)} + CH_3COCl_{(l)}$$

- **2nd way (reaction 2b)**, Through the following reaction:

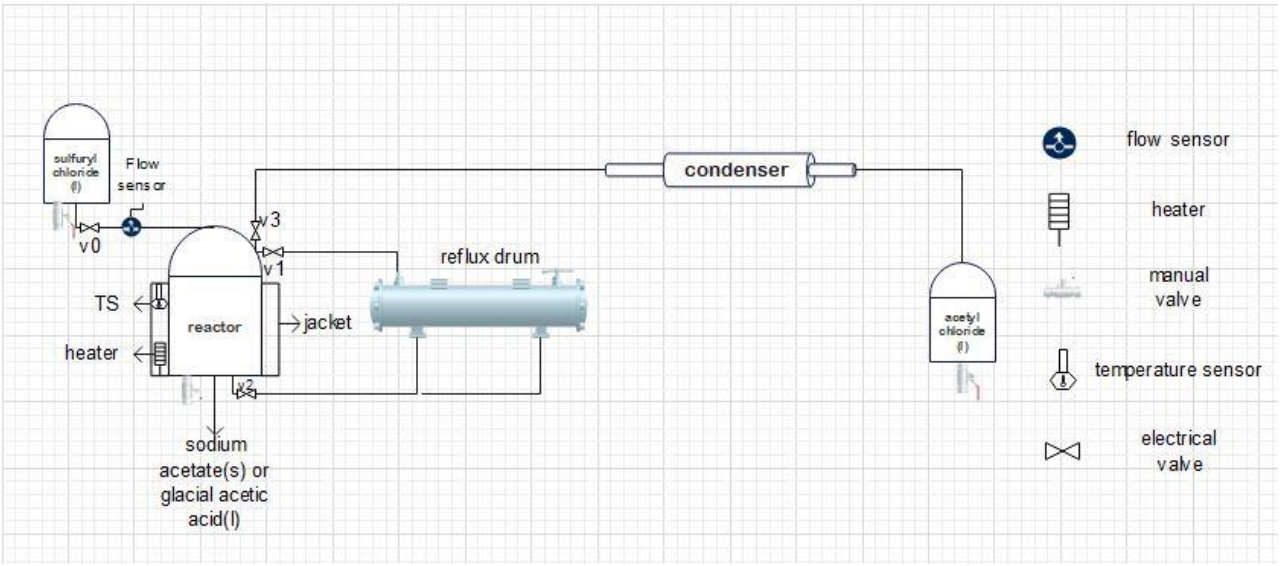
Sodium Acetate + Sulfuryl Chloride → Hydrochloric Acid + Acetyl Chloride + Sulfur Dioxide

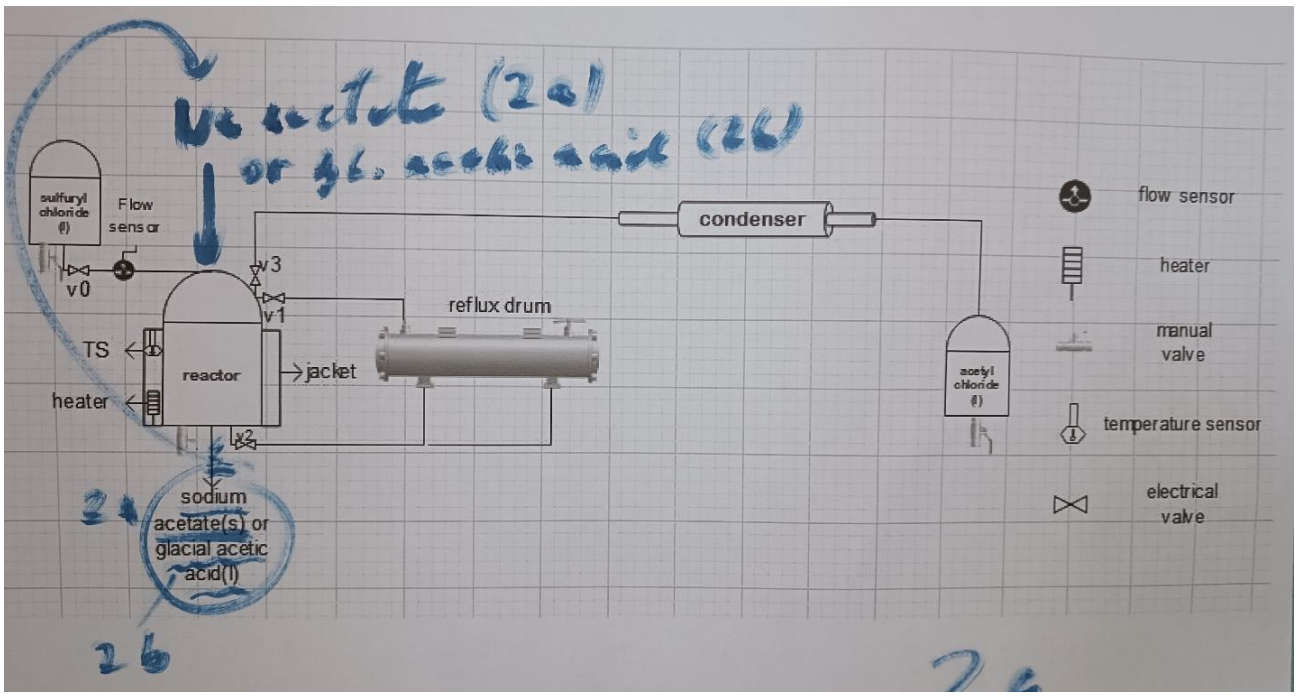
$$CH_3COONa_{(s)} + SO_2Cl_{2(l)} \rightarrow HCl_{(l)} + CH_3COCl_{(l)} + SO_{2(g)}$$

1.4.4.1 Lab Scale



1.4.4.2 Pilot Plant Scale

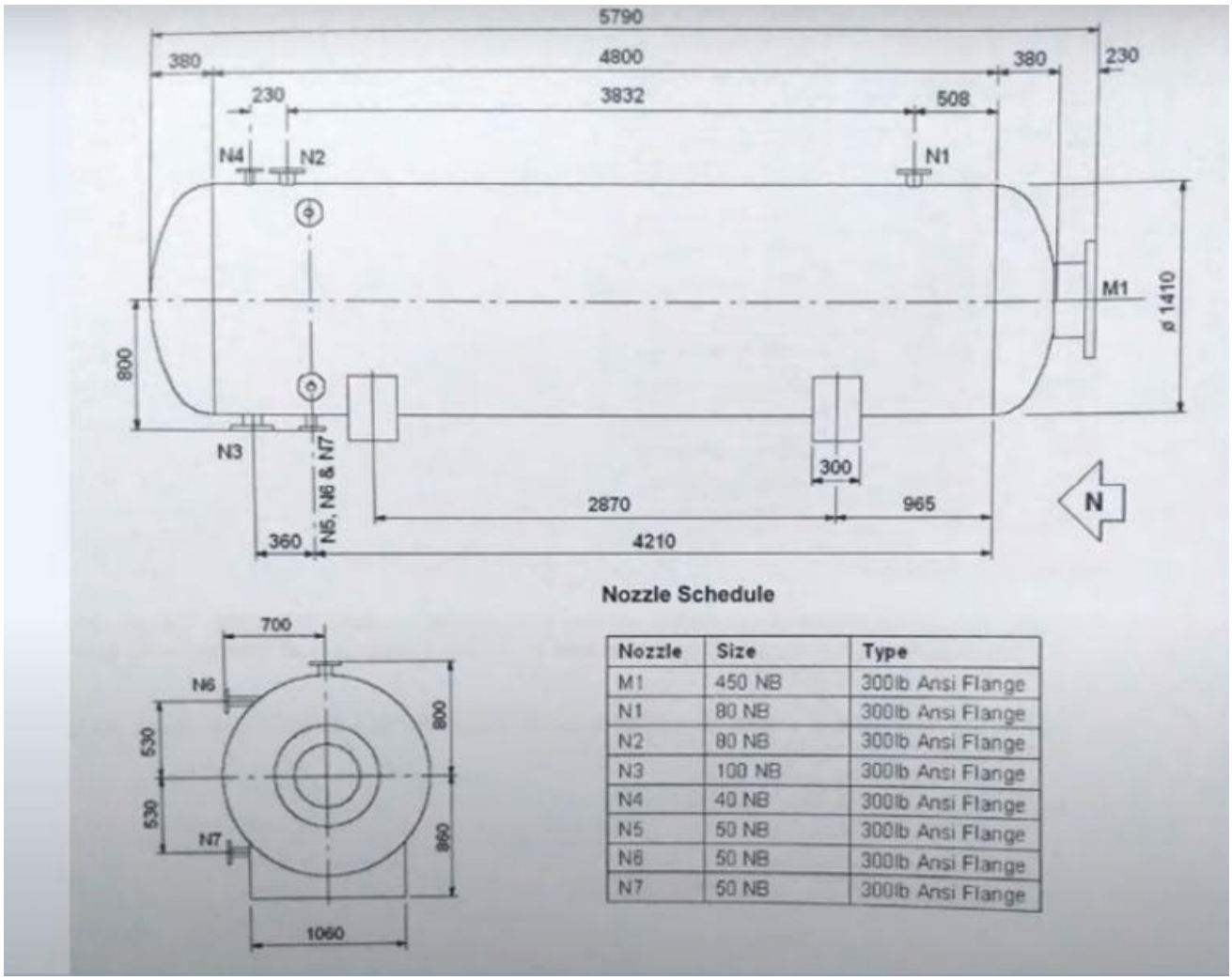




This reaction occurs under the following conditions:

1) Add Sulfryl Chloride periodically / ice bath
2) Reflux at 50-60°C, time: 2-3 hrs with distillation at 55°C

Reflux Drum:



Nozzle Schedule

Nozzle	Size	Type
M1	450 NB	300lb Ansi Flange
N1	80 NB	300lb Ansi Flange
N2	80 NB	300lb Ansi Flange
N3	100 NB	300lb Ansi Flange
N4	40 NB	300lb Ansi Flange
N5	50 NB	300lb Ansi Flange
N6	50 NB	300lb Ansi Flange
N7	50 NB	300lb Ansi Flange

1.4.5 Production of sodium acetate (reaction 3)

- Production of Sodium Acetate [CH_3COONa]:

We can obtain Sodium Acetate [CH_3COONa] through the following reaction:

Acetic Acid + Sodium Bicarbonate → Sodium Acetate + Water + Carbon dioxide

$CH_3COOH_{(l)} + NaHCO_{3(s)} \rightarrow CH_3COONa_{(s)} + H_2O_{(l)} + CO_{2(g)}$

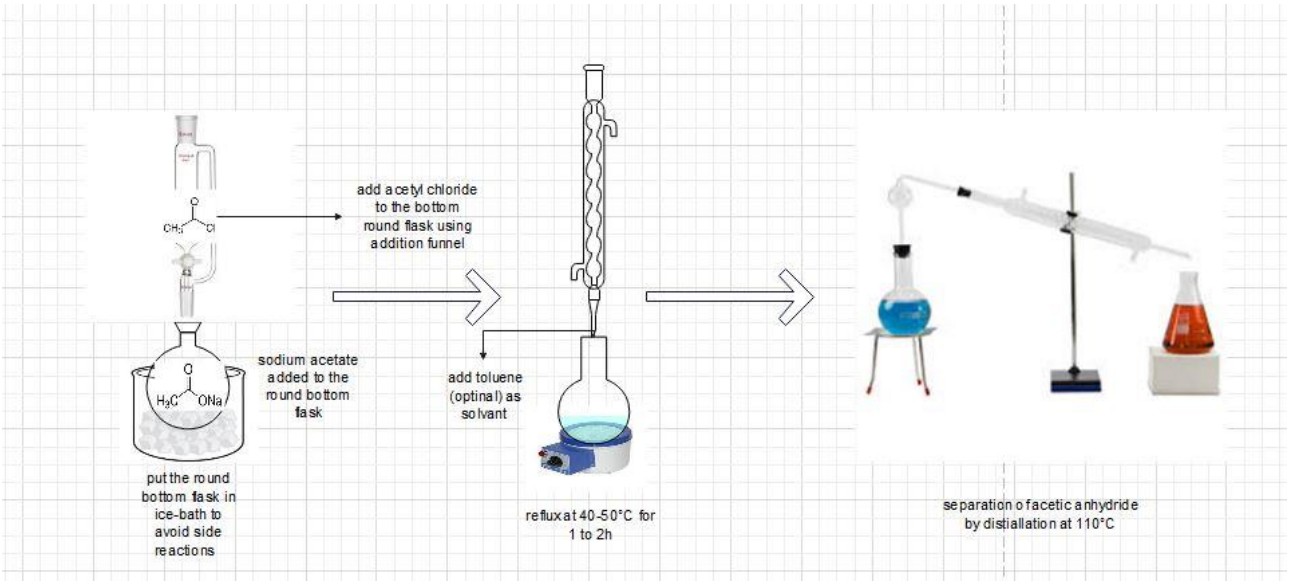
This reaction occurs under the following conditions:

1) T = +100°C, Fizzy reaction Stirring
2) pH=7 / time : several hours(3-4 hrs) if using vinegar

Acetic Acid and Sodium Bicarbonate must be obtained to achieve this reaction, or they are available.

1.4.6 Acetic Anhydride Production (reaction 4)

1.4.6.1 Lab Scale



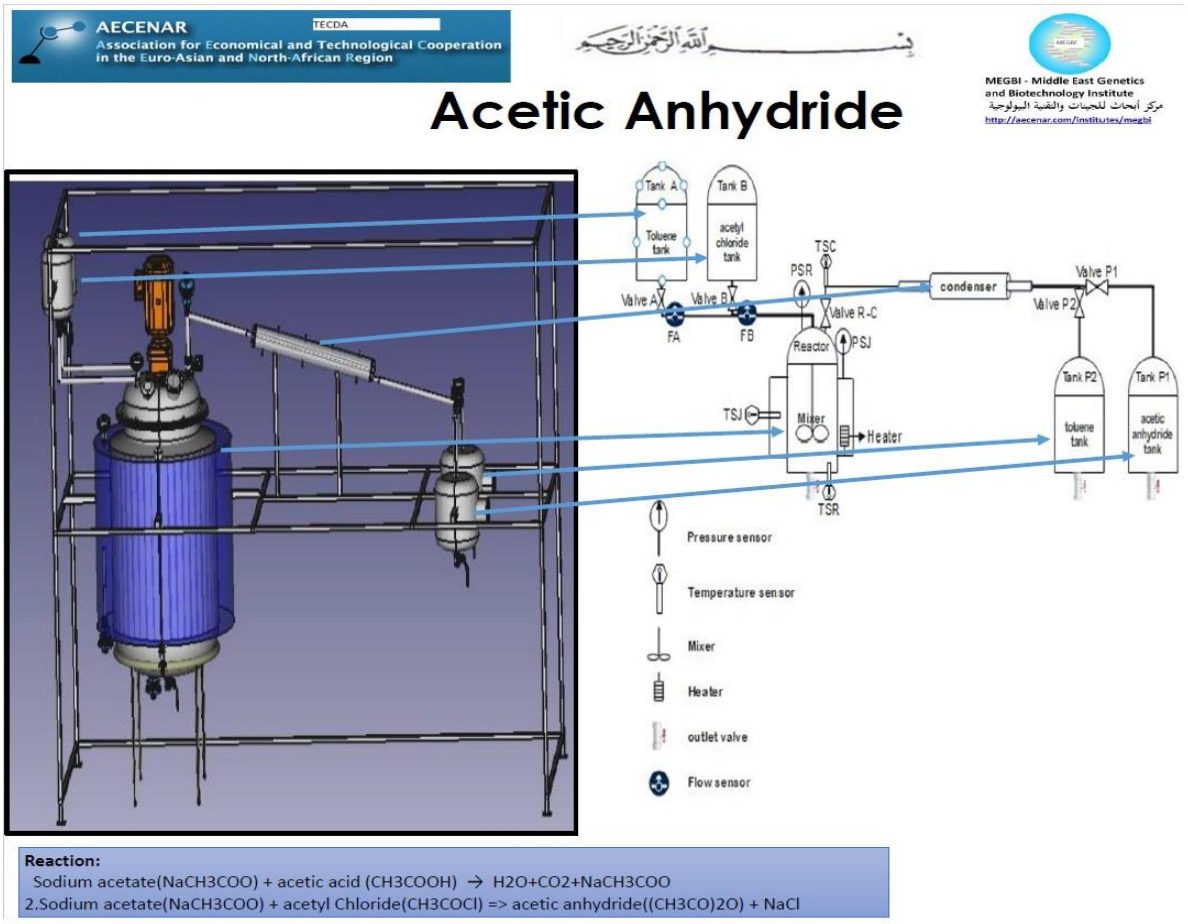
$$\text{Sodium Acetate} + \text{Acetyl Chloride} \rightarrow \text{Acetic Anhydride} + \text{Sodium Chloride}$$

$$CH_3COONa_{(s)} + CH_3COCl_{(l)} \rightarrow (CH_3CO)_2O_{(l)} + NaCl_{(s)}$$

This reaction occurs under the following conditions:

1) T = 0-10°C, Add Acetyl Chloride periodically, Stirring (not req.)
2) Simple distillation at T = 139°C / Toluene solvent needed

1.4.6.2 Pilot plant scale



1.5 Acetyl chloride with phosphor path (lab scale and pilot plant scale)

Acetic Acid + Phosphorus pentachloride → Acetyl Chloride + Phosphoryl Chloride + Hydrogen Chloride

$$CH_3COOH_{(l)} + PCl_{5(s)} \rightarrow CH_3COCl_{(l)} + POCl_{3(l)} + HCl_{(g)}$$

1.5.1 Procedure

1.5.1.1 Preparation:

- The reaction should be set up in a fume hood due to the release of HCl gas.
- A dry reaction flask is equipped with a stirring mechanism.
- The flask is placed in an ice bath to control the exothermic reaction.

1) Addition of PCl5
2) Reaction: The reaction mixture is allowed to warm to room temperature and is stirred until the reaction is complete.
3) Purification: By distillation, Acetyl chloride (at T = 51°C) and Acetic Acid (at T = 118°C).

1.5.1.2 Reaction Conditions

This reaction occurs under the following conditions:

1) Temperature: The reaction is exothermic and should be initiated at low temperature (0°C to 10°C) using an ice bath to control the heat and prevent excessive release of HCl gas
2) Reaction environment: To avoid hydrolysis of acetyl chloride, the reaction must be carried out in a water-free environment. To safely manage the HCl gas produced, it should be performed in a well-ventilated fume hood.
3) Reagents ratio: A typical molar ratio of 1:1 for Acetic Acid to PCl_5 is used, but excess PCl5 can be added to ensure complete conversion.
4) Stirring: Continuous stirring is crucial for proper mixing, smooth reaction progress, and effective heat dissipation.
5) Time: The reaction usually takes 30 minutes to 1 hour to complete, depending on the scale and specific conditions.

TECDA

AECENAR
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region

ACETYL CHLORIDE PRODUCTION

OVERVIEW

Chloride plays a crucial role in the manufacturing process of acetic anhydride. In this process, chloride ions often act as catalysts or intermediates, facilitating the reaction that converts raw materials into acetic anhydride, a key chemical used in the production of various industrial products such as acetyl compounds, cellulose acetate, and aspirin. The presence of chloride is essential for optimizing the reaction efficiency and ensuring the quality of the final product.

$$CH_3COOH + PCl_5 \rightarrow CH_3COCl + POCl_3 + HCl$$

MATERIALS

- Acetic acid (CH_3COOH)
- Phosphorus pentachloride (PCl_5)
- Dry reaction flask
- Ice bath
- Fume hood
- Glass stirrer
- Distillation apparatus
- Anhydrous drying agents (e.g., calcium chloride)

PROCEDURE

1. Preparation:
- The reaction should be set up in a fume hood due to the release of HCl gas.
- A dry reaction flask is equipped with a stirring mechanism.
- The flask is placed in an ice bath to control the exothermic reaction.

2. Addition of PCl_5:

3. Reaction:
- The reaction mixture is allowed to warm to room temperature and is stirred until the reaction is complete.

4. Purification:

CONDITIONS

1.Temperature:
The reaction is exothermic and should be initiated at low temperatures (0°C to 10°C) using an ice bath to control the heat and prevent excessive release of HCl gas.

2. Reaction Environment:
The reaction must be carried out in a water-free environment to avoid hydrolysis of acetyl chloride. It should be performed in a well-ventilated fume hood to safely manage the HCl gas produced.

3. Reagents Ratio:
A typical molar ratio of 1:1 for acetic acid to PCl_5 is used, but excess PCl_5 can be added to ensure complete conversion.

4. Stirring:
Continuous stirring is crucial for proper mixing, smooth reaction progress, and effective heat dissipation.

5.Time:
The reaction usually takes 30 minutes to 1 hour to complete, depending on the scale and specific conditions

Distillation: _ Acetyl chloride 51 °C
_Acetic acid 118 °C

Sep 03/2024

Ahmad Jawhar @MEGBI/AECENAR
Ali Foul @MEGBI/AECENAR

بسم الله الرحمن الرحيم

AECENAR
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region

TECDA

ACETYL CHLORIDE PRODUCTION

OVERVIEW

•**Background**: White phosphorus is a crucial material in various chemical processes, including chemical vapor deposition and semiconductor applications. The need for high purity white phosphorus drives the development of efficient preparation methods.

•**Objective**: To present a method for converting high purity red phosphorus to high purity white phosphorus.

$$CH_3COOH + PCl_5 \rightarrow CH_3COCl + POCl_3 + HCl$$

MATERIALS AND METHODS

Materials:

- High purity red phosphorus (≥ 99% purity)
- Pyrex glass apparatus (bulbs, tubes)
- Heating furnace
- Nitrogen gas
- Whatman #50 filter paper
- Gas-tight syringe
- Oxygen torch

PROCEDURE

1. Preparation:

- The reaction should be set up in a fume hood due to the release of HCl gas.
- A dry reaction flask is equipped with a stirring mechanism.
- The flask is placed in an ice bath to control the exothermic reaction.

2. Addition of PCl_5:

3. Reaction:

- The reaction mixture is allowed to warm to room temperature and is stirred until the reaction is complete.

4. Purification:

Distillation: _ Acetyl chloride 51 °C
_Acetic acid 118 °C

CONDITIONS

1.Temperature:
The reaction is exothermic and should be initiated at low temperatures (0°C to 10°C) using an ice bath to control the heat and prevent excessive release of HCl gas.

2. Reaction Environment:
The reaction must be carried out in a water-free environment to avoid hydrolysis of acetyl chloride. It should be performed in a well-ventilated fume hood to safely manage the HCl gas produced.

3. Reagents Ratio:
A typical molar ratio of 1:1 for acetic acid to PCl_5 is used, but excess PCl_5 can be added to ensure complete conversion.

4. Stirring:
Continuous stirring is crucial for proper mixing, smooth reaction progress, and effective heat dissipation.

5.Time:

The reaction usually takes 30 minutes to 1 hour to complete, depending on the scale and specific conditions

Ahmad Jawhar @MEGBI/AECENAR
Ali Foul @MEGBI/AECENAR

(Poster from Sep 2024)

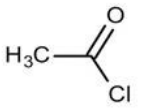
بِسْمِ ٱللَّهِ ٱلرَّحْمَٰنِ ٱلرَّحِيمِ

AECENAR TECDA
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region

Acetyl chloride production scale

Introduction: Acetyl chloride (CH_3COCl) is an acyl chloride used in various organic synthesis reactions, particularly for introducing acetyl groups. One common method of synthesis is by reacting acetic acid with phosphorus pentachloride (PCl_5), a strong chlorinating agent.

Alternate Name:
1.Acetic acid chloride
2.Ethanoyl Chloride
3.Acyl Chloride



Chemical structure

Objective: This poster explores the chemical synthesis process of acetyl chloride and the reaction conditions necessary for optimal production

Chemical reaction:

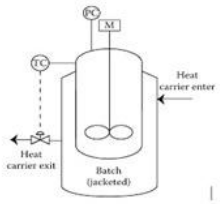
$$CH_3COOH+PCl_5 \rightarrow CH_3COCl+POCl_3+HCl$$

In this reaction, phosphorus pentachloride replaces the hydroxyl group (-OH) in acetic acid with a chlorine atom, forming acetyl chloride. The by-products are phosphorus oxychloride
($POCl_3$) and hydrogen chloride (HCl)

Parameter	condition	Explanation
Time	1to 2 hours	
Reaction type	Exothermic	Heat is released during the reaction, which needs to be controlled.
Temperature	20–40°C (room temperature to slightly elevated)	Optimal for the reaction, prevents decomposition of acetyl chloride.
Pressure	Optimal for the reaction, prevents decomposition of acetyl chloride.	No pressure requirements

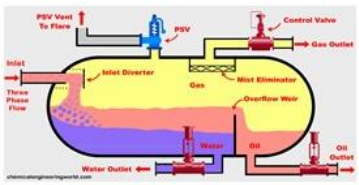
Unit operation needed:

Reactor : Since this is a relatively simple, exothermic reaction, a batch reactor may be appropriate for small-scale synthesis, allowing for careful control of reactant addition



3_phase separator :
3-phase separator in acetyl chloride synthesis separates hydrogen chloride gas (HCl), acetyl chloride (light liquid), and phosphorus oxychloride (heavy liquid). HCl gas is vented to a scrubber, while acetyl chloride and phosphorus oxychloride are separated based on density. This ensures efficient separation and safe handling of corrosive by-products.

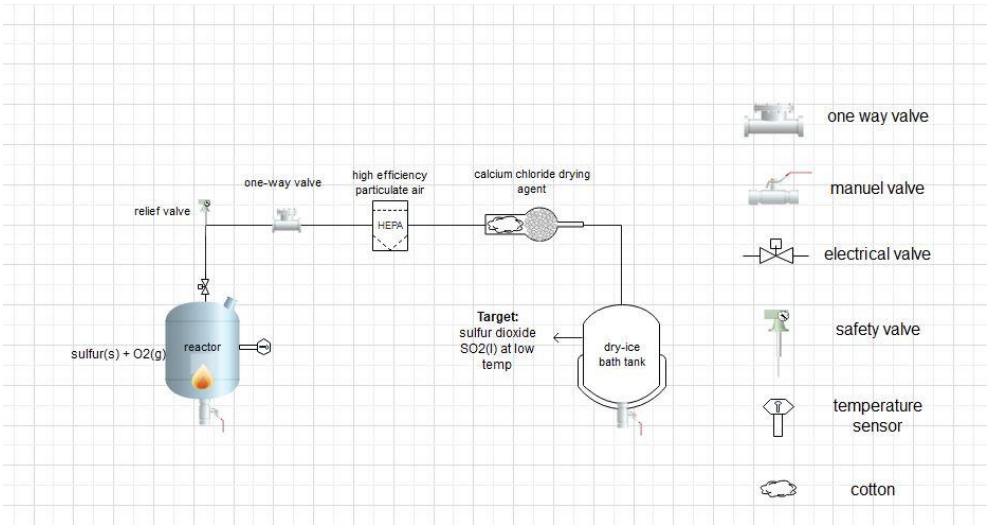
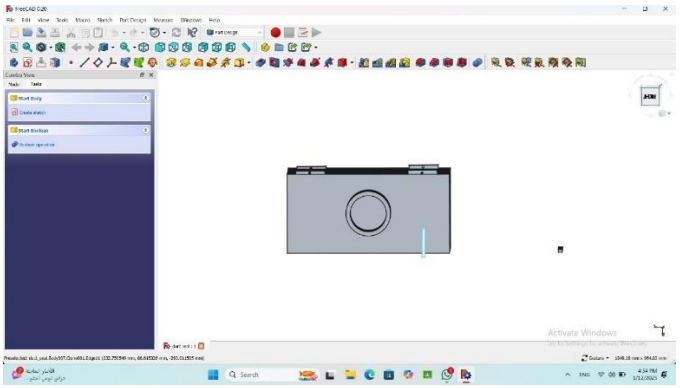
.

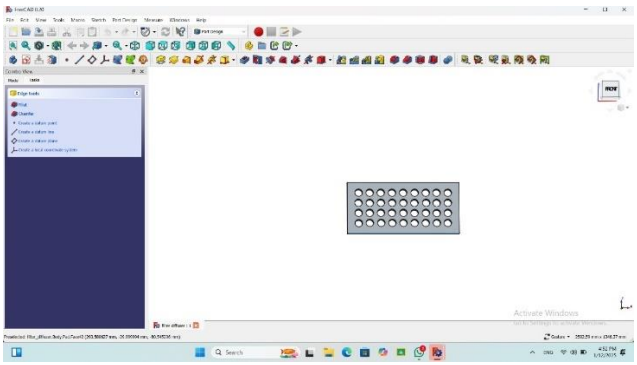
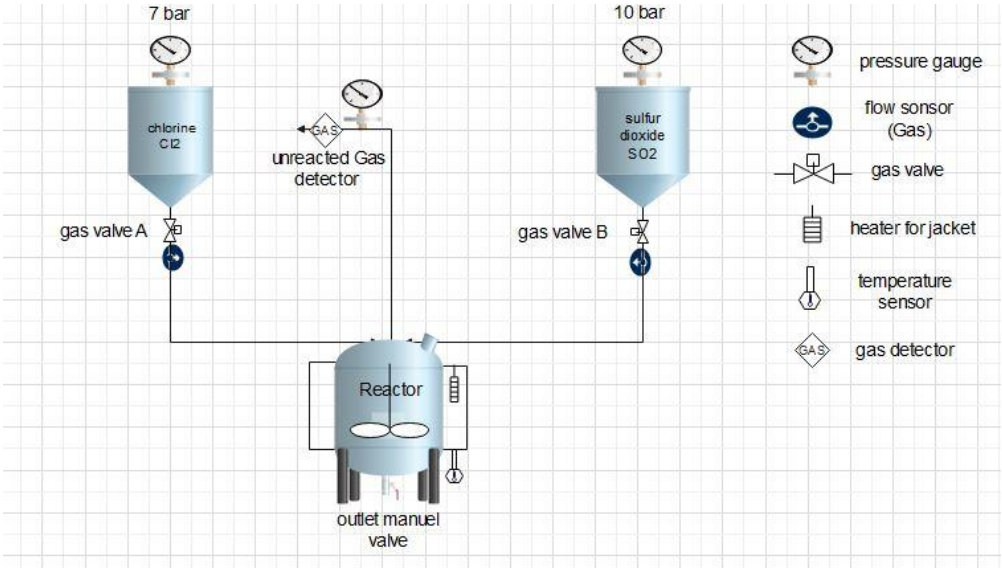
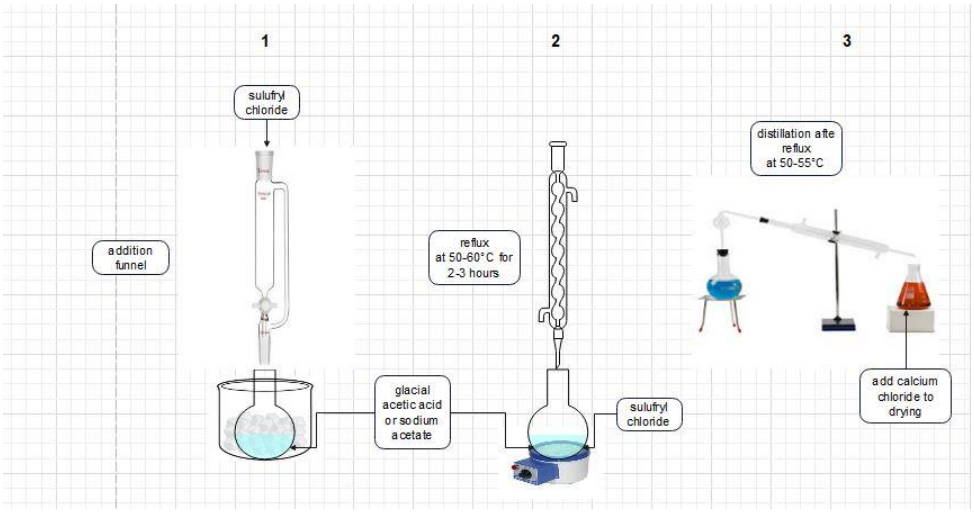


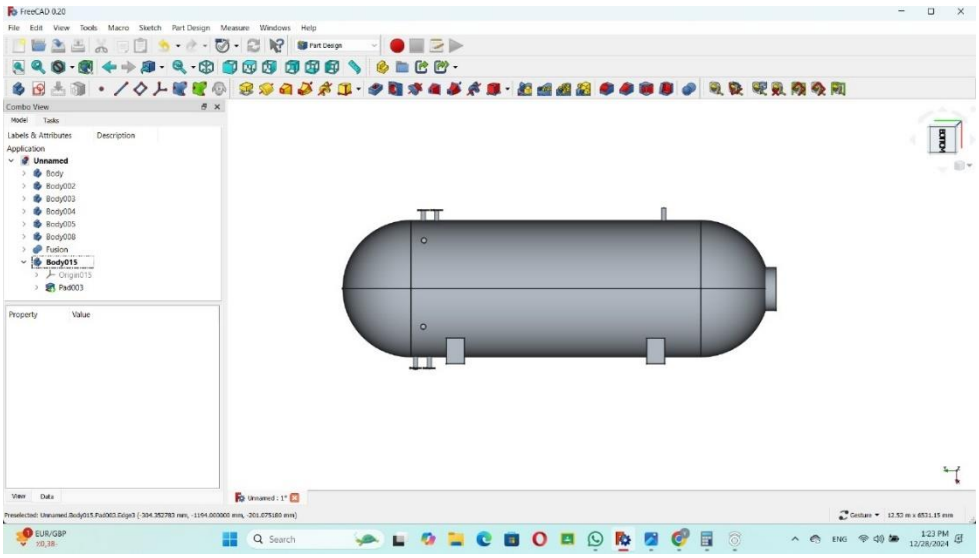
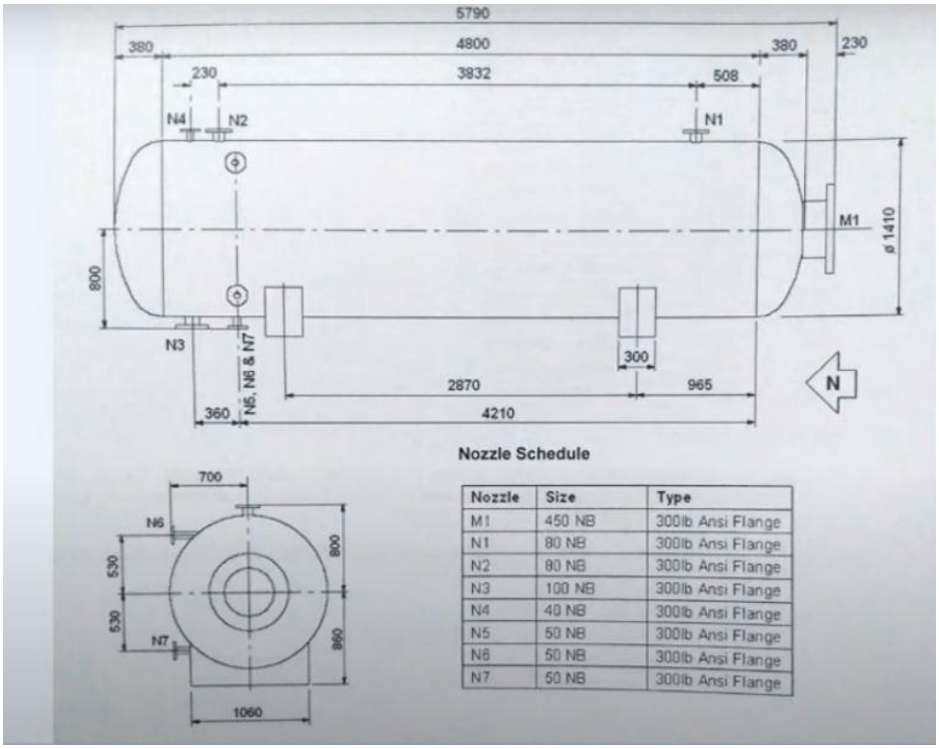
Rawan Abdelmajid @ AECENAR_Green chemistry /September 2024

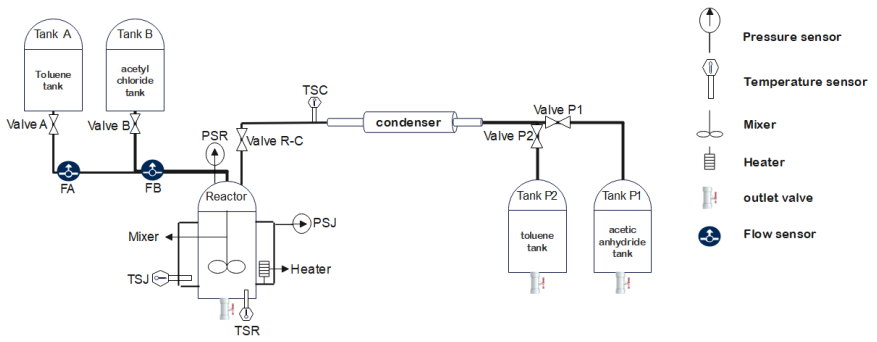
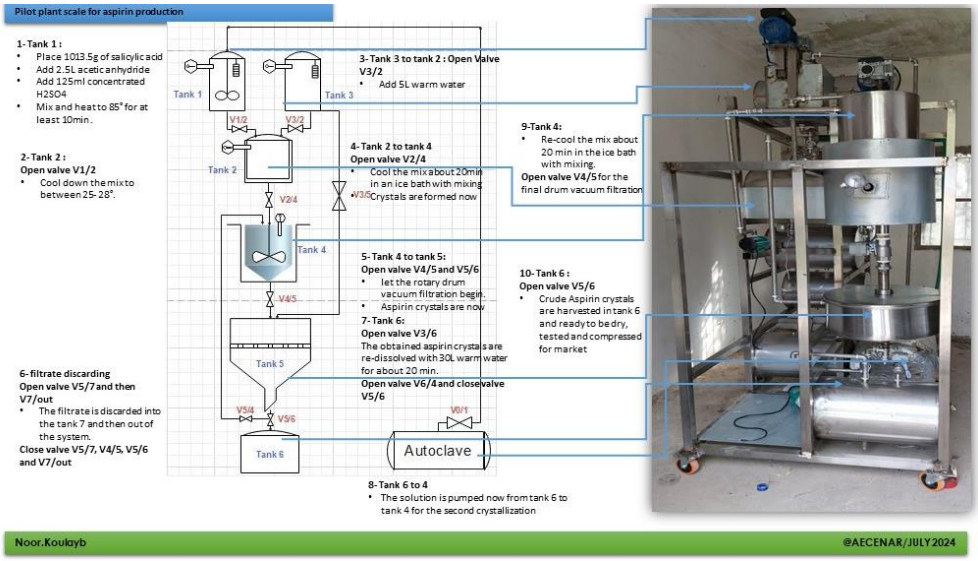
Poster of Acetyl chloride production scale [pptx file]:	10-09-24Acetyl chloride production

1.6 Needed Production Sections for Aspirin Production Plant

<table>
<tr><th>Needed Place</th><th>Production Section</th><th colspan="2"></th></tr>
<tr><td rowspan="2">3mx2m</td><td rowspan="2">(0) Sulfur Dioxid Production</td><td colspan="2"> 
</td></tr>
<tr><td>HEPA:
  </td><td>https://drive.google.com/file/d/1D4bzIMto5m_PWutzEXQCJMI58VJmh7fx/view?usp=drive_link (Duct seal)</td></tr>
</table>

			https://drive.google.com/file/d/1JgdSB8Hpipp6wvUrTj0-zpD-pHu8E9gB/view?usp=drive_link (Filter diffuser)
3mx2m	(1) Sulfuryl chloride Production		
3mx2m	(2a) Acetyl Chloride Production		

		 Reflux drum: https://drive.google.com/file/d/19OzRXjUFxW4LpvQ9NoZQcnv-FAnyDO2U/view?usp=drive_link  Nozzle Schedule Nozzle | Size | Type M1 | 450 NB | 300lb Ansi Flange N1 | 80 NB | 300lb Ansi Flange N2 | 80 NB | 300lb Ansi Flange N3 | 100 NB | 300lb Ansi Flange N4 | 40 NB | 300lb Ansi Flange N5 | 50 NB | 300lb Ansi Flange N6 | 50 NB | 300lb Ansi Flange N7 | 50 NB | 300lb Ansi Flange
3mx2m	(3) Sodium Acetat Production	

3mx2m	(4) Acetic Anhydride Production	
3mx2m	(5) Aspirin Production	
3mx12m	***Total Plant***	

2 Project 1: Aspirin Production Project

2.1 Poster concerning the Aspirin production pilot plant

AECENAR
TECDA
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region

بسم الله الرحمن الرحيم

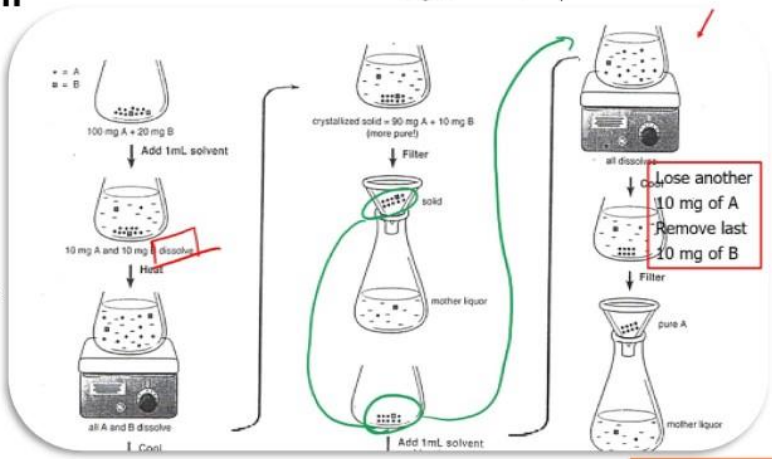
MEGBI - Middle East Genetics and Biotechnology Institute
مركز أبحاث للجينات والتقنية البيولوجية
http://aecenar.com/institutes/megbi

Material:
1. Salicylic acid
2. Acetic anhydride

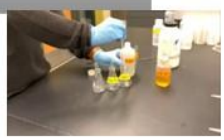
Recrystallization of aspirin:
aspirin + ethanol => add cold water => Ice water bath => vaccum filtration => weigh the aspirin

Purity of aspirin:
Measure the melting point => Qualitative test (formation iron Phenol.

