بسم الله الرحمن الرحيم

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مركز أبحاث تكنولوجيا العمليات الكيميائية
Institute for Chemical Process Technology (ICPT)
http://aecenar.com/institutes/icpt

طاقة الشمال
North Lebanon Alternative Power

NLAP-WEDC (Waste to Energy Demonstration Cycle)

Report 2023

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12.03.2025 00:12

Inhalt

Preface

This report contains details of the NLAP Institute projects that we carried out for the year 2023. These projects include the old ones that have been continued, such as the Waste Incineration Power Plant project, the Multistage Electrolyzer project, the Ashes Recycling project, the Mass Spectrometer, the Biogas, and the Gas Turbine of Methane. It also reviews new projects such as the Waste Separation Plant project and the Waste Smoke Filters (Chemical and electro filters).

1 Introduction

In 2022, the NLAP Institute was responsible for 8 projects; 6 of them were started in the past years and this year they were continued, and two new projects were attached to the institute this year.

For projects started in previous years (2022 and earlier) and continued in 2022: Waste Incineration Power Plant project, Electrolyser project, Ashes recycling project, Mass Spectrometer project, Biogas project, and Gas Turbine of Methane GTM.

For the projects added to the NLAP-WEDC this year (2023):

- **Mobile Waste Separation Plant**
- **Mobile Biogas Generation and Gas Turbine Plant**
- **Waste Incineration Flue Gas Filters (Chemical Filter, Electrostatic Filter (Electrofilter)**

In the following sections, we will talk about each project in detail. What is it, where did it arrive before 2023, details that were added this year, and finally what should be completed with it.

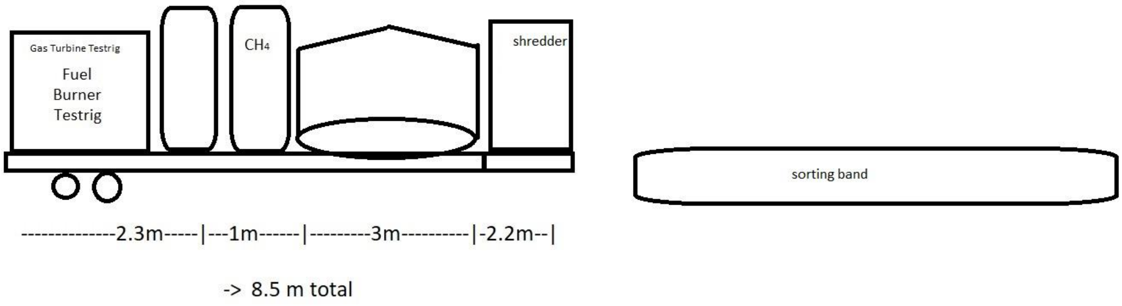
2 NLAP-WEDC (Waste to Energy Demonstration Cycle) Ras Maska 2023 - Overview [1]

2.1.1 تأمين الحائط من الانهيار

2.1.2 Panorama picture of whole plant (date: 04.10.2023):

From left to right: 1. Plant control system container 2. waste incineration plant 3. biogas (organic waste) plant 4. waste separation plant

2.1.3 Design with waste sorting band



Mobile Waste Separation Plant

https://www.aecenar.com/index.php/institute-projects/nlap-wedc/mobile-waste-separation-plant

Mobile Biogas Generation and Gas Turbine Plant

https://www.aecenar.com/index.php/institutes/icpt/mobile-biogas-generation-and-gas-turbine-testrig

Filter system improvement (Electrofilter)

https://www.aecenar.com/index.php/institute-projects/nlap-wedc/nlap-ipp-cad/nlap-ipp-filtersystem-design-cad/nlap-ipp-electrofilter-design

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[1] Ref : https://www.aecenar.com/index.php/institute-projects/nlap-wedc

Ref : NLAP-WEDC (Waste to Electricity Demonstration Cycle) (aecenar.com)

https://www.aecenar.com/index.php/institute-projects/nlap-wedc

3 Waste Separation System[2]

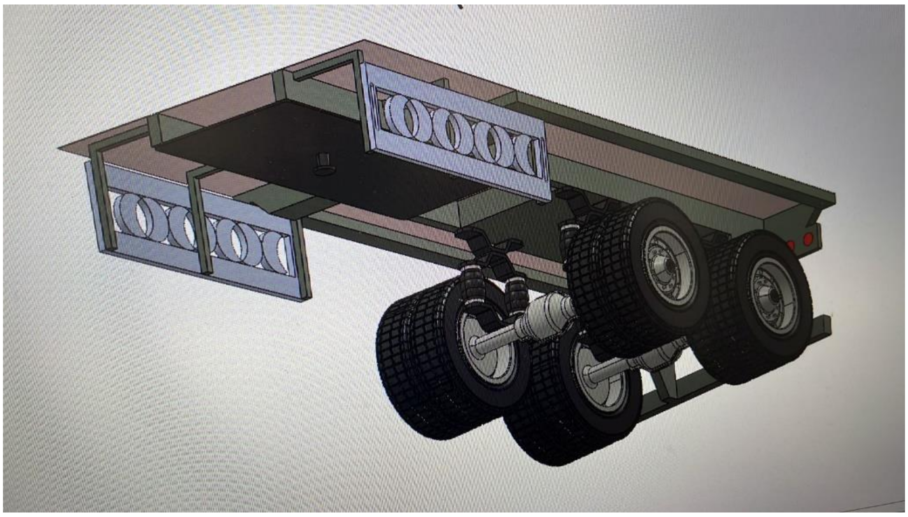
[Waste separation plant POSTER (pptx)](https://www.aecenar.com/index.php/downloads/send/5-nlap/1409-waste-separation-plant)

https://www.aecenar.com/index.php/downloads/send/5-nlap/1409-waste-separation-plant

Waste separation plant POSTER (pdf)

08.10.2023 Waste separation plant process Video 📽

https://www.aecenar.com/index.php/downloads/send/5-nlap/1429-waste-separation-process-video



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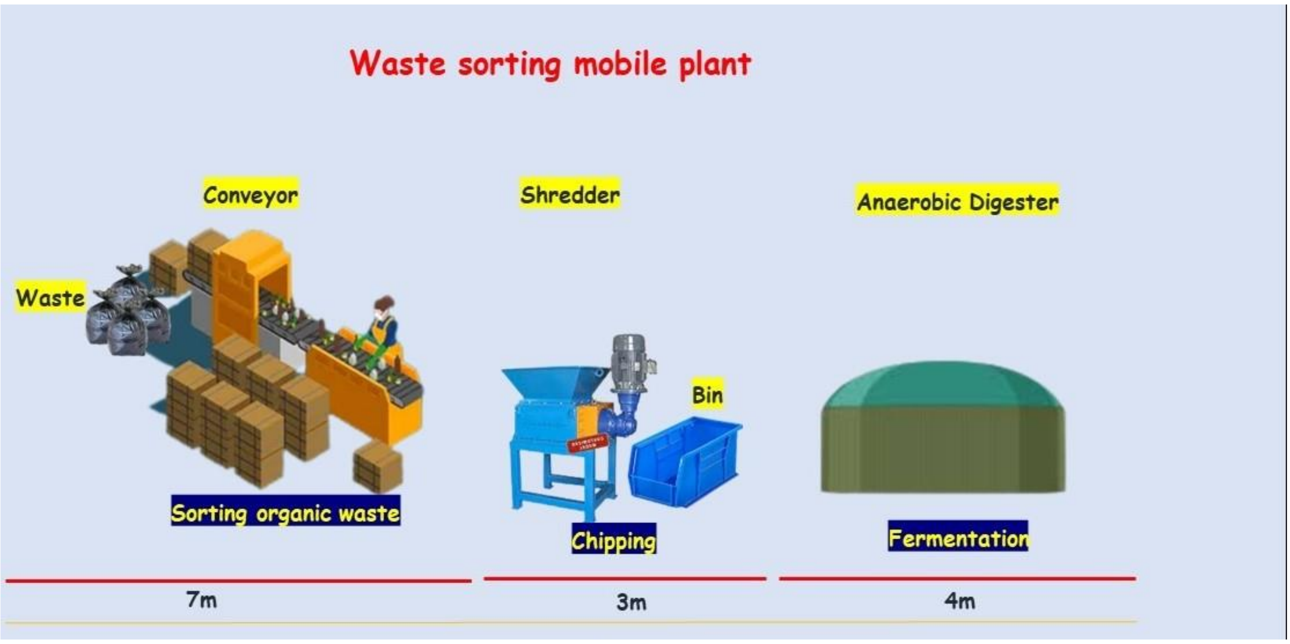
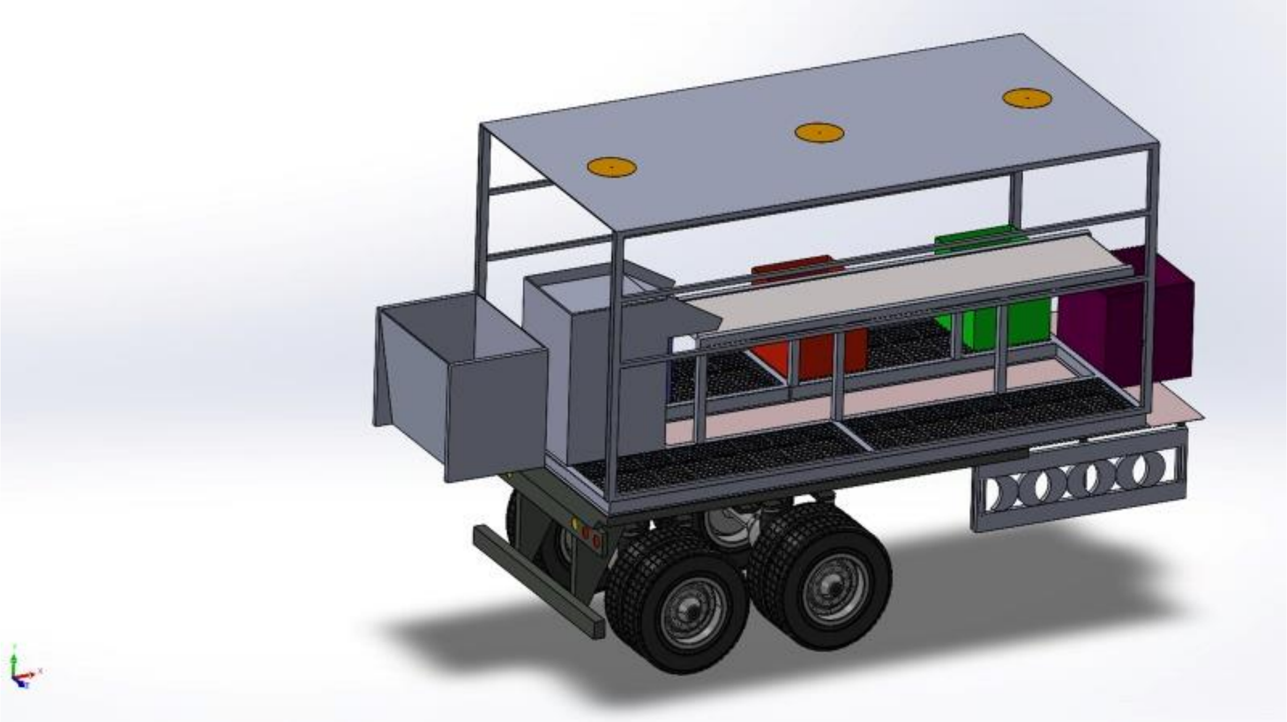
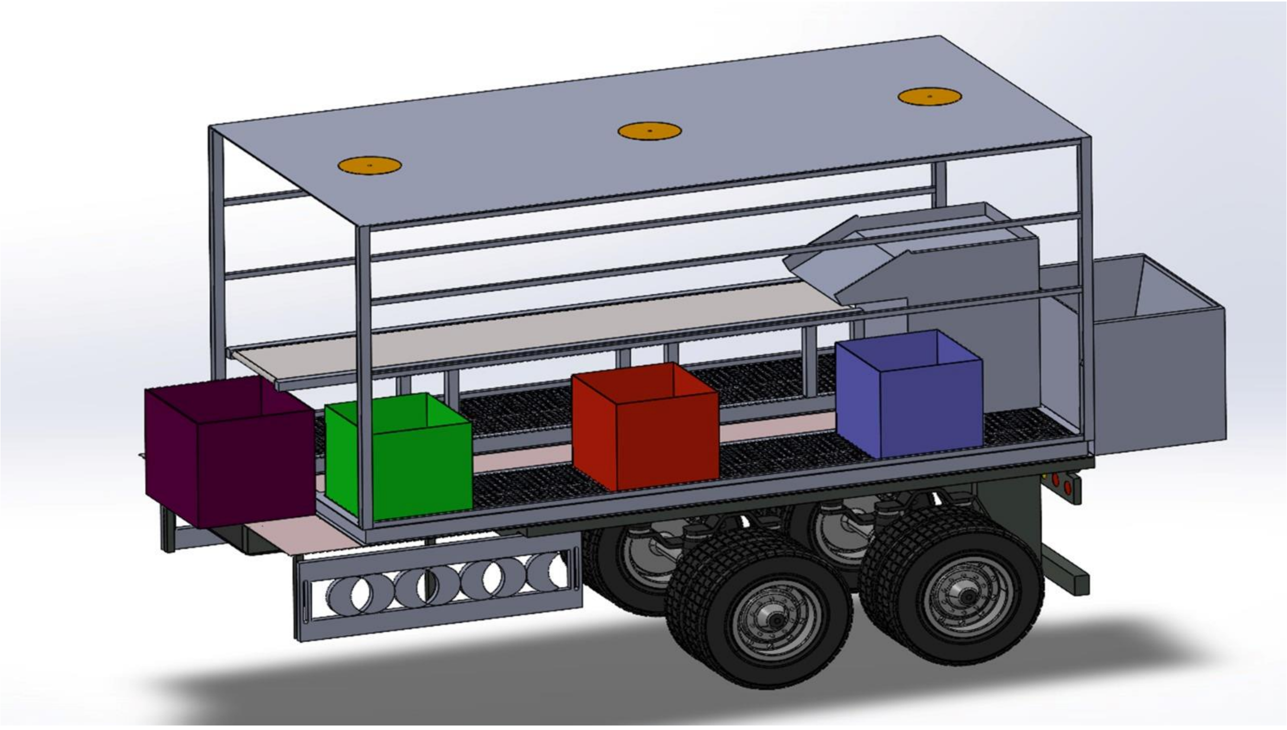
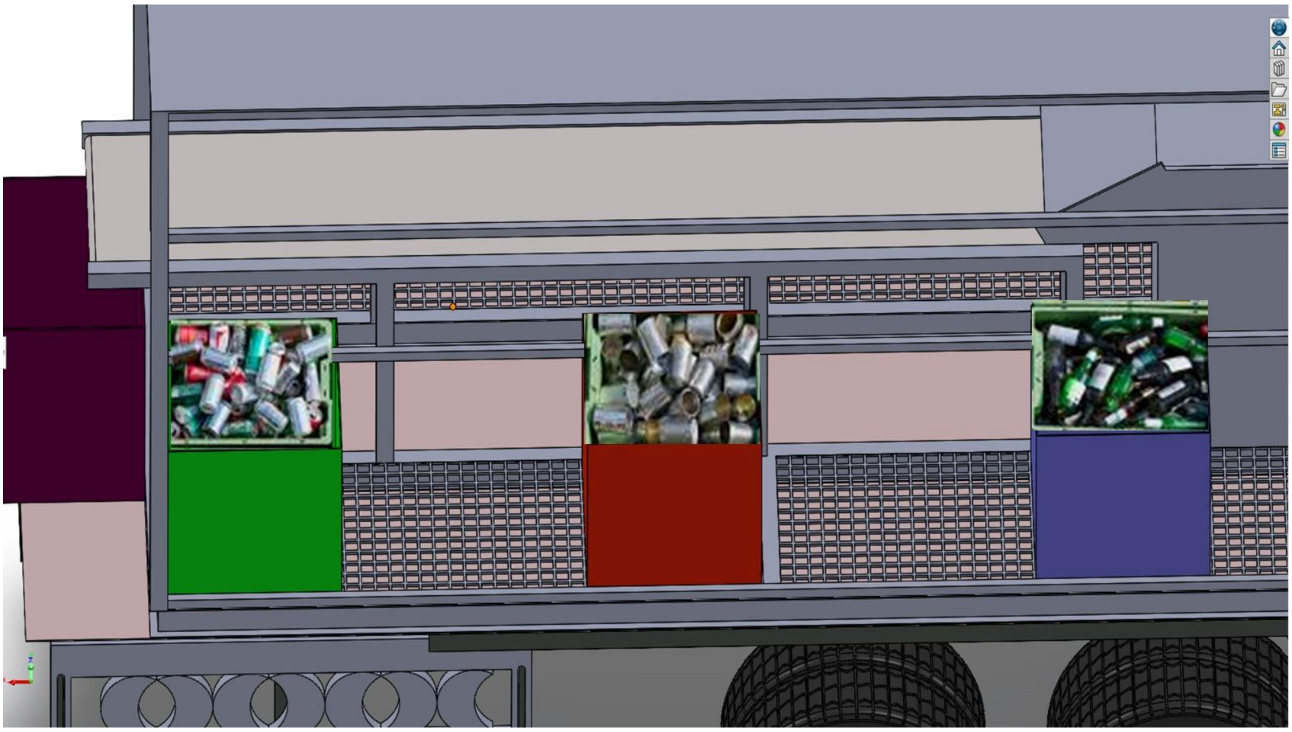
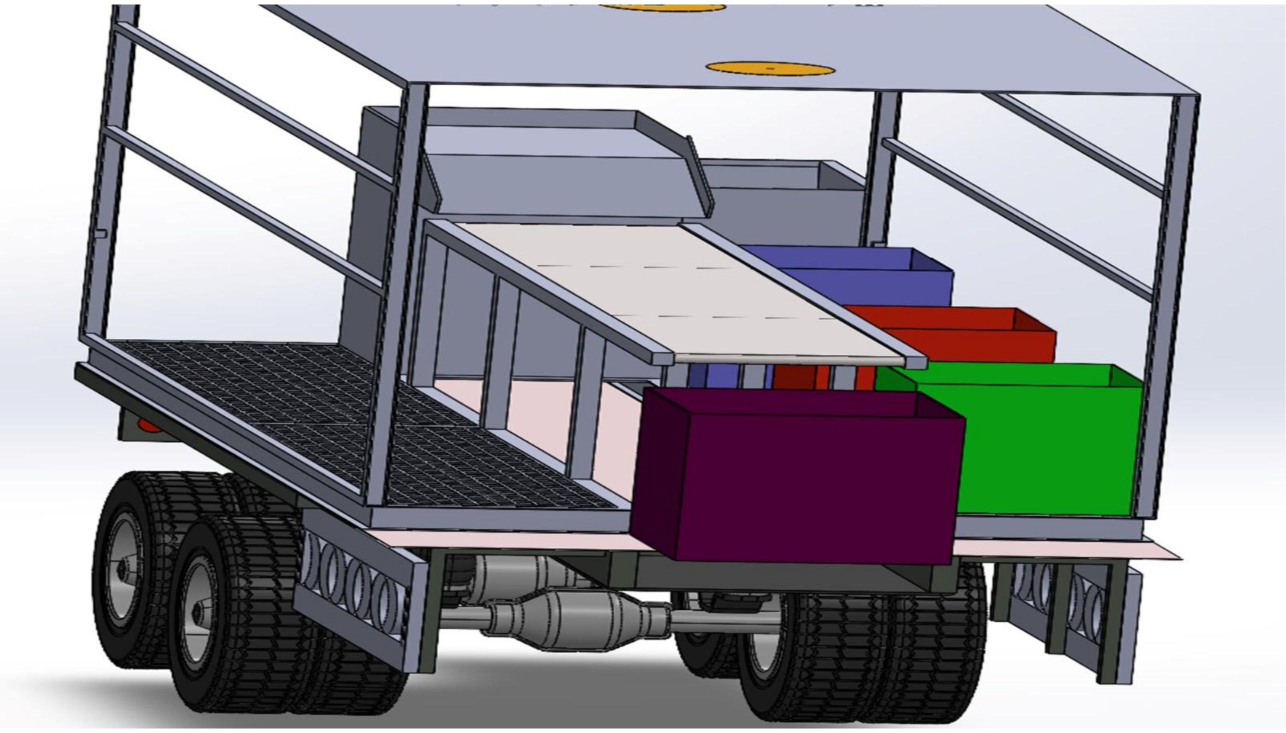
[2] Ref : https://www.aecenar.com/index.php/institute-projects/nlap-wedc/mobile-waste-separation-plant