Aspirin production pilot plant

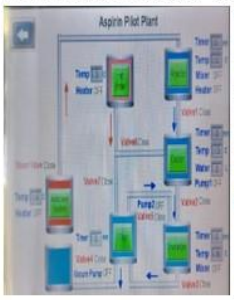


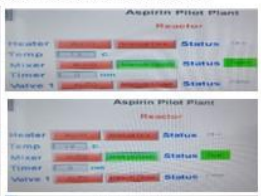

Take a very small amounts of the 3 products and put them in test tubes.

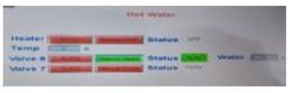

Add 1 mL of ethanol 95% to each test tube


Add 1 drop of FeCl3 Solution (2.5%)


Shake the test tubes and see the colors







Aspirin Quantification: Done by HPLC
1. Pure standard with Known concentration to get TR and W
2. Our sample to get TR and W
[ ] = sample area/(stand.area * [ ] of stand.

Note: To avoid obstructive problems
1. Repeat same production
2. Experiment shows to increase time of crystallization with mixing for 20 min

Pilot plant scale for aspirin production

1- Tank 1 :
- Place 1013.5g of salicylic acid
- Add 2.5L acetic anhydride
- Add 125ml concentrated H_2SO_4
- Mix and heat to 85° for at least 10min.

2- Tank 2 :
Open valve V1/2
- Cool down the mix to between 25- 28°.

3- Tank 3 to tank 2 : Open Valve V3/2
- Add 5L warm water

4- Tank 2 to tank 4
Open valve V2/4
- Cool the mix about 20min in an ice bath with mixing
- Crystals are formed now

5- Tank 4 to tank 5:
Open valve V4/5 and V5/6
- let the rotary drum vacuum filtration begin.
- Aspirin crystals are now

6- filtrate discarding
Open valve V5/7 and then V7/out
- The filtrate is discarded into the tank 7 and then out of the system.

Close valve V5/7, V4/5, V5/6 and V7/out

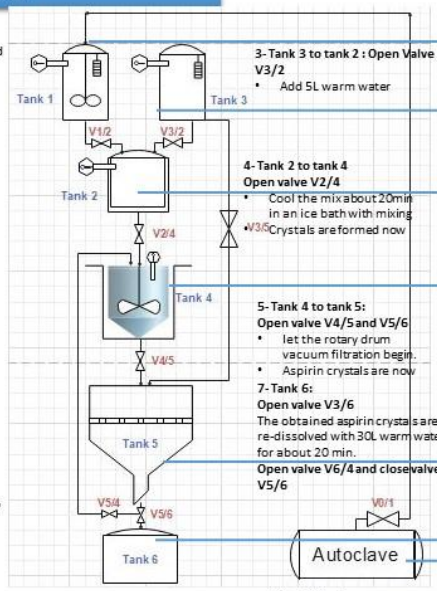
7- Tank 6:
Open valve V3/6
The obtained aspirin crystals are re-dissolved with 30L warm water for about 20 min.
Open valve V6/4 and close valve V5/6

8- Tank 6 to 4
- The solution is pumped now from tank 6 to tank 4 for the second crystallization

9- Tank 4:
- Re-cool the mix about 20 min in the ice bath with mixing.

Open valve V4/5 for the final drum vacuum filtration

10- Tank 6 :
Open valve V5/6
- Crude Aspirin crystals are harvested in tank 6 and ready to be dry, tested and compressed for market



Noor.Koulayb
@AECENAR/JULY2024

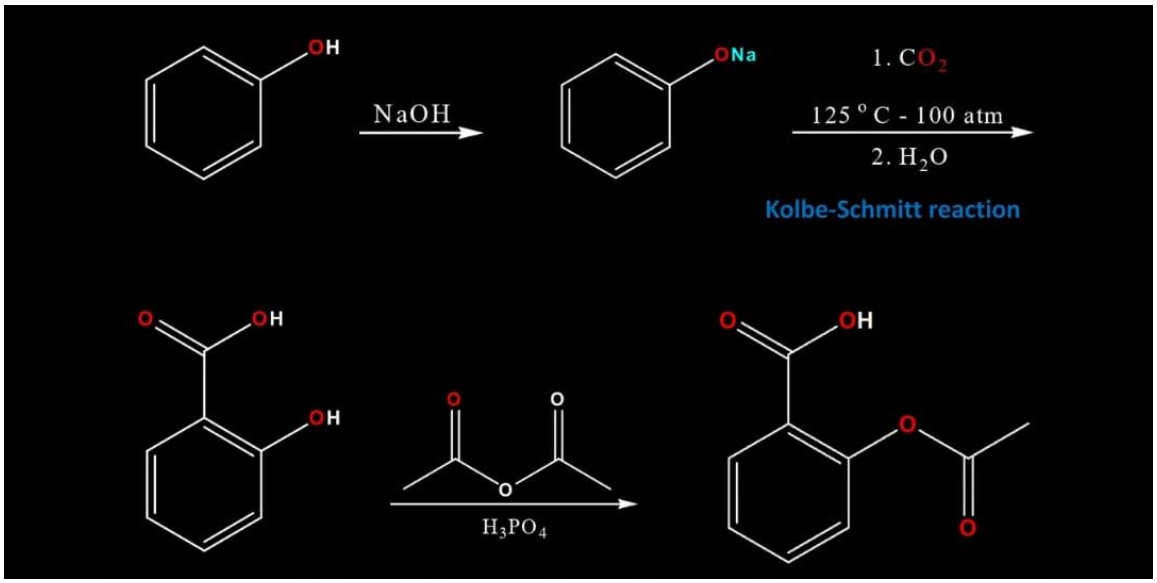
2.2 Aspirin production

2.2.1 Introduction

Aspirin, also known as acetylsalicylic acid (ASA), is a medication used to reduce pain, fever, or inflammation. Aspirin is used to treat specific inflammatory conditions including Kawasaki disease, pericarditis, and rheumatic fever. Aspirin given shortly after a heart attack decreases the risk of death.

Aspirin is also used long-term to help prevent further heart attacks, ischaemic strokes, and blood clots in people at high risk. It may also decrease the risk of certain types of cancer, particularly colorectal cancer.

2.2.2 Synthesis



2.2.3 Trademark

Bayer lost its trademark for Aspirin in the United States in actions taken between 1918 and 1921 because it had failed to use the name for its product correctly and had for years allowed the use of "Aspirin" by other manufacturers without defending the intellectual property rights. Today, aspirin is a generic trademark in many countries. Aspirin, with a capital "A", remains a registered trademark of Bayer in Germany, Canada, Mexico, and in over 80 other countries, for acetylsalicylic acid in all markets, but using different packaging and physical aspects for each.

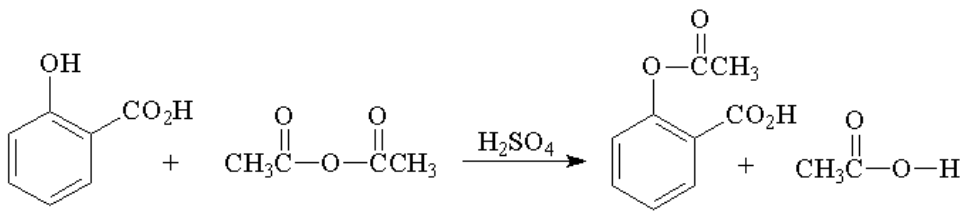
2.2.4 Adverse effects

In October 2020, the U.S. Food and Drug Administration (FDA) required the drug label to be updated for all nonsteroidal anti-inflammatory medications to describe the risk of kidney problems in unborn babies that result in low amniotic fluid. They recommend avoiding NSAIDs in pregnant women at 20 weeks or later in pregnancy. One exception to the recommendation is the use of low-dose 81 mg aspirin at any point in pregnancy under the direction of a health care professional.

2.2.5 References

- "Aspirin". Drugs.com. American Society of Health-System Pharmacists. 6 June 2016. Archived from the original on 25 April 2017.
- "FDA Warns that Using a Type of Pain and Fever Medication in Second Half of Pregnancy Could Lead to Complications". U.S. Food and Drug Administration (FDA)(Press release). 15 October 2020. Retrieved 15 October 2020. ‹See TfM› This article incorporates text from this source, which is in the public domain.
- "NSAIDs may cause rare kidney problems in unborn babies". U.S. Food and Drug Administration. 21 July 2017. Retrieved 15 October 2020. ‹See TfM› This article incorporates text from this source, which is in the public domain.
- Bayer Co. v. United Drug Co., 272 F. 505, p.512 (S.D.N.Y 1921).
- "Has aspirin become a generic trademark?". genericides.org. 25 March 2020. Retrieved 17 February 2021.
- Huth EJ, et al. (CBE Style Manual Committee) (1994). Scientific style and format: the CBE manual for authors, editors, and publishers. Cambridge University Press. p. 164. Bibcode:1994ssfc.book.....S. ISBN 978-0-521-47154-1. Archived from the original on 15 October 2015.
- "Aspirin: the versatile drug". CBC News. 28 May 2009. Archived from the original on 6 November 2016.
- Cheng TO (2007). "The history of aspirin". Texas Heart Institute Journal. 34 (3): 392–3. PMC 1995051. PMID 17948100.

Aspirin Synthesis (Acetylsalicylic acid)



2.2.6 Procedure

Procedure 1st part

1. Place 2.0 g (0.015 mole) of salicylic acid in a 125-mL Erlenmeyer flask.
2. Add 5 mL (0.05 mole) of acetic anhydride, followed by 5 drops of conc. H_2SO_4 (*use a dropper, H_2SO_4 is highly corrosive*) and swirl the flask gently until the salicylic acid dissolves.
3. Heat the flask gently on the steam bath for at least 10 minutes.
4. Allow the flask to cool to room temperature. If acetylsalicylic acid does not begin to crystallize out, scratch the walls of the flask with a glass rod. Cool the mixture slightly in an ice bath until crystallization is completed. The product will appear as a solid mass when crystallization is completed.
5. Add 50 mL of water and cool the mixture in an ice bath. Do not add the water until crystal formation is complete.
6. Vacuum filter the product using a Buchner funnel. You can use some of the filtrate to rinse the Erlenmeyer flask if necessary.

Rinse the crystals several times with small portions (5 mL) of cold water and air dry the crystals on a Buchner funnel by suction until the crystals appear to be free of solvent. Test this crude product for the presence of unreacted salicylic acid using the ferric chloride test. Record the weight of the crude solid which probably contains water.

Precedure 2nd part

1. Stir the crude solid with 25 mL of a saturated aqueous sodium bicarbonate solution in a 150 mL beaker until all signs of reaction have ceased (evolution of CO_2 ceases).
2. Filter the solution through a Buchner funnel to remove any insoluble impurities or polymers that may have been formed. Wash the beaker and the funnel with 5 to 10 mL of water.
3. Carefully pour the filtrate with stirring, a small amount at a time, into an ice cold HCl solution (*ca* 3.5 mL of conc. HCl in 10 mL of water) in a 150-mL beaker and cool the mixture in an ice bath. Make sure that the resulting solution is acidic (blue litmus paper) and that the aspirin has completely precipitated out.
4. Filter the solid by suction and wash the crystals 3X with 5 mL of *cold* water each. Remove all the liquid from the crystals by pressing with a clean stopper or cork. Air dry the crystals and transfer them to a watch glass to dry. Test a small amount of the product for the presence of unreacted salicylic acid using the ferric chloride solution.
5. When the product is completely dry, weigh the product, determine its melting point (lit mp 135-136 °C) and calculate the percentage yield.

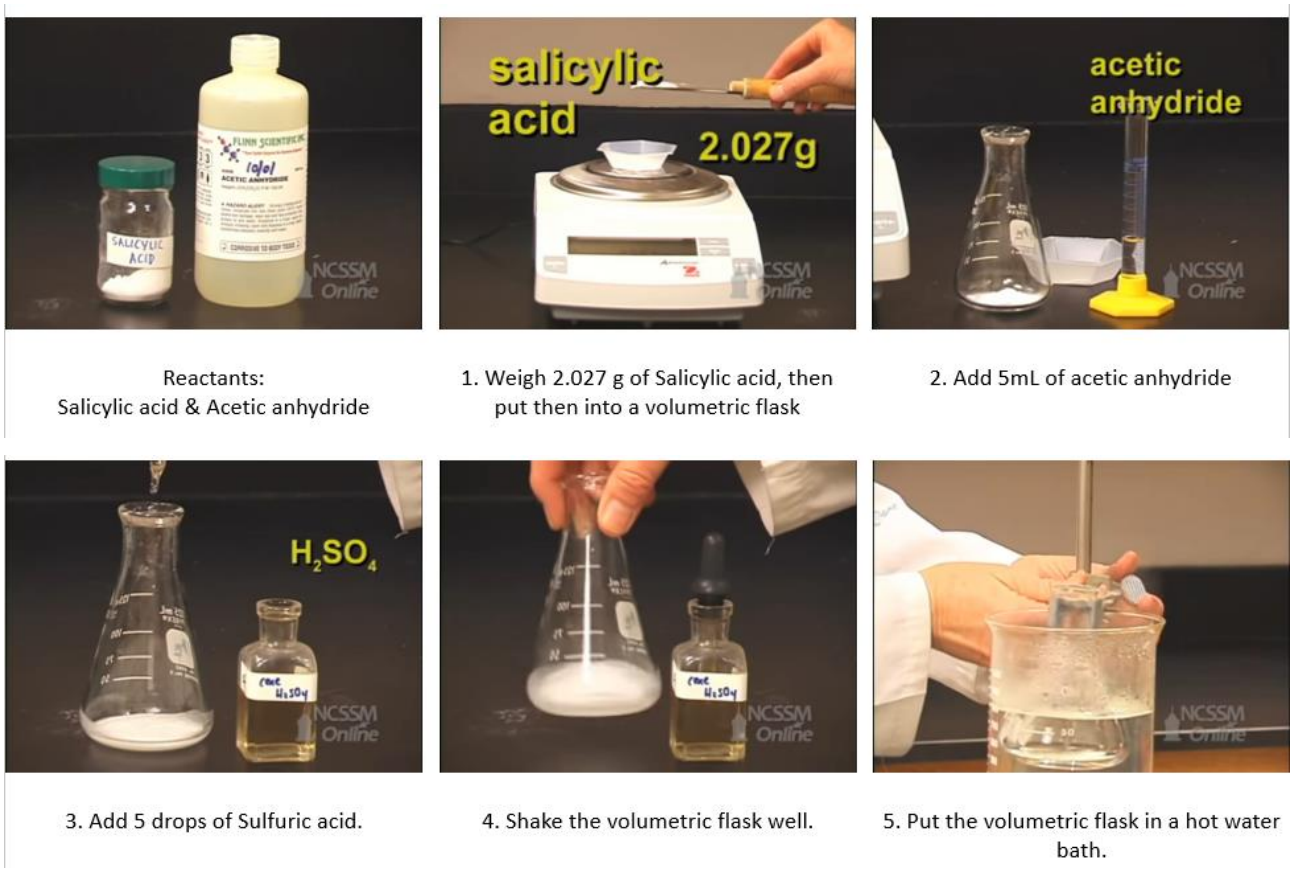
6. Dissolve the final product in a minimum amount (no more than 2-3 mL) of *hot* ethyl acetate in a 25 mL Erlenmeyer flask. Make sure that the product is completely dissolved while gently and continuously heating on a steam bath.
7. Cool the solution to room temperature and then in a ice-bath. Collect the product by vacuum filtration and rinse out of the flask with a few milliliters of cold petroleum ether.

When the product is completely dry, weigh its weight, determine its melting point (lit mp 135 °C) and calculate the percentage yield of this recrystallized product. Calculate the % recovery of recrystallized material from crude material. Submit the crystalline sample in a small vial with proper labeling to your instructor.

Aspirin production presentation [PPtx file]:

Aspirin.pptx

2.3 Aspirin Procedure in LAB Scale



Reactants:
Salicylic acid & Acetic anhydride

1. Weigh 2.027 g of Salicylic acid, then put then into a volumetric flask

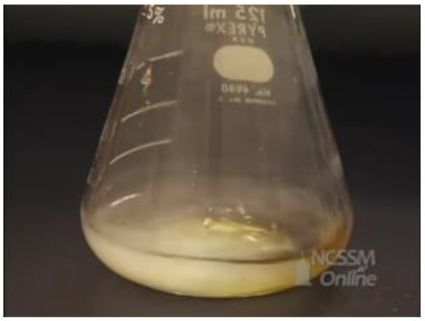
2. Add 5mL of acetic anhydride

3. Add 5 drops of Sulfuric acid.

4. Shake the volumetric flask well.

5. Put the volumetric flask in a hot water bath.

6. After 10 minutes, let the volumetric flask cool down



7. Add 10 mL of warm water.

8. Put the volumetric flask in ice, so aspirin can start precipitating.

9. 10 minutes later, the aspirin should be precipitated.

10. Now, we filtrate the aspirin by a vacuum filtration.

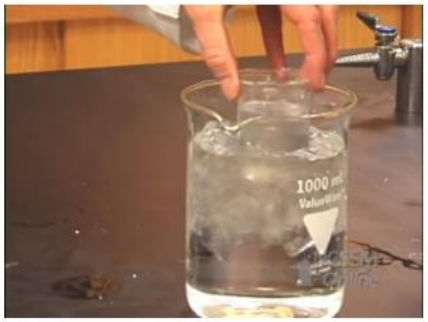
11. Add small amount of cold water on the filter paper.

12. Add the solution.

13. Pour the crystals into a beaker.

14. Dissolve them again by adding water.

15. Add 60 mL of warm water.

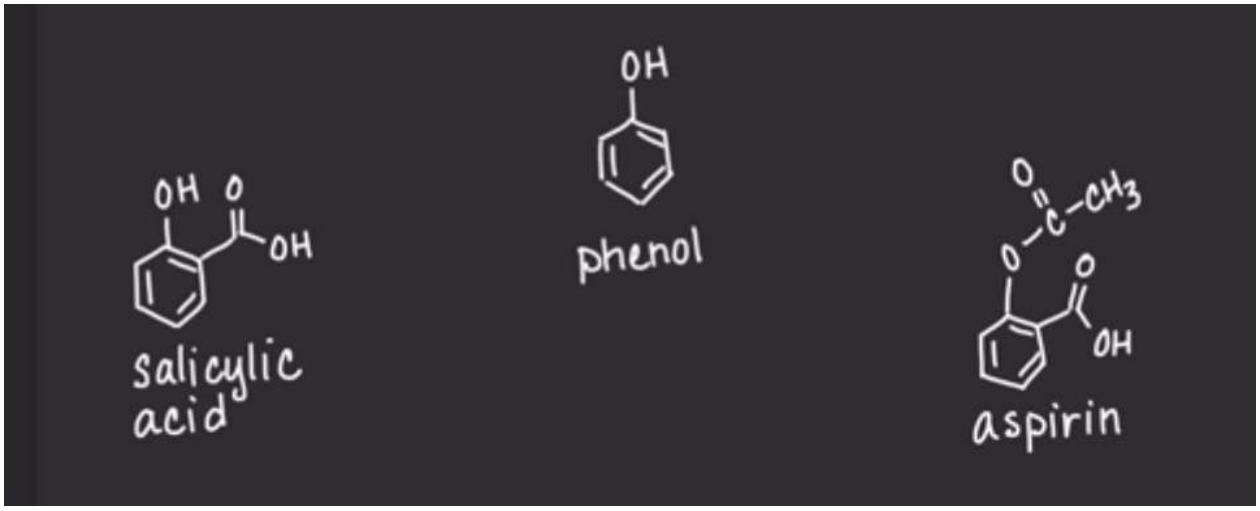


16. Put the beaker in ice to recrystallize.

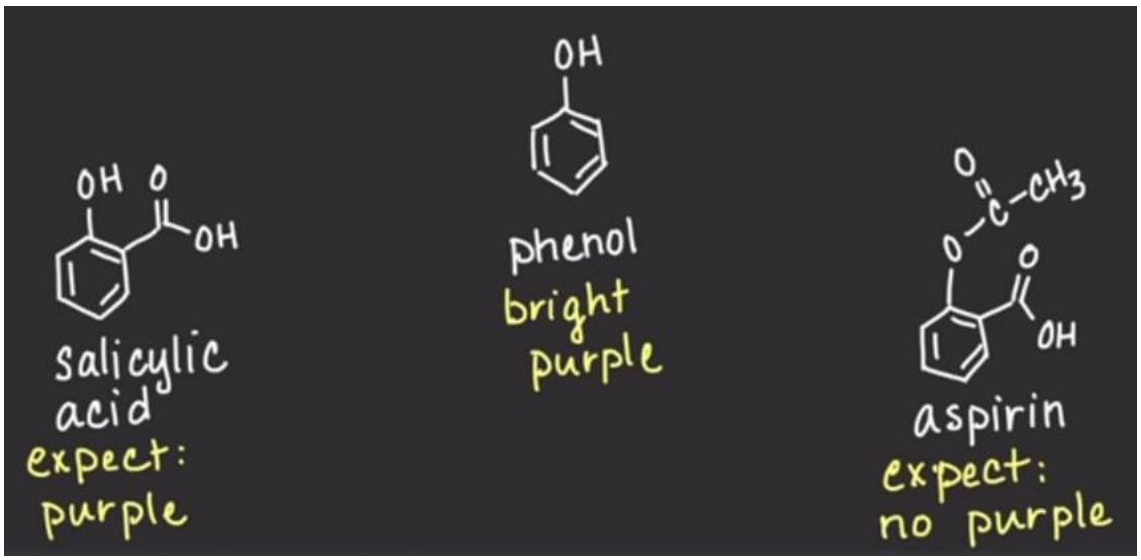
17. Last step, filter the product again.

Note: **In step 11 and 17 we used standard filtration, not vacuum filtration**

Synthesis of Aspirin – FeCl3 Test

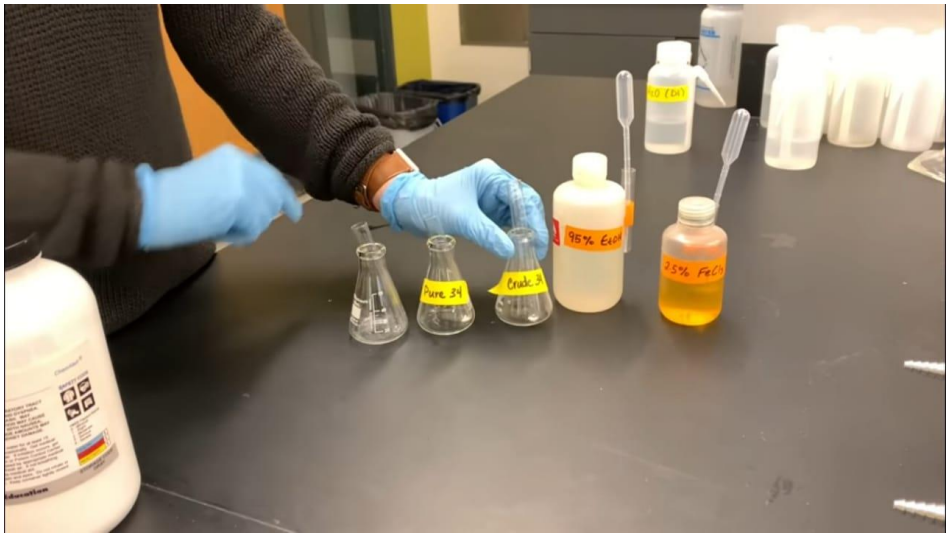


FeCl3 test, which is a qualitative test

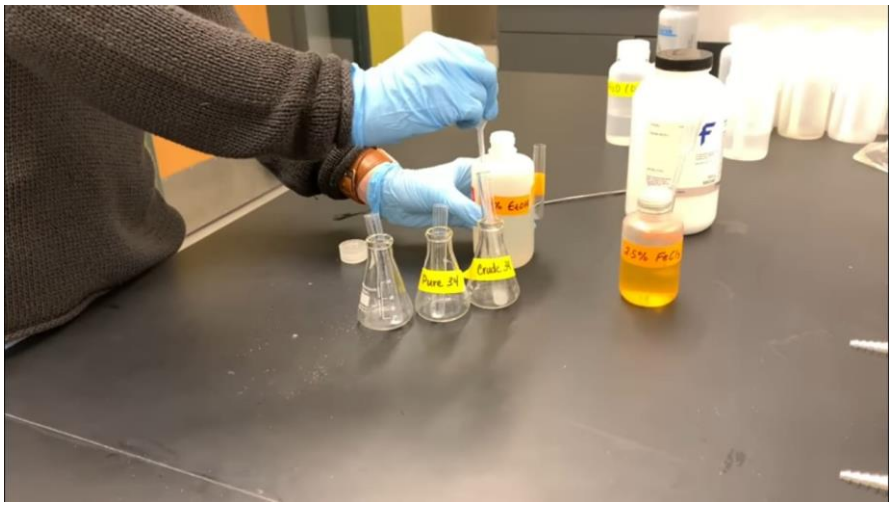


Expected colors! Since FeCl3 reacts with the OH group

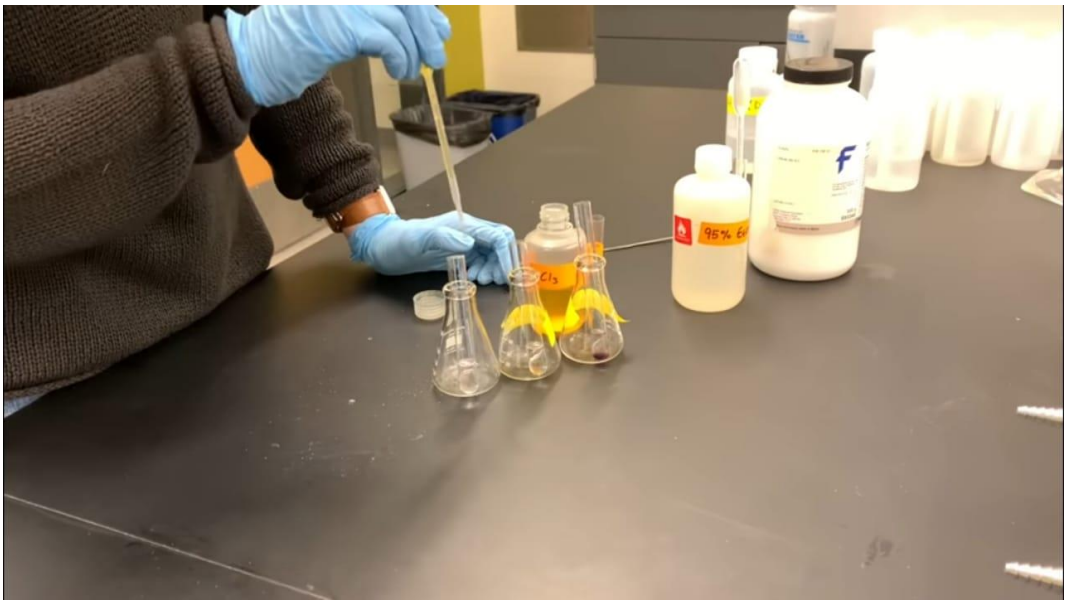
We can do the test for the crude product, pure product, and the salicylic acid



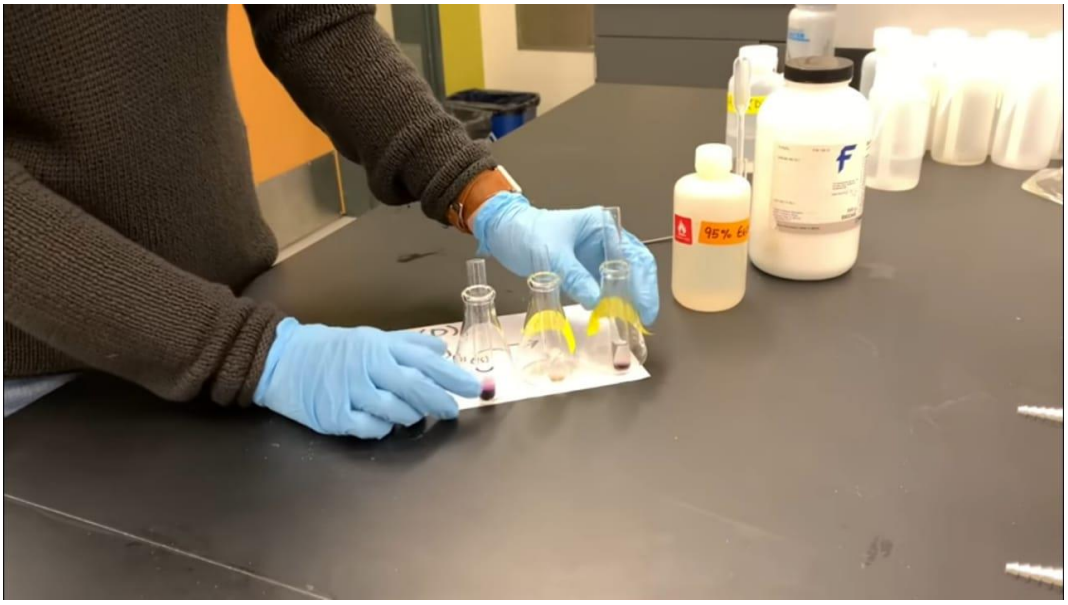
1. Take a very small amount of the 3 products and put them in test tubes.



2. Add 1 mL of ethanol 95% to each test tube



3. Add 1 drop of FeCl3 Solution (2.5%)



4. Shake the test tubes and see the colors.

3 Project 2: Acetic Anhydride Production Project

AECENAR
TECDA
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region

ACETIC ANHYDRIDE PRODUCTION

Acetic Anhydride: A Versatile Chemical Intermediate Acetic anhydride is a key organic compound with diverse applications in various industries. This poster explores its synthesis, properties, and key applications, highlighting its importance in modern chemical manufacturing.

$$CH_3COCl(l) + CH_3COONa(s) \rightarrow (CH_3CO)_2O(l) + NaCl(s)$$


Add sodium acetate in the round bottom flask

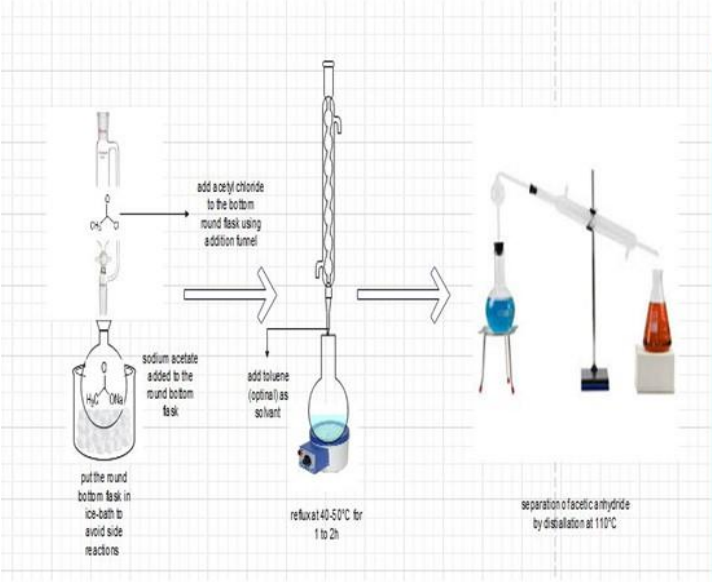

Add acetyl chloride using addition funnel


Put gas safety outlet


Cooling the reaction in the round bottom flask by an ice bath


Reflux at 40-50 °C for 1-2h


Distillation of acetic anhydride at 110 °C



MATERIALS AND METHODS

Apparatus Setup:

•Set up a dry, well-ventilated reaction apparatus equipped with:

- A round-bottom flask with a magnetic stirrer
- A reflux condenser
- A dropping funnel
- A thermometer

PREPARATION OF REAGENTS

•**Dry Sodium Acetate:** Anhydrous sodium acetate is crucial. If not available, dry sodium acetate trihydrate in an oven at 120-140°C for several hours. Store in a desiccator.

•**Dry Solvent:** If using a solvent (e.g., anhydrous diethyl ether or dichloromethane), ensure it is properly dried over a suitable drying agent (e.g., calcium chloride) and distilled.

Reaction:

•**Charge:** Place the calculated amount of dry sodium acetate in the round-bottom flask.

•**Solvent Addition:** If using a solvent, add the appropriate amount.

•**Cool Down:** Cool the flask in an ice bath.

•**Acetyl Chloride Addition:** Slowly add acetyl chloride dropwise from the dropping funnel while maintaining the temperature below 10°C. Stir continuously.

•**Warm Up:** After complete addition, slowly warm the reaction mixture to room temperature.

•**Reflux:** Gradually increase the temperature to 40-50°C and reflux for 1-2 hours.