Waste sorting mobile plant
Conveyor
Shredder
Anaerobic Digester
Waste
Bin
Sorting organic waste
Chipping
Fermentation
7m
3m
4m









CAD File (Solidworks/FreeCAD/...) (in STEP format) (Version 18.07.2023)

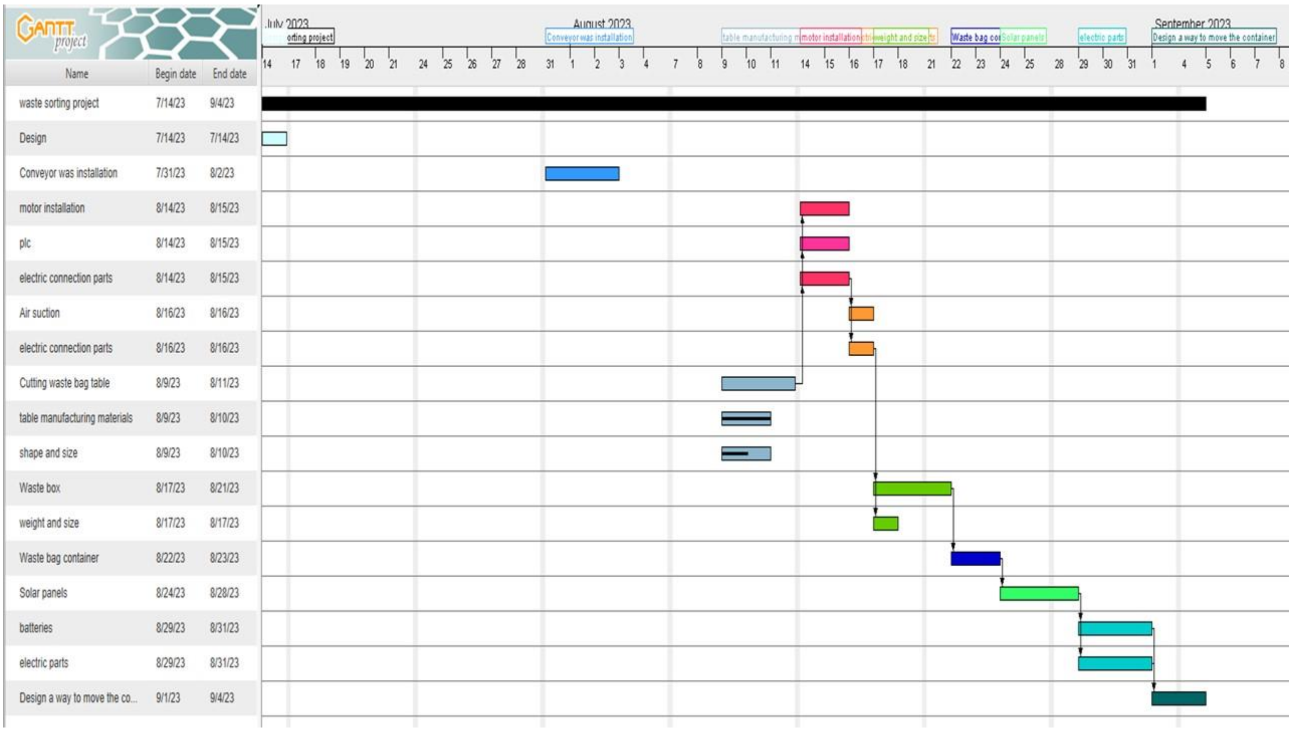
https://www.aecenar.com/index.php/downloads/send/5-nlap/1338-waste-separation-trailer-2023-cad-file-step-format

18.7.2023_0313_trailer (1).STEP

3.1 Realization (02.08.23)

Schedule

Realization (02.08.23)

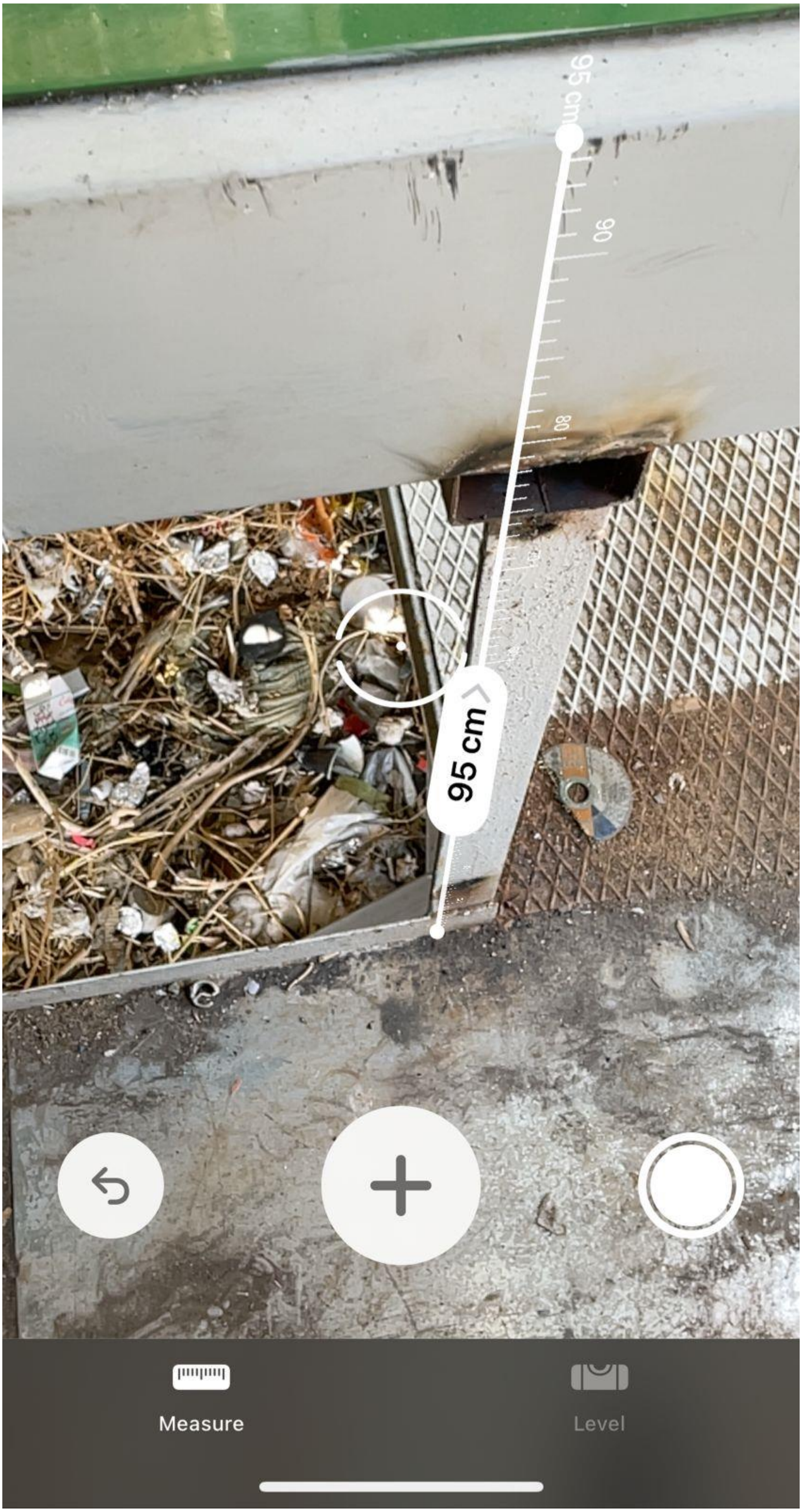


Name	Begin date	End date
waste sorting project	7/14/23	9/4/23
Design	7/14/23	7/14/23
Conveyor was installation	7/31/23	8/2/23
motor installation	8/14/23	8/15/23
plc	8/14/23	8/15/23
electric connection parts	8/14/23	8/15/23
Air suction	8/16/23	8/16/23
electric connection parts	8/16/23	8/16/23
Cutting waste bag table	8/9/23	8/11/23
table manufacturing materials	8/9/23	8/10/23
shape and size	8/9/23	8/10/23
Waste box	8/17/23	8/21/23
weight and size	8/17/23	8/17/23
Waste bag container	8/22/23	8/23/23
Solar panels	8/24/23	8/28/23
batteries	8/29/23	8/31/23
electric parts	8/29/23	8/31/23
Design a way to move the co...	9/1/23	9/4/23

Waste Sorting Project - Gantt chart

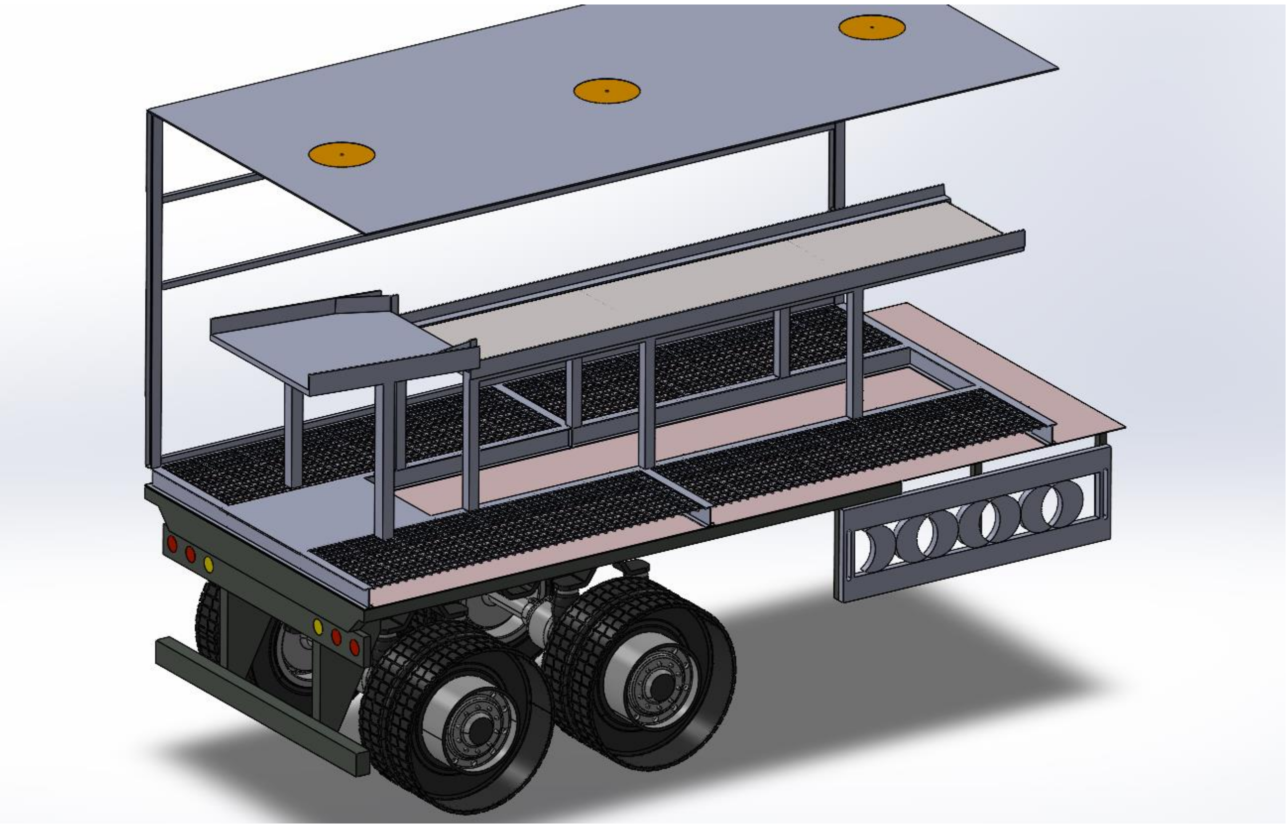
04082023_Waste Sorting Project.gan

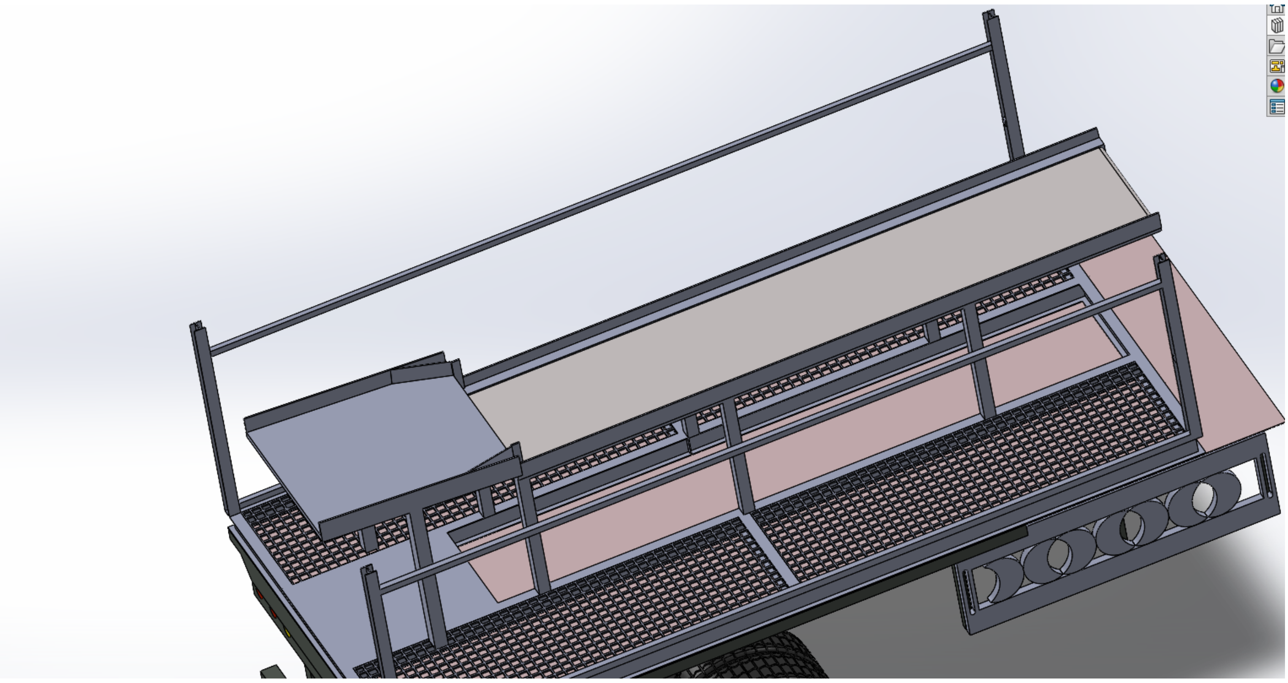
in this section we make the height of the conveyor 95cm tall and we put borders for the conveyor and the Waste cutting table


95 cm
90
80
95 cm
Measure
Level

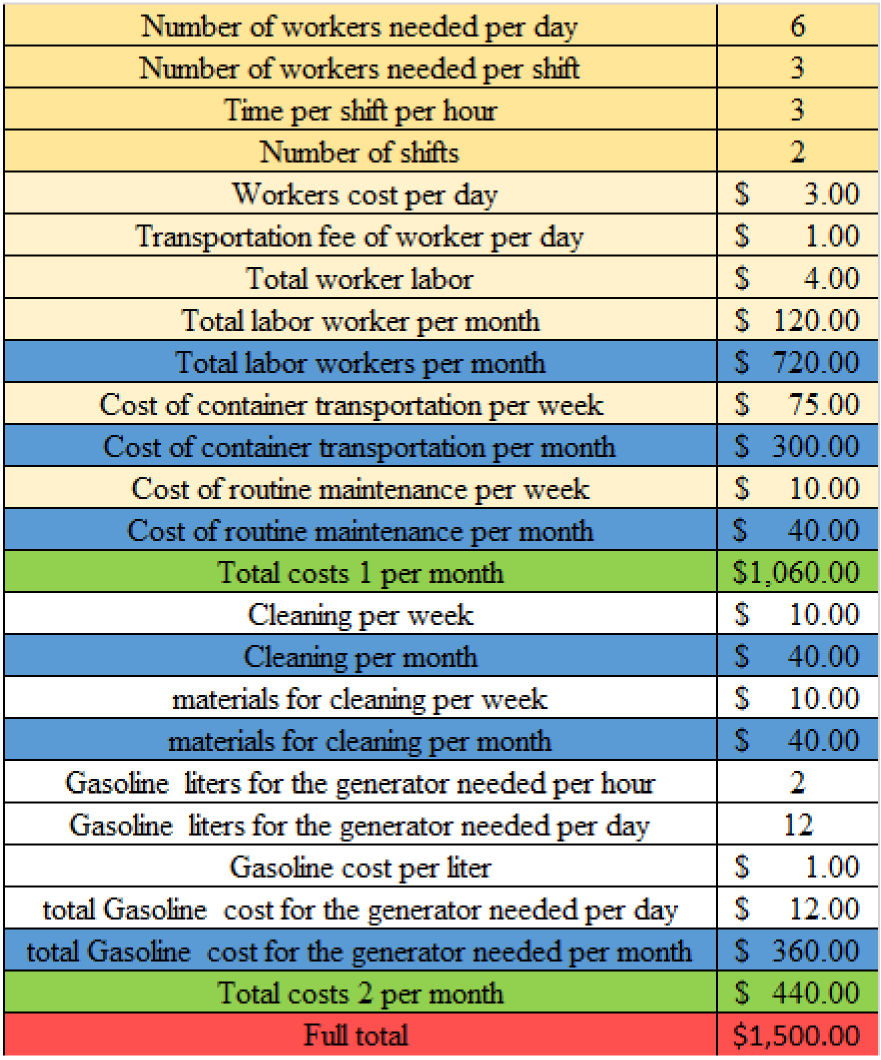

AYMORE

the model has been modified depending on the present work done





Costs of the waste separation process monthly



Number of workers needed per day	6
Number of workers needed per shift	3
Time per shift per hour	3
Number of shifts	2
Workers cost per day	$ 3.00
Transportation fee of worker per day	$ 1.00
Total worker labor	$ 4.00
Total labor worker per month	$ 120.00
Total labor workers per month	$ 720.00
Cost of container transportation per week	$ 75.00
Cost of container transportation per month	$ 300.00
Cost of routine maintenance per week	$ 10.00
Cost of routine maintenance per month	$ 40.00
Total costs 1 per month	$1,060.00
Cleaning per week	$ 10.00
Cleaning per month	$ 40.00
materials for cleaning per week	$ 10.00
materials for cleaning per month	$ 40.00
Gasoline liters for the generator needed per hour	2
Gasoline liters for the generator needed per day	12
Gasoline cost per liter	$ 1.00
total Gasoline cost for the generator needed per day	$ 12.00
total Gasoline cost for the generator needed per month	$ 360.00
Total costs 2 per month	$ 440.00
Full total	$1,500.00

excel-sheet of the prices on 1034_12092023

separation.xlsx

The roof top of the container has been changed on 19/09/2023

- First the old roof top of the container removed

- The connecting rod of the roof top was removed and replaced by 4 rods to make it more durable and stable

- A new T.O.T sheets were added to the roof top


Galarie


Galerie Paul Steeve

Realization (02.08.23)