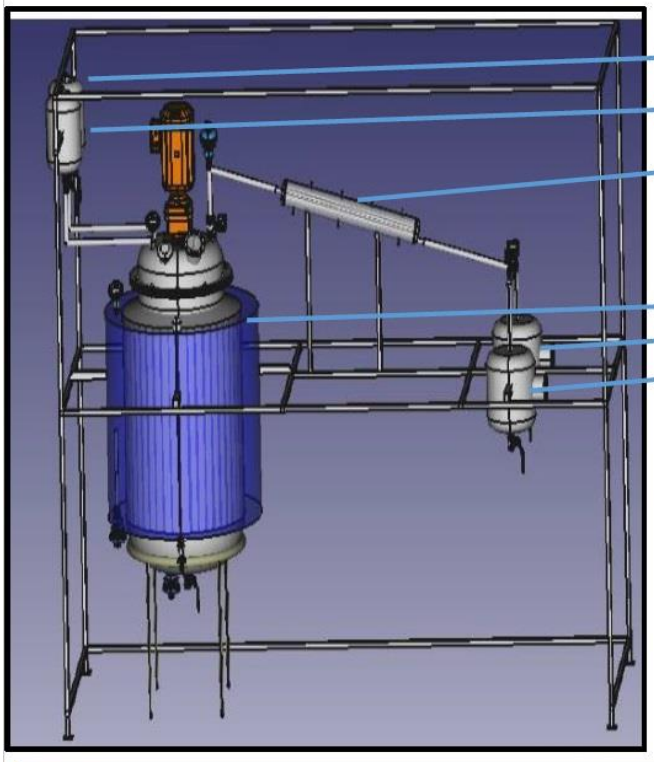
•**Distillation:** After the reaction is completed, turn ON the water circulation in the condenser to start the distillation by turning ON the heating to 110°C to evaporate the acetone
increase the temperature to 140°C to evaporate the Acetic Anhydride

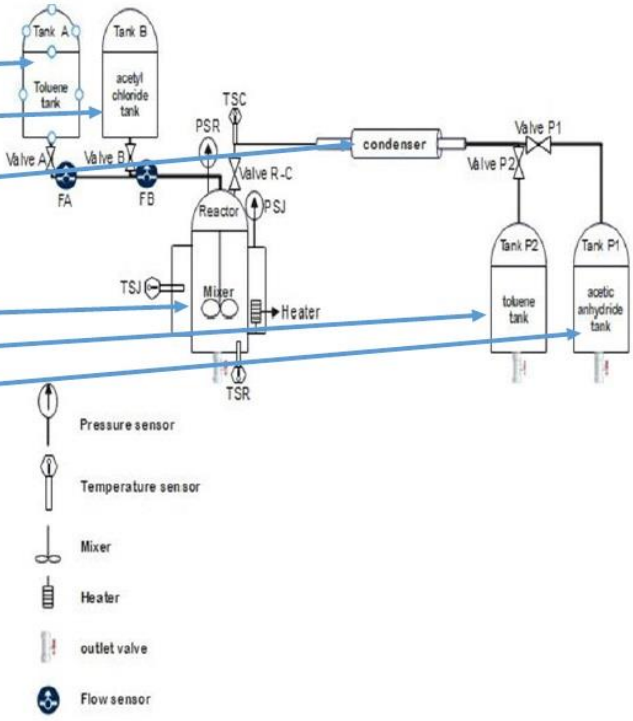
AECENAR
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region
TECDA

بسم الله الرحمن الرحيم

MEGBI - Middle East Genetics and Biotechnology Institute
مركز أبحاث للجينات والتقنية البيولوجية
http://aecenar.com/institutes/megbi

Acetic Anhydride

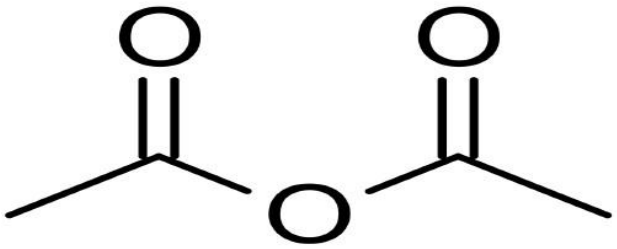




Reaction:
Sodium acetate(NaCH3COO) + acetic acid (CH3COOH) → H2O+CO2+NaCH3COO
2.Sodium acetate(NaCH3COO) + acetyl Chloride(CH3COCl) => acetic anhydride((CH3CO)2O) + NaCl

Material:
- Vinegar
- Sodium bicarbonate
- Acetyl chloride (C2H3ClO)

Safety:
1.Toluene: Sweet aroma,
2. Acetyl chloride: Corrosive, flammable
3.Acetic anhydride: Highly corrosive, inhalation damages lungs,



Procedure:
1. Mixer activation
2. Reagent addition for reaction 1"vinger + Na + C2H3ClO"
3. Heat to 120°C
4. Cool Down to 50-80°C
5. Add 5L of toluene as "solvent"
6. Distillation
7. Test purity and yield of acetic anhydride.

Requirements:

1. Physical: Pipes, tanks, and autoclave must withstand high temperatures (T = 300°C) and pressures (P = 260 bar).
2. Chemical: - Tanks, autoclaves, pipes, valves shall be withstand corrosion with acetic anhydride, toluene , acetic acetate and acetyl chloride
3. Mechanical:
system: stain less steel 316, close
Valve: close compound
Sensor: road data from system

Noor.Koulayb @AECENAR/JULY 2024

For aspirin we need as raw material acetic anhydride. So the goal of is to produce Acetic Anhydride [$(CH_3CO)_2O$] according to the following reaction:

Sodium Acetate + Acetyl Chloride → Acetic Anhydride + Sodium Chloride

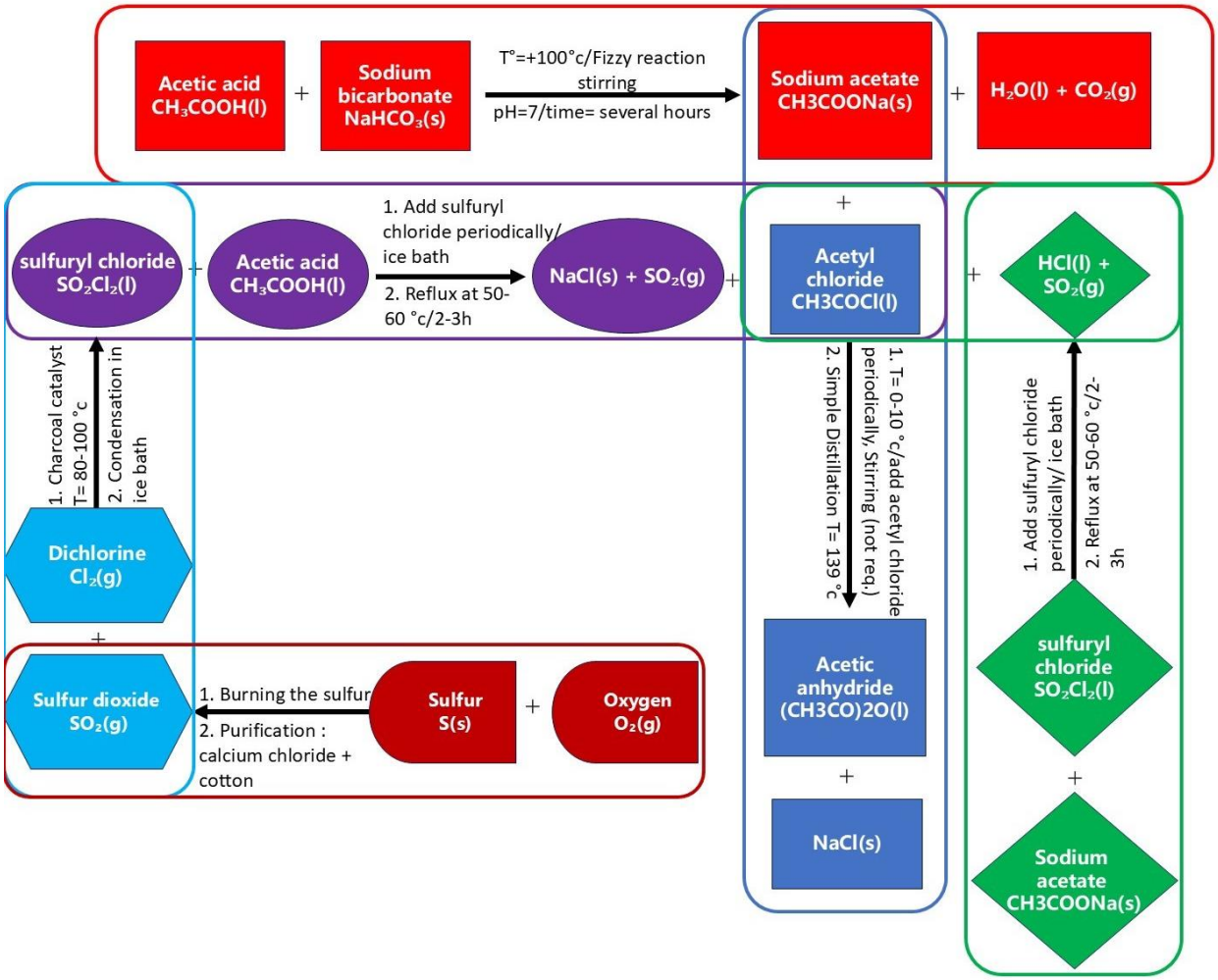
$CH_3COONa_{(s)} + CH_3COCl_{(l)} \rightarrow (CH_3CO)_2O_{(l)} + NaCl_{(s)}$

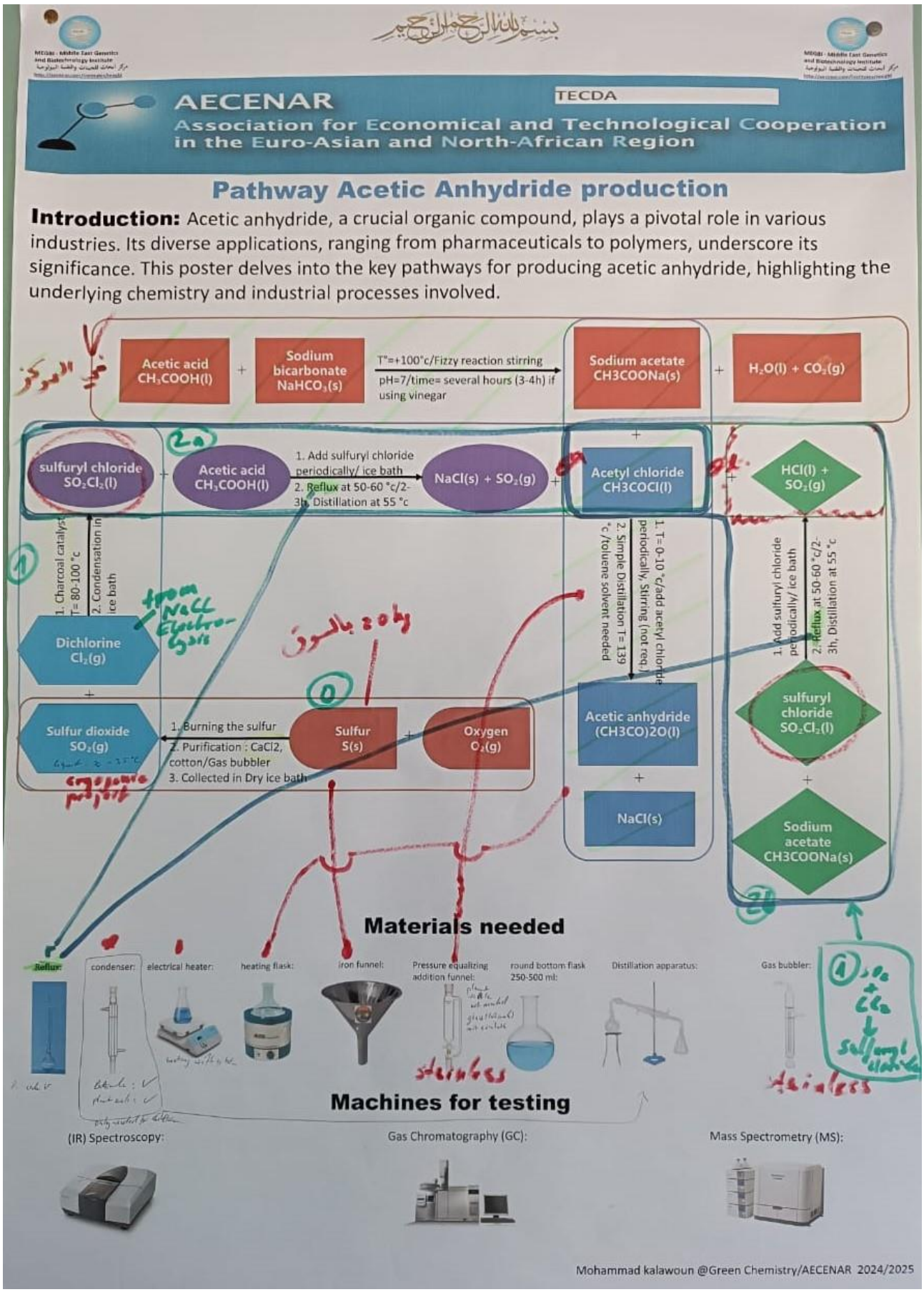
This reaction occurs under the following conditions:

1) T = 0-10°C, Add Acetyl Chloride periodically, Stirring (not req.)
2) Simple distillation at T = 139°C / Toluene solvent needed

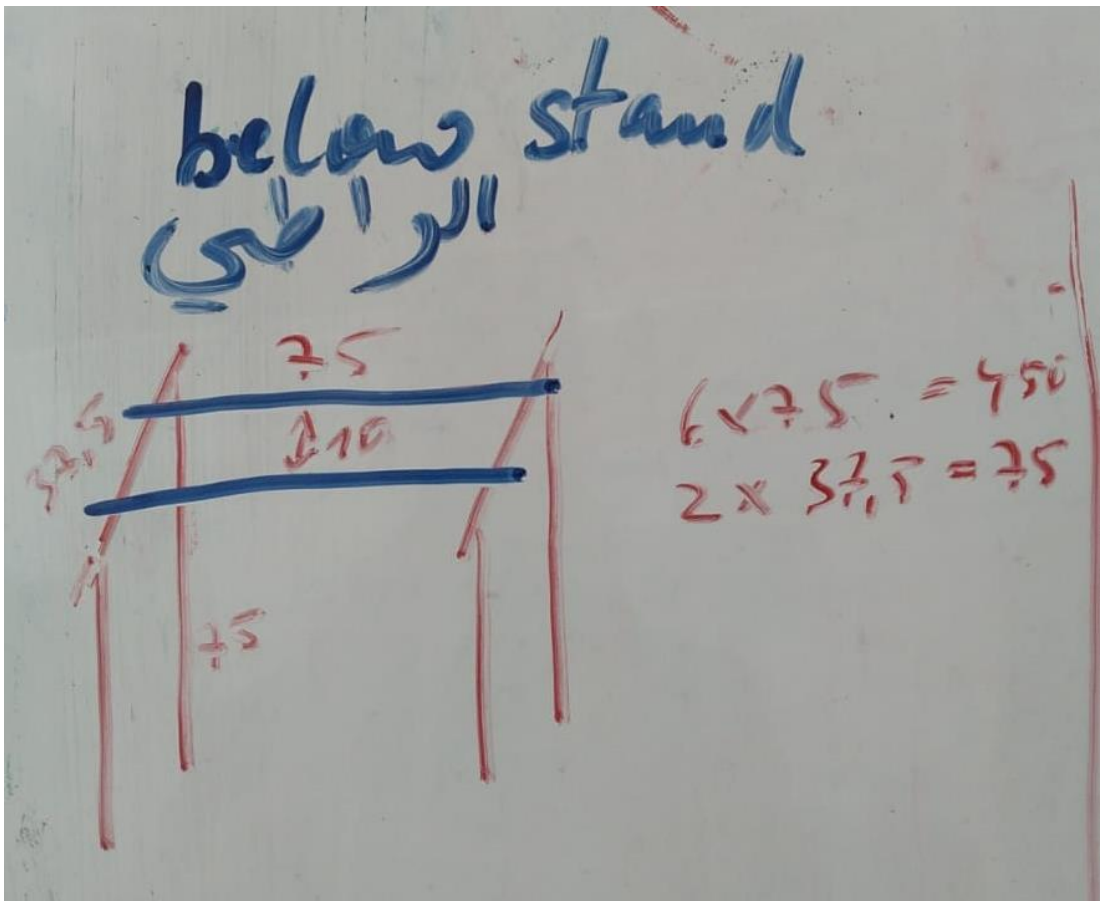
To achieve this reaction, Sodium Acetate and Acetyl Chloride must be obtained.

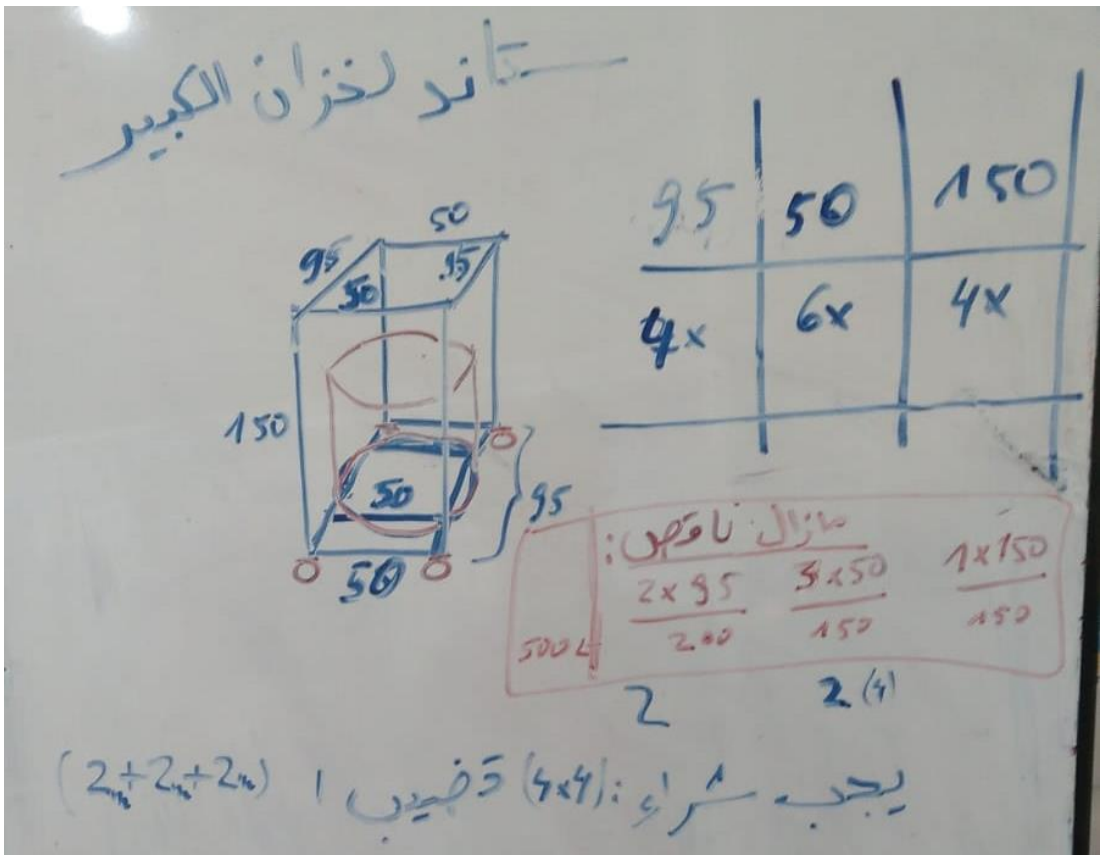
And therefore we need to produce Acetyl Chloride.

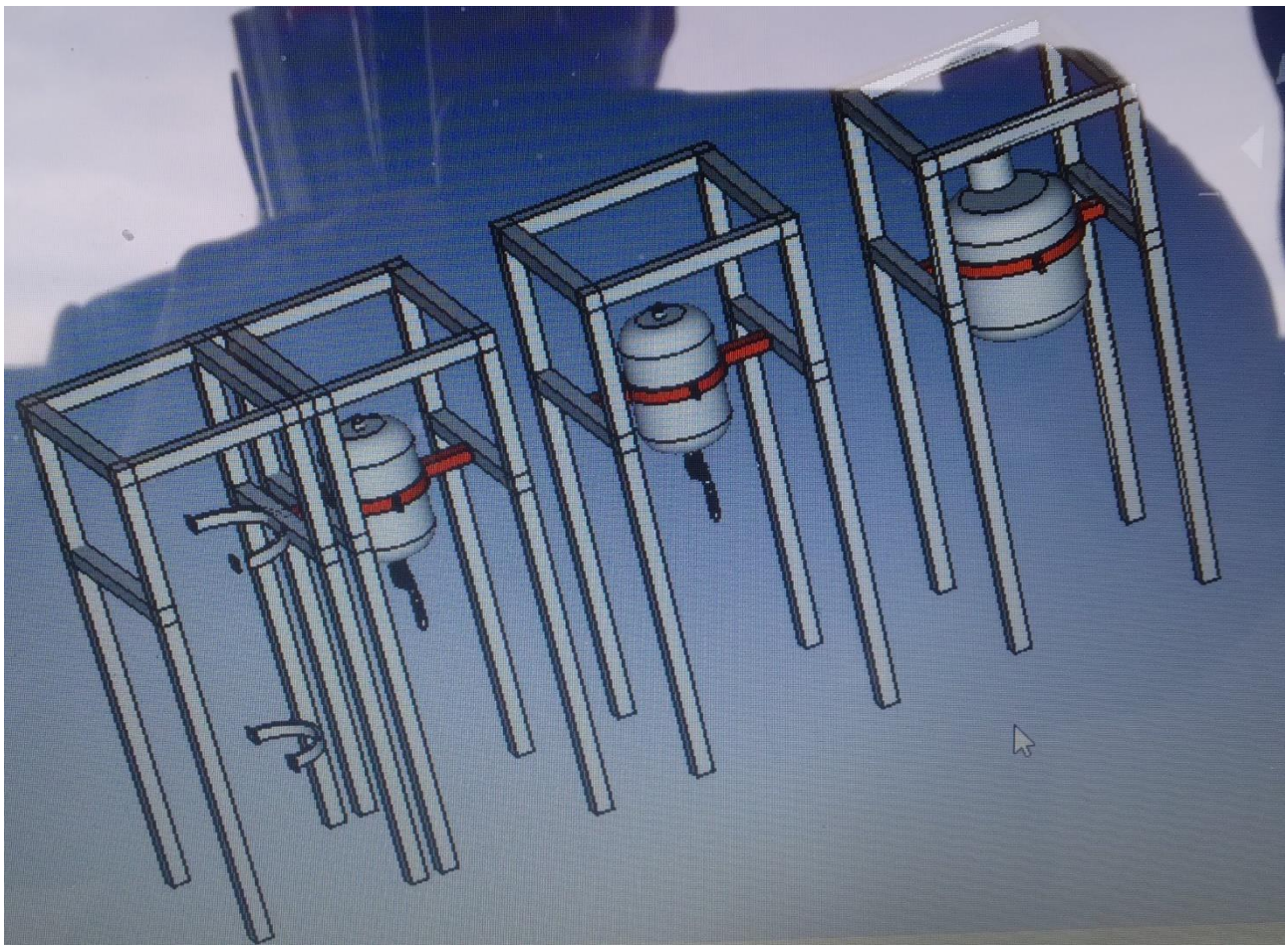



بسم الله الرحمن الرحيم
AECENAR
TECDA
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region
Pathway Acetic Anhydride production
Introduction: Acetic anhydride, a crucial organic compound, plays a pivotal role in various industries. Its diverse applications, ranging from pharmaceuticals to polymers, underscore its significance. This poster delves into the key pathways for producing acetic anhydride, highlighting the underlying chemistry and industrial processes involved.
Acetic acid CH3COOH(l)
Sodium bicarbonate NaHCO3(s)
T°=+100°c/Fizzy reaction stirring
pH=7/time= several hours (3-4h) if using vinegar
Sodium acetate CH3COONa(s)
H2O(l) + CO2(g)
sulfuryl chloride SO2Cl2(l)
Acetic acid CH3COOH(l)
1. Add sulfuryl chloride periodically/ ice bath
2. Reflux at 50-60 °c/2-3h, Distillation at 55 °c
NaCl(s) + SO2(g)
Acetyl chloride CH3COCl(l)
HCl(l) + SO2(g)
1. Charcoal catalyst T= 80-100 °c
2. Condensation in ice bath
Dichlorine Cl2(g)
from NaCl Electro-Gas
1. T= 0-10 °c/add acetyl chloride periodically, Stirring (not req.)
2. Simple Distillation T= 139 °c /toluene solvent needed
1. Add sulfuryl chloride periodically/ ice bath
2. Reflux at 50-60 °c/2-3h, Distillation at 55 °c
sulfuryl chloride SO2Cl2(l)
Sulfur dioxide SO2(g)
1. Burning the sulfur
2. Purification : CaCl2, cotton/Gas bubbler
3. Collected in Dry ice bath
Sulfur S(s)
Oxygen O2(g)
Acetic anhydride (CH3CO)2O(l)
NaCl(s)
Sodium acetate CH3COONa(s)
Materials needed
Reflux:
condenser:
electrical heater:
heating flask:
iron funnel:
Pressure equalizing addition funnel:
round bottom flask 250-500 ml:
Distillation apparatus:
Gas bubbler:
Machines for testing
(IR) Spectroscopy:
Gas Chromatography (GC):
Mass Spectrometry (MS):
Mohammad kalawoun @Green Chemistry/AECENAR 2024/2025

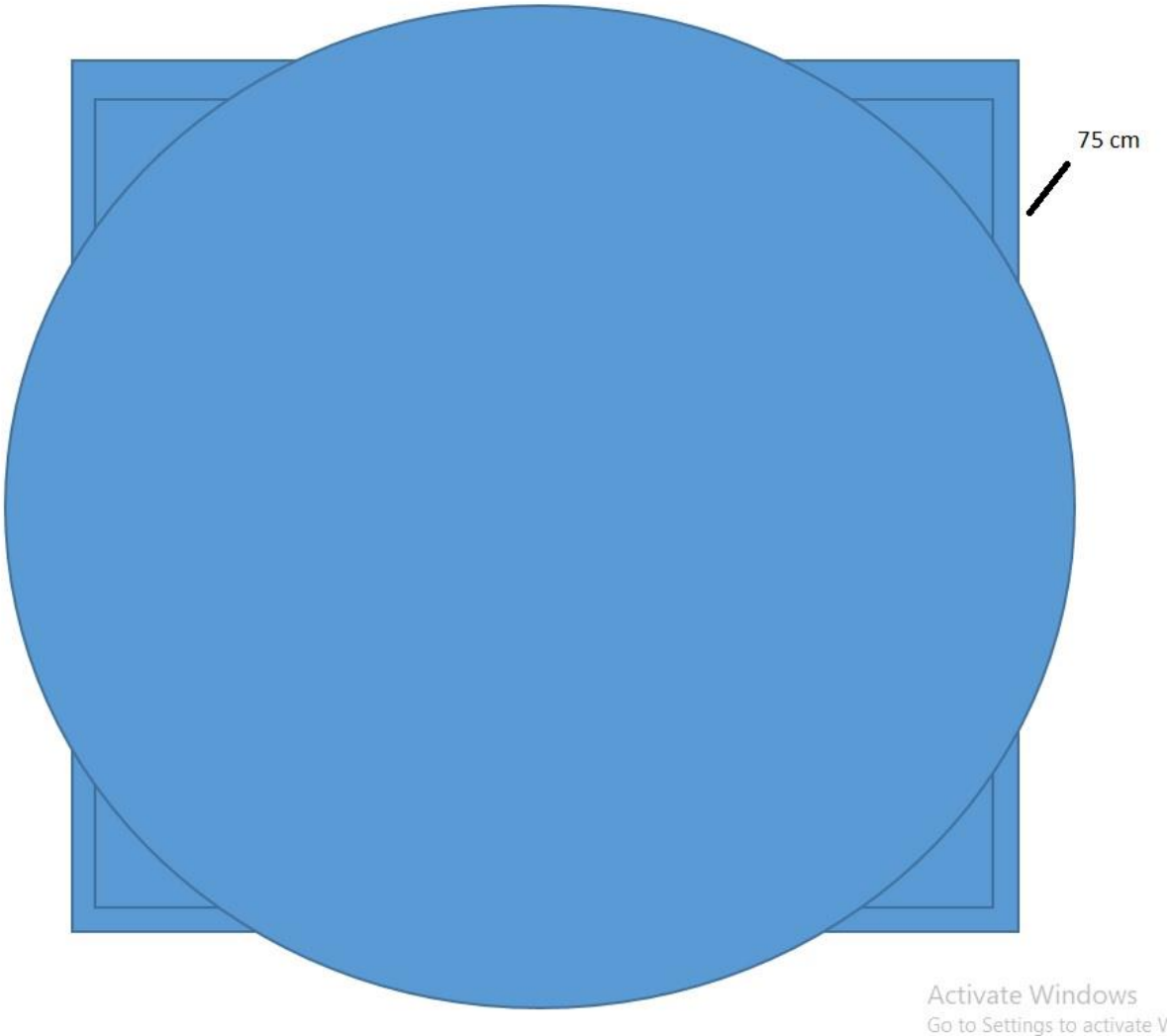
3.1 Plant Scale Devices

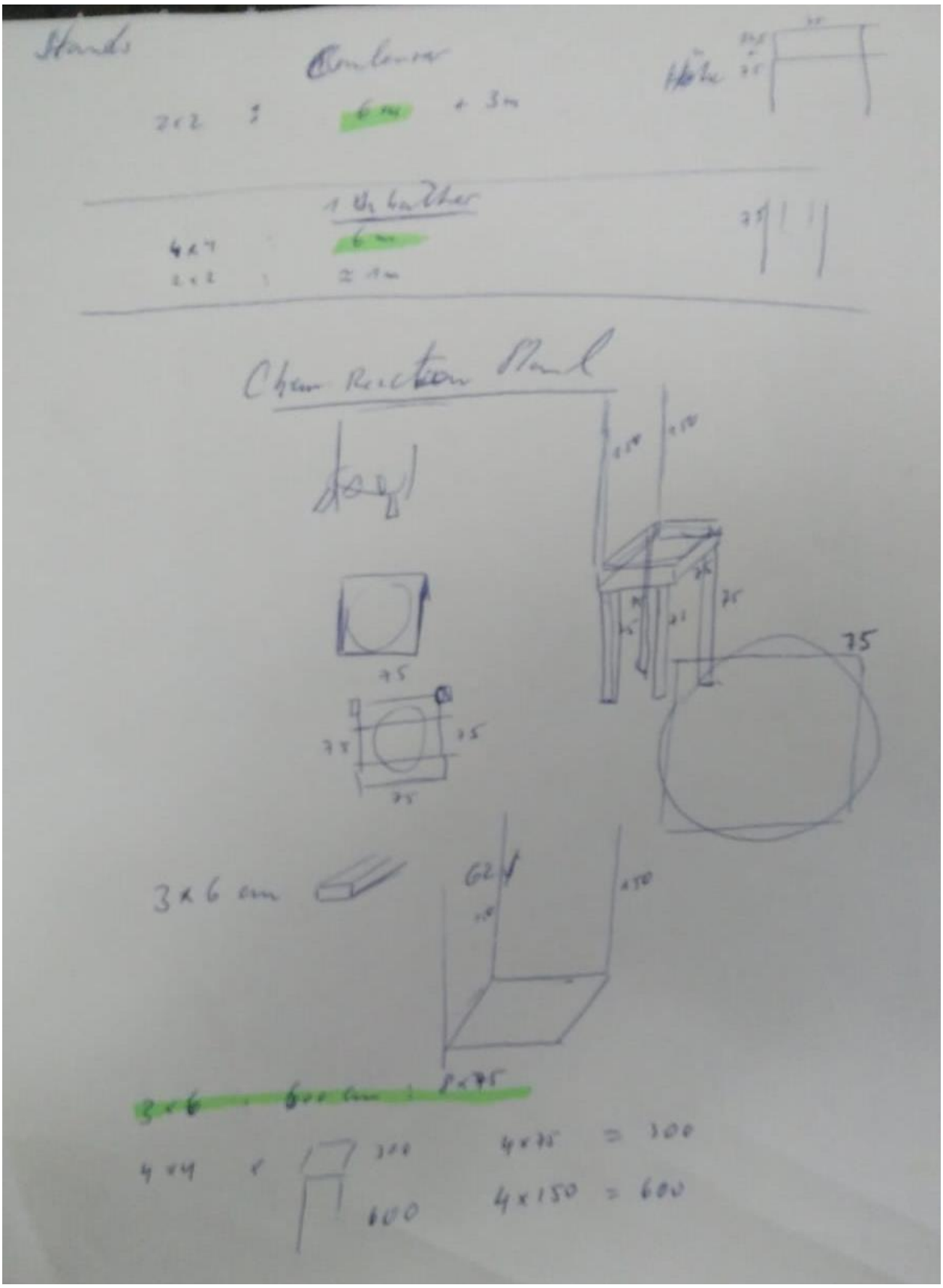

below stand
الراطي
75
37,5
110
75
6x75 = 450
2x37,5 = 75


ستاند لخزان الكبير
95
50
150
4x
6x
4x
50
95
150
50
95
50
نازل ناقص:
2x95
200
3x50
150
1x150
150
500 L
2
2 (4)
يجب شراء: (4x4) قطع 1 (2+2+2)




LASER FILM
LASER FILM
LASER FILM
LASER FILM





3.1.1 Needed Materials for Stands

1. Chemical React. Vessel:

3cm x 6cm (62$ per 6m): 8x75

Chemical React. Vessel	3cm x 6cm (62$ per 6m): 8x75	600 cm	62$
	4cm x 4cm (farigh)/ edges (zawiya) 4x4	4x150cm = 600cm	42$
	4 cm x 4cm (farigh)	4x75 cm = 300cm	21$

3.2 Production of acetic anhydride (chemical reactions and calculations)

3.2.1 Reactions

1. Sodium acetate + Acetic Acid → water + Carbone Dioxide + Sodium acetate
- $NaHCO_3(s) + CH_3COOH(l) \rightarrow CO_2(g) + H_2O(l) + CH_3COONa(s)$

2. Sodium acetate + Acetyl chloride → Acetic anhydride + Sodium chloride
- $CH_3COONa(s) + CH_3COCl(l) \rightarrow (CH_3CO)_2(l) + NaCl(s)$

3.2.2 Calculations

Acetic anhydride needed for 1 trial in aspirin process production = 5 Liters.

Theory calculation to know the amount of the following reagents needed for acetic anhydride production: (Acetic Acid "vinegar", Sodium acetate, Sodium Bicarbonate and Acetyl Chloride).

Acetyl chloride

Acetic anhydride { $C_4H_6O_3$, $\rho = 1.08$ g/ml, $M = 102.089$ g/mol

$\rho = m/V \Rightarrow m = \rho.V \Rightarrow 1.08 \times 5000 = 5400$ g.

$n = m/M \Rightarrow 5400/102.089 = 52.896$ moles.

Calculate the volume of Acetyl Chloride:

$CH_3COONa(s) + CH_3COCl(l) \rightarrow (CH_3CO)_2(l) + NaCl(s)$ according to the reaction the ratio between Acetyl Chloride and Acetic Anhydride is (1:1), that's mean they have the same molar number.

{ CH_3COCl

Acetyl Chloride ρ = 1.1 g/ml
M = 78.49 g/mol

ρ = m/V & m = n.M ⇒ V = n.M/ρ ⇒ 52.896 x 78.49 / 1.1 = 3.77 L.

Calculate the mass of Sodium Acetate:

In the same reaction mentioned above, Sodium acetate should be used it in excess to ensure the reaction is completed.

Sodium Acetate
- CH_3COONa
- ρ = 1.53 g/ml
- M = 82.0343 g/mol

Here, the ratio of the excess should be a little higher; we choose according to the reference, the following ratio (1.1:1)

n = 52.895 x 1.1 = 58.1845 moles

n = m/M ⇒ m = n.M ⇒ 58.1845 x 82.0343 = 4.77 kg.

Calculate the mass of Sodium Bicarbonate:

$NaHCO_3(s) + CH_3COOH(l) \rightarrow CO_2(g) + H_2O(l) + CH_3COONa(s)$ according to the reaction the ratio between sodium bicarbonate and sodium acetate is (1:1), that's mean they have the same molar number.

Sodium Bicarbonate
- $NaHCO_3$
- ρ = 2.2 g/ml
- M = 84.007 g/mol

m = n.M ⇒ 58.145 x 84.007 = 4.88 kg.