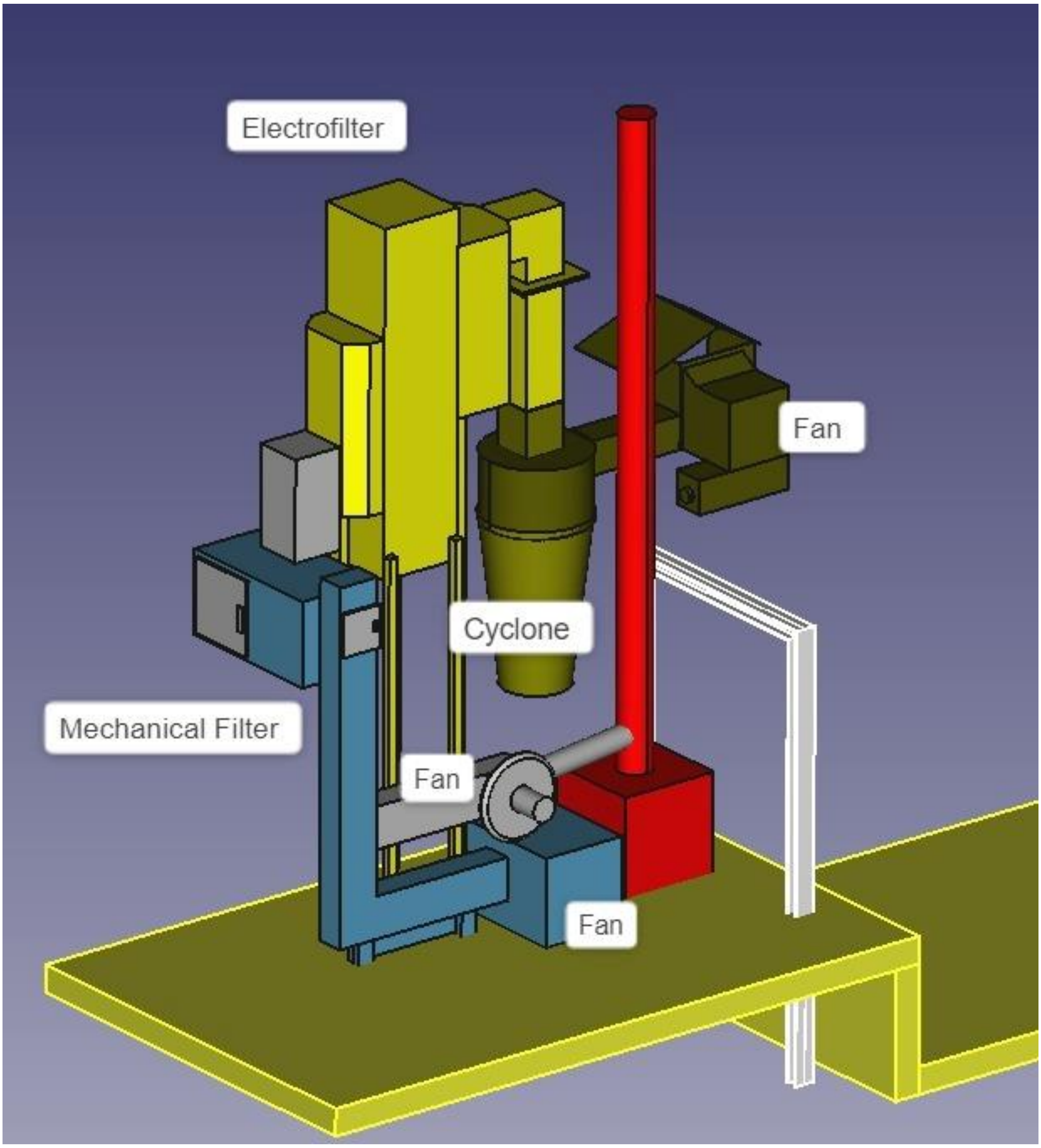
https://www.aecenar.com/index.php/institute-projects/nlap-wedc/mobile-waste-separation-plant

4 Environment Friendly Treatment

4.1 NLAP-IPP Incinerator Filter System[3]

NLAP-IPP Filtersystem Design (CAD)

4.1.1 Filter System 2022



The current design of the filtration system without the electrofilter (11.8.2022):

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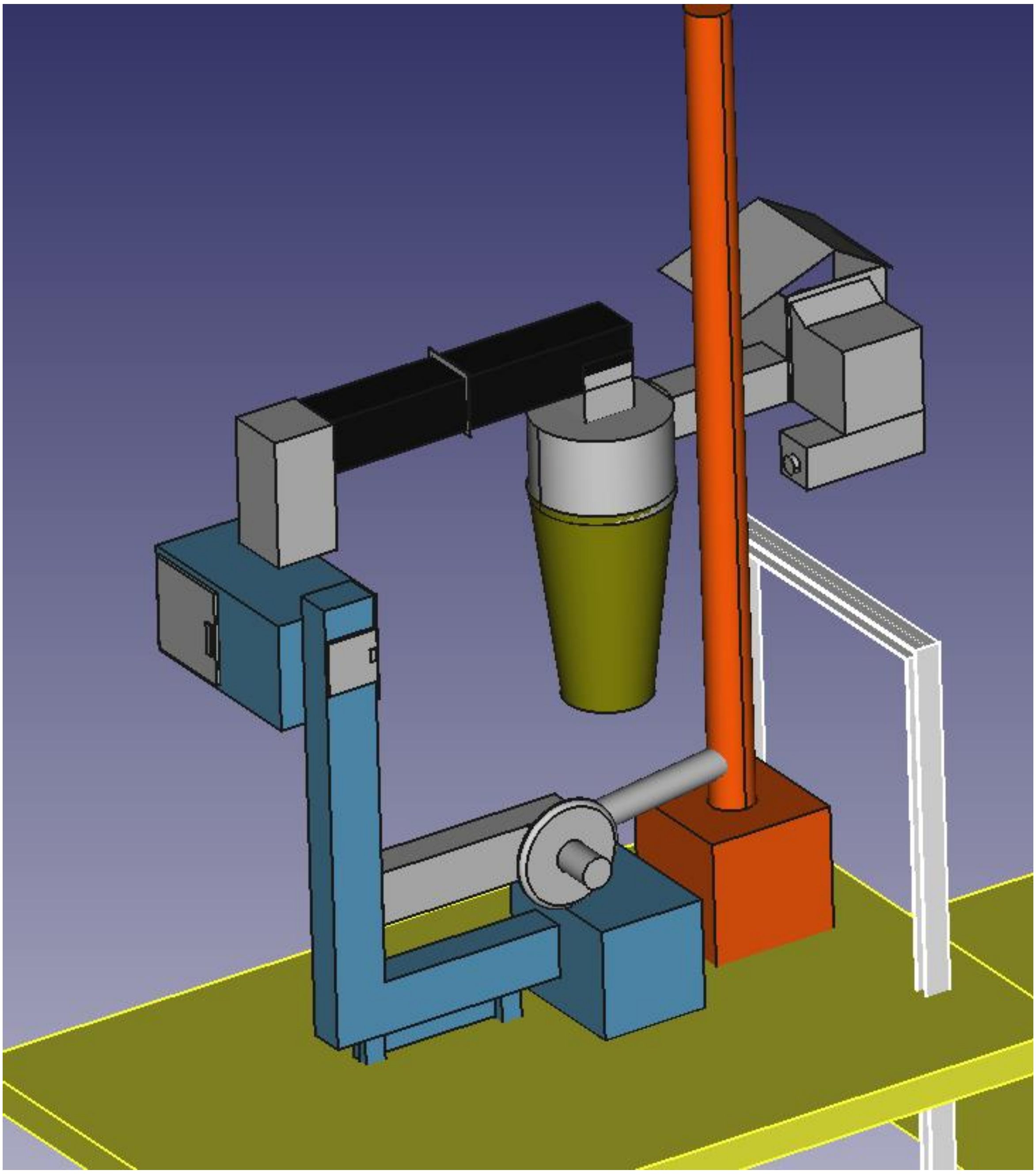
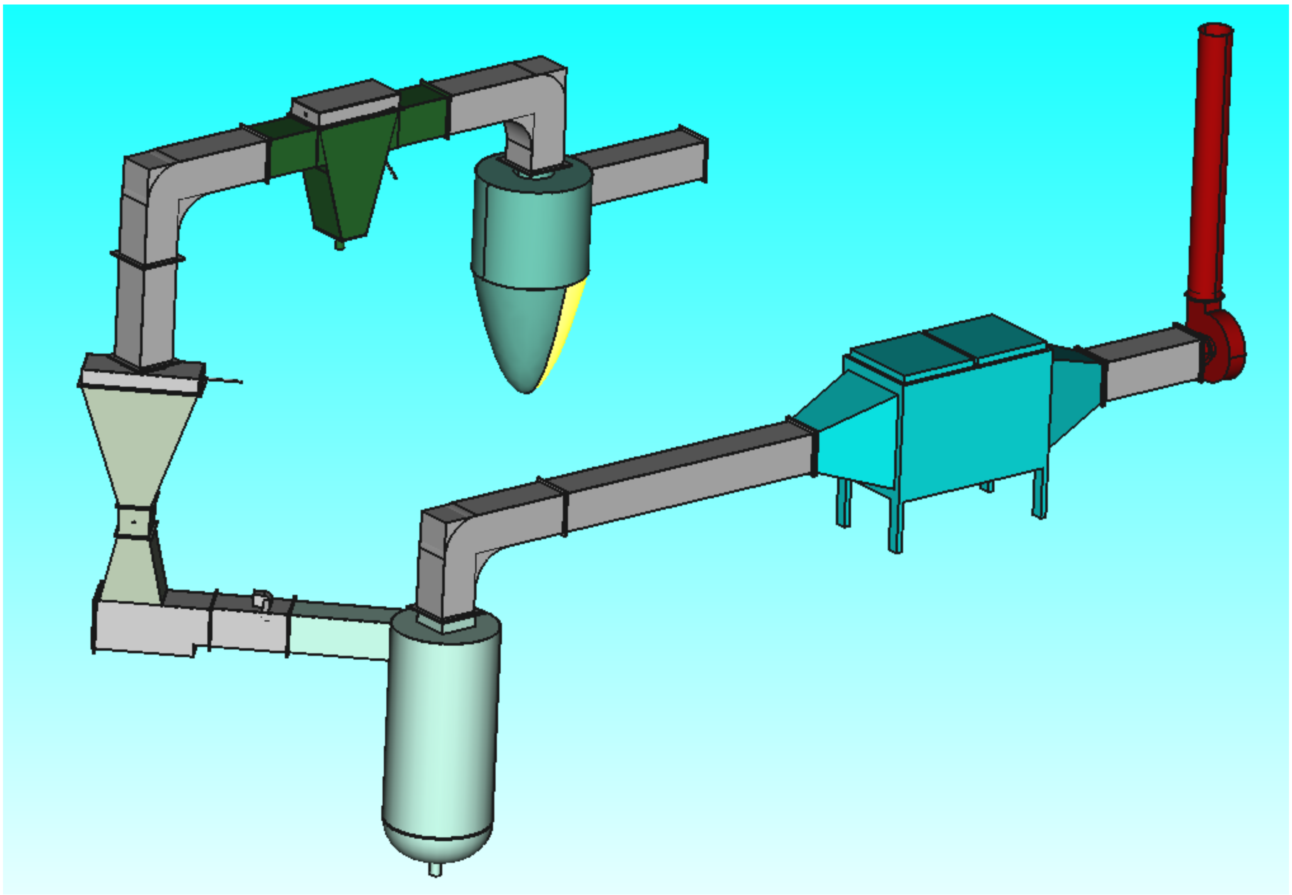
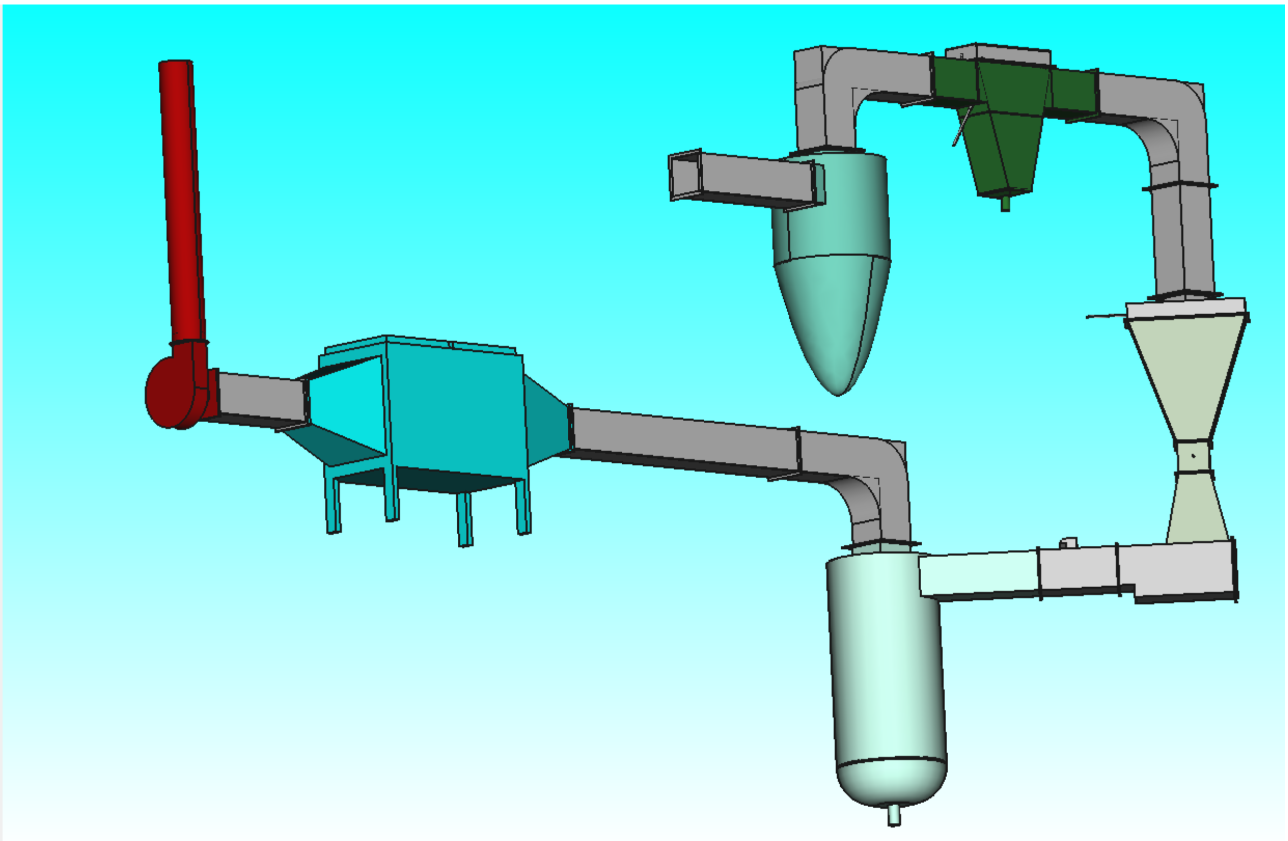
[3] Ref : https://www.aecenar.com/index.php/institute-projects/nlap-wedc/nlap-ipp-cad/nlap-ipp-filtersystem-design-cad



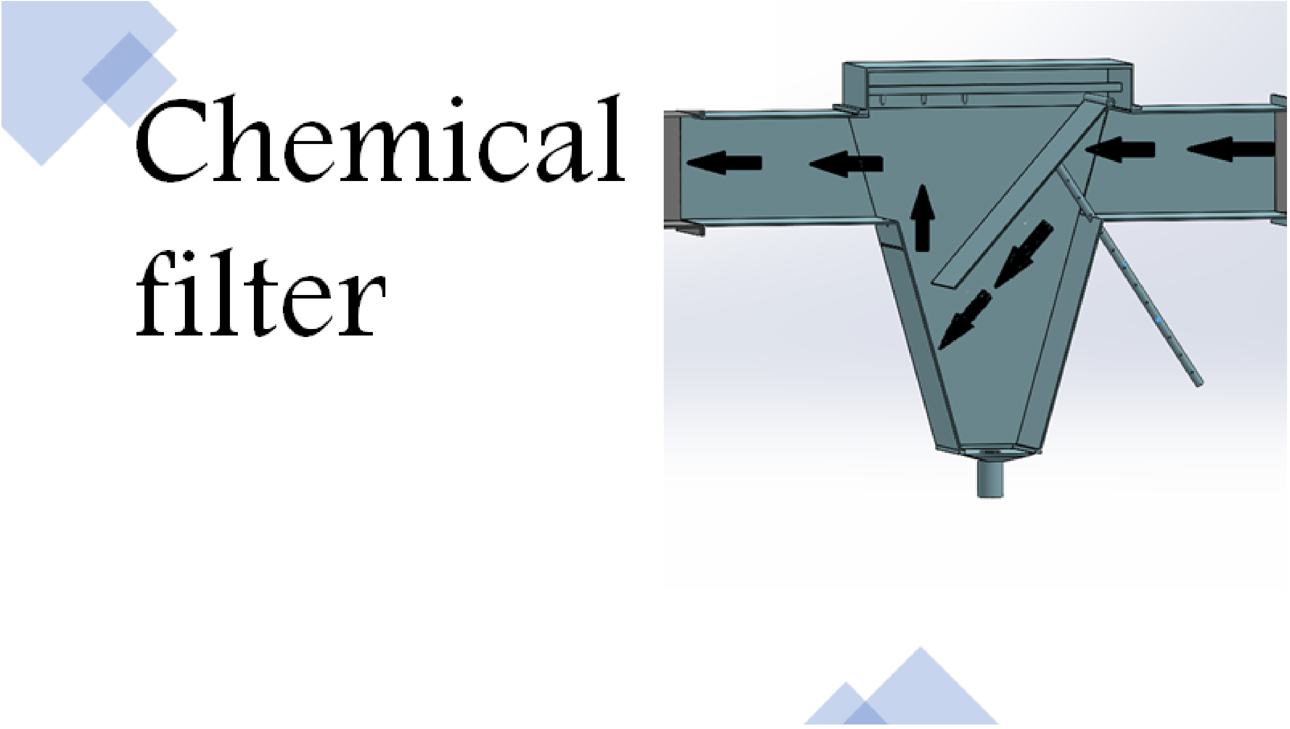
4.1.2 Power Plant Filtration System Version 21.10.2023