Calculate the volume of acetic acid 5% (vinegar):

In the same reaction mentioned above, Sodium acetate should be used it in excess to ensure the reaction is completed

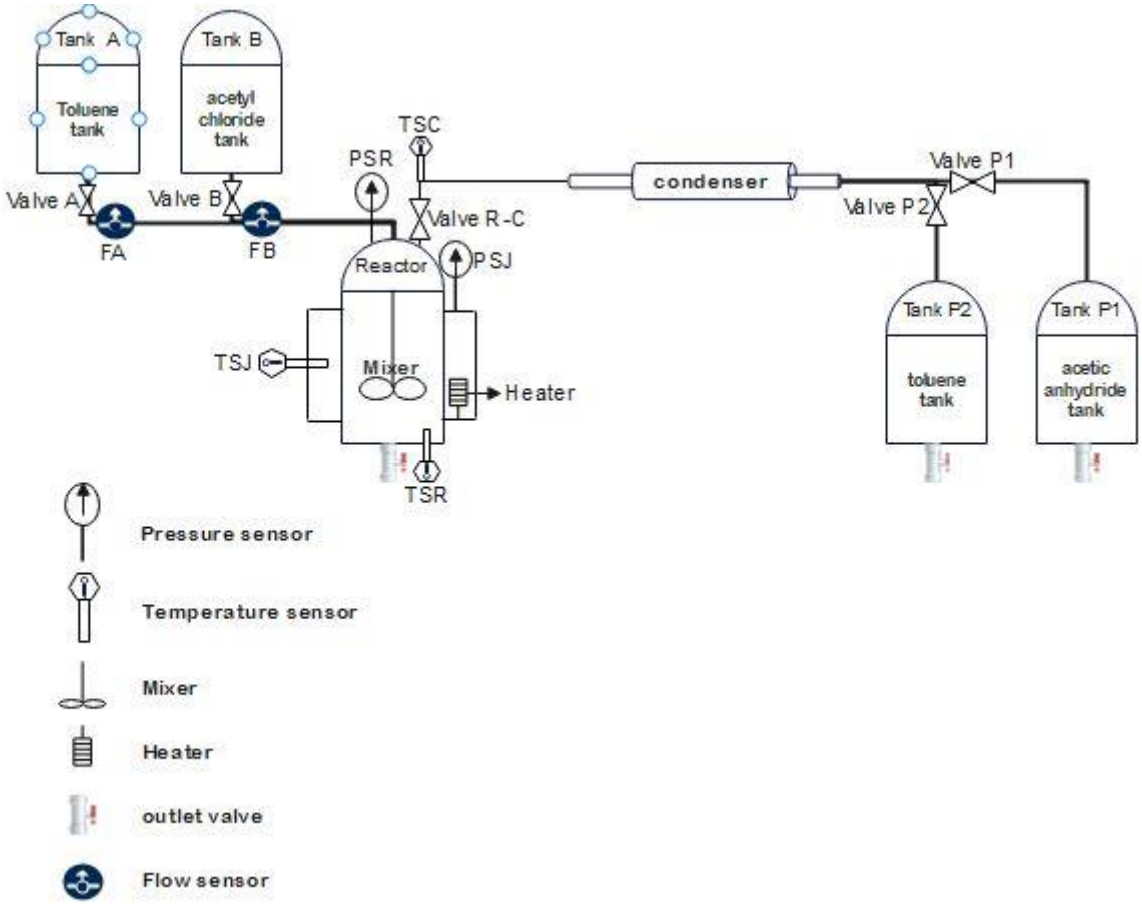
Acetic Acid
- CH_3COOH
- ρ = 1.0005 g/ml
- C = 0.86 mol/l
- M = 60.05 g/mol

Here, the ratio of the excess should be a little higher; we choose according to the reference, the following ratio (1.1:1)

The number of moles needed n x 1.1 = 58.1845 x 1.1 = 64.00295 moles.

C = n/V⇒ V= n/C = 64.00295/0.86 = 74.4 L.

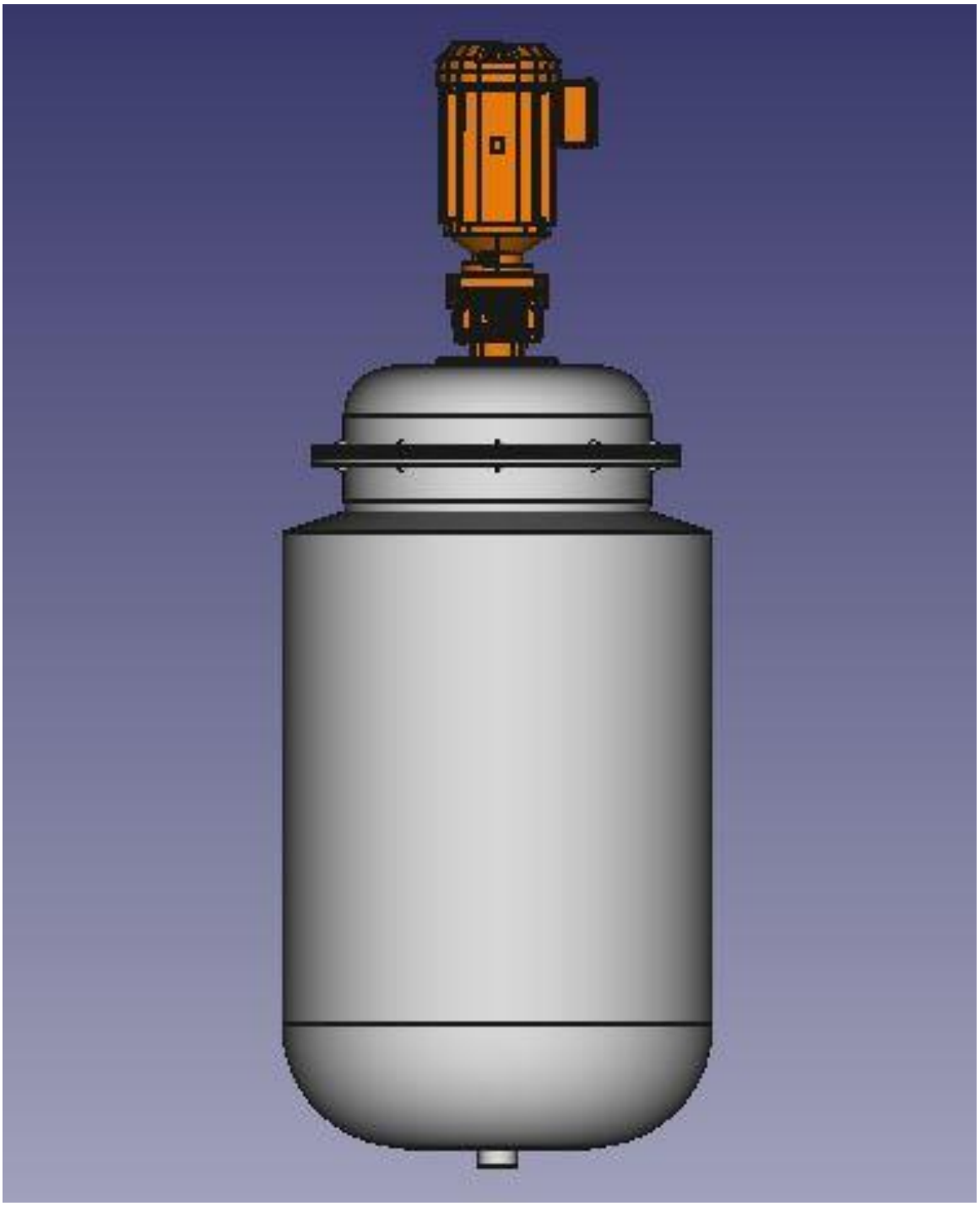
3.3 System design/system concept (Acetic Anhydride Pilot Plant productoin)



Approximate View for acetic anhydride pilot plant [Edraw file]:

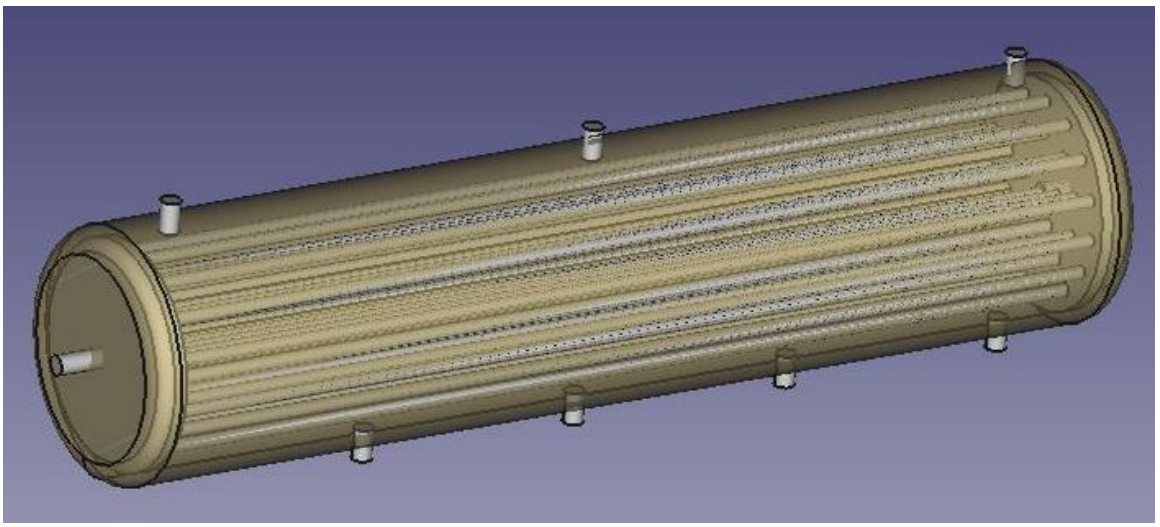
Edraw file acetic anhydride MAP for s

3.3.1 Mechanical design (Acetic Anhydride Pilot Plant)



Reactor for acetic anhydride pilot plant [FreeCAD file]:

reactor for acetic anhydride.FCStd

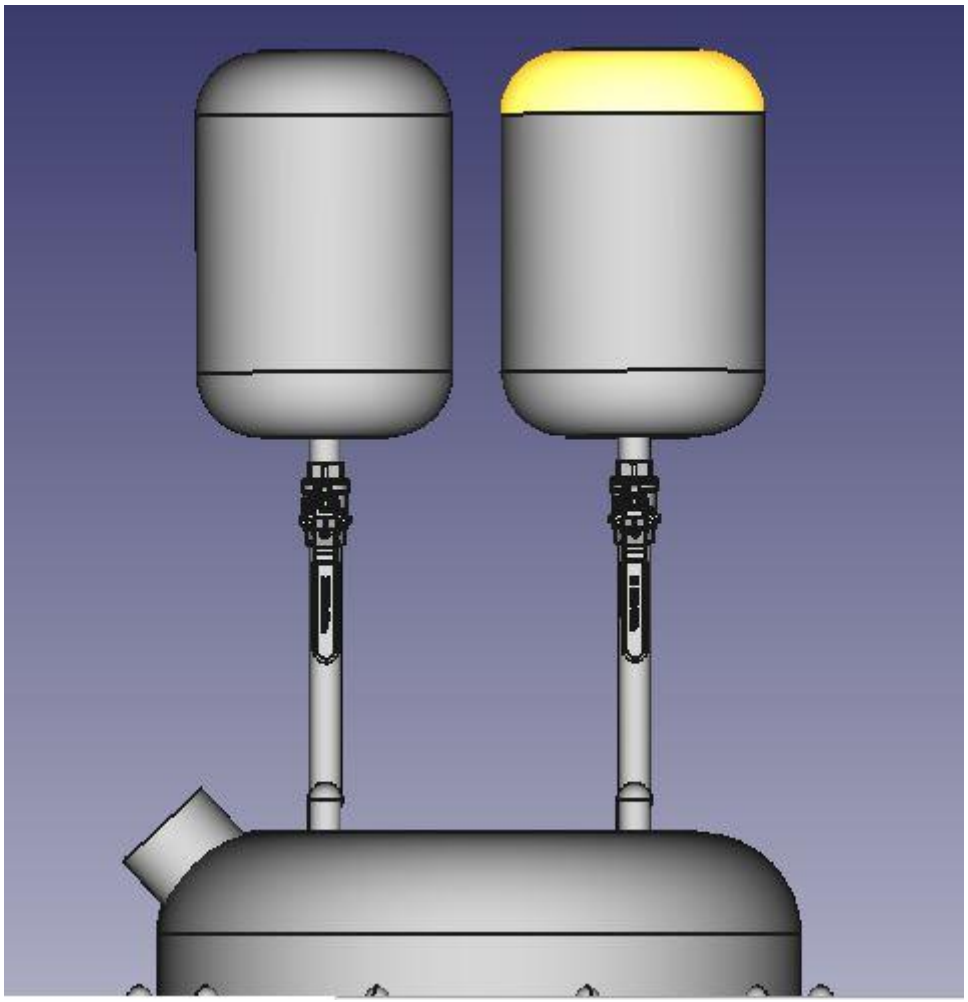


Condenser for acetic anhydride pilot plant [FreeCAD file]:

25122023_Condenser.FCStd

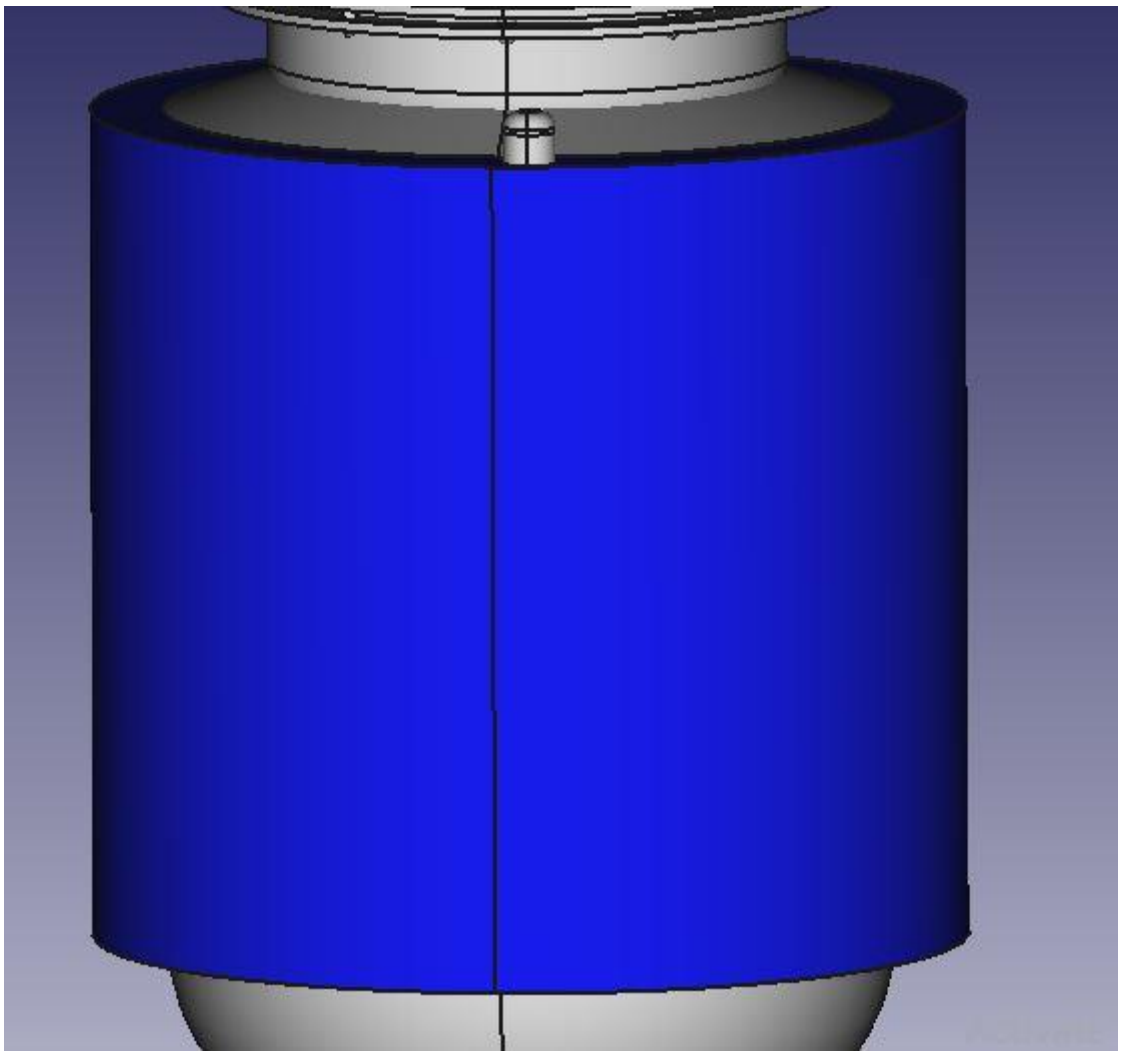
Mixer for acetic anhydride pilot plant [FreeCAD file]:

agitator 3.FCStd



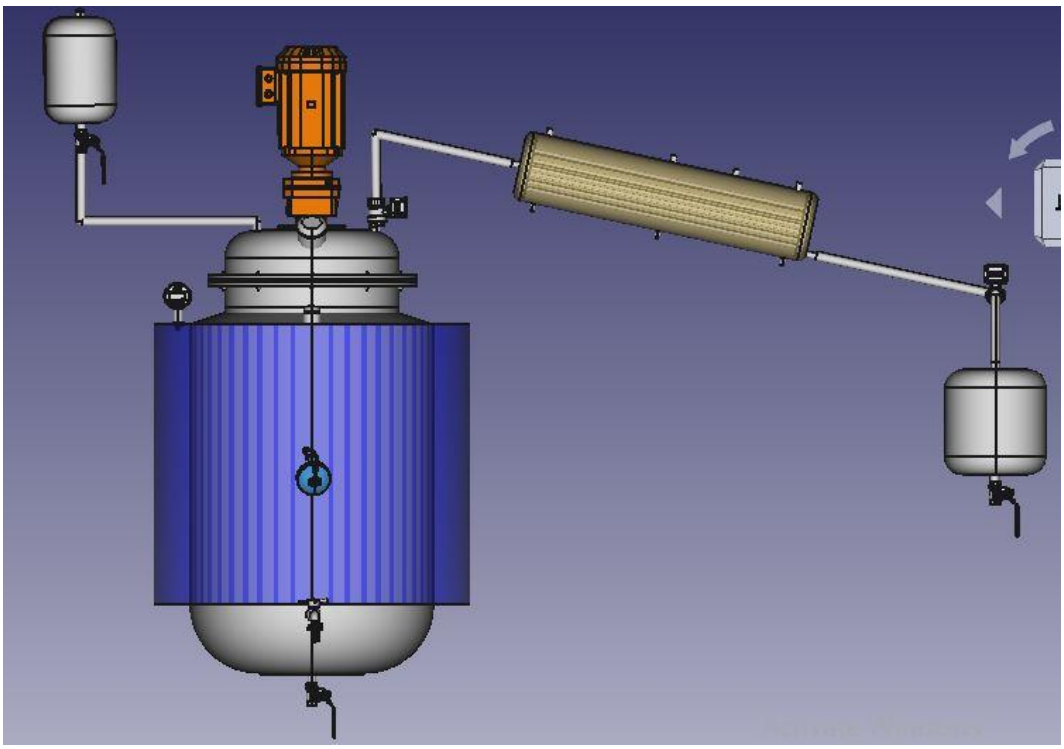
External tanks for acetic anhydride pilot plant

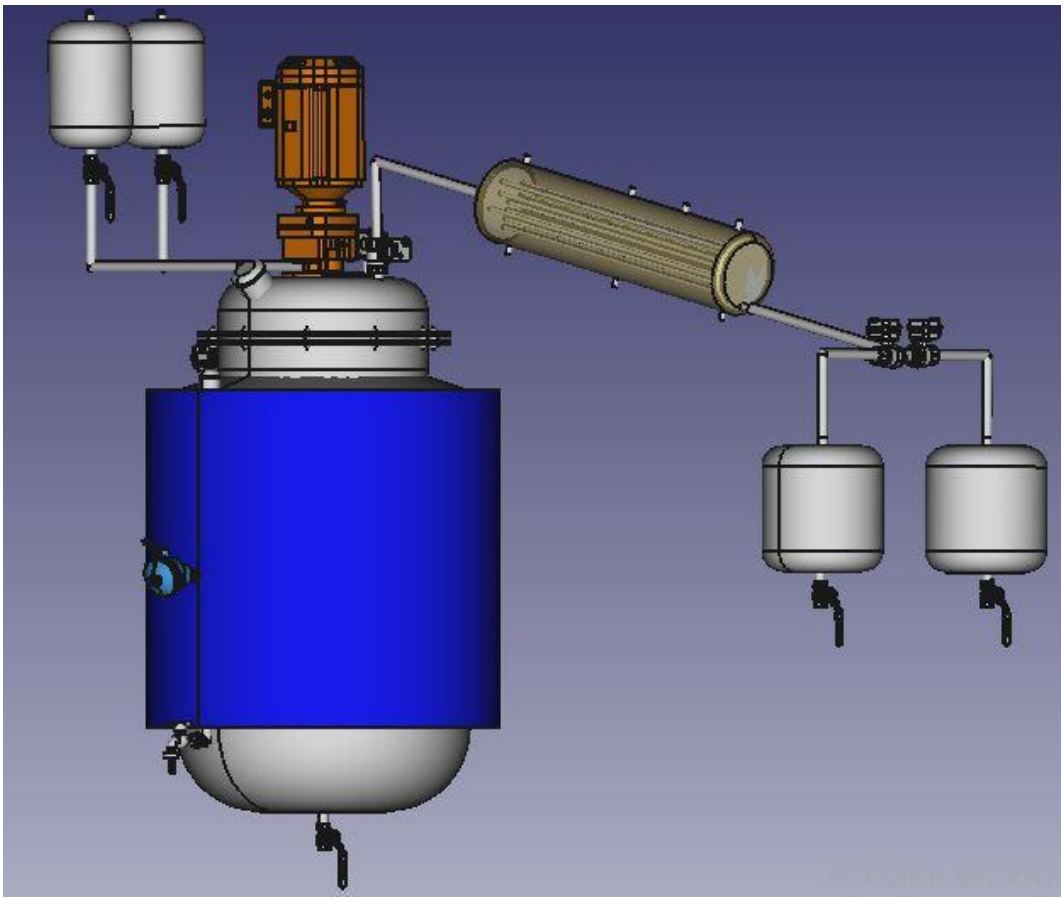
reactor for acetic anhydride0.FCStd



Jacketed reactor for acetic anhydride pilot plant [FreeCAD file]:

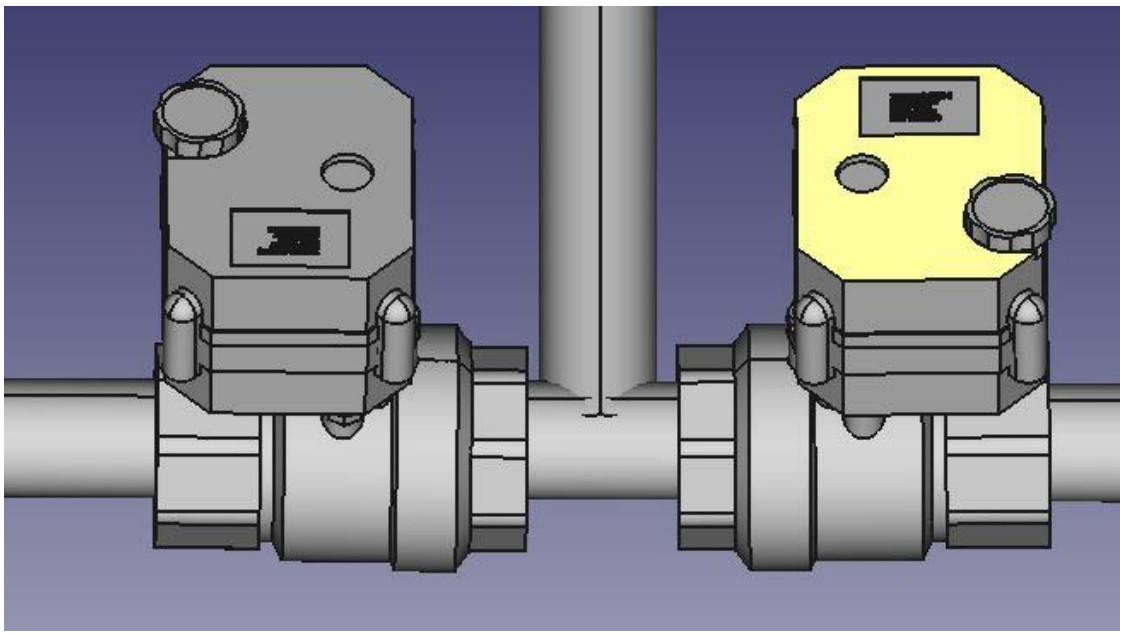
reactor for acetic anhydride1.FCStd



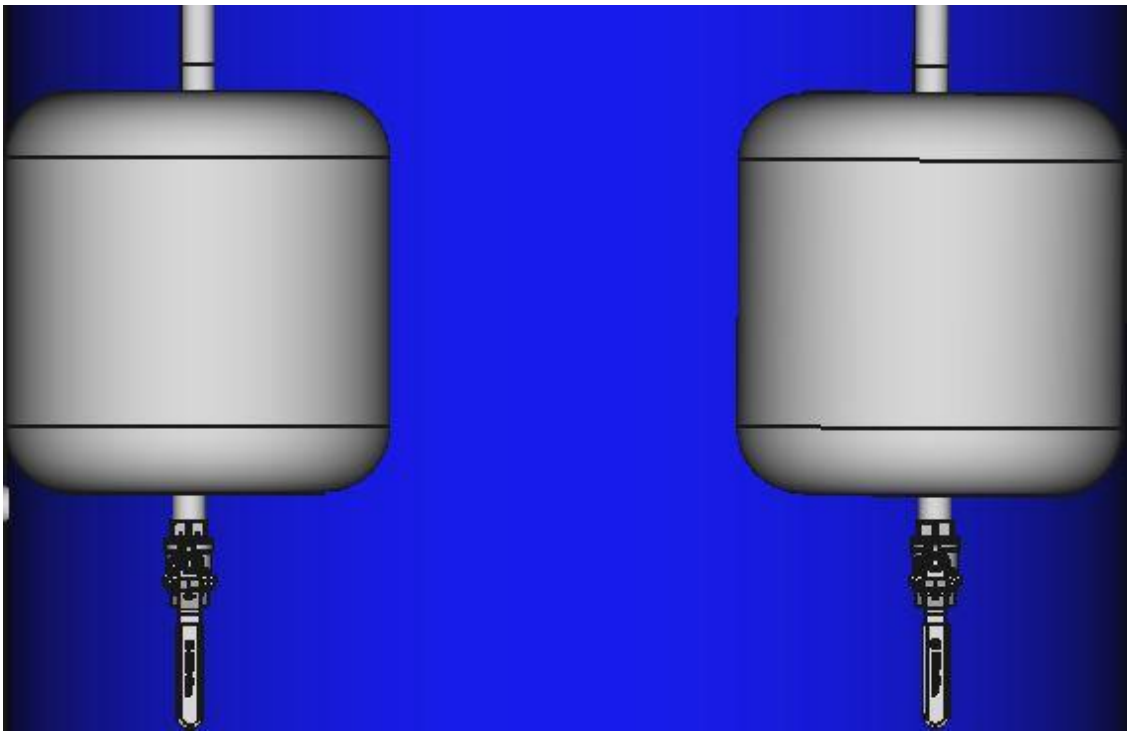


Pilot Plant for Acetic Anhydride [FreeCAD file]:

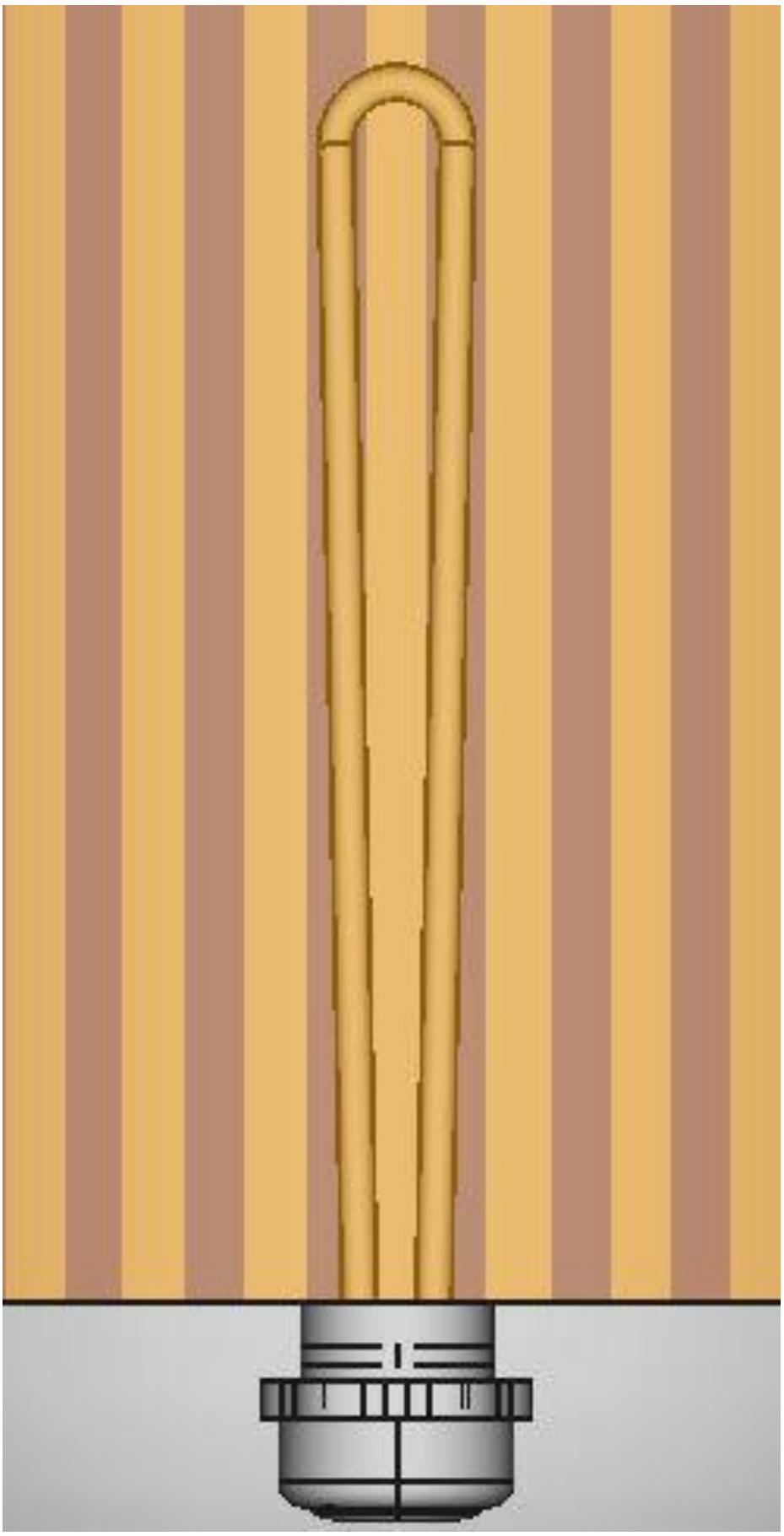
Pilot Plant for acetic anhydride productic



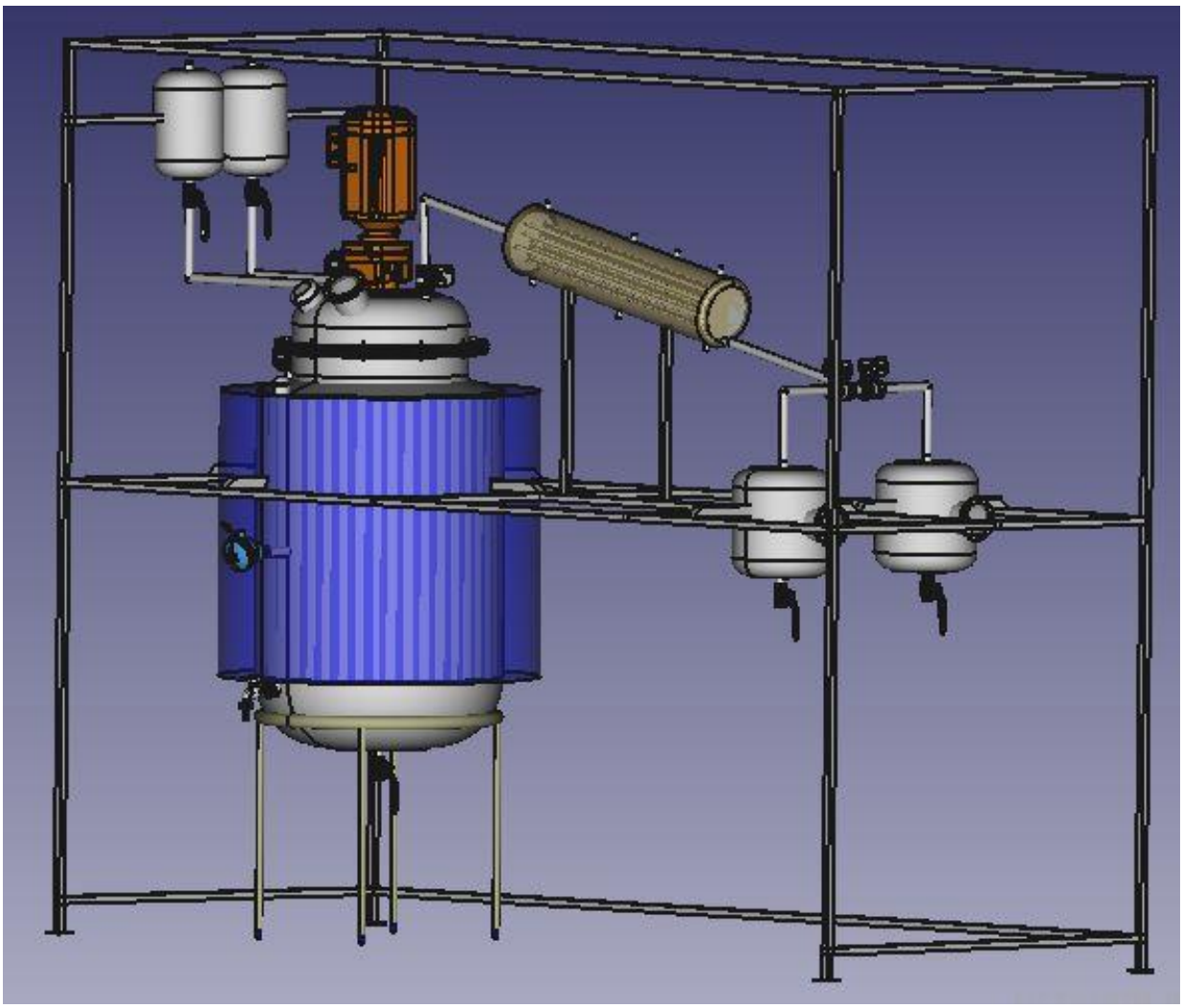
Electrical valve for Acetic Anhydride Pilot Plant



Products tanks (acetic anhydride tank and solvent tank) for Acetic Anhydride Pilot Plant



Electrical Heater for Acetic Anhydride Pilot Plant



Stand for Acetic Anhydride Pilot Plant - 07.02.2024 [FreeCAD file]:

reactor for acetic anhydride2.FCStd

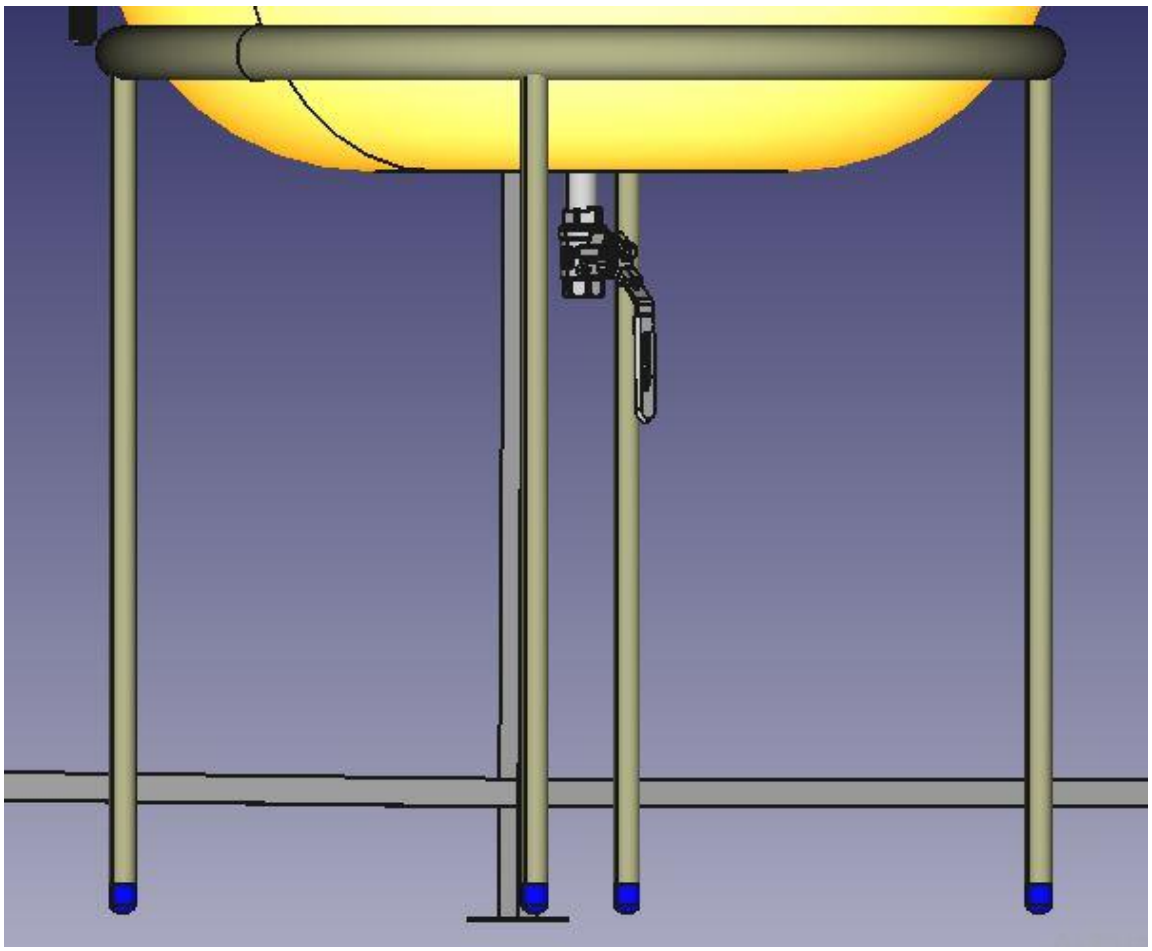


Image 4: Support Stand for the "Reactor of Acetic Anhydride" 07.02.2024

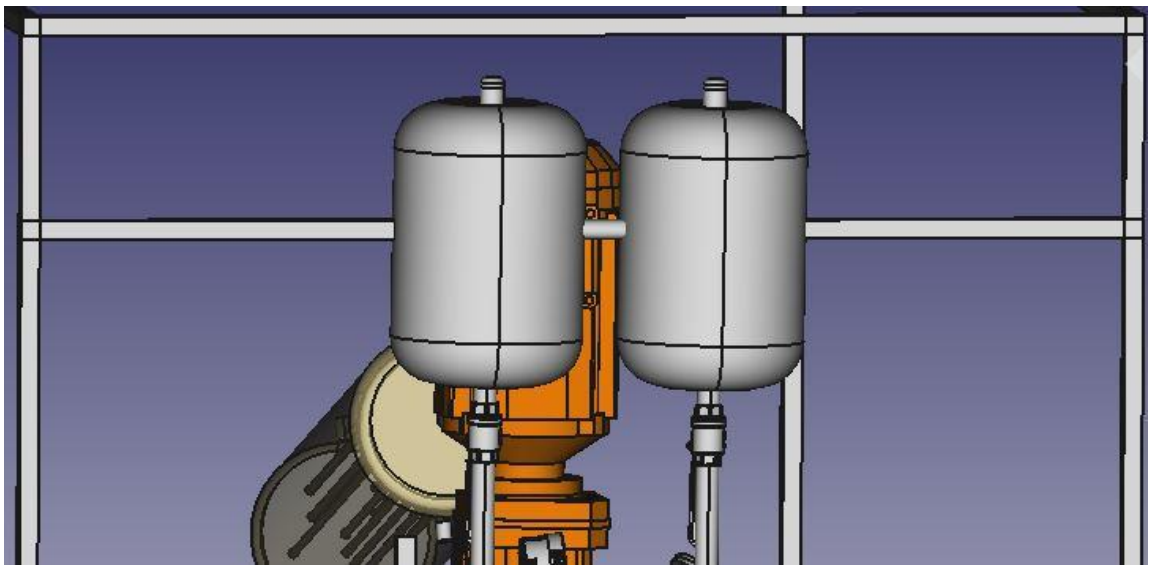


Image 5: support for 2 reagents tanks 07.02.2024

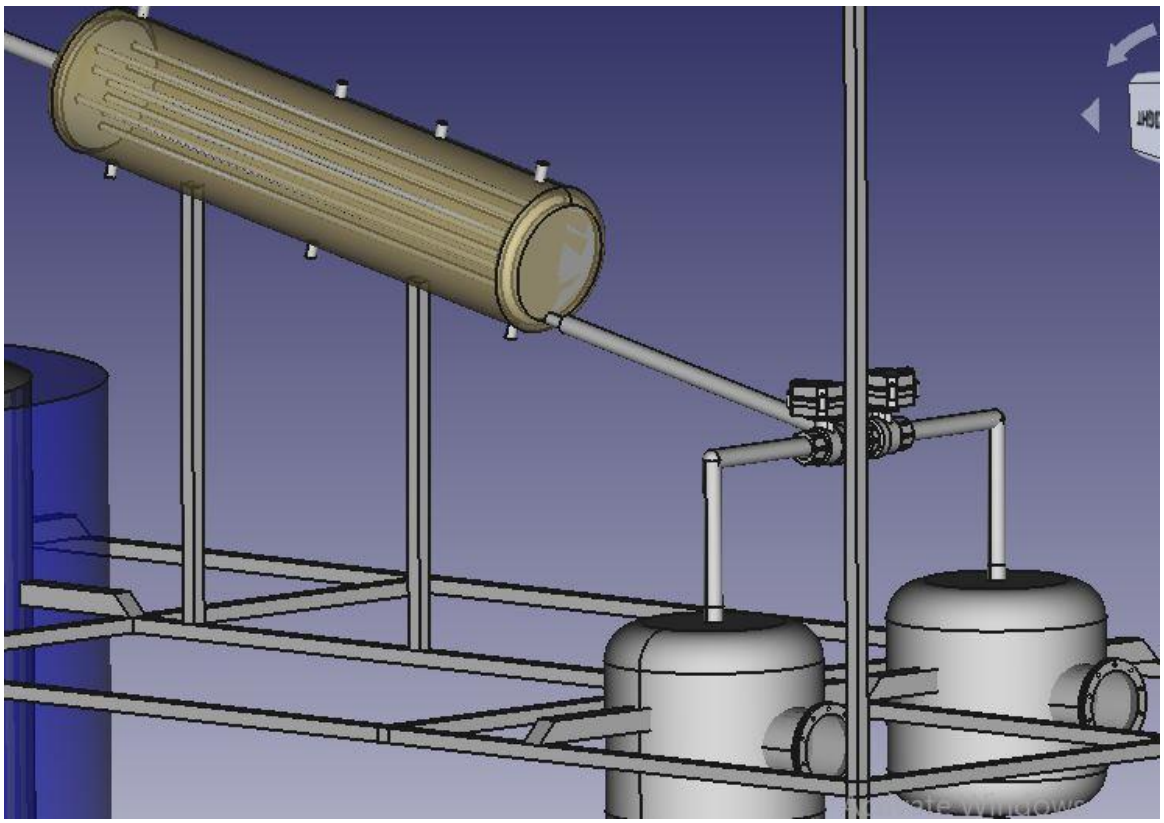
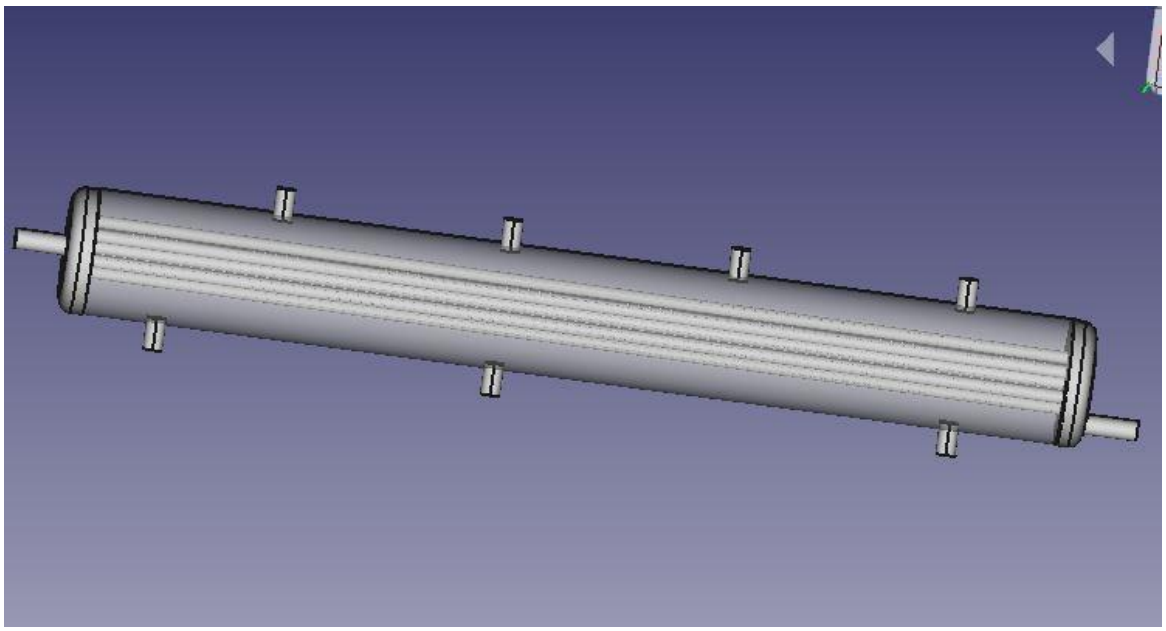


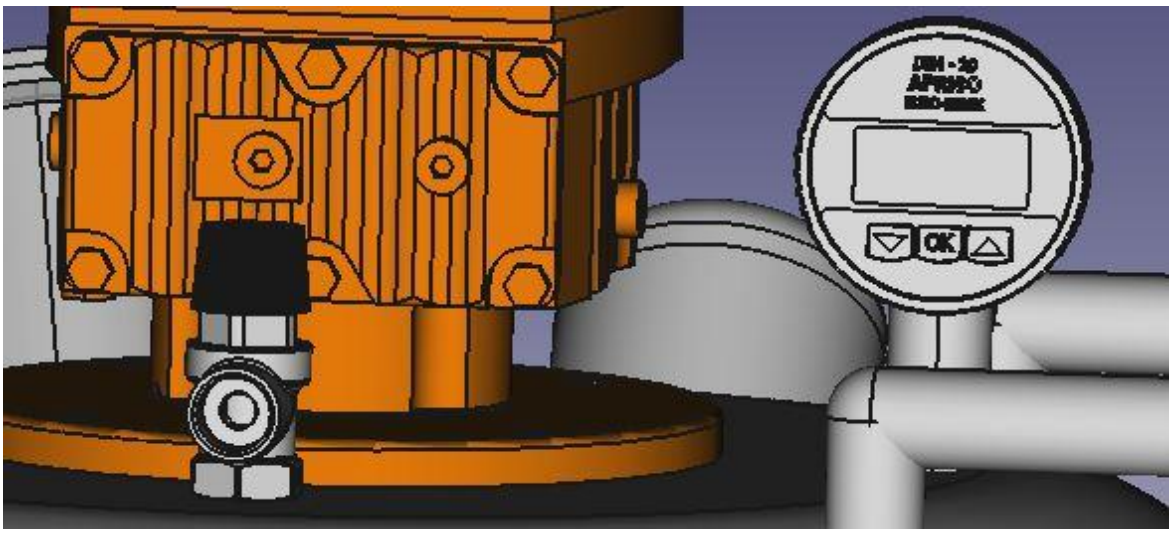
Image 6: Support for 2 products tanks and condenser 07.02.2024



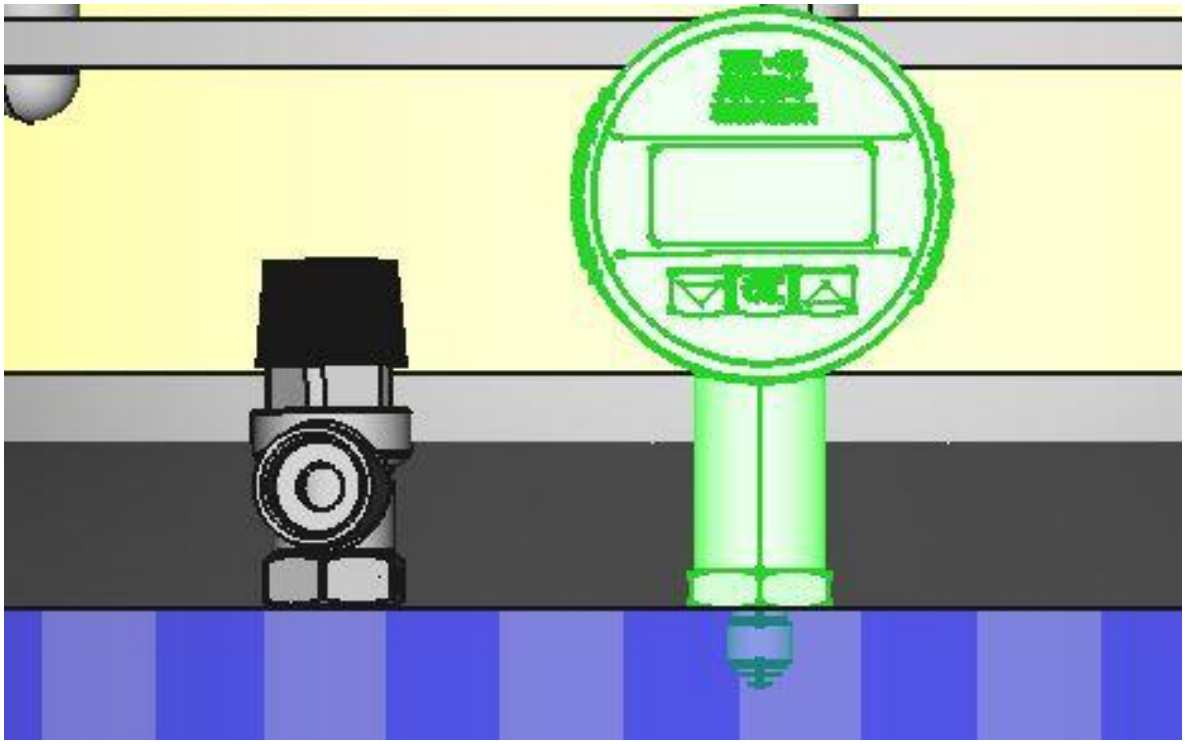
New condenser for acetic anhydride pilot plant 12.02.2024

Last update condenser acetic anhydride pilot plant" 12.02.2024 [FreeCAD file]:

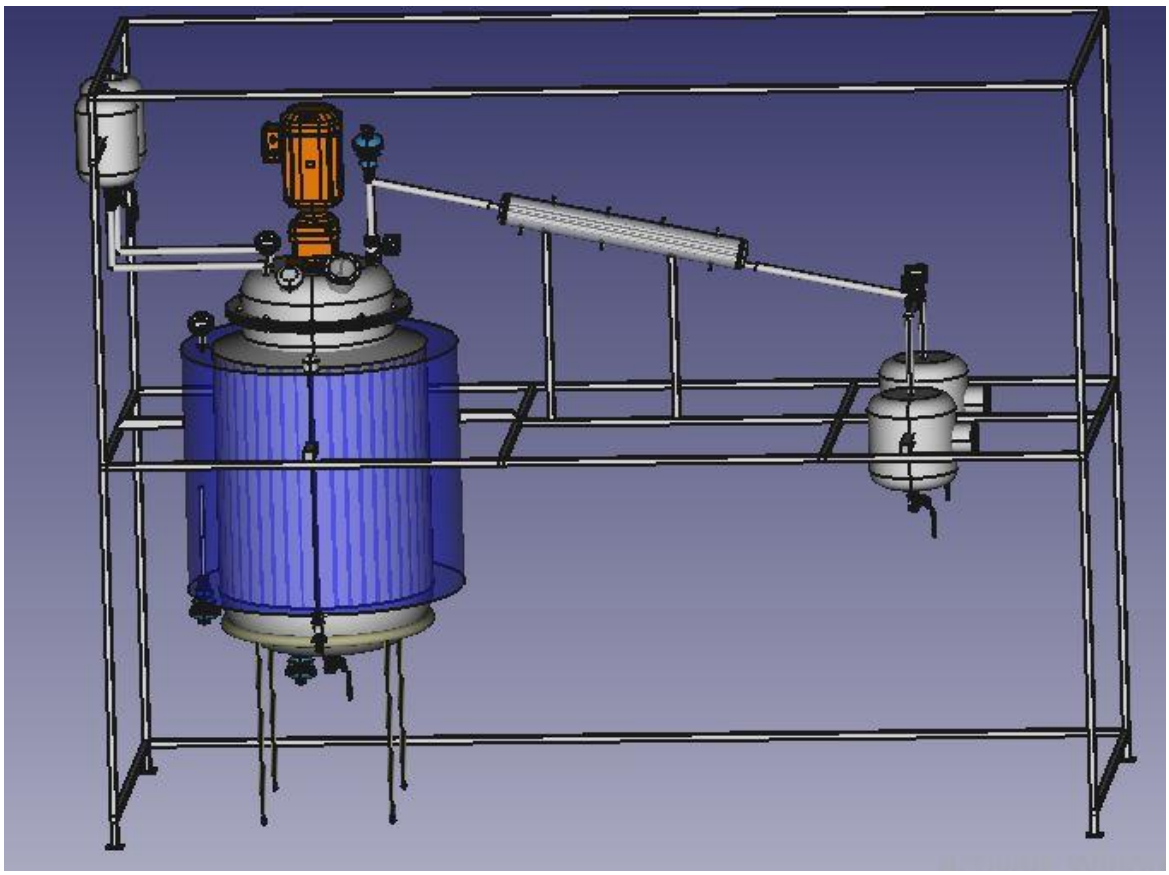
Condenser
MK.FCStd



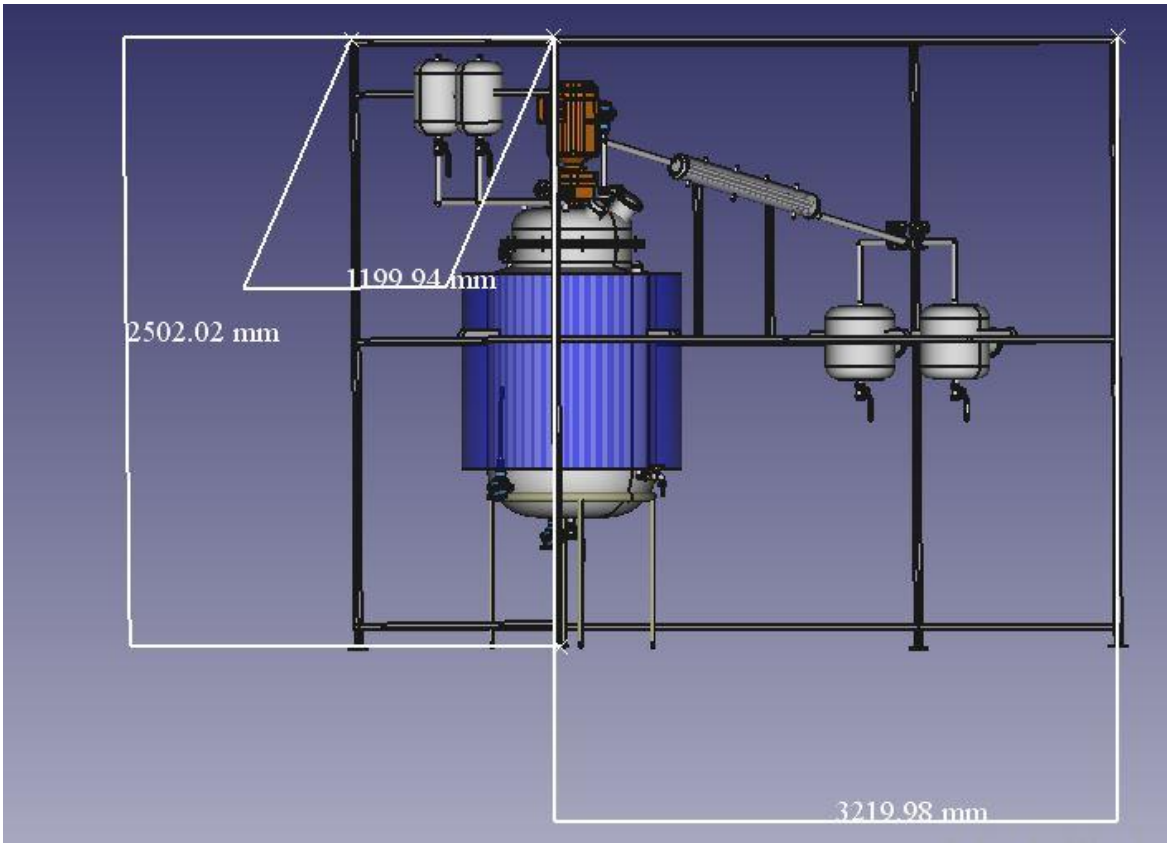
Safety pressure valve in reactor for acetic anhydride pilot plant 22.02.2024

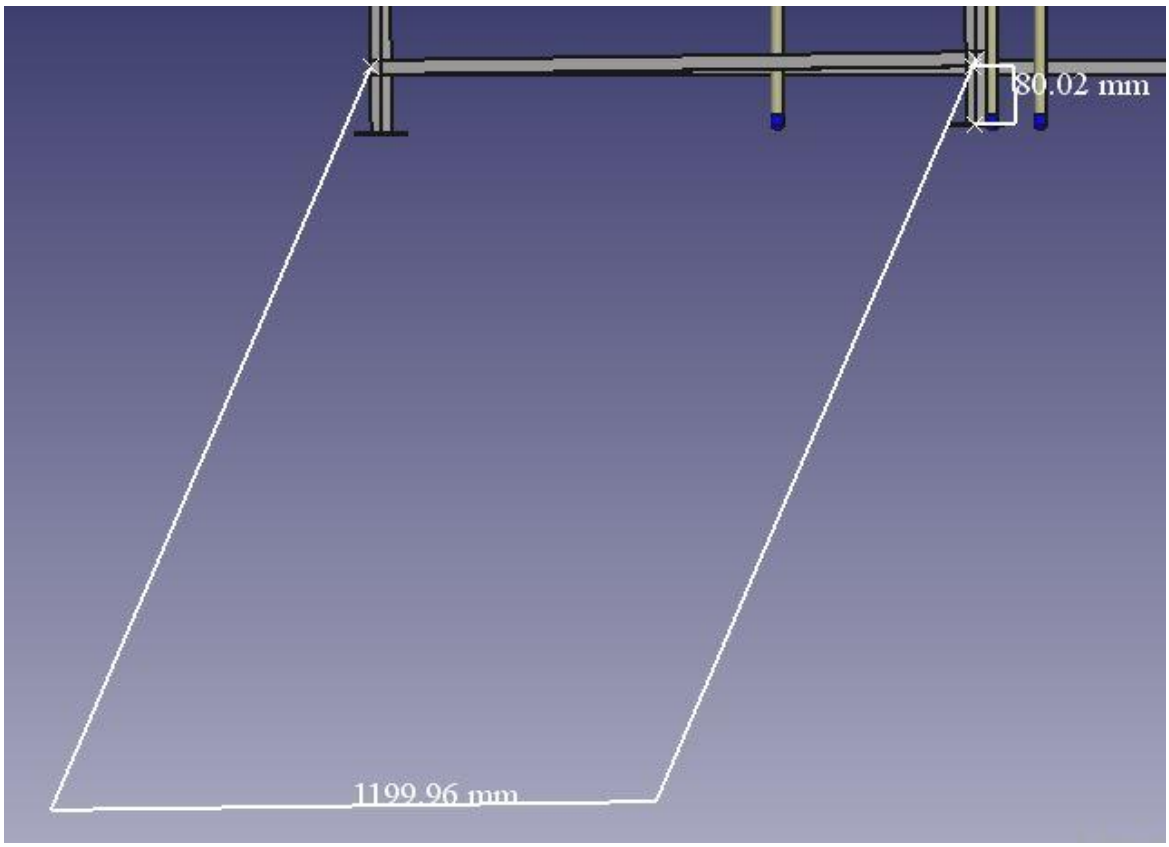


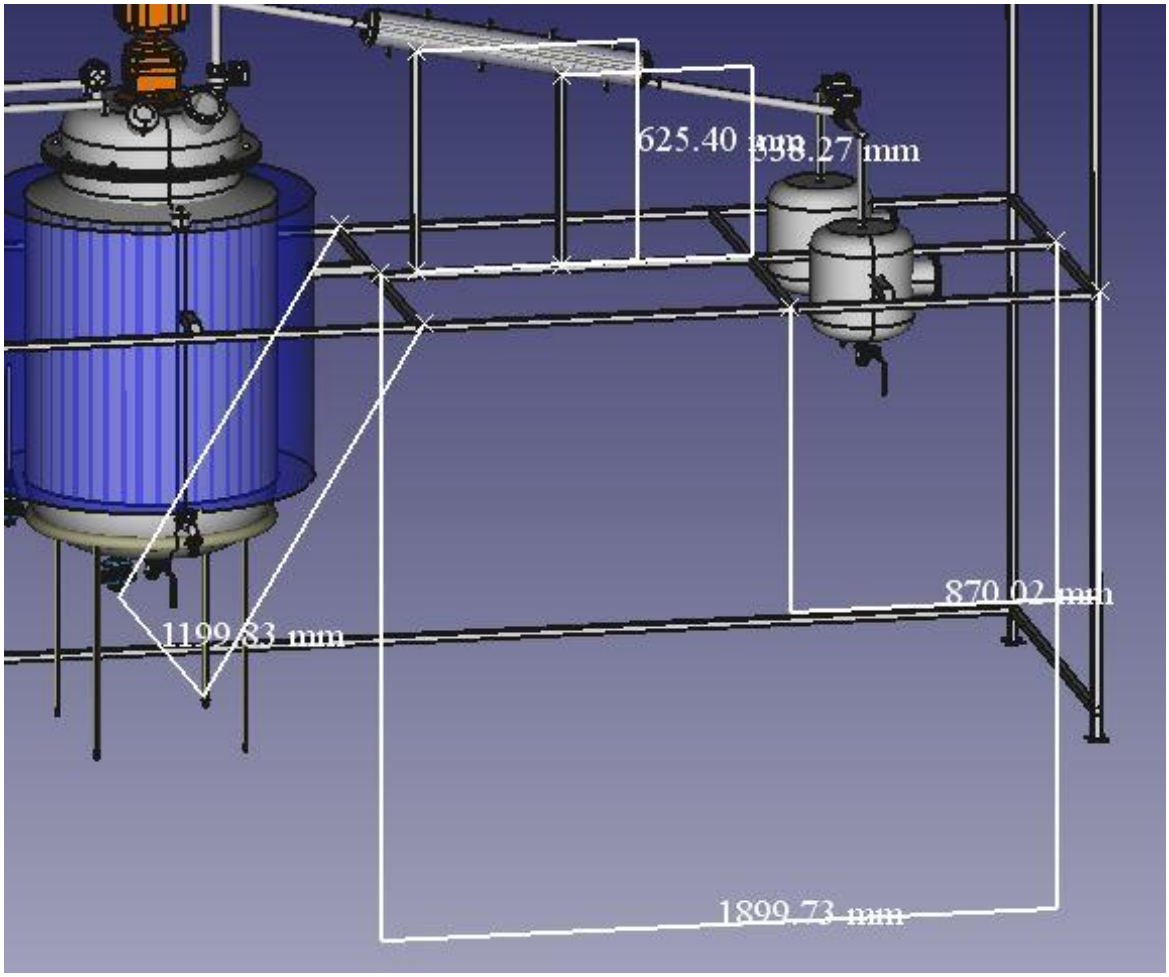
Safety pressure valve in jacket for acetic anhydride pilot plant 22.02.2024

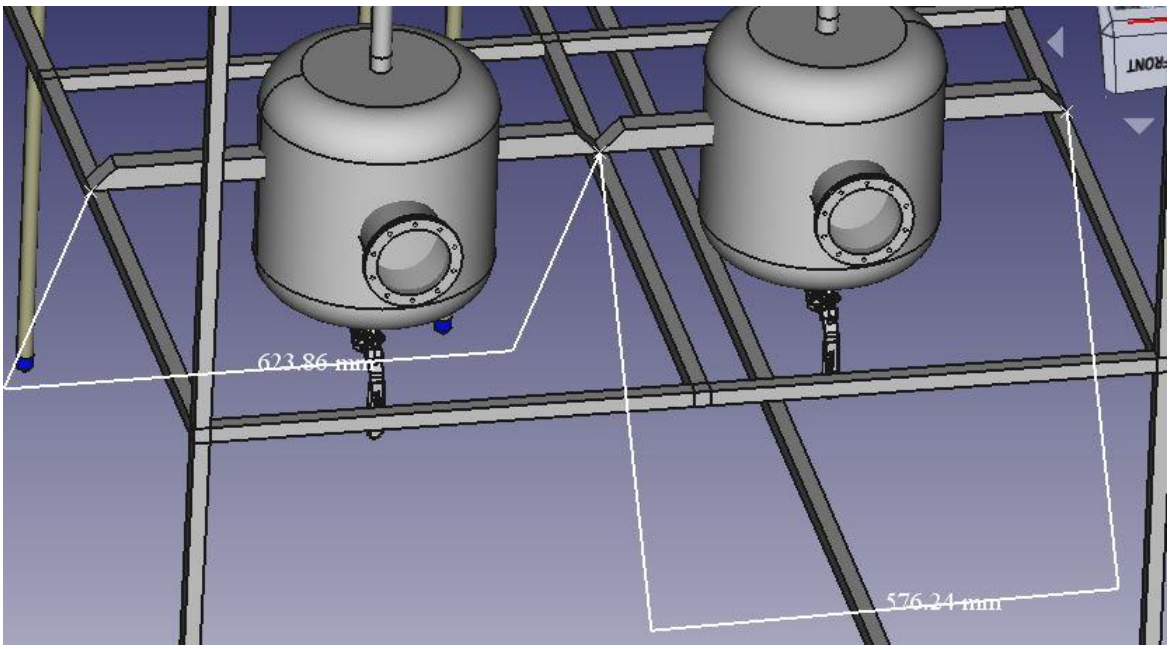


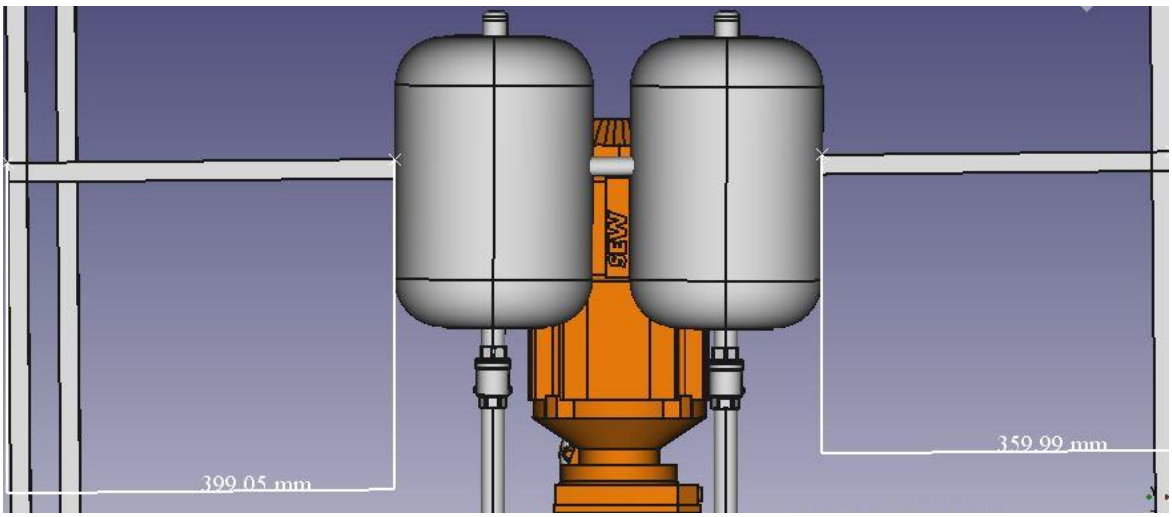
Measurements from the Acetic Anhydride pilot plant