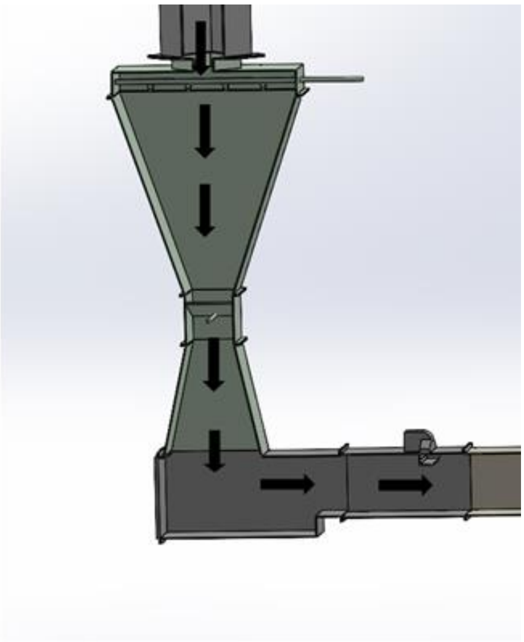
4.1.2.1 Here is how the cycle works

First Cyclone

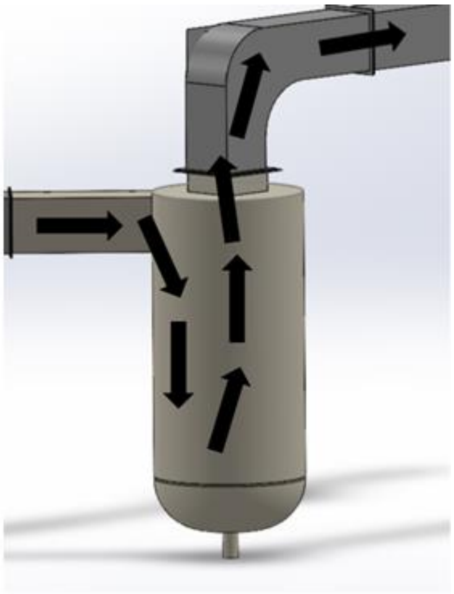


Chemical filter

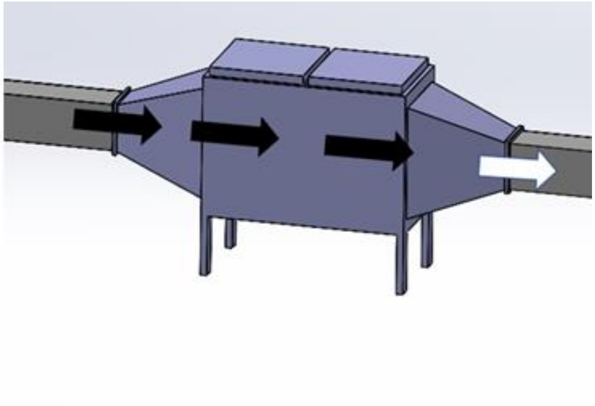
Scrubber



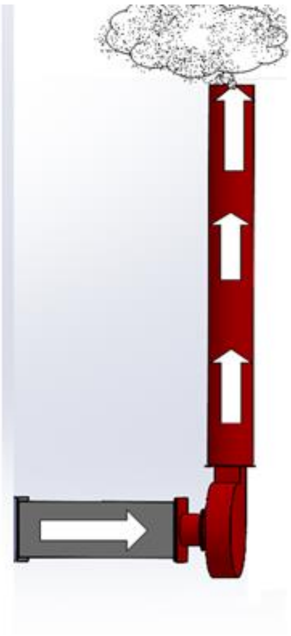
Second Cyclone

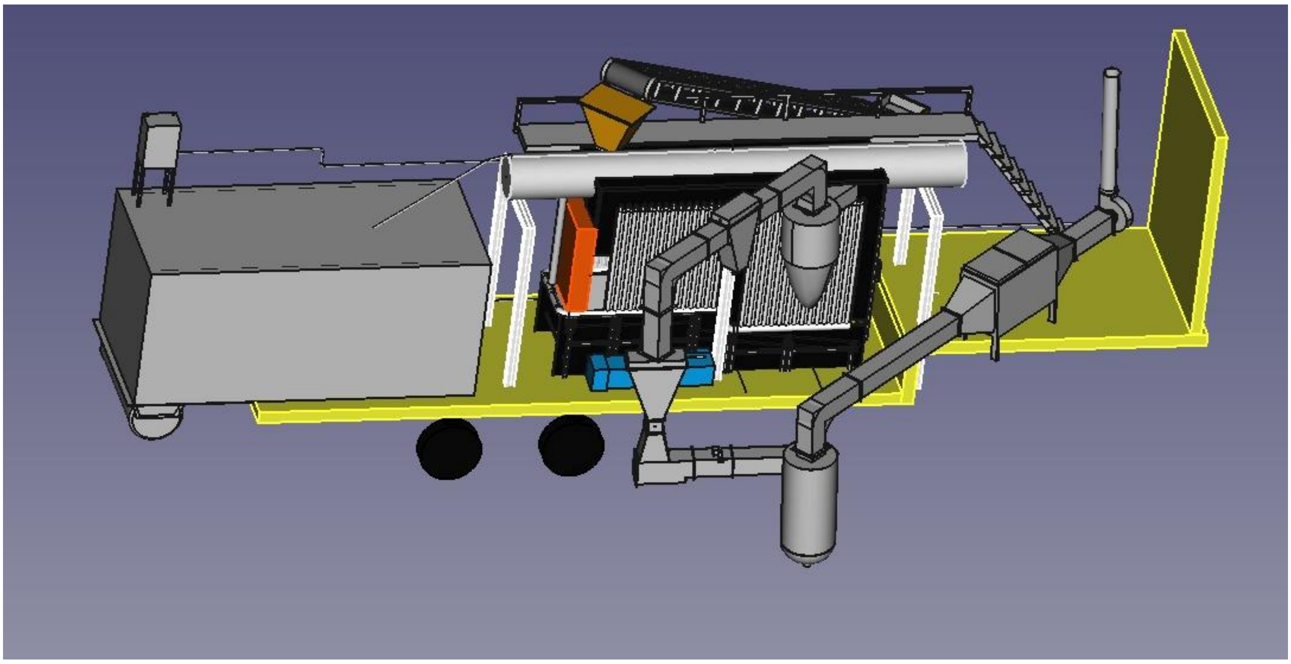


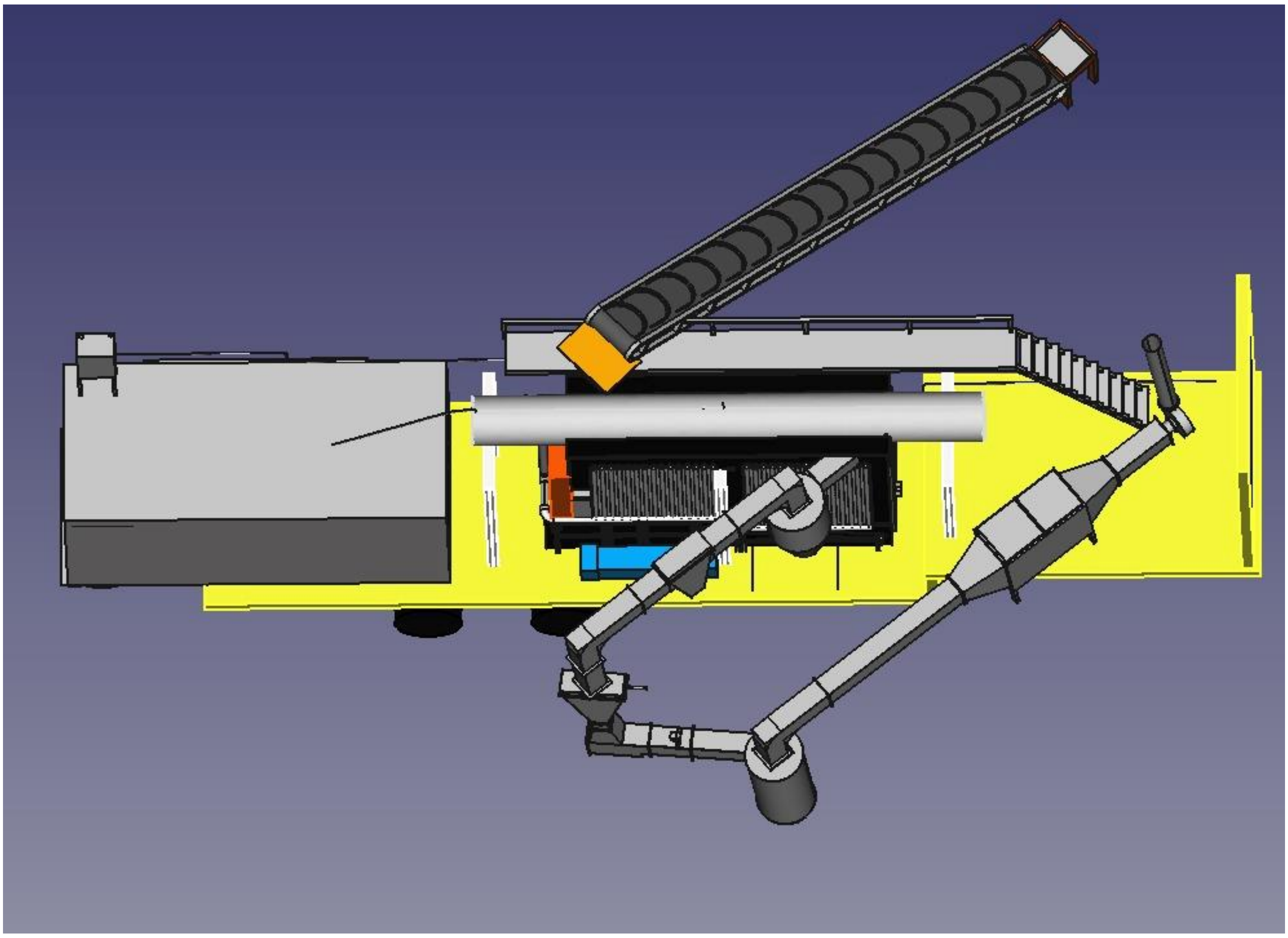
Electro filter

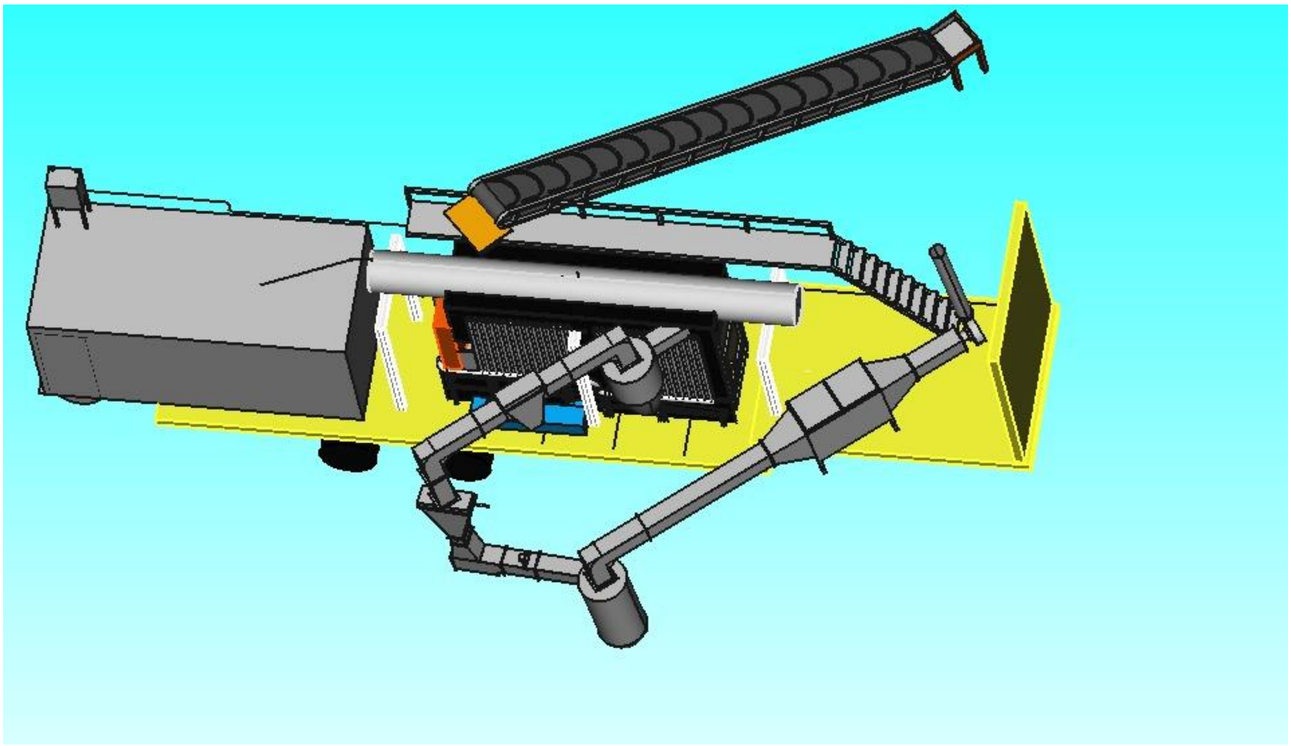


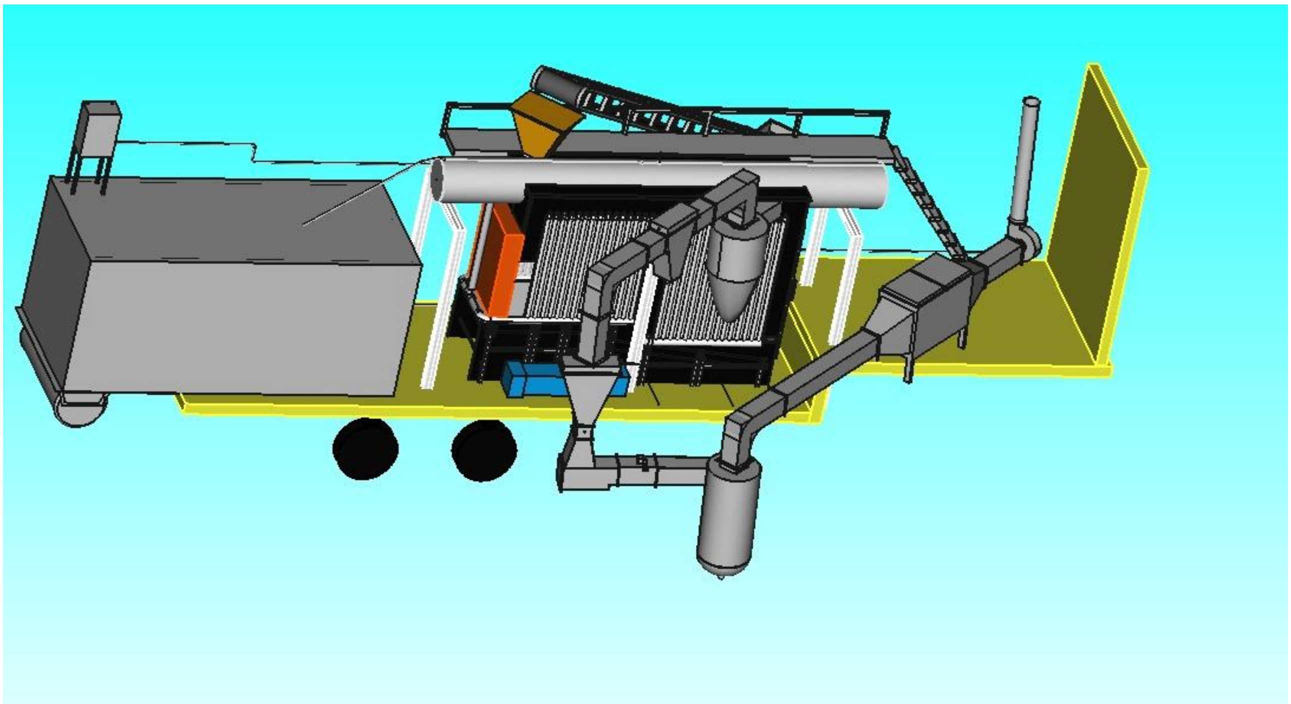
Chimney







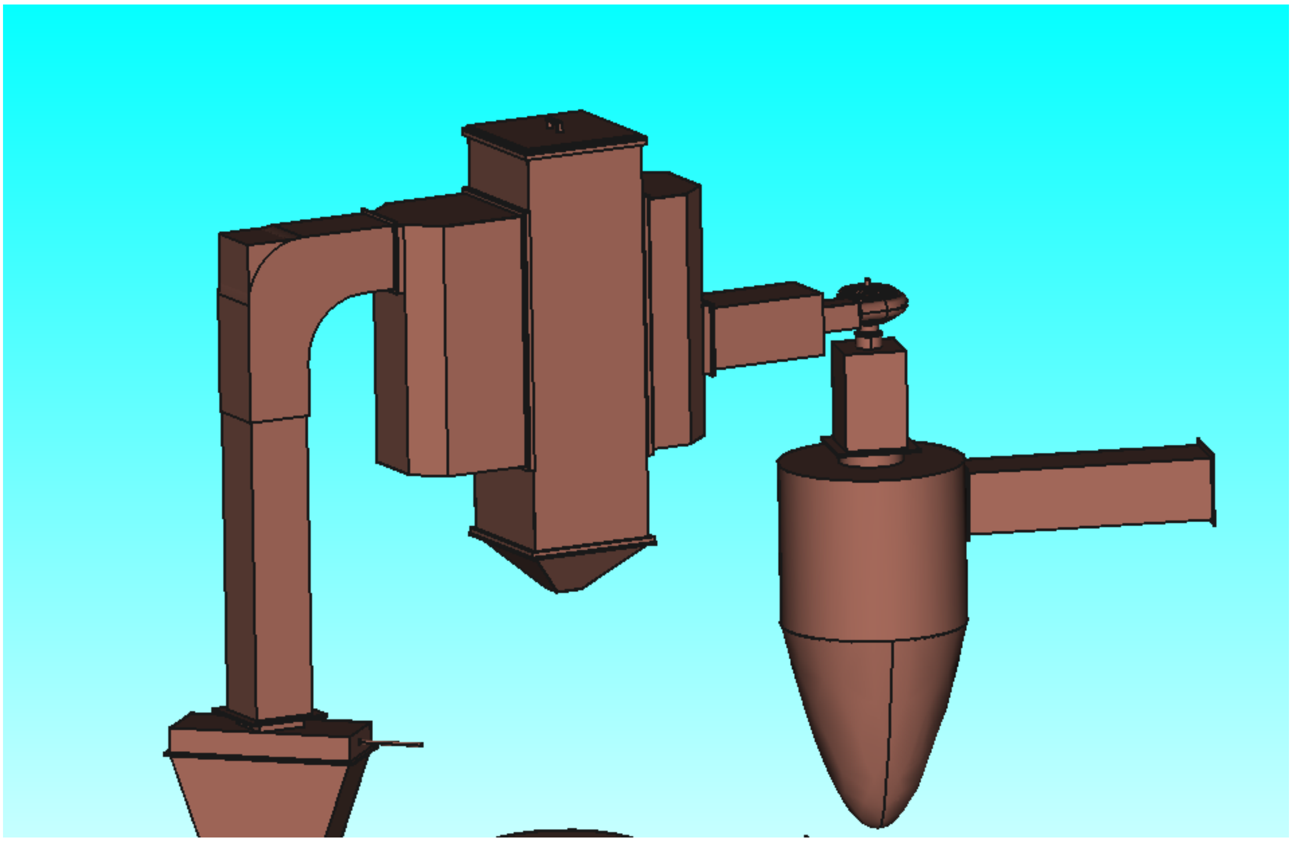


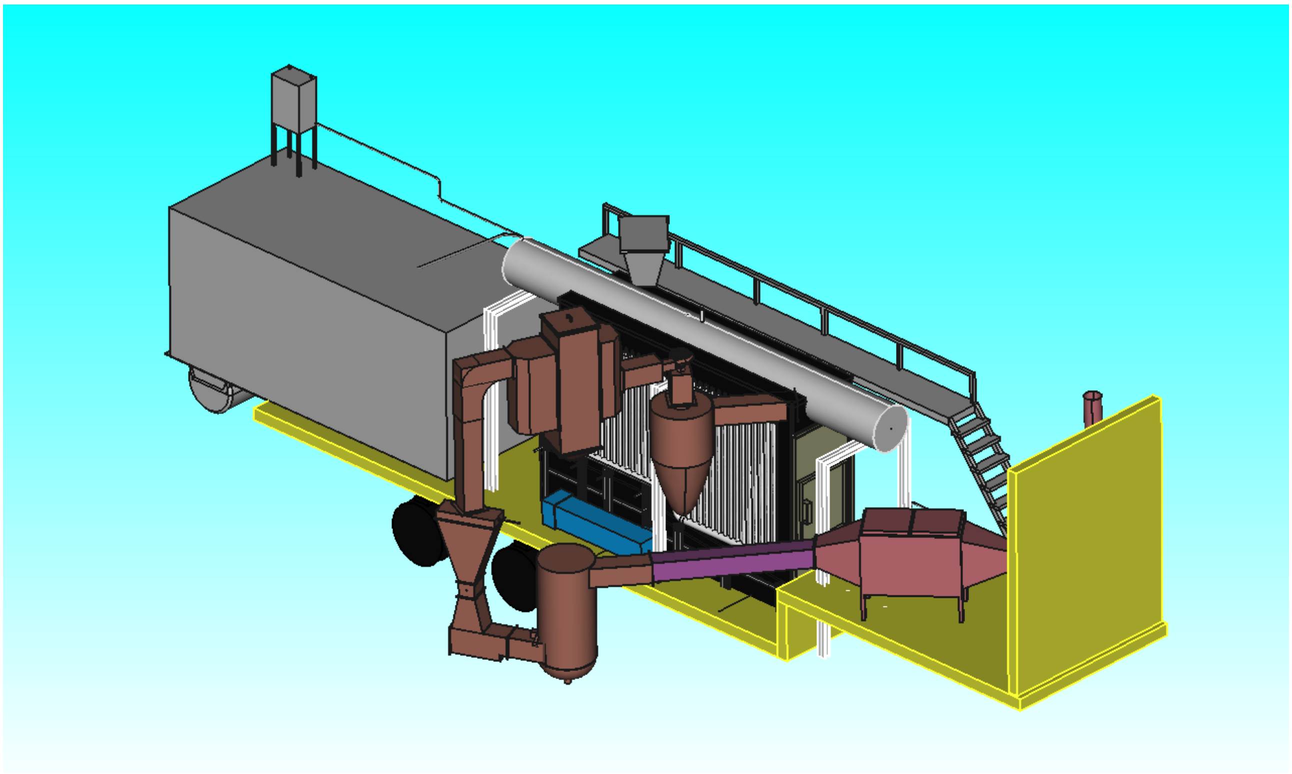


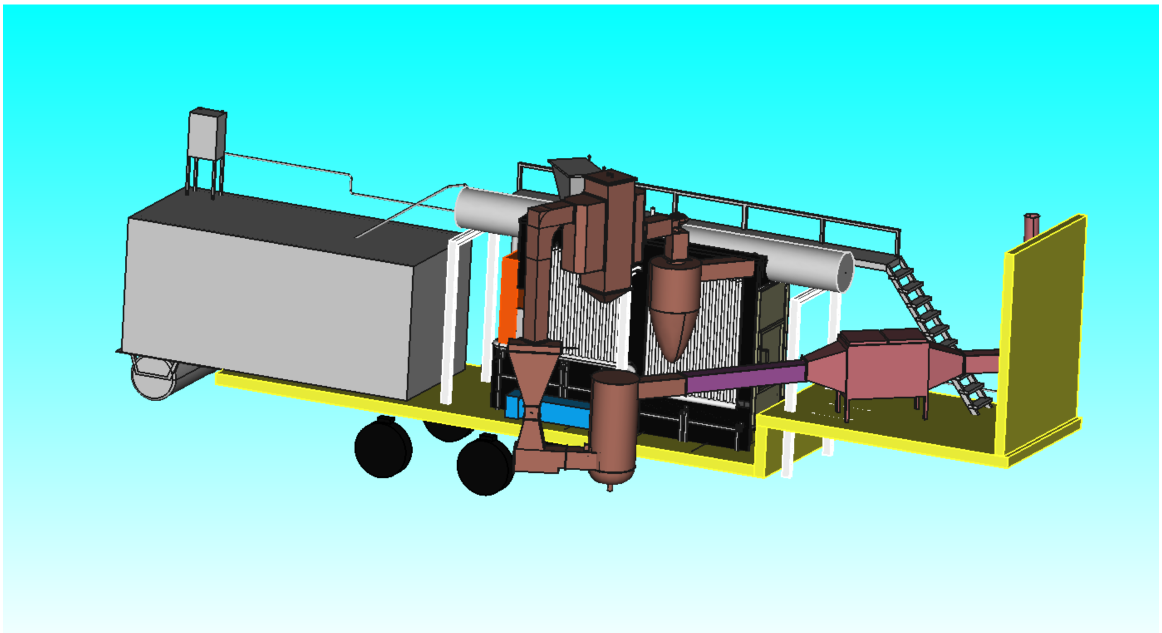
4.1.2.2 On November 27, 2023, the Flue Gas System was updated

This system has been modified by adding the old chemical filter and the old cyclone only, and nothing else has been modified.