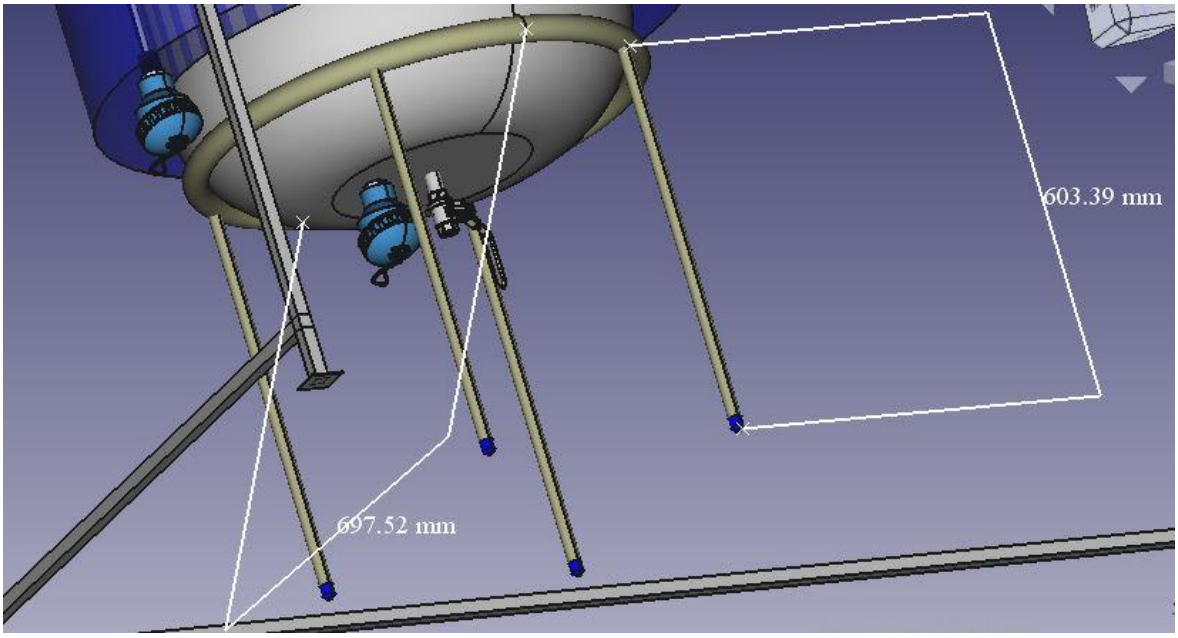


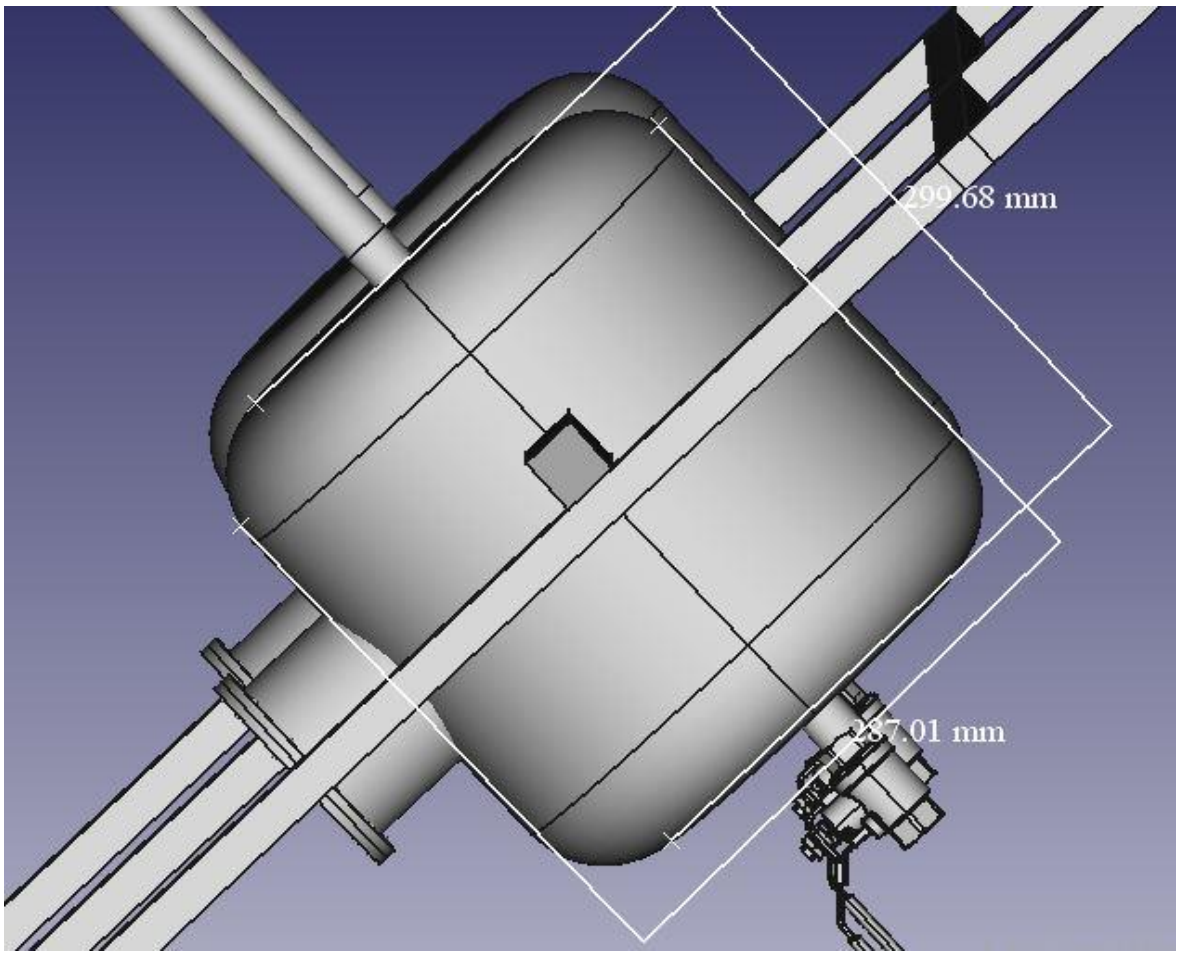

80.02 mm
1199.96 mm

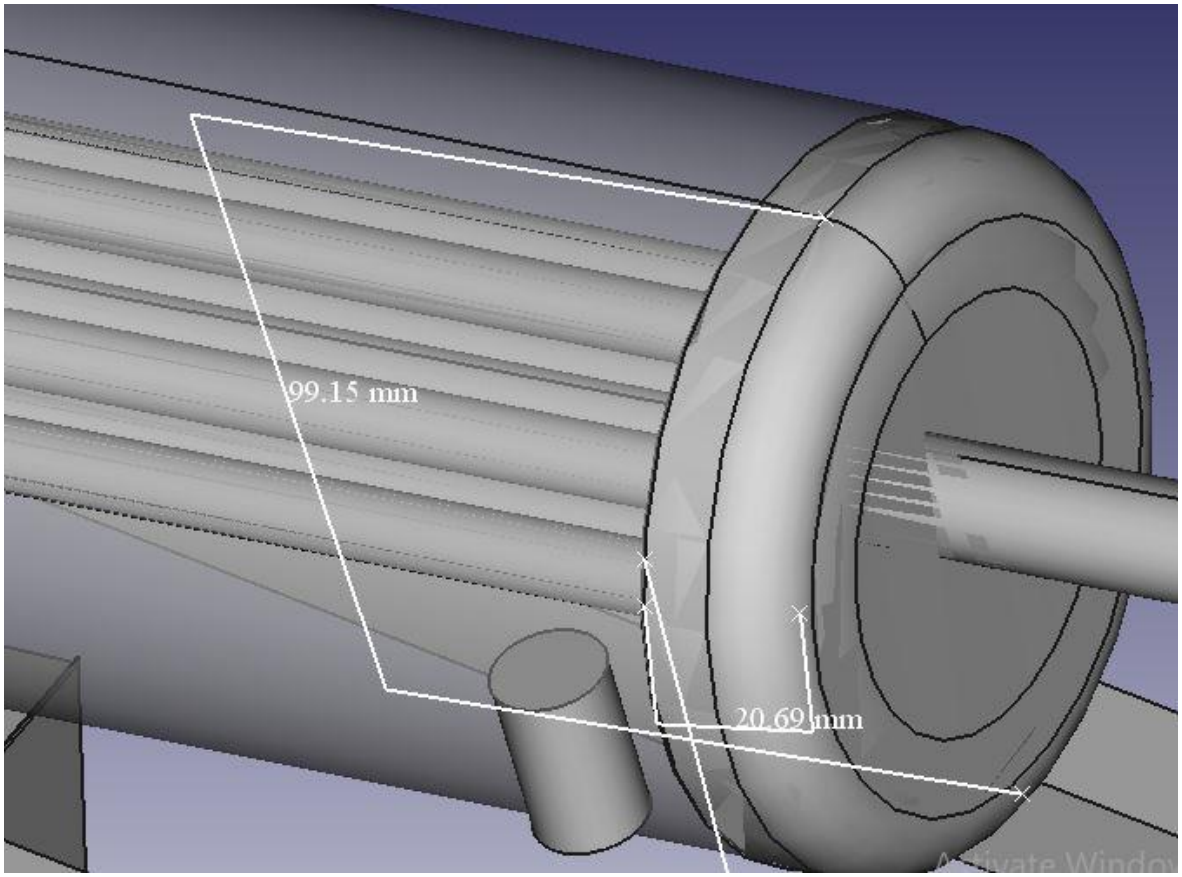

625.40 mm
870.02 mm
1199.83 mm
1899.73 mm

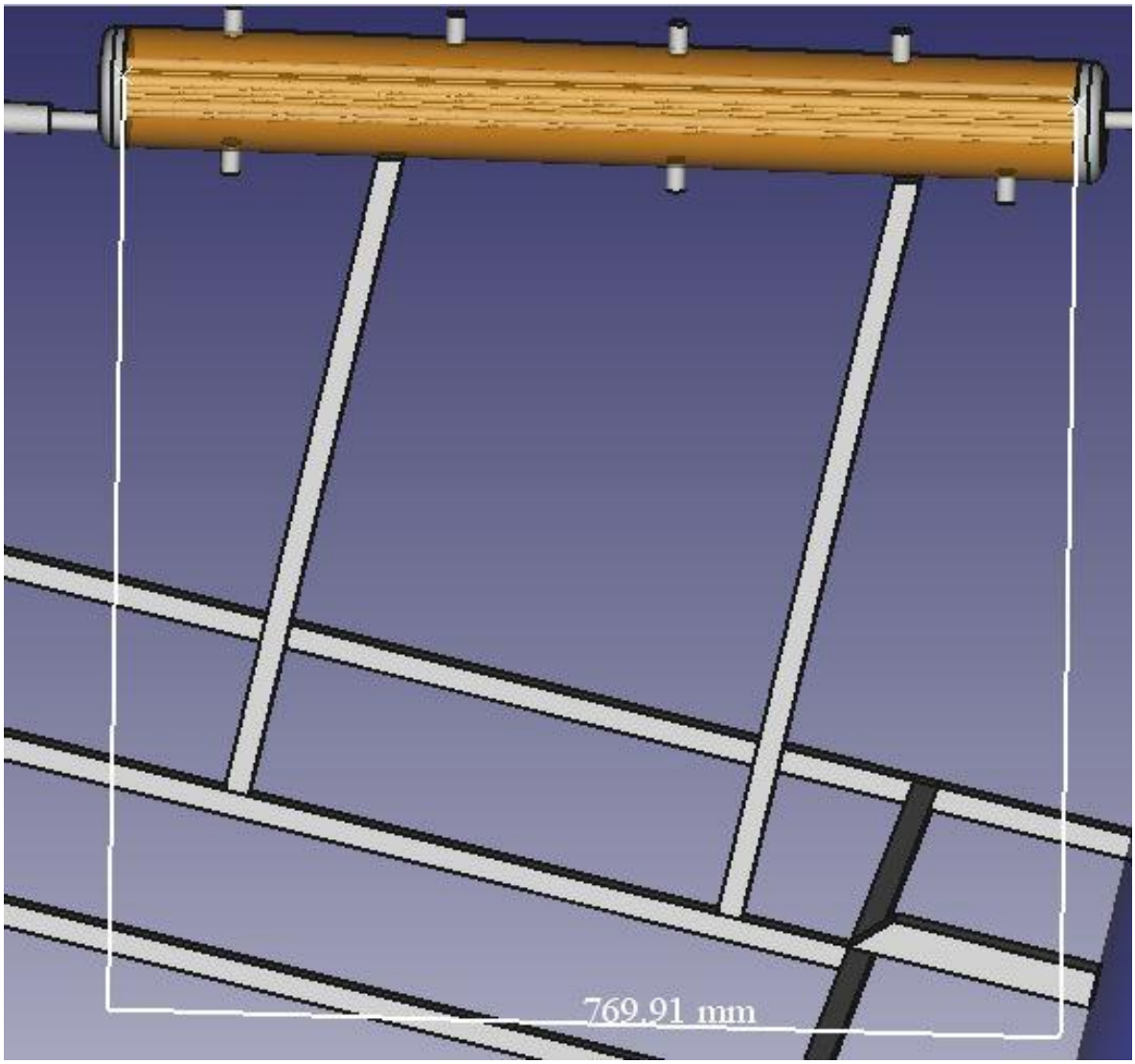

623.86 mm
576.24 mm

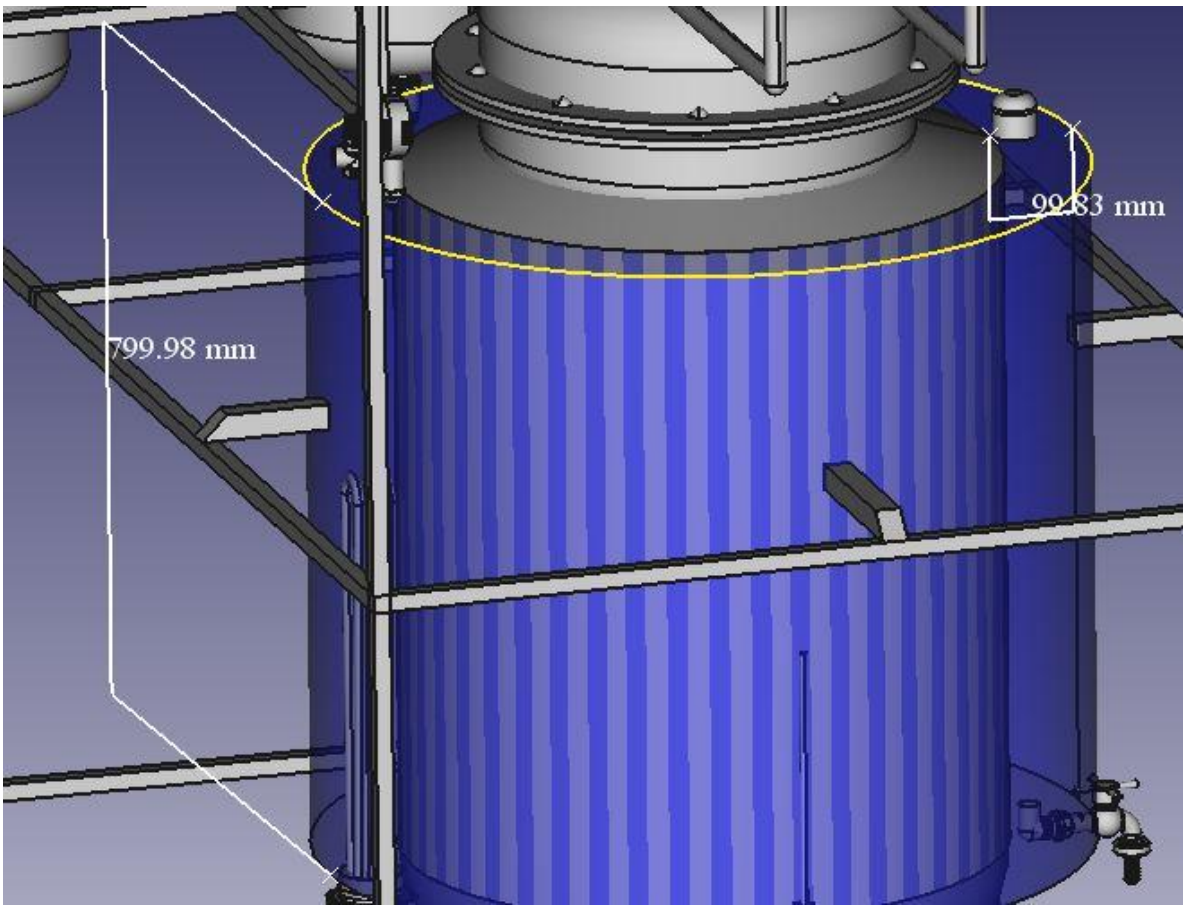

SEW
399.05 mm
359.99 mm

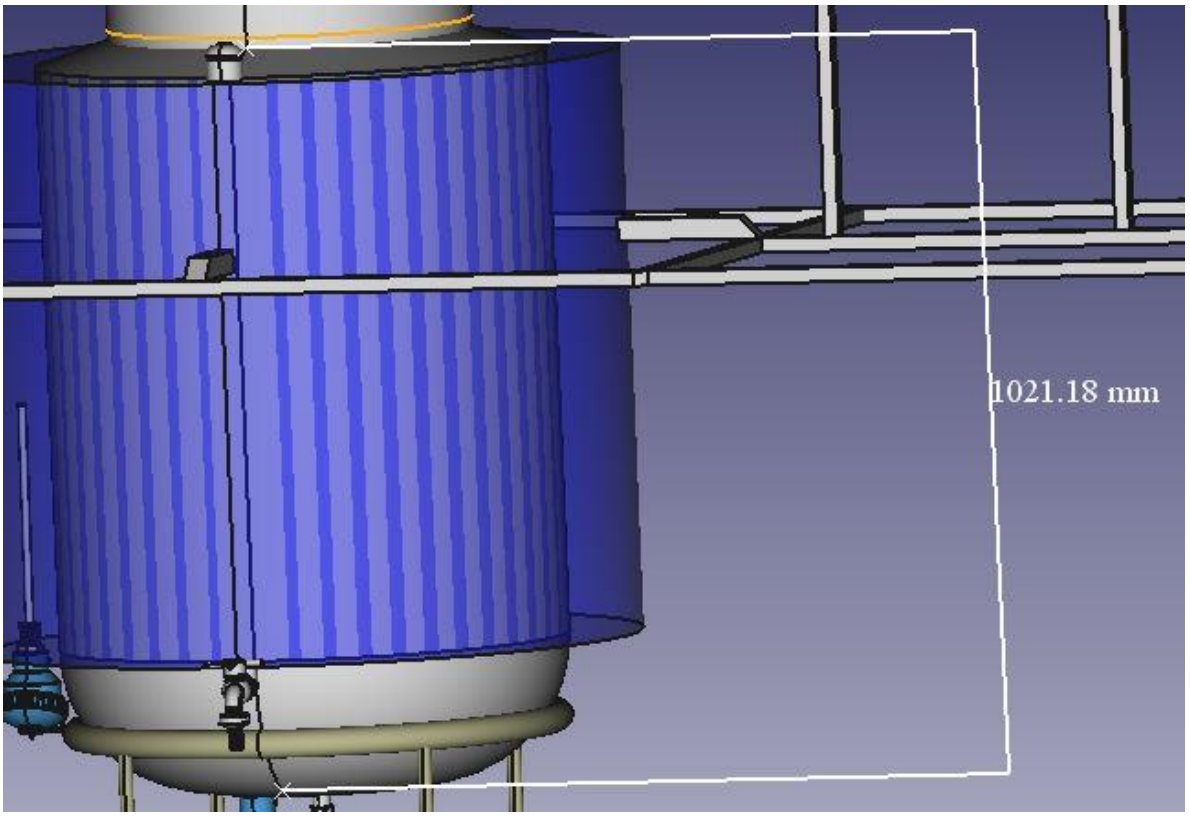

603.39 mm
697.52 mm

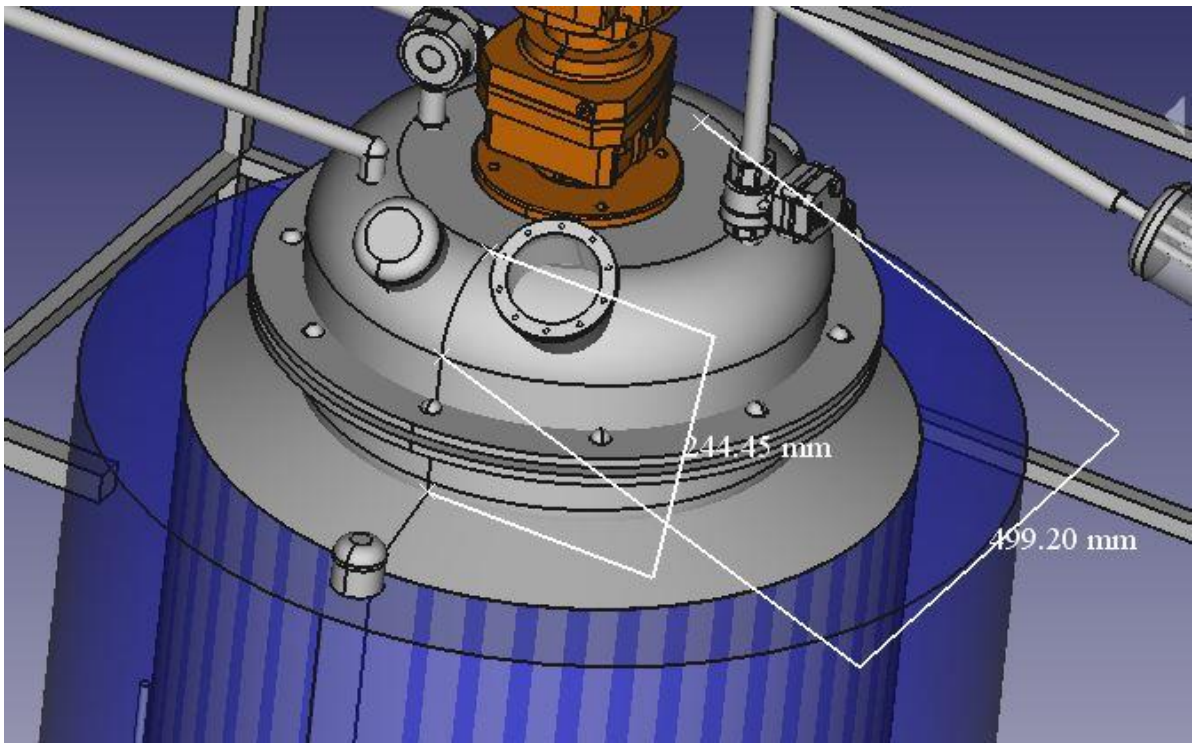

299.68 mm
237.01 mm

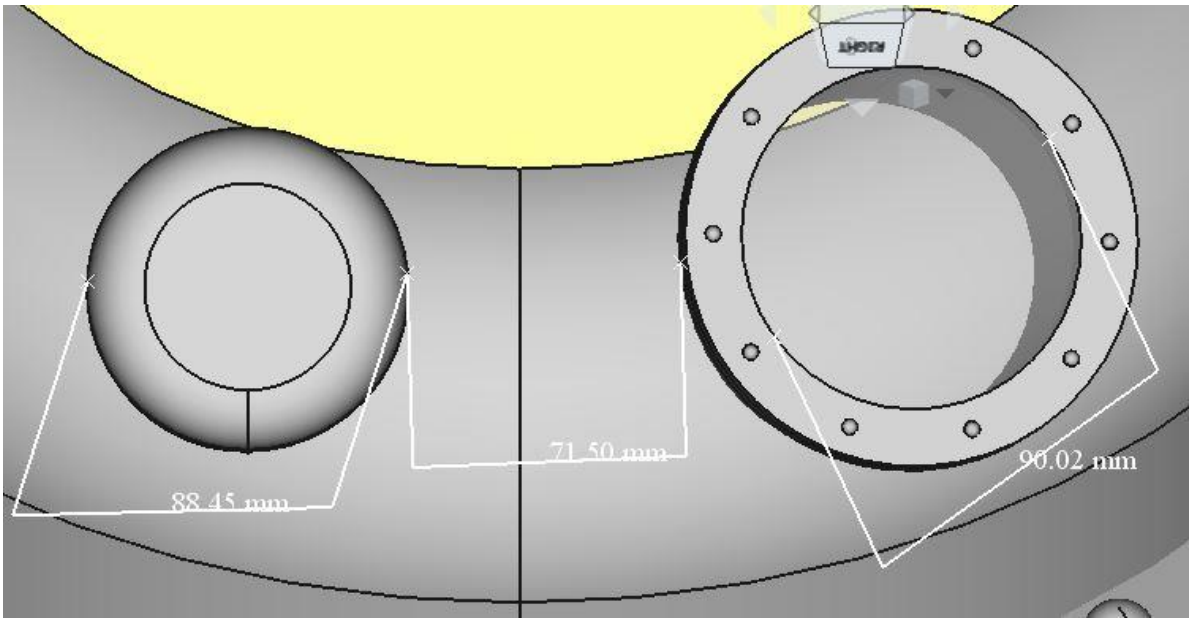

99.15 mm
20.69 mm

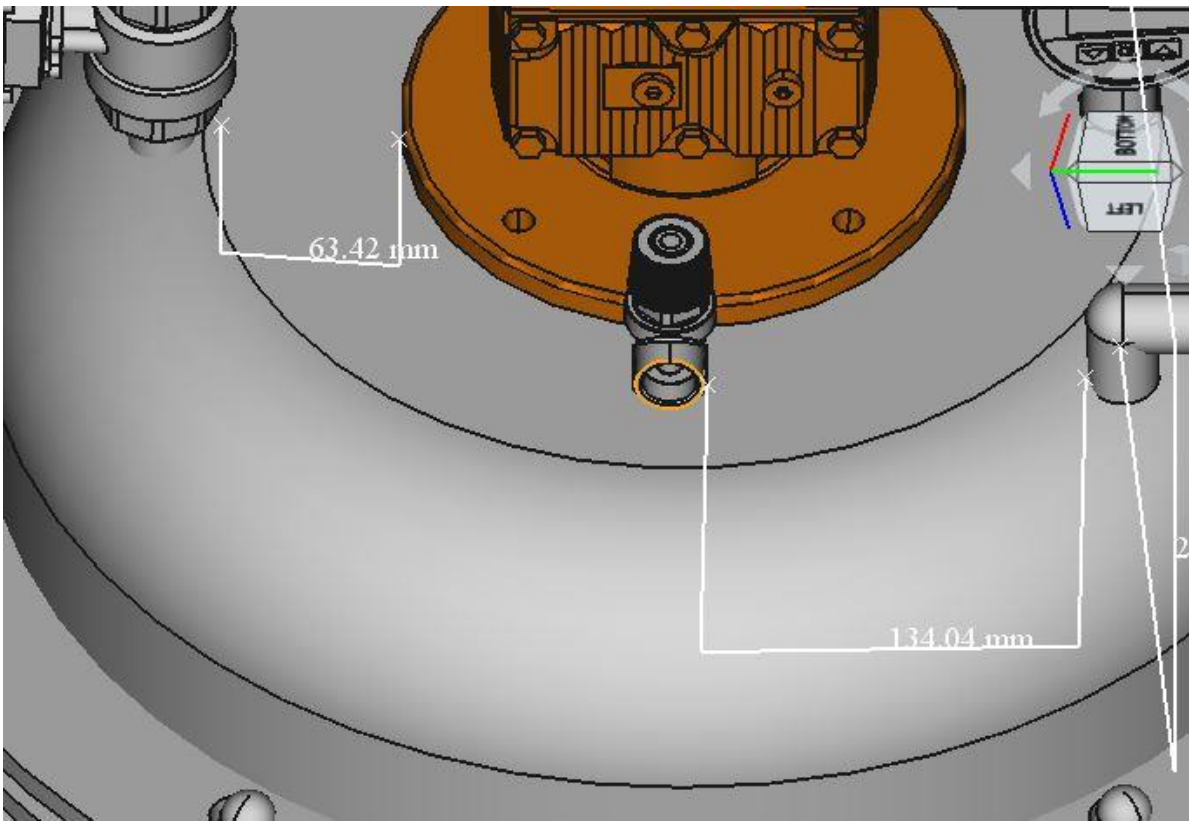

769.91 mm

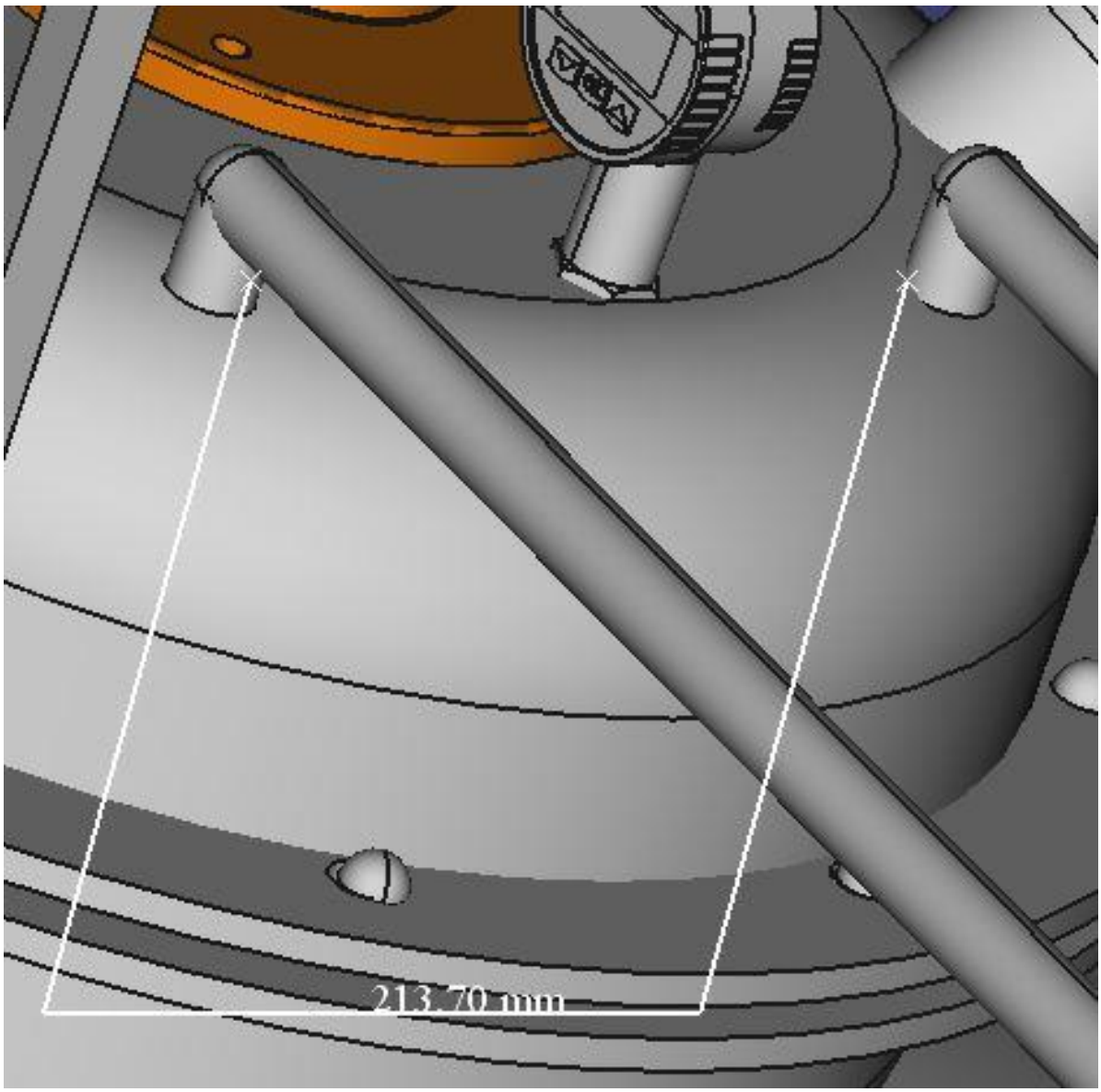

99.83 mm
799.98 mm

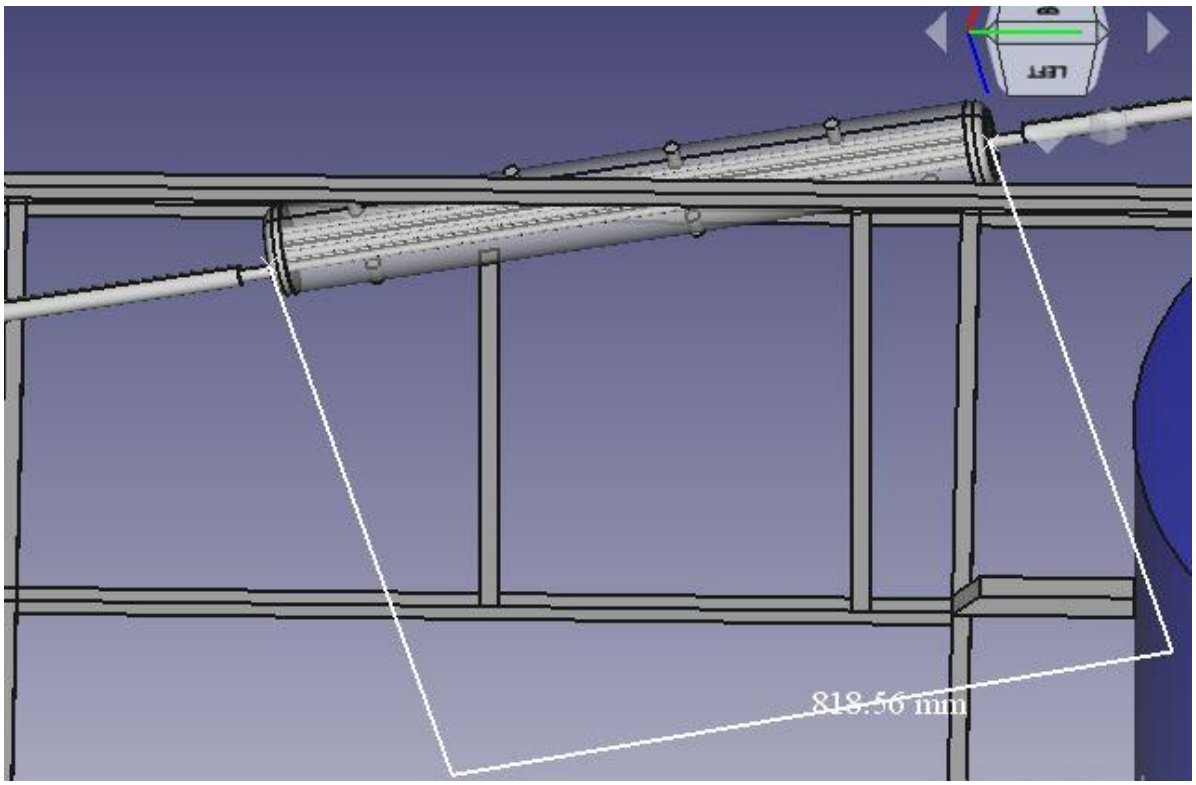

1021.18 mm

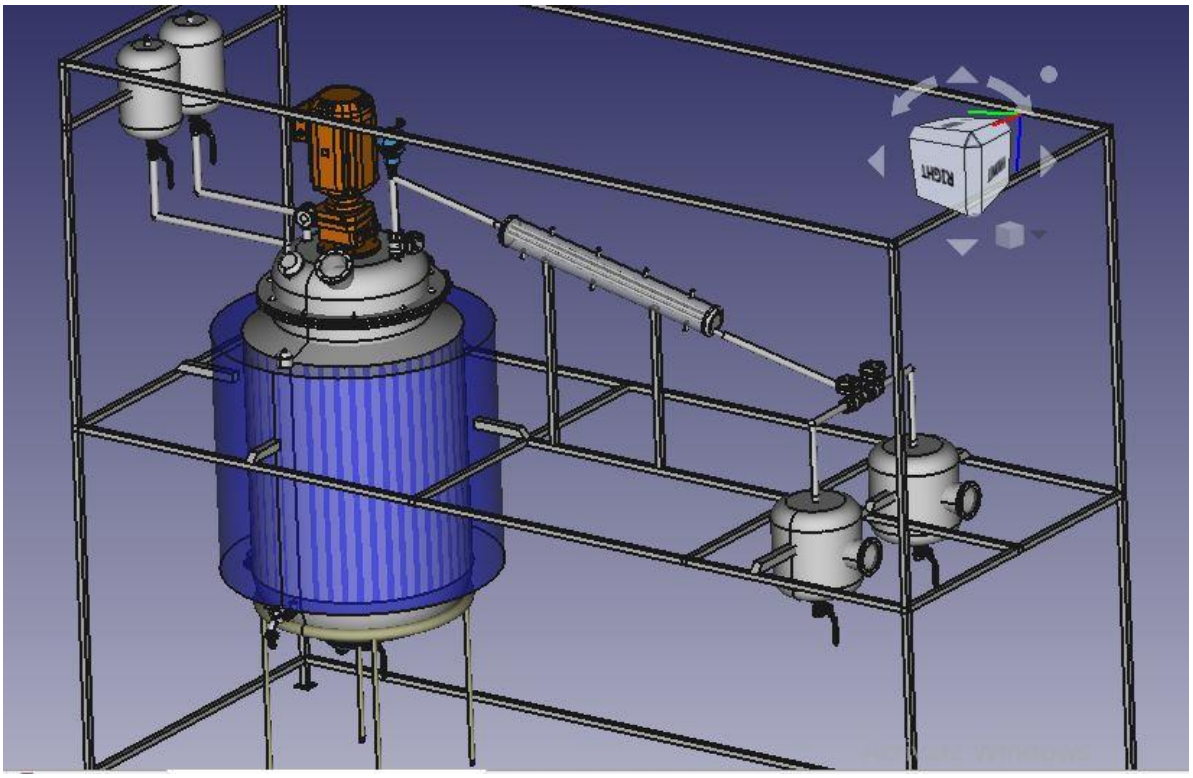

244.45 mm
499.20 mm

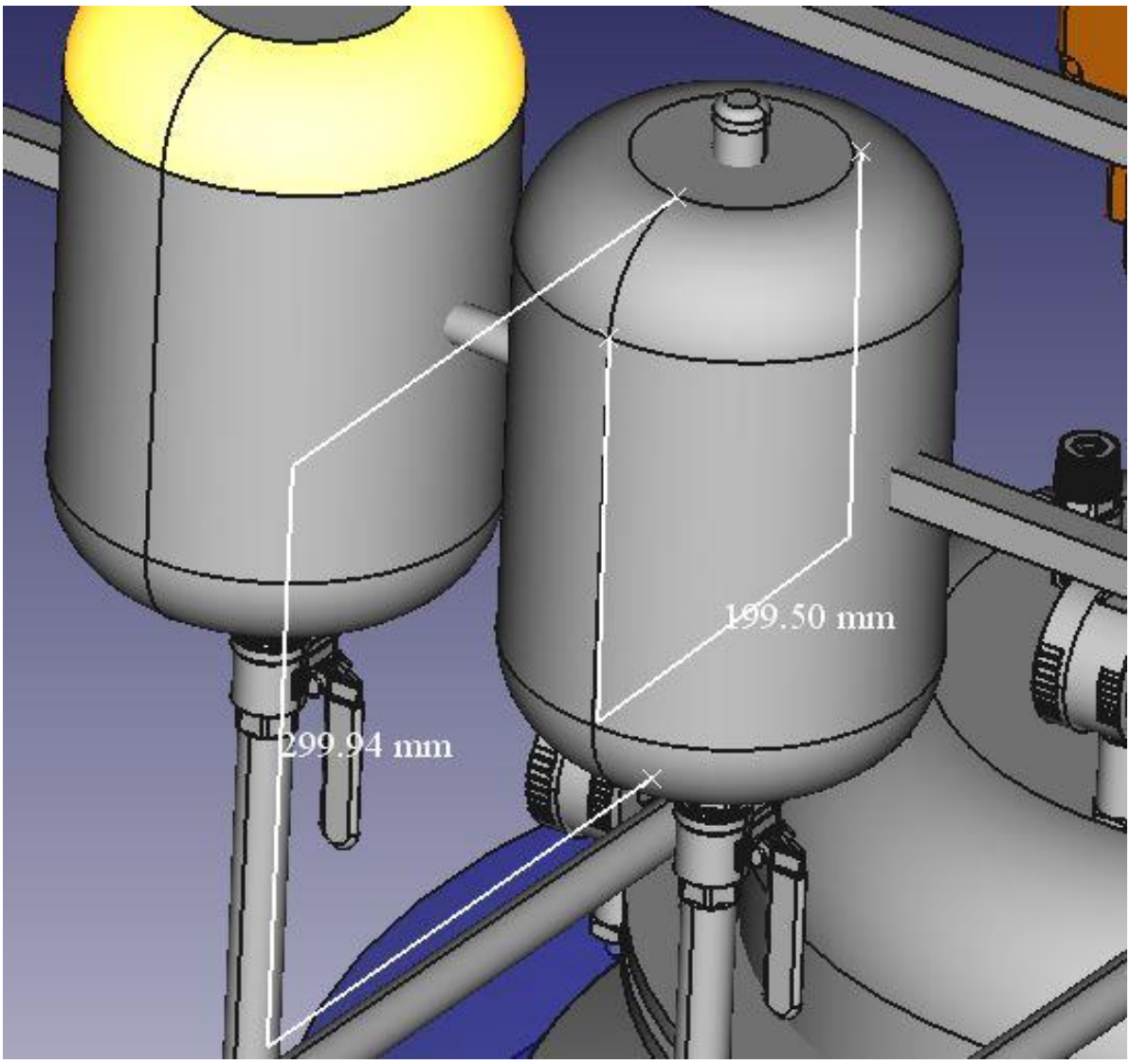

71.50 mm
88.45 mm
90.02 mm


63.42 mm
134.04 mm
BOTTOM
LEFT


213.70 mm

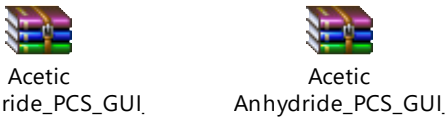

LEFT
818.56 mm


RIGHT



3.3.2 Acetic anhydride PCS implementation

- PCS_AceticAnhydrideProduction_250225 - GUI



- PCS_AceticAnhydrideProduction_250225 - GUI

- Acetic Anhydride PCS _PLC Modbus addresses_201224

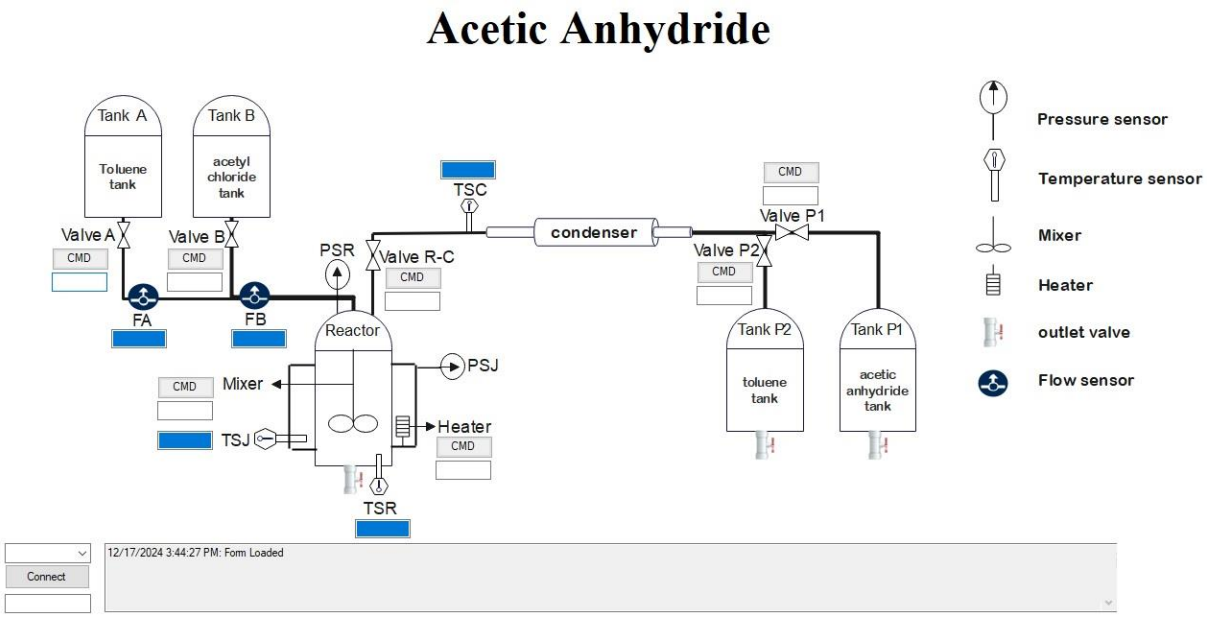
- Acetic_Anhydride_PCS_PLC_24.12.24

Acetic_Anhydride_P
CS_PLC_24.12.24.dvp

- All files concerning the process control system

PCS_AceticAnhydrideProduction_250225.zip

Graphical user interface



3.4 Requirements For Acetic Anhydride Pilot Plant Production

3.4.1 System requirements

- Acetic Anhydride Pilot Plant shall be able to produce the Acetic anhydride.
- The control panel shall be able to control all valves, mixer and read the data of the sensors (Temperature-pressure-Heater).