This test will be done to see the effectiveness of the old cyclone, the chemical filters, the air turbine, and the new sprinklers with the new water system to cool and clean the flow gas.







This system will be tasted after connecting the new water tank and the two chemical tanks with water pump for each one

and this is the full FREECAD system that was modified on November 27, 2023:

https://www.aecenar.com/index.php/downloads/send/5-nlap/1437-nlap-ipp-ver-nov2023-with-chem-filter-e-filter-fcstd-file

27112023_NLAP-IPP_filtration systemCyclon

Filter System	21102023NLAP-IPP-FilterSystem.FCSTd (last Version: 21.10.2023)	filter system0.FCStd
	FreeCAD Model of NLAP-IPP Filtration System (FCStd file) (Version: 3.8.2022)	030822NLAP-IPP_Filters.FCStd
Filter System without the Electrofilter	FreeCAD Model of NLAP-IPP Filtration System without the Electrofilter(FCStd file) (Last version: 11.8.2022)	120822NLAP-IPP_Filters.FCStd
Filter System without the Electrofilter :	FreeCAD Model of NLAP-IPP Filtration System without Electrofilter(FCStd file) (last version: 27.11.2023)	27112023_NLAP-IPP_filtration systemCyclon

Ref : https://www.aecenar.com/index.php/institute-projects/nlap-wedc/nlap-ipp-cad/nlap-ipp-filtersystem-design-cad

4.1.3 Filtration test (18.12.2023)[4]

In this test, we adhere to the test specification (01 Test Specification of the Filtration System) by inspecting each and every component of the filter system to ensure its functionality.

First, we double-checked the mechanical components, such as the ball valves, ducts, and piping system from the chemical filter and scrubber to the chemical and water supply tanks, to ensure that everything was properly linked.

Second, we used the GUI software to inspect the electrical connections for each item, such as pumps, air supply fans, and exhaust fans. Each device was checked for functionality for 10 seconds

Third, we discovered that the garbage bags had become wet as a result of the rain; therefore, instead of the waste bags, we used bags filled with wood.

Fourth, we examined the manual flame igniter and discovered that it was not working properly due to a malfunction in its electric parts, and the trailer of the powerplant was also not steady and was inclined, preventing the tank from pushing the Dizel to the flame igniter.

Fifth, we used the GUI software to open the exhaust fans and the air supply fans, then we manually started the fire through the main gate, and when the fire started, we closed the main gate.

Sixth, we begin collecting data for the temperature at the incinerator outlet, the temperature between the chemical filter and the scrubber, and finally the temperature in the electro-filter inlet.

Seventh, we collected data on T1, T2, and T3 sensors when P1 and P2 were closed, when P1 and P2 were opened, when P2 was closed, and finally when both P1 and P2 were opened together again.

Eighth, we used the GUI software to close the pumps P1 and P2, the exhaust fans E1 and E2, and the air supply fans AF1 and AF2, while manually closing the ball valves BV1, BV2, BV3, BV4, and BV5. to put out the fire, and we double-checked that all systems were turned off.

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[4] https://www.aecenar.com/index.php/institute-projects/nlap-wedc/nlap-ipp-system-tests/filtration-test-18-12-2023

4.1.3.1 Table of System Test

Step	Step description	Expected result	result		result description
Precondition			✗	✓	Why?
check out the system	System is off	all system is off		✓	
Initial Condition					
check out steps	check the mechanical filtration connections: Ducts, BV1, BV2, BV3 ,BV4, BV5, P1, P2, E1 and E2.	All Mechanical connections are good		✓	

	check the electrical filtration connections: wires. T1, T2, T3, P1, P2, E1 and E2 (By the Gui Software) for 10 seconds each one.	All Electrical connections are good and temperature between 20 and 30 degrees Celcius	✓	
	Check the Air Supply fans AF1 and AF2 for 10 seconds (By the Gui Software).	Air fan Supply working good	✓	
	check the flame igniter manually for the incinerator	Flame igniter working good	✗	the flame igniter didn't work
putting waste bags	put the waste bags inside the incinerator	no obstacle	✗	we use wood instead of

					the wet waste bags
add Dizel	add Dizel inside the dizel-tank presented above the turbine room	no obstacle	✗		the power plant trailer is inclined and the dizel not moving in the pipes
Turn on E1 and E2	for 10 Seconds Turn on the Exhaust fans presented Presented above Cyclone #1 and below the chimney (By the Gui Software).	the Exhaust fan working		✓	

Turn on P1 and P2	for 10 Seconds Turn on the liquid pumps for the chemical filter and the Scrubber (By the Gui Software).	the Liquid pumps working	✓		
open BV1, BV2, BV3 ,BV4 and BV5	open the ball valves for the chemical filter, scrubber, cyclone #2, the Water tank, and the chemical tank. Manually	no obstacle	✓		
Starting Test					
check out T1, T2 and T3	check the temperature at its initial condition before starting	the Temperature must be between 20 and 30	✓		T_1 16 T_2 25 T_3 17

	(by the GUI Software).	degrees celcius		
Turn on E1, E2, AF1 and AF2	Turn on the exhaust fans (E1, E2) and the air supply fans (AF1, AF2) of the incinerator (By the Gui Software).	air start flow out of chimney.	✓	
	Start the fire using the Flame igniter to the waste bags (manually).	igniter start fire	✗	the fire started manually and igniter doesn't work
	Close the main incinerator gate	it close well	✓	

check out T1, T2 and T3	Check out T1 Sensor to get the max Temparature	the max temperature above 400 degrees Celcius	✓		T_1	125	140	200	226
	Check out T2 Sensor to see the Effectiveness of the Chemical filter chamber and the Cyclone #1	no obstacle	✓		T_2	60	70	95	120
	Check out T3 Sensor to see the effectiveness of the Cyclone #2	no obstacle	✓		T_3	46	51	67	80
open P1 and P2	Turn on the liquid pump for the chemical filter to start filtration	the pumps start working well	✓						

	and the scrubber to start cooling															
check out T1, T2 and T3	Check out T1 Sensor to get the max Temparature	temperature increasing or stay steady		✓		T_1	335	350	400	435	456	456	453	478	513	503
	Check out T2 Sensor to see the Effectiveness of the Chemical filter and Cyclone #1 cooling	temperature decrease		✓		T_2	131	130	128	133	150	152	161	171	178	185
	Check out T3 Sensor to see the effectiveness of the system and especially the scrubber cooling	temperature decrease below 60 degrees Celcius	✗		the temperature is between 74 degrees Celcius and 94 degrees Celcius	T_3	79	73	71	74	81	82	85	89	91	94

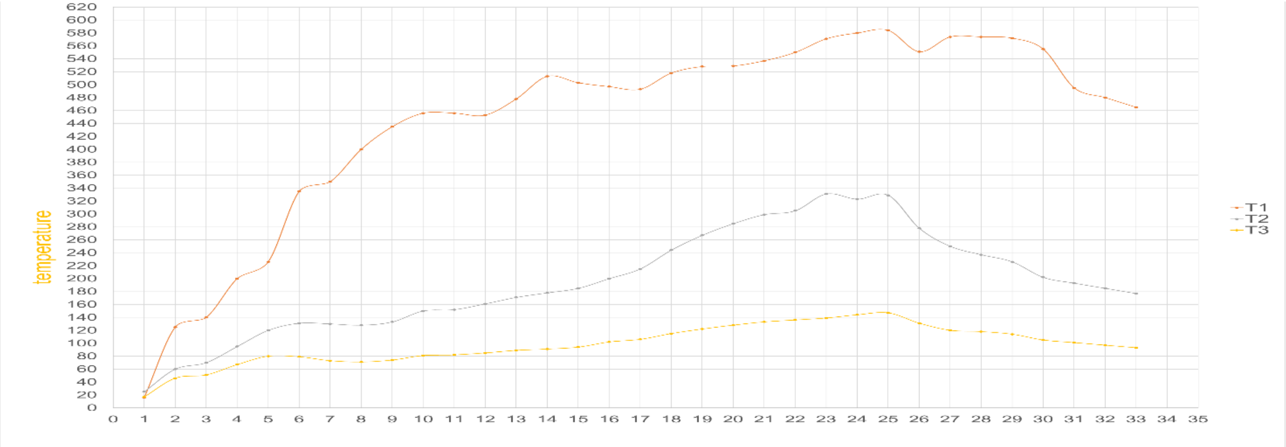
open the Chemical tanks	check out the filtration Cycle	the color of water starts to change	✓												
close P2 pump	close the chemical liquid pump to check the effectiveness of the cooling process by the scrubber	the pump P2 close normally	✓												
check out T1, T2 and T3	Check out T1 Sensor to get the max Temparature	temperature increasing or stay steady	✓		T_1	497	493	518	528	529	537	550	571	580	584
	Check out T2 Sensor	temperature start to raise up	✓		T_2	200	215	244	267	285	299	305	331	323	329
	Check out T3 Sensor to see the effectiveness of	difference in the temperature	✗	the temperature	T_3	102	106	115	122	128	133	136	139	144	147

	the scrubber cooling compared to the initial process without both Pumps	compared to the initial data		is increasing									
open P2 pump	open the chemical liquid pump	the pump P2 open normally	✓										
check out T1, T2 and T3	Check out T1 Sensor to get the max Temparature	temperature increasing or stay steady	✗	the temperature started to decrease because the wood bags are mostly finished	T_1	551	574	574	572	555	495	480	465
	Check out T2 Sensor	temperature start to decrease	✓		T_2	278	250	237	226	202	193	185	177

	Check out T3 Sensor to see the effectiveness of the filters cooling compared to the condition when P2 was closed	difference in the temperature compared to the above condition	✓		T_3	131	120	118	114	105	101	97	93
close P1 and P2	disconnect the water and chemical towards the filters	system kills the fire	✓										
close BV1, BV2, BV3 ,BV4 and BV5	close the ball valves to close the filtration system		✓										
close E1 and E2	close the exhaust fan (E1, E2)		✓										

Close AF1 and AF2	close the Air supply fan (AF1, AF2)		✓	
Postcondition				
check out the system	System is off	all system is off	✓	

Graph of the Temperature Sensors



Guide:

From 0 to 1, the Table system was in rest.

From 1 to 5, Pumps P1 and P2 were closed.

From 5 to 15, Pumps P1 and P2 were opened.

From 15 to 25, pump P2 was closed and P1 was opened.

From 25 to 33, P2 was opened again when P1 was already opened.

4.1.3.2 Test Analysis

- The flame igniter had difficulty since the trailer became inclined due to rain and sank backward to the ground, preventing the fuel from feeding the flame igniter.
- Both pumps P1 and P2 assisted the flue gas in lowering the temperature when it reached values between 74 and 94 degrees Celsius; it was not enough, but it did a nice job in comparison to the previous test results.
- The chemical filter performed well in the filtration process and in cooling the flue gases, but this was not permanent because the drainage of the chemical filter was connected to the main hole of the chemical, so it was fed again, causing its temperature to rise to 60 degrees Celsius, reducing the cooling process of the flue gases.
- The new sprinklers did a better job of chilling the flue gas with less water and chemical supplies than the old standard method.
- The cleaning procedure was improved by the addition of cyclone number one.
- Adding an extra temperature sensor to the filtering cycle improves data collection accuracy.
- Connecting the GUI software in the room close to the engine trailer rather than the far-distant center improves data transmission speed.

4.1.3.3 What should we do next?

- Repairing the flame igniter and connecting it to the graphical user interface software.
- Maintain the trailer's stability on the ground.
- Increase the cooling process with the chemical filter and get a bigger water tank.
- Re-cool the flue gas with the scrubber drainage water.
- Including additional methods to get the flue gas to the required temperature.
- Using fewer trash bags instead of 12 bags to avoid a dramatic spike in temperature.