3.4.2 Physical requirements

- The pipes shall be able to withstand the temperatures and pressures that exist at the points.
  - Temperature that shall be withstood: +100°C.
  - Pressure that shall be withstood: 2 bars.
- The tanks shall be able to withstand the temperature exchanges, pressures, and mechanical forces that exist at the points.
  - Temperature that shall be withstood: +100°C.
  - Pressure that shall be withstood: 2 bars
  - mechanical force: mixer movements and rotation.

- The sensors (Temperature, Pressure, and Flow) shall be able to withstand the temperatures and pressures that exist at the points.
  - Temperature that shall be withstood: +100℃.
  - Pressure that shall be withstood: 2 bars.

3.4.3 Chemical requirements

- The Tanks system shall be able to insulate the chemical reagents.
- The Tanks system shall be able to withstand the corrosion with organic reagents acetic anhydride, toluene, acetyl chloride and acetic acid.
- The pipe system used shall be able to withstand the corrosion with acetic anhydride, toluene, acetyl chloride and acetic acid.
- The valves shall be able to withstand the corrosion with acetic anhydride, toluene, acetyl chloride, and acetic acid.
- The sensors (Temperature, Pressure, and flow) shall withstand corrosion with acetic anhydride, toluene, acetyl chloride, and acetic acid.

3.4.4 Mechanical requirements

- The Tank system shall be made of Stainless Steel 316.
- The Tank system shall be able to close the system completely.
- The pipes shall be made of stainless steel 316.
- The pipes shall be able to resist the pressure without letting gas or vapor exit through.
- The valves shall be made of stainless steel 316.
- The valves shall be able to close completely.
- The valves shall be able to open or close with independent pressure.
- The sensors shall be made of stainless steel 316.
- The sensors shall be able to close the system completely.
- The sensors shall be able to read the data from the system.
- The Acetic Anhydride pilot plant shall be designed according to the *mechanical design*.

3.5 Pilot Plant test specification

3.5.1 Pre-Starting

Please read these instructions thoroughly. This will make sure you obtain full safe use, Keep this instruction manual in a handy place for future reference.

3.5.2 Prepare the reactor

1. Make sure all valves are closed
2. Make sure the power is turned off
3. Connect the reagent valve to the reactor
4. Reaction nb 1: Put the reagents amount needed in the reactor (amount of reagents: 4.88 kg Sodium bicarbonate, 74.4L vinegar)
5. Reaction nb 2: Put the new reagents amount needed in the reactor (amount of reagents: 3.77 L acetyl chloride, **5**L Toluene-Solvent)

6. Closed the valve for reagent filling.

3.5.3 Safety precaution

- The hot water (+100 °c) could suffer some burns (tank number 1 = Reactor)
- Toluene: Sweet aroma, hidden dangers - inhalation, skin, and fire risks; long-term impacts on organs, development, and nervous system.
- Acetyl chloride: corrosive, flammable, explosive, lung irritant, potential carcinogen.
- Acetic anhydride: highly corrosive, burns eyes and skin, inhaling damages lungs, potential carcinogen, flammable, and reacts violently with many substances.

(Wear protective gloves/protective clothing/eye protection/face protection).

3.5.4 Acetic Anhydride Production Operation

1. Ensure all sanitary connections
2. Put the dangerous reagents in the "reagents tanks" (tank A= Acetyl chloride and tank B= Acetone)
3. Put the reagents in the reactor (vinegar and sodium bicarbonate)
    1) Reaction 1: (vinegar or acetic acid 5%, sodium bicarbonate)
    2) Reaction 2: "solvent: toluene", sodium acetate and Acetyl chloride)
4. Plug the control system
5. Check the control system if it's working properly
6. Operate to boiling up the water (+100°c) in the "Jacket Reactor"
7. Operate the mixer to mix the reagents in the "Reactor"
8. Operate the reagents tanks (tank A and tank B) to transfer the reagents (from tank A and tank B to the "Reactor")
9. Operate to warm the water (50-80°c) in the "Jacket Reactor"
10. Operate to circling the water in the condenser
11. After finishing, operate the pipe to close.

3.5.5 001: ACETIC ANHYDRIDE PRODUCTION SYSTEM TEST

Step	**Step Description**	**Expected Result**
Precondition	System is OFF	
TURNING ON the system	Turn ON the GUI	The system is ON

Switch ON the heater (Jacket Reactor)	Turn ON the heater from the GUI	Reaction 1 THE HEATER is heating the water in the jacket till it reaches +100°C indicated on the TSJ (temperature sensor of the jacket)
Switch ON the mixer (Reactor tank)	Turn ON the mixer from the GUI	Mixing the reagents (manual added) to obtain the mixture in the "Reactor" till the water evaporated (creating anhydrous conditions)
Switch OFF the heater (Jacket Reactor)	Switch OFF the heater from the GUI	The water in the "Reactor jacket" and the mixture in the "Reactor" is cooled till it reaches 20-30°C, indicated on the TSJ (temperature sensor of the jacket)
1- Open the valve B (tank B: tank acetyl chloride) 2- Close the valve B (tank B: tank acetyl chloride)	1- Open the valve VB to transfer 3.77L acetyl chloride from Tank B to "Reactor" 2- Close the valve VB after transferring 3.77L acetyl chloride from tank B to "Reactor"	Reaction 2 1- 3.77L of Acetyl chloride is transferred to "Reactor" indicated on the FB (flow sensor tank B) 2- Valve B is closed after 3.77L is transferred to "Reactor" indicated on the FB (flow sensor tank B)
1- Open the valve A (tank A: tank Toluene) 2- Close the valve A (tank A: tank toluene)	1- Open the valve VA to transfer 5L toluene from Tank A to "Reactor" 2- Close the valve VB after transferred 5L toluene from tank A to "Reactor"	1- 5L of Toluene is transferred to "Reactor" indicated on the FA (flow sensor tank A) 2- Valve B is closed after 5L is transferred to "Reactor" indicated on the FA (flow sensor tank A)
Turn ON the water circulation in the condenser	Turn ON the water circulation in the condenser to start the distillation	The condenser is filled with water

Switch ON the heater (Jacket Reactor)	Turn ON the heater from the GUI	THE HEATER is heating the water in the jacket till it reaches 50-60°C indicated on the TSJ and TSR (temperature sensor of the jacket/Reactor)
After the reaction has completed Increase the Temperature of the heater (Jacket Reactor)	Increase the temperature of the heater from the **GUI**	Distillation (Toluene) The HEATER is heating the water in the jacket till it reaches 110°C indicated TSC (temperature sensor between the reactor and condenser)
1- Open the valve R-C (Reactor: Condenser) 2- Open the Valve P2 (tank P2: tank toluene)	1- Open the valve V R-C to transfer the vapor from Reactor to the condenser 2- Open the valve VP2 to transfer the liquid after distillation from the condenser to the tank P2	1- The vapor is transferred to "condenser" 2- The liquid is transferred to "Tank P2"
Increase the Temperature of the Heater (Jacket Reactor)	Increase the temperature of the Heater from the GUI	Distillation (Acetic Anhydride) THE HEATER is heating the water in the jacket till it reaches 139°C indicated TSC (temperature sensor between the reactor and condenser)
1. Open the valve R-C (Reactor: Condenser) 2. Open the Valve P1 (tank P1: tank acetic anhydride)	1. Open the valve V R-C to transfer the vapor from Reactor to condenser 2. Open the valve VP1 to transfer the liquid after distillation from condenser to tank P1	1. The vapor is transferred to "condenser" 2. The liquid is transferred to "Tank P1"
Switching OFF the system	Switch OFF the system	The system is OFF

Postcondition	System is OFF	

3.6 Mechanical Realization / Implementation Acetic Anhydride Pilot Plant

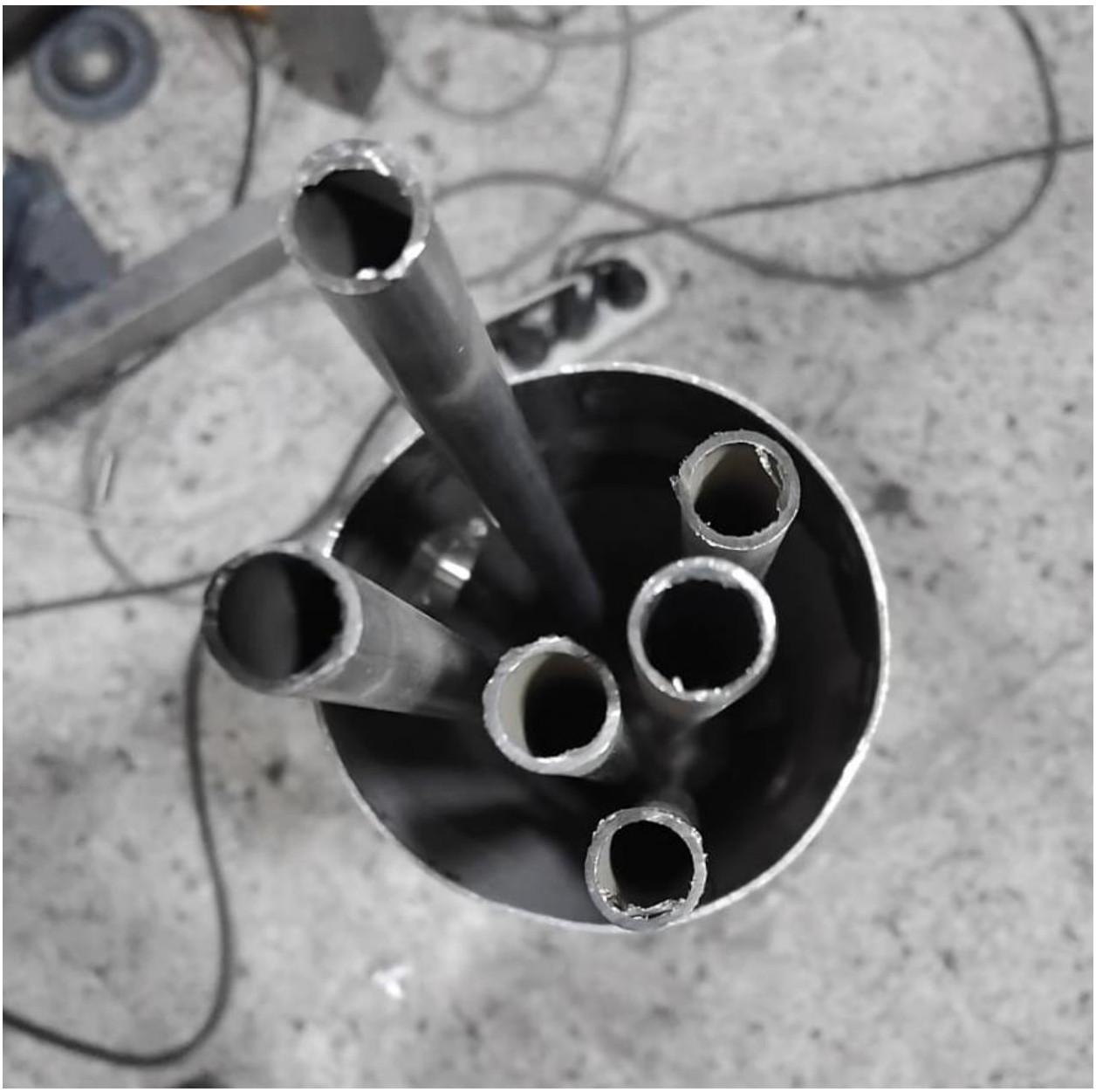
Condenser stainless-steel (inside view 1) 09.08.2024

Condenser stainless-steel (inside view 2) 09.08.2024

Stainless-steel 316 female connector (condenser inlet) 14.08.2024

View 1 View 2

Outside condenser inlets and jacket 20.08.2024



Whole condenser inlets tubes and jacket (view 1) 10.09.2024

Whole condenser inlets tubes and jacket (view 2) 10.09.2024

Whole condenser inlets tubes and jacket (view 3) 10.09.2024

3.6.1 List of instruments and extensions for Acetic Anhydride Pilot Plant

- 2 Flow sensors (1/2 inches) – (stainless steel 316)
- 3 Temperature sensors (+100 °C) – (stainless steel 316)
- 5 electrical valves (1/2 inches) – (stainless steel 316)
- 2 Pressure sensors + 2 safety valves (1/2 inches) – (stainless steel 316)
- 1 mixer + axe (stainless steel 316)
- 4 manual valves stainless steel 316
- Heater

Price $???

3.7 Protocol Acetic Anhydride Production 16.02.2024

3.7.1 Introduction

"Acetic anhydride, a key ingredient in aspirin production, is a versatile chemical used in various industries." In this experiment, we will synthesize acetic anhydride from acetic acid and sodium bicarbonate.

3.7.2 Materials and Equipment

- Safety goggles
- Gloves
- Lab coat
- Reactor
- Condenser
- Temperature sensor x3
- Pressure sensor x2
- Flow sensor x2
- Heater
- Mixer
- Stainless steel tanks x4
- Vinegar (74.5L)
- Sodium bicarbonate (4.88kgs)
- Acetyl chloride (3.77L)

3.7.3 Procedure

1. In the "Reactor", Turn ON the mixer

   Add 74.5L vinegar and 4.88kgs sodium bicarbonate periodically to avoid fizzing reaction [Rx 1]

2. Turn ON the heating system in the jacket to reach 120 °C until the formation of sodium acetate to create anhydrous conditions
3. Cooldown between 50-80 °C, add 3.77L acetyl chloride and Turn ON the mixer [Rx 2]
4. Add 5L of toluene as "solvent" acts as a reaction medium and facilitator, enhancing interaction, controlling temperature, and aiding product isolation.
5. After the reaction completed, turn ON the water circulation in the condenser to start the distillation by turning ON the heating to 110 °C to evaporate the toluene (tank P1).
6. increase the temperature to 140 °C to evaporate the Acetic Anhydride (tank P2).
7. Turn OFF the system, test the purity and the yield of acetic anhydride

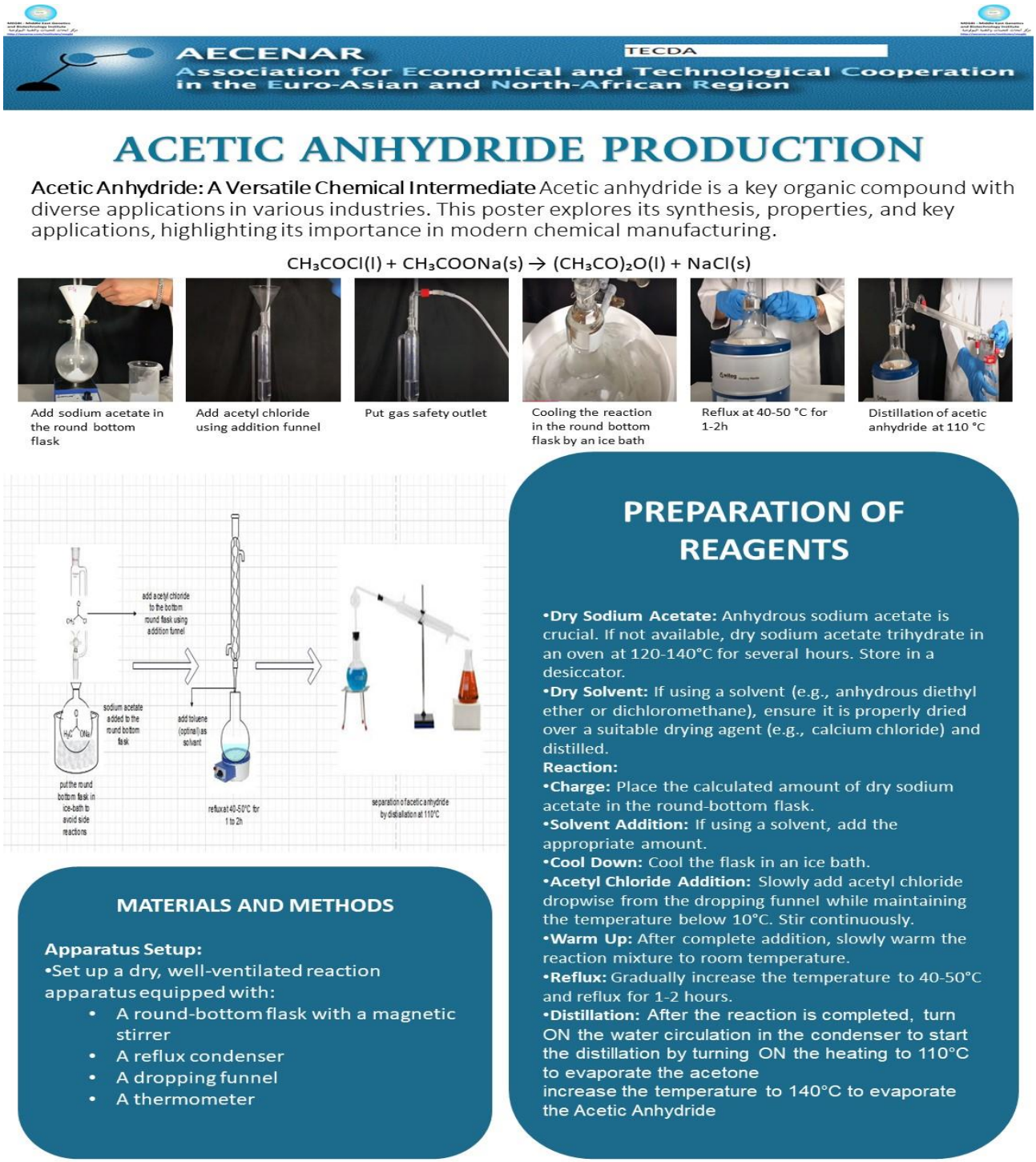
3.7.4 Safety

- -Toluene: Sweet aroma, hidden dangers - inhalation, skin, and fire risks; long-term impacts on organs, development, and nervous system.
- -Acetyl chloride: corrosive, flammable, explosive, lung irritant, potential carcinogen.
- -Acetic anhydride: highly corrosive, burns eyes and skin, inhaling damages lungs, potential carcinogen, flammable, and reacts violently with many substances.

⚠ N.B.: Always wear gloves and safety goggles when handling these chemicals

3.7.5 Result

3.8 Acetic anhydride Lab Scale Production



3.8.1 Synthesis of Acetic anhydride (PROTOCOL)

Aim: an experiment aims to provide a comprehensive understanding of both the theoretical and practical aspects of synthesizing acetic anhydride, which is a valuable intermediate in many industrial and laboratory processes.

- Material:

1. Vinegar (5%) or glacial acetic acid (CH_3COOH, 98%)
2. Sodium bicarbonate ($NaHCO_3$)
3. Acetyl chloride (C_2H_3ClO, 98%)
4. Toluene ($C_6H_5CH_3$) - optional

- Equipment:

1. Round-Bottom Flask
2. Beaker
3. Erlenmeyer flask
4. Digital balance
5. Spatula
6. Reflux Condenser
7. graduated cylinder
8. Aluminum
9. Hot plate
10. Distillation Apparatus
11. Droppers or Pipettes
12. Magnetic Stirrer
13. Thermometer
14. Ice Bath
15. Fume hood
16. Bunsen Burner
17. Protective Gear: Lab coat, gloves, and safety goggles for personal protection

- Reaction:

1. $NaHCO_3(s) + CH_3COOH\ (aq) \rightarrow CH_3COONa\ (s) + H_2O(l) + CO_2(g)$ "exothermic reaction"
2. $CH_3COONa(s) + CH_3COCl(aq) \rightarrow (CH_3CO)_2O(aq) + NaCl(s)$

- Procedure:

1. Setup the apparatus
2. Add 600ml of vinegar (5%) in a beaker under a Hot plate at T= 120°C and put a magnetic stirrer to mix the reaction
3. Weigh 42g using digital balance, then add gradually to the beaker to prevent excessive foaming
4. Start boiling to remove water, the liquid has crystals and becomes solid powder, which is sodium acetate, then the solution turns into a gooey paste
5. To fully dry the solid, melt it
6. Stop heating and the liquid was mixed by using a spatula that prevented it from solidifying
7. After mixing and cooling between 50-80°C, add 98.18 ml acetyl chloride
8. Add 1L of Toluene acts as a reaction medium and facilitator, enhancing interaction, controlling temperature, and aiding product isolation.
9. After the reaction is completed, turn ON the water circulation in the condenser to start the distillation by turning ON the heating to 110°C to evaporate the acetone
10. Increase the temperature to 140°C to evaporate the Acetic Anhydride
11. Turn OFF the system, test the purity and the yield of acetic anhydride.

3.8.2 HPLC Quantitative Analysis

HPLC: is a high-performance liquid chromatography that is widely used in analytical techniques and quantitative methods in the industry. Also, it provides accuracy and reliability.

Procedure:

1. We set up the equipment,
2. The mobile phase passes through a Gradient acetonitrile 'MeCN' – 20-100%, water, and phosphoric acid 15 min to degasser, that is, removing gas dissolve.
3. Then the mobile phase passes to the pump that maintains constant flow of mobile phase through the HPLC, following the sample injector, whereas the stationary phase is silica gel
4. Passing to column of Newcrom R1 is a special reverse-phase column with low silanol activity. It is based on spherical silica particles with 100 Å pores size and particle sizes of 3 μm and 5 μm. The stationary phase has advanced proprietary end-capping and is generally stable at basic pH values, with a recommended pH range of 1.0 to 10.0. Also, the principle of elution is dependent on the affinity, which is that the less polar will elute first and the more polar will elute second, whereas the flow rate is 5 ml/min
5. The elution will be entering the detector, 'Lambert Law," and will be LED light which passes to the filter, then to the sample and the absorbance value is detected at UV 200, 275nm
6. The light that passes through the sample will provide info of a particular wavelength that is converted to a digital signal and displayed in a monitor.
7. Monitor graph is absorbance versus retention time and shows chromatogram peaks.

3.8.3 INFRA-RED Method

IR: is infra-red, which is most useful in providing information about the presence or absence of specific functional groups. It also provides a molecular fingerprint that can be used when comparing samples. If two pure samples display the same IR spectrum, it can be argued that they are the same compound. Also, it is electromagnetic radiation

Preparation:

1. Set up the instrumentation by using a liquid cell with sodium chloride (NaCl) windows. Ensure the cell is clean and dry to prevent interference, followed by applying a thin film of the sample between the NaCl windows.

2. Then prepare a KBr pellet by grinding a small amount of KBr to a fine powder and mixing a small amount of your sample with the KBr powder, then pressing the mixture into a pellet using a hydraulic press.
3. IR radiation is generated by fitting a light source and directing it to the sample, "acetic anhydride synthesis "
4. Some light is reflected; the sample absorbs the specific amount of passing light.
5. The part of the light that is transmitted and carries the molecular information of the sample will be collected in acetic anhydride by a detector to produce electronic signals
6. To transfer the electronic signals into a spectrum, the light should first be directed to a diffraction grating, splitting into several beams traveling in a different direction
7. These beams were mechanically directed to the sample and each wavelength was examined individually
8. The Fourier Transform (FT) in FTIR spectroscopy is essential for converting the raw data collected from the sample (interferogram) into a usable IR spectrum. This process enables rapid, sensitive, and accurate analysis of the sample's molecular structure.
9. Analyze the IR spectrum. The two carbonyl groups in acid anhydrides give rise to two carbonyl stretching peaks. The vibrations involved are a symmetric C=O stretch where the two carbonyl groups stretch in phase with each other and an asymmetric C=O stretch where the two carbonyl groups stretch out of phase with each other

V = 1810-1750 cm^{-1}

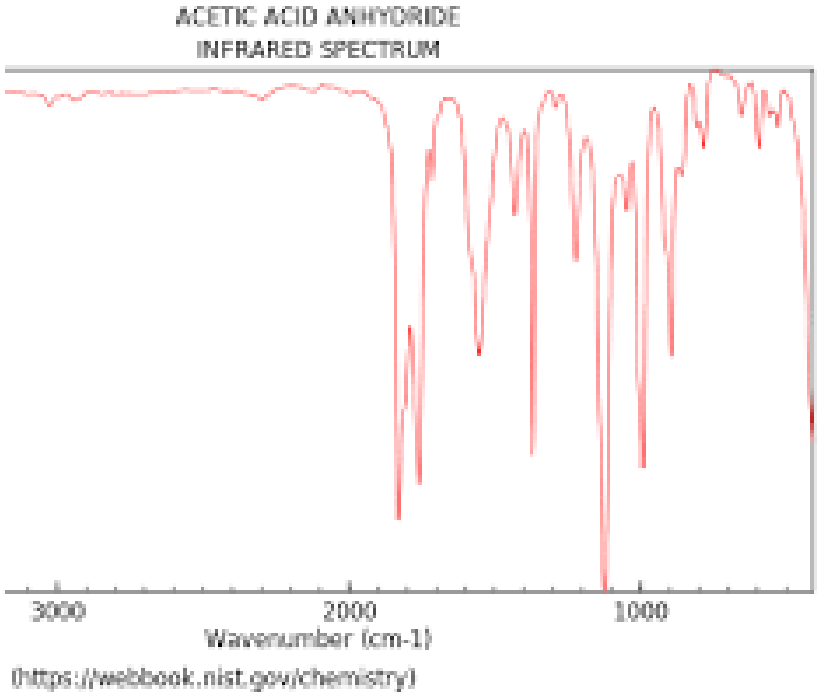
Additional Considerations

Solvent effects: If using a solvent, ensure it doesn't interfere with the target peaks.

Sample purity: Impurities can introduce additional peaks, complicating the spectrum interpretation.

Spectrum quality: Ensure a good signal-to-noise ratio for accurate peak identification.

By following these steps and carefully analyzing the IR spectrum, you should be able to confirm the presence of acetic anhydride in your sample.



4 Project 3: Sulfuryl Chloride Production Project

4.1 Objective of process control for Sulfuryl Chloride production

To produce sulfuryl chloride (SO_2Cl_2) by reacting sulfur dioxide (SO_2) with chlorine (Cl_2) in a controlled environment.

4.2 Materials and Equipment

4.2.1 Reactants

- Sulfur Dioxide (SO_2) - stored in pressurized tanks
- Chlorine (Cl_2) - stored in pressurized tanks

4.2.2 Catalyst (if applicable)

- Sodium fluoride-carbon catalyst (prepared as per the specifications)

4.2.3 Equipment

- 50-gallon stainless steel reaction vessel
- Stirring mechanism (mechanical stirrer)
- Cooling coils (for temperature control)
- Pressure gauge
- Off-gas line with a pressure relief valve
- Sampling apparatus
- Filtration system
- Storage containers for sulfuryl chloride

4.3 Safety Precautions

- Ensure all personnel are wearing appropriate personal protective equipment (PPE), including gloves, goggles, and lab coats.
- Work in a well-ventilated area or fume hood to avoid inhalation of gases.
- Have emergency equipment (eyewash station, safety shower, fire extinguisher).
- Be aware of the properties of chlorine and sulfur dioxide, as both are toxic and corrosive.

4.4 Preparation of Catalyst (if applicable)

Impregnation:

1. Boil activated carbon (e.g., Darco G-60) with a 4% aqueous solution of sodium fluoride.
2. Ensure that the sodium fluoride is thoroughly impregnated into the carbon particles.
3. Allow the catalyst to dry before use.

4.5 Setup of Reaction Vessel

4.5.1 Reactor Preparation

- Clean the stainless steel reaction vessel thoroughly.
- Install the stirring mechanism and cooling coils.

- Connect the off-gas line to a safe venting system.

4.5.2 Pressure and Temperature Control

- Ensure that the pressure gauge is calibrated and functioning.
- Set up the cooling system to maintain the reaction temperature below 55 °C.

4.6 Reaction Procedure

4.6.1 Cooling of Reactants

- Cool the chlorine and sulfur dioxide to their respective boiling points:
  - Chlorine: below -34.04 °C
  - Sulfur Dioxide: below -10 °C

4.6.2 Pressurization

- Pressurize the tanks containing chlorine to approximately 5-6 atmospheres.
- Pressurize the tanks containing sulfur dioxide to approximately 2-3 atmospheres.

4.6.3 Addition of Reactants

- Begin stirring the contents of the reaction vessel.
- Slowly add liquid chlorine and liquid sulfur dioxide in equimolar proportions (approximately 64 lbs. of SO_2 and 71 lbs. of Cl_2 per hour).
- Monitor the pressure and temperature continuously during the addition.

4.6.4 Monitoring the Reaction

- Analyze the reaction mixture periodically for unreacted gases using sampling apparatus.
- If unreacted chlorine is detected, add additional sulfur dioxide; if unreacted sulfur dioxide is detected, add additional chlorine.
- Maintain the temperature below 55 °C using the cooling coils.

4.6.5 Completion of Reaction

- After approximately 4-5 hours of continuous addition, stop the stirring.
- Allow the reaction mixture to settle for 1-2 hours.

4.7 Separation and Filtration

4.7.1 Withdrawal of Product

- Carefully withdraw the supernatant liquid (sulfuryl chloride) from the bottom of the reactor.
- Use a filtration system to remove any suspended solids or catalyst residues.

4.7.2 Storage of Sulfuryl Chloride

- Transfer the filtered sulfuryl chloride to appropriate storage containers.
- Ensure that the containers are sealed and labeled correctly.

4.8 Post-Reaction Cleanup

4.8.1 Cleaning the Reactor

- Cleaning the Reactor: Clean the reaction vessel and all equipment used in the process to remove any residual chemicals.
- Dispose of any waste materials according to local regulations.

4.8.2 Safety Check

- Conduct a safety check of the area to ensure no leaks or residual gases are present.

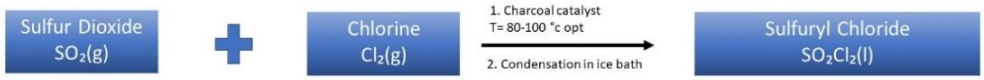
4.9 Sulfuryl Chloride Lab Scale production

بسم الله الرحمن الرحيم

AECENAR
TECDA
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region

Sulfuryl Chloride Production

Introduction: **Acetyl sulfuryl chloride (CH_3COSO_2Cl)** is a valuable reagent in organic synthesis, offering unique reactivity profiles for various transformations. This poster will explore the synthesis of acetyl sulfuryl chloride, highlighting its significance and potential applications in chemical research.




2x Gaz cylinders for $SO_2(g)$ And $Cl_2(g)$


Pressure gauge


Magnetic stirrer

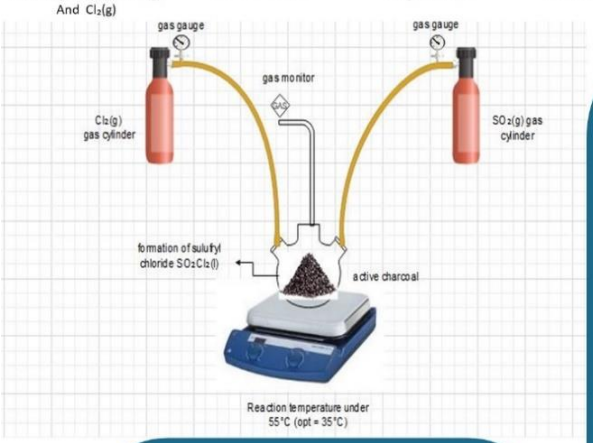

condenser


PPE safety


spectrophotometer


Gas chromatography


Round bottom flask multi neck



MATERIALS AND METHODS

Materials:

Materials and Methods

1.Catalyst Preparation:

1. Use activated carbon (e.g., Darco G-60).
2. Impregnate with a solution of sodium fluoride (NaF) or other metal salts.
3. Example preparation: Boil a 4% sodium fluoride solution with carbon for 1-2 hours, then filter and dry.

2.Experimental Setup:

1. Reactor setup for liquid phase or vapor phase reactions.
2. Ensure proper handling of toxic gases (SO_2 and Cl_2) in a fume hood.

TESTING

1. Gas Chromatography (GC)

•**Purpose**: To analyze the composition of the gas phase and detect unreacted sulfur dioxide (SO_2) and chlorine (Cl_2).

3. Infrared Spectroscopy (IR)

•**Purpose**: To identify and quantify the presence of sulfuryl chloride and other reactants/products in the liquid phase.

REACTION CONDITIONS

1.Reactor Setup:

1. **Type**: 1-liter round-bottom flask or small stainless-steel reactor.
2. **Stirring**: Magnetic stirrer with a stir bar to ensure thorough mixing of reactants.
3. **Cooling**: Cooling bath (ice or water) to maintain the reaction temperature.

2.Temperature:

1. **Optimal Range**: Maintain the temperature of the reaction mass below 55 °C.
2. **Preferred Range**: Ideally, operate within 30-40 °C for better control and efficiency.

3.Pressure:

1. **Initial Pressure**: Atmospheric pressure (1 atm) is typically sufficient.
2. **Maximum Pressure**: Monitor and prevent pressure buildup; vent excess gases if necessary.
3. **Operating Pressure**: Can be adjusted up to 25 lbs. or higher if needed, but this is not typically required for laboratory-scale reactions.

4.Reactant Proportions:

1. **Equimolar Ratio**: Introduce sulfur dioxide (SO_2) and chlorine (Cl_2) in equimolar proportions.
2. **Example Quantities**: For a small-scale reaction, approximately 10 mL of each gas can be used.

5.Catalyst Preparation:

1. **Type**: Sodium fluoride-activated carbon catalyst.
2. **Amount**: Approximately 5 grams of the prepared catalyst.
3. **Preparation Method**: Impregnate activated carbon (e.g., Darco G-60) with a 4% aqueous solution of sodium fluoride, then dry.

6.Reaction Time:

1. **Duration**: Continuous addition of SO_2 and Cl_2 for about 1-2 hours, depending on the desired yield.
2. **Monitoring**: Regularly check for unreacted gases and adjust the addition rate accordingly.

7.Gas Flow Rates:

1. **Controlled Addition**: Use gas flow meters to control the rate of SO_2 and Cl_2 addition.
2. **Example Flow Rates**: Approximately 64 lbs. of SO_2 and 71 lbs. of Cl_2 per hour for larger setups; adjust for smaller laboratory-scale reactions.

8.Post-Reaction Conditions:

1. **Settling Time**: Allow the reaction mass to settle for 30 minutes to 1 hour after the reaction is complete.
2. **Filtration**: Filter the supernatant liquid to remove any suspended catalyst particles.

Mohammad Kalawoun@ AECENAR_Green chemistry /December 2024

Poster of Sulfuryl Chloride Production [pptx file]:	sulfuryl chloride production.pptx

4.9.1 Reaction Equation

$Cl_2(g) + SO_2(g) \rightarrow SO_2Cl_2(l)$

4.9.2 Laboratory Setup:

1. **Gas Cylinders:** Obtain gas cylinders of chlorine and sulfur dioxide. Ensure that the cylinders are equipped with pressure regulators and flow meters.
2. **Glassware:**
    - **Reaction Flask:** A round-bottom flask with a capacity of 250-500 mL is suitable.
    - **Condenser:** A reflux condenser to condense the sulfuryl chloride vapor.
    - **Drying Tube:** A drying tube filled with anhydrous calcium chloride or concentrated sulfuric acid to remove any moisture from the gases.
    - **Gas Delivery Tubes:** Glass tubes with rubber tubing connections to connect the gas cylinders to the reaction flask.
3. **Catalyst:** Prepare a charcoal catalyst with 4% fluorine. This can be done by impregnating activated charcoal with a fluorine-containing compound, such as ammonium fluoride or hydrofluoric acid.
4. **Heating Source:** A hot plate or heating mantle to heat the reaction flask.
5. **Ice Bath:** A beaker filled with ice to cool the condenser and collect the sulfuryl chloride.

4.9.3 Experimental Procedure:

1. **Set Up the Apparatus:**
    - Connect the gas cylinders to the reaction flask using the gas delivery tubes.
    - Insert the condenser into the reaction flask, and connect the drying tube to the condenser.
    - Place the ice bath around the condenser.
2. **Add the Catalyst:**
    - Weigh the desired amount of the prepared charcoal catalyst and add it to the reaction flask.
3. **Introduce the Gases:**
    - Slowly open the valves on the gas cylinders to allow a steady flow of chlorine and sulfur dioxide into the reaction flask.
    - The flow rates can be adjusted using the flow meters.
4. **Heat the Reaction:**

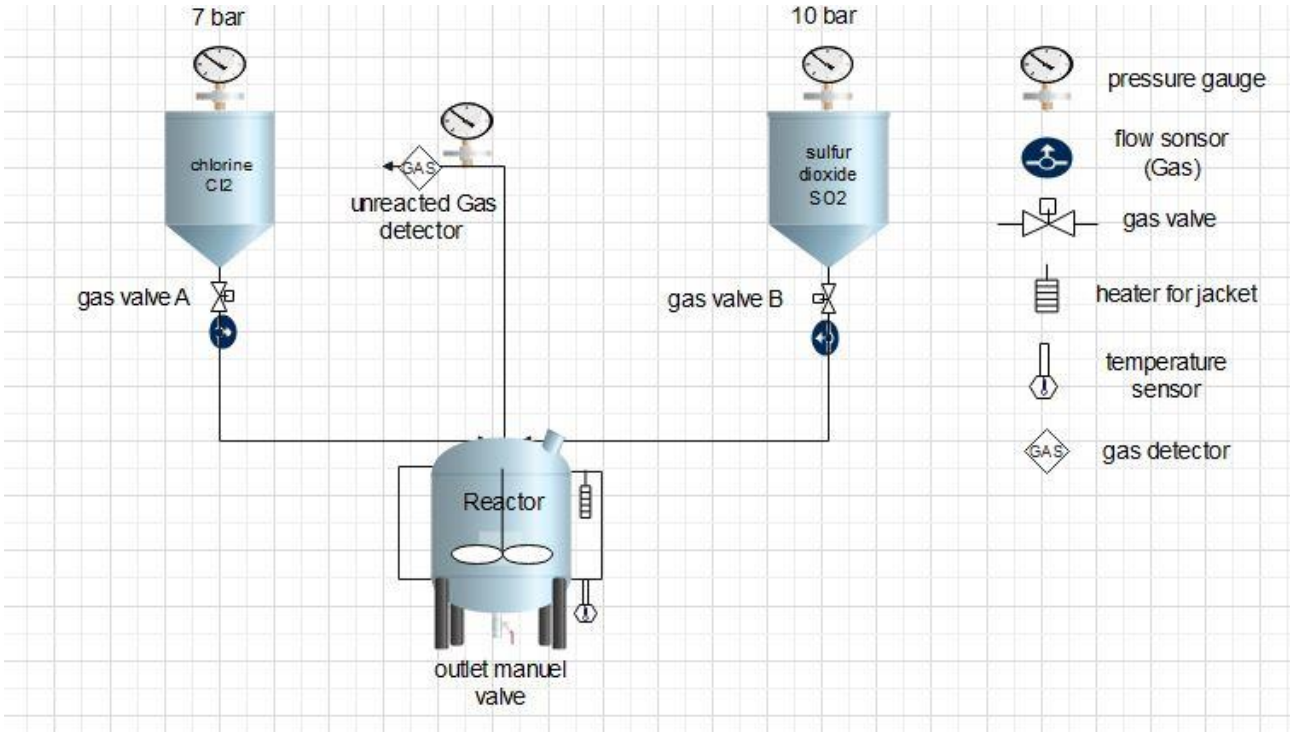
- Turn on the heating source to heat the reaction flask to a temperature of 80-100°C.
- The reaction will proceed at this temperature.

5. **Collect the Product:**
   - The sulfuryl chloride product will condense in the condenser and collect in the ice bath.
6. **Monitor the Reaction:**
   - Periodically check the flow rate of the gases and the temperature of the reaction.
   - Once the reaction is complete, the flow of gases can be stopped.
7. **Isolate the Product:**
   - Remove the condenser from the ice bath and carefully transfer the sulfuryl chloride to a suitable container.
   - The product can be further purified by distillation, if necessary.

4.9.4 Safety Precautions:

- Chlorine and sulfur dioxide are toxic gases. Work in a well-ventilated area and wear appropriate respiratory protection.
- Hydrofluoric acid is a highly corrosive and dangerous chemical. Handle it with extreme caution and wear protective gloves and eye protection.
- The reaction should be carried out in a fume hood to prevent the release of toxic gases.

4.10 Sulfuryl chloride Pilot Scale production



Flow chart of sulfuryl chloride [Edraw file]:

map sulfuryl chloride.eddx

4.11 Compressor for Cl_2 and SO_2

Q1: Can we use a normal air compressor to compress $Cl_2(g)$ and SO_2 into the gas cylinders?

R1:

Chlorine gas Cl2:

No, you should **not** use a normal air compressor to compress chlorine gas (Cl2) into a gas cylinder. Here's why:

1. Compatibility: Normal air compressors are designed for air, which is a mixture of primarily nitrogen and oxygen. Chlorine gas is a highly reactive and corrosive substance that can damage the internal components of a standard air compressor.

2. Safety Hazards: Chlorine gas is a toxic and potentially lethal substance. If a **leak** occurs during compression, it can pose a **serious health risk to anyone** in the vicinity. Standard air compressors are not designed to handle hazardous gases and may not have the necessary safety features to prevent leaks or contain a release.

3. Equipment Requirements: Compressing chlorine gas requires specialized equipment designed to handle its corrosive properties and ensure safe operation. This includes:

    - Chlorine-specific compressor: These compressors are made from materials resistant to chlorine, such as stainless steel or special alloys.
    - Safety features: These may include leak detection systems, emergency shutoff valves, and containment measures to minimize the risk of exposure in case of a leak.
    - Proper ventilation: Adequate ventilation is crucial to prevent the buildup of chlorine gas, which can be dangerous at high concentrations.

If you need to compress chlorine gas, it is essential to consult with experts in gas handling and safety. They can advise you on the appropriate equipment, procedures, and safety precautions.

Sulfur dioxide Gas SO2:

No, you should not use a normal air compressor to compress sulfur dioxide (SO2) gas into a gas cylinder. Here's why:

1. Compatibility: Normal air compressors are designed for air, which is a mixture of primarily nitrogen and oxygen. Sulfur dioxide is a corrosive gas that can damage the internal components of a standard air compressor.

2. Safety Hazards: Sulfur dioxide is a toxic gas that can irritate the respiratory system. If a leak occurs during compression, it can pose a serious health risk to anyone in the vicinity.

Standard air compressors are not designed to handle hazardous gases and may not have the necessary safety features to prevent leaks or contain a release.

3. Equipment Requirements: Compressing sulfur dioxide gas requires specialized equipment designed to handle its corrosive properties and ensure safe operation. This includes:

    - SO2-specific compressor: These compressors are made from materials resistant to sulfur dioxide, such as stainless steel or special alloys.
    - Safety features: These may include leak detection systems, emergency shutoff valves, and containment measures to minimize the risk of exposure in case of a leak.
    - Proper ventilation: Adequate ventilation is crucial to prevent the buildup of sulfur dioxide gas, which can be dangerous at high concentrations.

Solution:

For compressing chlorine (Cl2) and sulfur dioxide (SO2) gases, you should use specialized compressors designed to handle these corrosive and hazardous substances.

Here are some suitable options:

- Diaphragm compressors: These compressors use a flexible diaphragm to separate the gas from the compressor's internal components, minimizing the risk of corrosion and contamination.
- Liquid ring compressors: These compressors use a rotating impeller to displace the gas, with a liquid seal to prevent leakage and corrosion.
- Screw compressors: Some specially designed screw compressors with corrosion-resistant materials and coatings can be used for certain applications involving these gases.

Important Considerations:

- Materials of Construction: The compressor and its components must be made from materials resistant to the specific gas (e.g., stainless steel, Hastelloy, Teflon).
- Safety Features: The compressor should incorporate safety features such as leak detection systems, emergency shutoff valves, and pressure relief devices.
- Ventilation: Adequate ventilation is crucial to prevent the buildup of hazardous gases in the work area.

5 Project 5: Sulfur dioxide Production Project

5.1 Sulfur dioxide Lab Scale Production

MEGBI - Middle East Genetics and Biotechnology Institute
مركز ابحاث للجينات والتقنية البيولوجية
http://aecenar.com/institutes/megbi

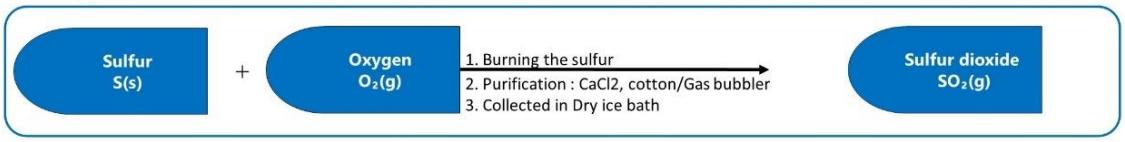
بسم الله الرحمن الرحيم

MEGBI - Middle East Genetics and Biotechnology Institute
مركز ابحاث للجينات والتقنية البيولوجية
http://aecenar.com/institutes/megbi

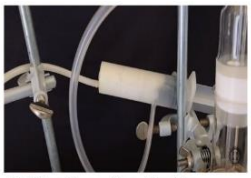
AECENAR TECDA
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region

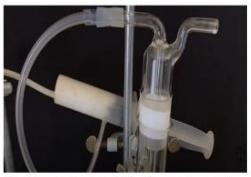
Sulfur Dioxide Production

Introduction: Sulfur dioxide (SO_2) is an important industrial chemical with various applications. One of the most common methods for producing SO_2 is by directly burning elemental sulfur in the presence of oxygen.
This reaction readily occurs at elevated temperatures, yielding sulfur dioxide gas as the primary product.
This poster will explore the key aspects of this production method, including the reaction mechanism, process conditions, and industrial applications of sulfur dioxide.



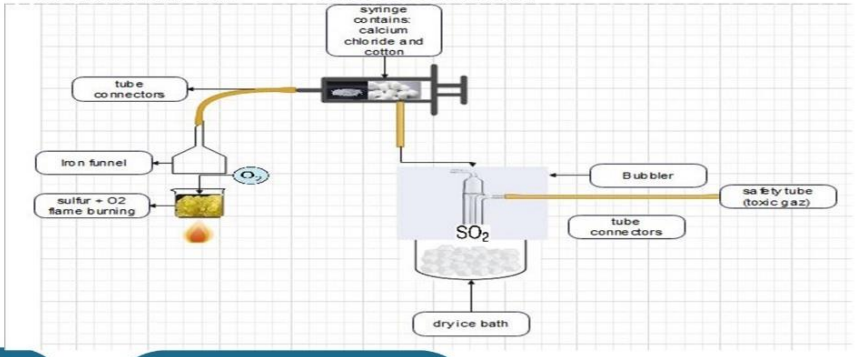

1. Burning the Sulfur powder in presence of O_2 with fire gun.


2. Filter the sulfur dioxide through a syringe contain cotton and calcium chloride $CaCl_2$.


3. After the purification step the gas should go through the bubbler connected to collection tube.


4. Put the collection tube in dry ice bath.


5. Sulfur dioxide is transformed to liquid form



MATERIALS AND METHODS

Materials:

- Elemental sulfur (powdered or chunks)
- Oxygen gas source
- Heat source (Bunsen burner or fire gun)
- Glassware (bubbler gas, iron funnel, collection tubes, delivery tubes, dry ice bath, Large Syringe, tyau caoutchouc)

CONDITIONS

Conditions Needed for Sulfur Combustion to Produce SO2
To ensure the efficient combustion of sulfur and the production of sulfur dioxide (SO2), the following conditions are essential:

1.Sufficient Oxygen Supply:
- •A plentiful supply of oxygen is crucial for the reaction to proceed.
- •Air, which contains approximately 21% oxygen, can be used as the oxygen source.

2.Ignition Source:
- •A heat source, such as a flame or a spark, is required to initiate the combustion process.

3.Adequate Temperature:
- •The reaction is exothermic, meaning it releases heat. However, an initial input of heat energy is necessary to start the reaction.
- •Once initiated, the reaction sustains itself due to the heat released.

TESTING

1.Water Test:
- •Bubble the gas through water.
- •SO2 reacts with water to form sulfurous acid, which is acidic.
- •Dip blue litmus paper into the solution. A color change to red confirms the presence of an acid, indicating the presence of SO2.

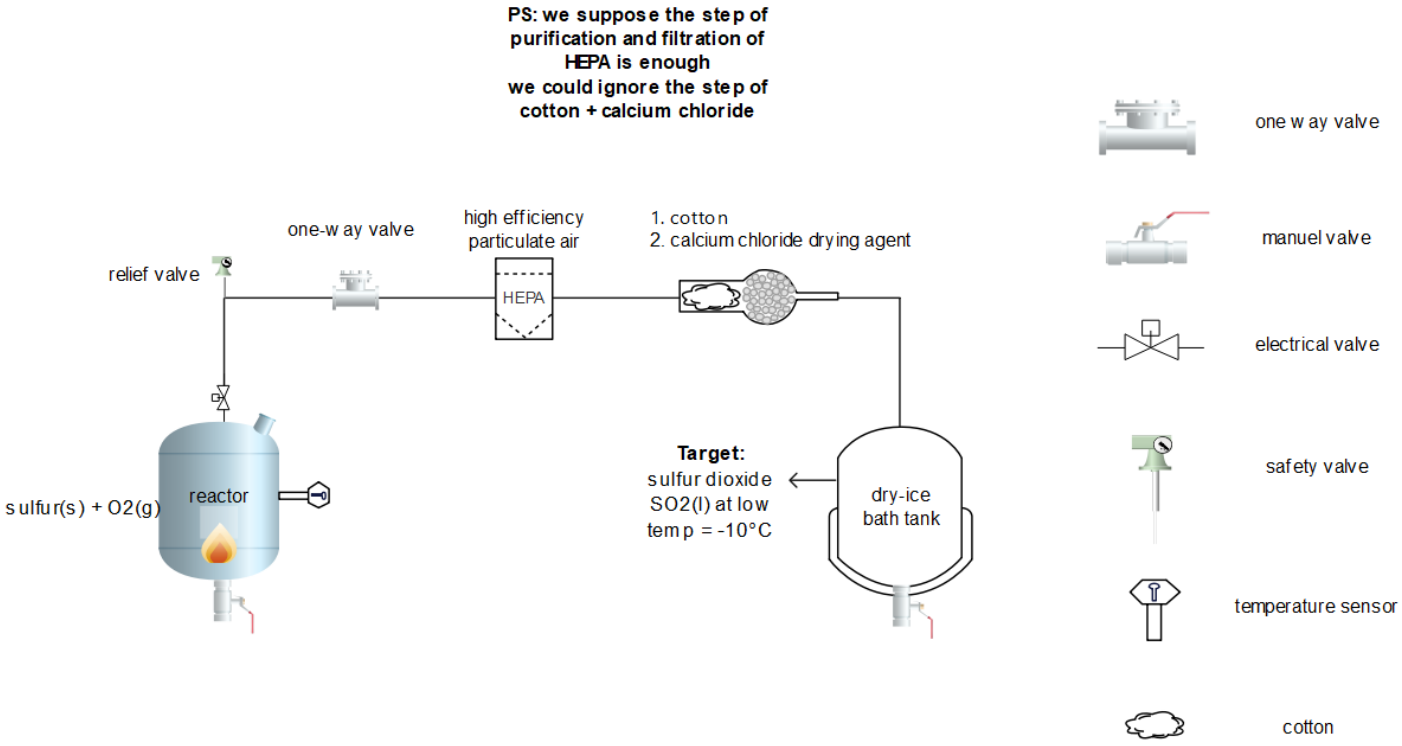
2.Potassium Permanganate Test:
- •Bubble the gas through a potassium permanganate solution.
- •SO2 is a reducing agent and will decolorize the purple potassium permanganate solution.

3.Iodine-Starch Paper Test:
- •Moisten a piece of starch-iodine paper with water.
- •Expose the moistened paper to the gas.
- •SO2 will react with iodine, causing the paper to lose its blue-black color.

Mohammad kalawoun @Green Chemistry/AECENAR 2024/2025

Poster of Sulfur Dioxide Lab Scale production [pptx file]:	sullfur dioxide production poster.p

5.2 Sulfur dioxide pilot plant scale production



Flow chart of Sulfur Dioxide [Edraw file]:

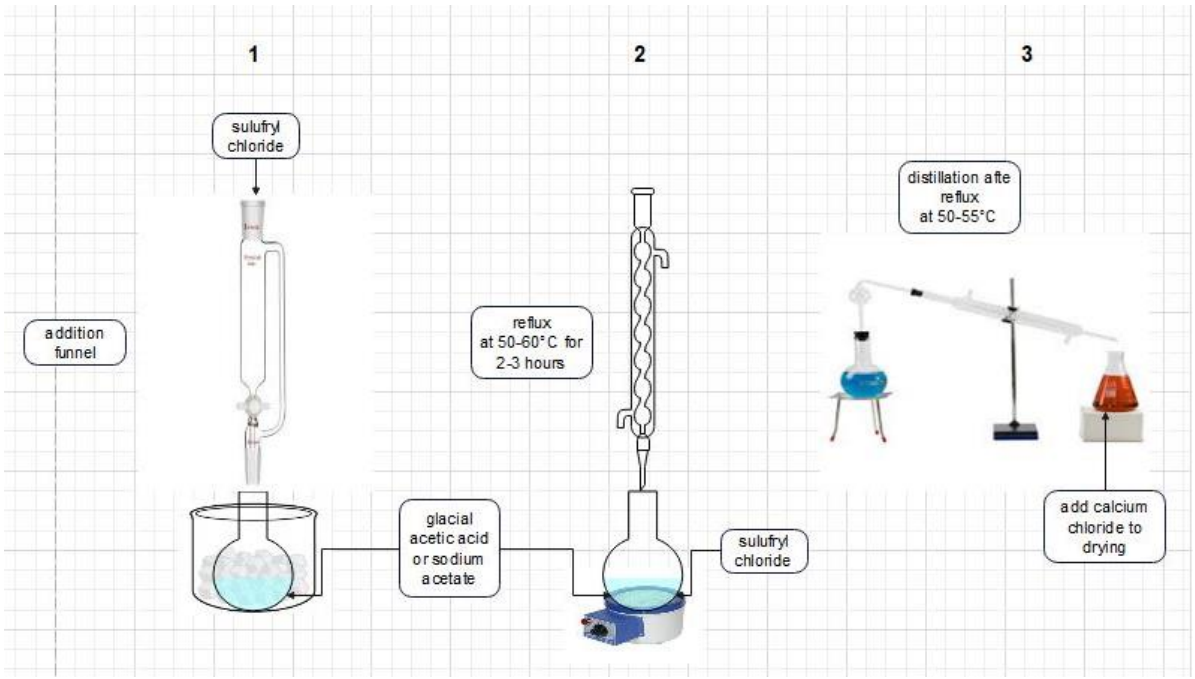
map sulfur dioxide.eddx

6 The two pathways for Acetyl Chloride Production

6.1 Acetyl Chloride Production with sulfuryl chloride (sulfur) as basic matrial (Reaction 2a and 2b)

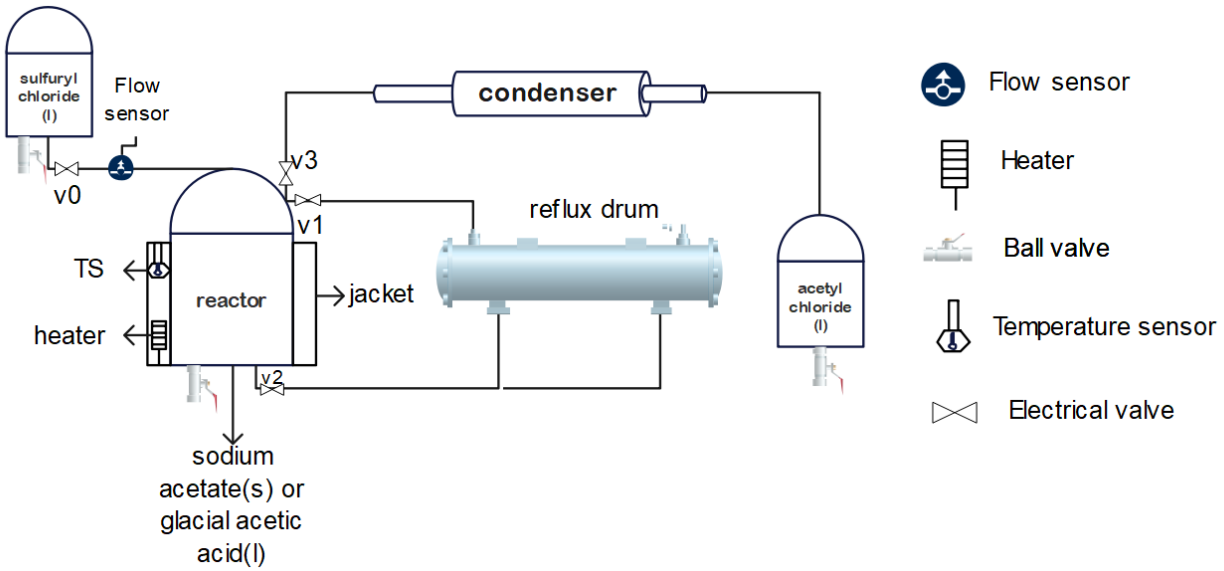
6.1.1 Lab scale reaction (2a)

23.12.2024: Flow chart production of acetyl chloride



map acetyl chloride.eddx

6.1.2 Pilot Plant scale reaction (2a)

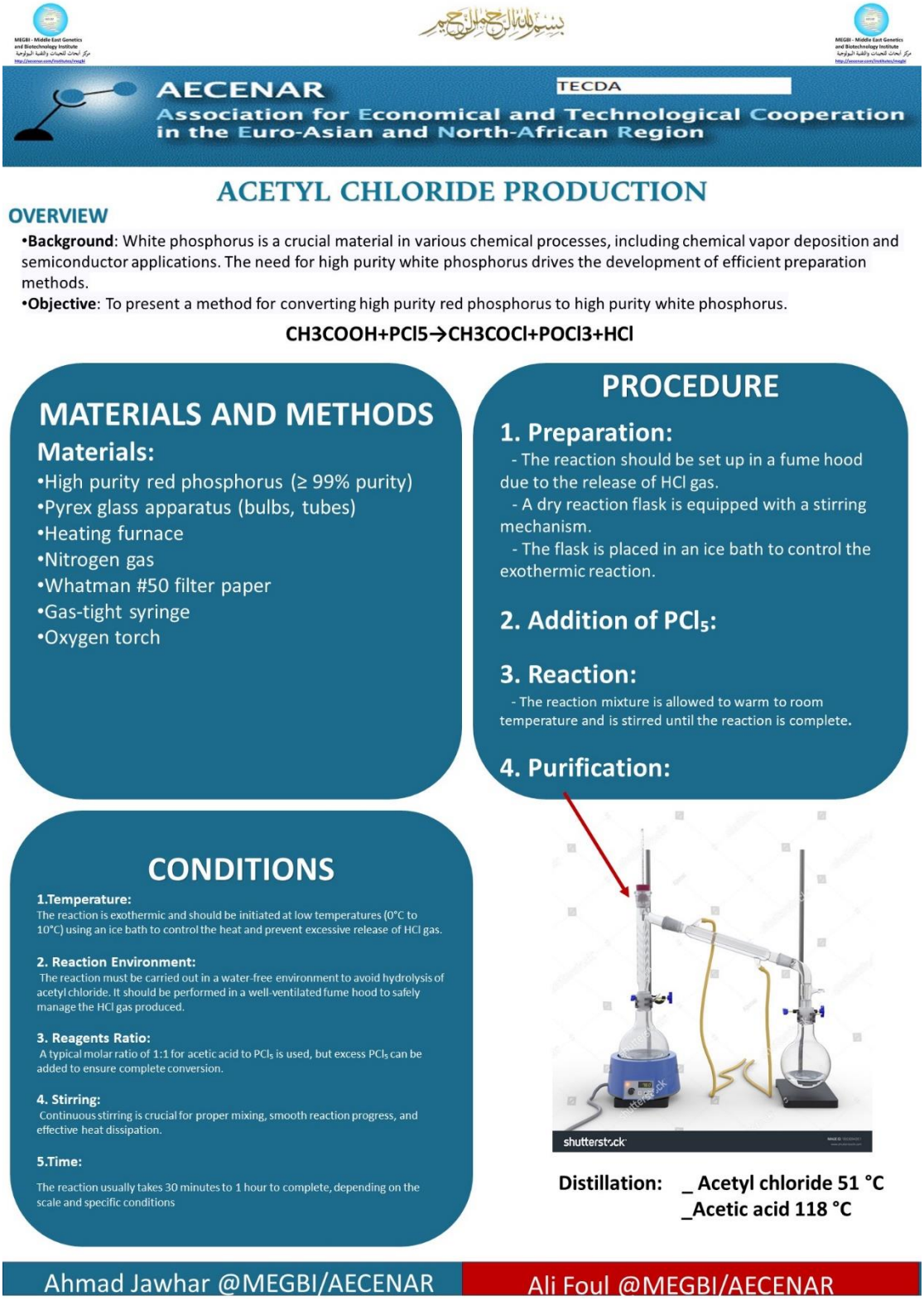


Flow Chart of Acetyl Chloride [Edraw file]:

The two pathways for Acetyl Chloride Production

6.2 Acetyl Chloride Lab Scale Production with Phosphor as basic material

6.2.1 Phosphor based Acetyl chloride Lab scale Production



Poster of Acetyl chloride [pptx file]:	W.P. Poster.pptx

The two pathways for Acetyl Chloride Production

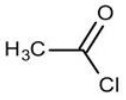
6.2.2 Phosphor based Acetyl chloride Pilot Plant Scale Production

AECENAR TECDA
Association for Economical and Technological Cooperation in the Euro-Asian and North-African Region

Acetyl chloride production scale

Introduction: Acetyl chloride (CH_3COCl) is an acyl chloride used in various organic synthesis reactions, particularly for introducing acetyl groups. One common method of synthesis is by reacting acetic acid with phosphorus pentachloride (PCl_5), a strong chlorinating agent.

Alternate Name:
1.Acetic acid chloride
2.Ethanoyl Chloride
3.Acyl Chloride



Chemical structure

Objective: This poster explores the chemical synthesis process of acetyl chloride and the reaction conditions necessary for optimal production

Chemical reaction:

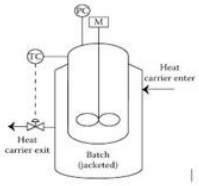
$$CH_3COOH + PCl_5 \rightarrow CH_3COCl + POCl_3 + HCl$$

In this reaction, phosphorus pentachloride replaces the hydroxyl group (-OH) in acetic acid with a chlorine atom, forming acetyl chloride. The by-products are phosphorus oxychloride
($POCl_3$) and hydrogen chloride (HCl)

Parameter	condition	Explanation
Time	1to 2 hours	
Reaction type	Exothermic	Heat is released during the reaction, which needs to be controlled.
Temperature	20–40°C (room temperature to slightly elevated)	Optimal for the reaction, prevents decomposition of acetyl chloride.
Pressure	Optimal for the reaction, prevents decomposition of acetyl chloride.	No pressure requirements

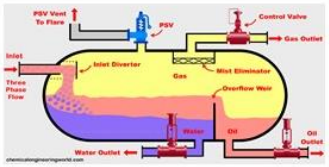
Unit operation needed:

Reactor : Since this is a relatively simple, exothermic reaction, a batch reactor may be appropriate for small-scale synthesis, allowing for careful control of reactant addition



3_phase separator :
3-phase separator in acetyl chloride synthesis separates hydrogen chloride gas (HCl), acetyl chloride (light liquid), and phosphorus oxychloride (heavy liquid). HCl gas is vented to a scrubber, while acetyl chloride and phosphorus oxychloride are separated based on density. This ensures efficient separation and safe handling of corrosive by-products.

.

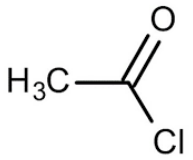


Poster of *Acetyl chloride production scale* [pptx file]:

10-09-24Acetyl chloride production

The two pathways for Acetyl Chloride Production

6.2.2.1 Chemical structure of Acetyl chloride:



6.2.2.2 Chemical and physical properties of Acetyl chloride

Molecular formula	CH3COCl
Density	1.1 g/cm³
Molar mass	78.94 g/mol
Boiling point	51℃
flash point	4 ℃

6.2.2.3 Alternative Names of Acetyl chloride

- Acetic acid chloride
- Ethanoyl Chloride
- Acyl Chloride

6.2.2.4 First reaction and typically used in the formation at AECENAR

CH3COOH +PCl5 → CH3COCl +PCl3 +HCl

6.2.2.5 Second reaction of Acetyl chloride

3CH3COOH+PCl5→ CH3COCl +POCl3 +HCl (exothermic reaction)

Acetyl chloride is toxic and corrosive.

6.2.2.6 Storage of Acetyl chloride

- Store in dry, well-ventilated area
- Take all necessary precautions to avoid the accidental
- keep container tightly closed
- Shelf life is 24 months

6.2.2.7 Unit operation needed :

Acetyl chloride needs

1. Reactor (Batch or CSTR), Batch reactor is better in this case
2. Distillation column or phase separator

The table shows the difference in the type of reactor and the price:

The two pathways for Acetyl Chloride Production

3000L jacketed	10800$
800L	7200$
50L emulsifying	3200$

Determining the exact price:

- Reactor size Volume
- Materials of construction
- operating condition
- Design specification

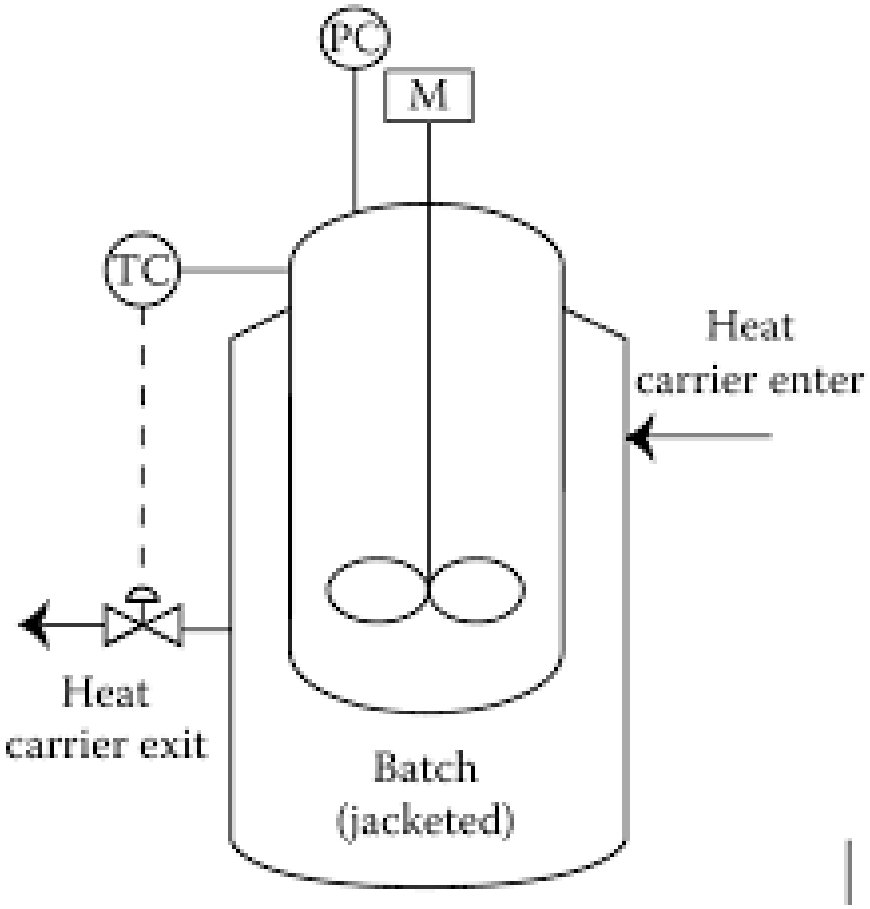
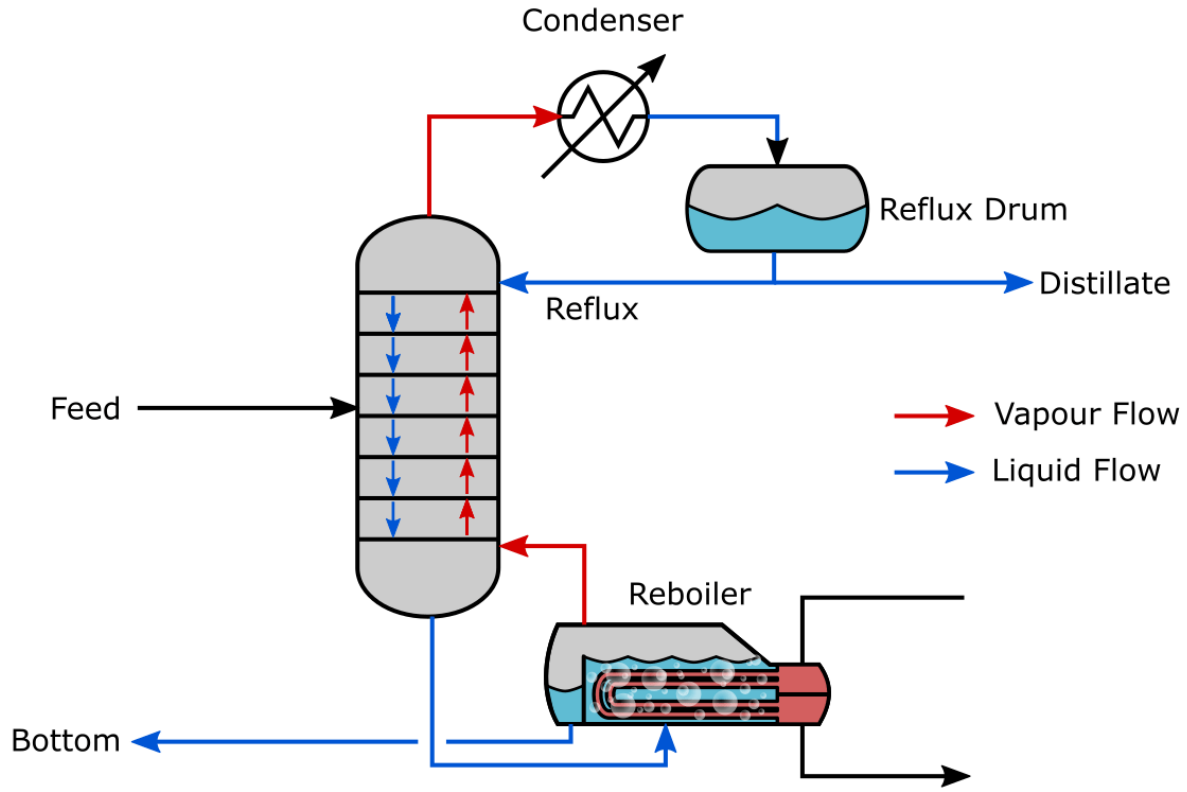


Figure 2: Batch jacketed reactor

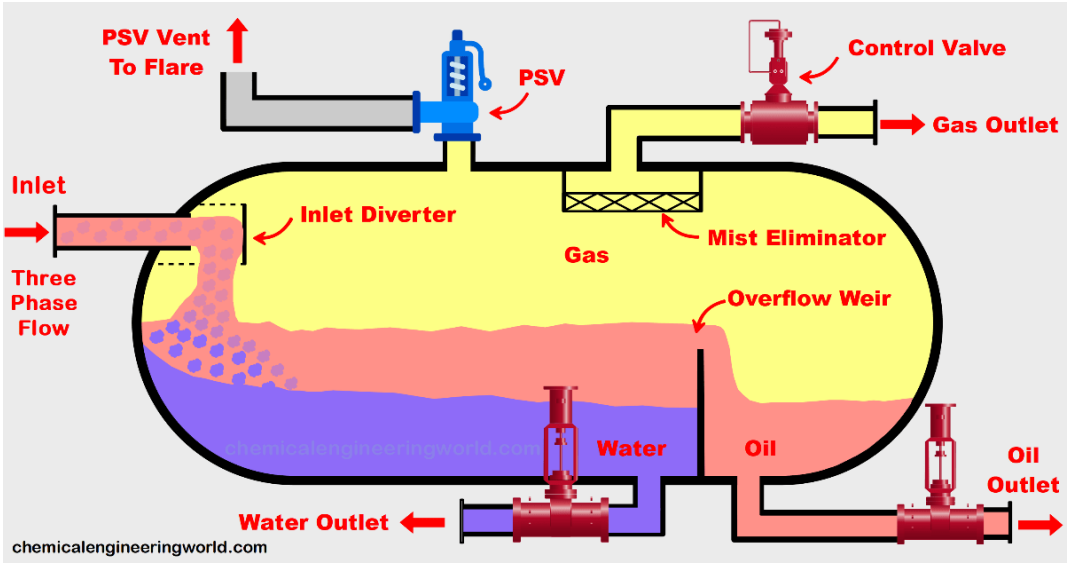
Distillation column or phase separator:

- Use a Distillation Column: A distillation column would be the better choice if your goal is to obtain high-purity acetyl chloride, especially in a continuous production process.

The two pathways for Acetyl Chloride Production



- Use a Phase Separator: If the mixture has clear immiscible phases and purity is less critical, or if you are looking for a more energy-efficient and simpler process, a phase separator might suffice.



6.2.2.8 Unit operation needed in the chemical industry

Momentum transport operation: pumps, pipes, compressor.

Heat transfer operation: change temperature

Mass transfer operation: distillation, absorption, extraction.

Chemical reaction operation: batch reactor, tubular.

Mechanical operation

References

https://www.google.com/url?sa=t&rct=j&q=&esrc=s&source=web&cd=&cad=rja&uact=8&ved=2ahUKEwi0ssvO86iIAxVJAvsDHcI4Lf8QFnoECBQQAw&url=https%3A%2F%2Fwww.sciencedirect.com%2Ftopics%2Fchemistry%2Ftablet-coating%23%3A~%3Atext%3DTablet%2520coating%2520is%2520the%2520process%2520where%2520coating%2520material%2520is%2520applied%2Cand%2520color%2520of%2520the%2520drug&usg=AOvVaw3i_tbYpeGn_jsifDP6qFzv&opi=89978449

https://www.yenchen.com.tw/en/product/tablet_coating_machine-171.html