4.2 Waste Incineration Ashes Recycling

4.2.1 Mechanical Realization[5]

Please put one overview photo

4.2.2 Requirements of Ashes recycling

System requirement

- The ashes recycling system shall be able to extract different metals from ashes.

Physical requirements

- In the column system, the rotator shall be able to mix the two phases well enough to transfer the metal ions from the aqueous phase to the organic phase.

- The mixer shall be able to dissolve all soluble in the ash.

Chemical requirements

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[5] https://www.aecenar.com/index.php/institutes/icpt/icpt-ashes-recycling/ashes-recycling-system-realization/ashes-recycling-mechanical-realization

- The column system shall be able to transfer the metallic salts from the solution to the solvent.

- The column material shall be able to withstand the corrosion with acid.

- In the column system, the rotator shall be able to mix the two phases well enough to transfer the metal ions from the aqueous phase to the organic phase.

- In the column system, the solvent used shall be able to extract a specific ion from the solution (e.g., LIX® 984N for copper ions).

- The mixer shall be able to dissolve all soluble in the ash into the acid.

- The pipes shall be able to withstand the corrosion with acid.

- The valves shall be able to withstand the corrosion with acid.

- The mixer shall be able to withstand the corrosion with acid.

Mechanical requirements

- The column shall be made of stainless.

- The mixer shall be rotate in 375 rpm in continuous and dispersed phase flow rate.

- The filter shall be able to avoid the solid particles to intel the column

- Time of soaking the ashes solution with acid shall be enough to dissolve all ashes in the acid solution.

- The material of pipes shall be made of PPR.

- The pumps shall be able to pump the liquids up to the column.

Automation requirements

- The sensors shall be able to control from the GUI.

- The valves shall be able to control from the GUI.

- The inflows and outflows of the fluids shall be able to harmonize with each other.

4.2.3 Ashes Recycling Solvents[6]

Recovery of valuable metals such as copper, nickel and zinc from synthetic sulfate solutions using LIX 984N as the extractant dissolved in kerosene was investigated. The metals get extracted at different equilibrium pH of the aqueous phase and thus the pH of the aqueous solutions forms the basis of their separation. Copper was extracted at an equilibrium pH of 3.5, while nickel and zinc get extracted at an equilibrium pH of 7.3 and 8.8 respectively. The extraction isotherms of copper, nickel and zinc were obtained by contacting the respective metal solutions and 0.05 M LIX 984N at different A:O phase ratio

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[6] Ref : https://www.aecenar.com/index.php/institutes/icpt/icpt-ashes-recycling/ashes-recycling-solvents

at their respective equilibrium pH of maximum extraction. A two-stage counter- current extraction simulation was done to achieve quantitative extraction of the metals. Furthermore, a two-stage counter-current stripping simulation yielded quantitative recovery of the individual metals, from their loaded organic phases. On the basis of the results, a flow sheet of the process has been developed.

Introduction

Chalcopyrites, pentaldite, spent catalyst, batteries, brass ash, zinc ash are some of the major sources for the production of copper, nickel and zinc1 . Apart from sulfides, significant resources for these metals are available as oxide/silicate deposits. These raw materials are subjected to various hydrometallurgical treatments which include atmospheric/pressure/bioleaching of sulfate/chloride solutions. The leach liquor, thus obtained contains copper, nickel, zinc along with some impurities such as iron, chromium, aluminium, silicon etc. Liquid-liquid extraction has been applied to many solutions to obtain either metals or their salts in highly pure form, as these metals are industrially and economically more important and therefore there is a great need to separate and recover them. Solvent extraction has been carried out extensively for the separation of cobalt and nickel with alkyl phosphorus reagents like Cyanex 272 and D2EHPA2,3. Similarly Chelating reagents like LIX 87QN, LIX 54, LIX 973, LIX 34, LIX 984,

LIX 984N and LIX 84I have been used for the extraction, separation and recovery of copper, nickel and zinc from ammonium carbonate / ammonium sulfate solutions4-10. Extraction of copper, nickel and zinc from their sulfate solutions, using LIX 84I was found to depend on the equilibrium pH of the aqueous phase11,12. It was found that LIX 984N was very selective for copper extraction. In this paper, we report the solvent extraction separation of copper, nickel and zinc from synthetic solution using LIX 984 N as the extractant dissolved in kerosene, with an aim to separate these metals efficiently by single stage extraction. On the basis of this, a flow sheet of the process has been developed.

Experimental

Copper, nickel and zinc solutions were prepared by dissolving their corresponding sulfate salts in double distilled water. The chelating extractant, LIX 984N, a 1:1 mixture of LIX 84 and LIX 860N (mixture of 5-nonylsalicylaldoxime and 2-hydroxy 5-nonylacetophenone oxime) was used as received from Cognis – Ireland. Deodourised kerosene (bp, 160-200 0 C) was used as a diluent. All other chemicals and reagents used were of analytical grade.

Extractions were carried out by equilibrating a suitable aliquot of the solution containing metal ions at the desired pH with an equal volume of LIX 984N diluted in kerosene for 20 minutes, in glass-stopper bottles using a mechanical shaker. After phase disengagement, the aqueous phase was separated and metal concentrations were analysed by using chemito AA 203 atomic absorption spectrophotometer. The pH of the metal solutions was adjusted by adding dilute sulfuric acid or sodium hydroxide and using equiptronic pH meter model EQ-611. Experimental data were obtained in triplicate and those achieving a 98% mass balance were only accepted. The metal concentration in the organic phase was determined by stripping the loaded organic phase with acid. All experiments were carried out at room temperature.

Results and Discussion

Effect of equilibrium pH

Extraction studies were carried out with individual metal solutions containing 0.0025 M copper, nickel and zinc sulfate solutions. The extraction of these metals with 0.005 M LIX 984N as a function of pH was studied. The extraction of these metals depends mainly on the equilibrium pH of the aqueous phase as represented in Figure 1. Copper shows quantitative extraction at equilibrium pH 3.5, while nickel and zinc get extracted quantitatively at pH 7.3 and 8.8, respectively. Based on the data pertaining to the extraction behaviour of these metals, a synthetic solution containing 0.150 g/L copper, 0.140 g/L nickel and 0.163 g/L zinc was prepared and used.

Recovery of copper

The extraction isotherm for copper was obtained by contacting the metal solution and 0.05 M LIX 984N at different A:O phase ratios at an equilibrium pH of 3.5. The McCabe-Thiele plot (Figure 2) shows that quantitative copper extraction is achievable in two stages at A:O phase ratio of 5.5:1. This was confirmed by a two-stage counter- current extraction simulation experiment carried out at an A:O phase ratio of 5.5:1. Aqueous and organic phases were separated after second cycle onwards and analysed for metal values. The raffinate was found to contain 0.001 g/L copper, 0.140 g/L nickel and 0.163 g/L Zinc. The loaded organic phase contained 0.820 g/L copper. A two-stage stripping was carried out at A:O ratio of 1 with 190 g/L of sulfuric acid to strip copper from the loaded organic phase and the stripping efficiency was > 99%.

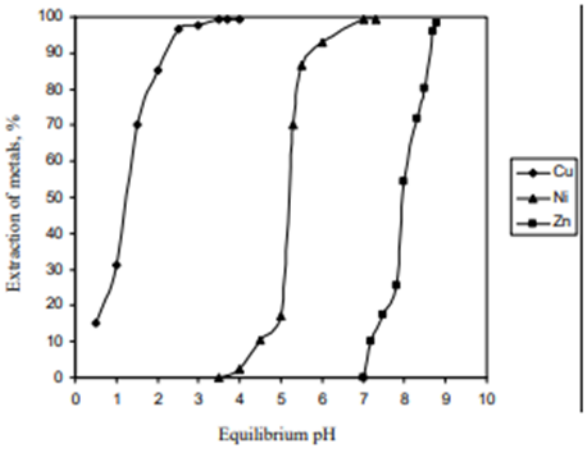


Figure 1. Influence of Equilibrium pH on the extraction of metals Aqueous phase: 0.0025 M each of Cu(II), Ni(II) and Zn(II). Organic phase:0.005 M LIX 984N. Contact time: 20 min. Temperature: Room temperature

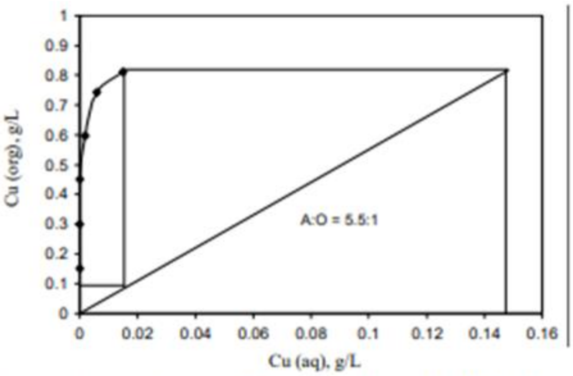


Figure 2. Extraction isotherm of copper. Aqueous phase: Cu(II): 0.150 g/L; Ni(II): 0.140 g/L; Zn(II): 0.163 g/L. Organic phase: 0.05 M LIX 984N. Equilibrium pH: 3.5 at room temperature.

Recovery of nickel

The raffinate after copper extraction was used for the separation of nickel. Extraction data for nickel was obtained by performing extraction at different A:O phase ratios at an equilibrium pH of 7. 3. Figure 3. Shows that quantitative nickel extraction is achievable in two stages at A:O phase ratio of 4:1. This was confirmed by a two-stage counter-current extraction simulation carried out at an A:O phase ratio of 4:1. The raffinate was found to contain 0.001g/L of nickel and 0.163 g/L of zinc and the loaded organic phase contained 0.500 g/L nickel. A two-stage stripping was carried out to strip nickel from the loaded organic phase, using 190 g/L of sulfuric acid at A:O phase ratio 1 and the stripping efficiency was > 99%.

Recovery of zinc

The raffinate after nickel extraction was used for the separation of Zinc. Extraction isotherm of zinc was obtained by performing extractions at different A:O phase ratios at an equilibrium pH of 8.8. A two-stage counter -current extraction simulation was carried out at an A:O phase ratio of 2:1 as obtained from McCabe-Thiele plot (Figure 4). The loaded organic phase was found to contain 0.310 g/L of zinc. A two-stage stripping was carried out to strip zinc from the loaded organic phase, using 150 g/L of sulfuric acid, at A:O ratio of 1:1 and the stripping efficiency was > 99% .

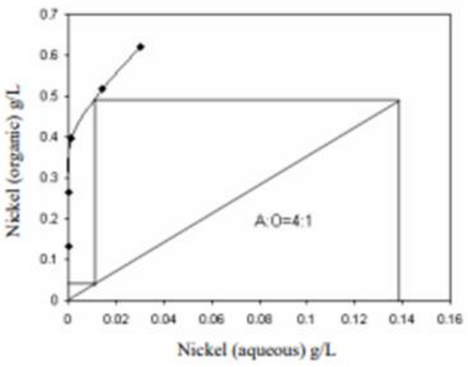


Figure 3. Extraction isotherm of nickel. Aqueous phase: Copper raffinate. Organic phase: 0.05 M LIX 984N. Equilibrium pH: 7.3. Contact time: 20 min. at ambient temperature

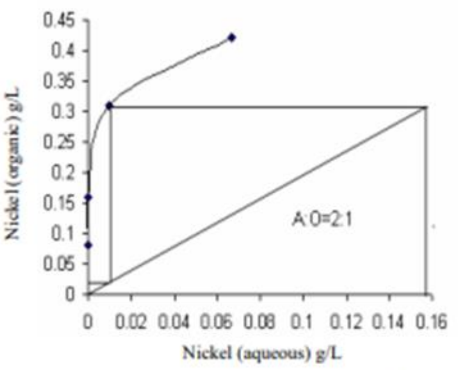


Figure 4. Extraction Isotherm of Zinc. Aqueous phase: Nickel raffinate. Organic phase: 0.05 M LIX 984N. Equilibrium pH=8.8. Contact time: 20 min

Flow sheet for the recovery of copper, nickel and zinc

Based on the extraction behaviour of these metals, a flow sheet for the separation of copper, nickel and zinc has been developed using LIX 984N and is shown in the Figure 5.

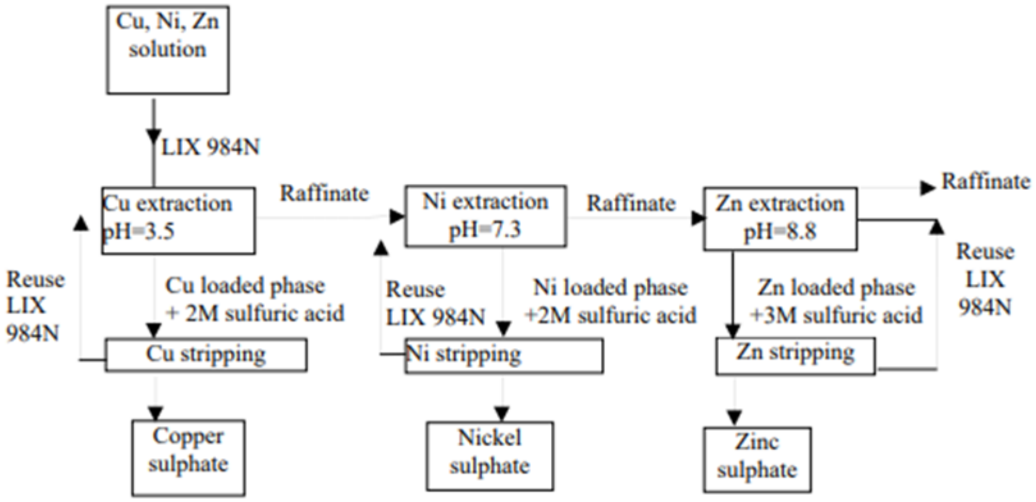


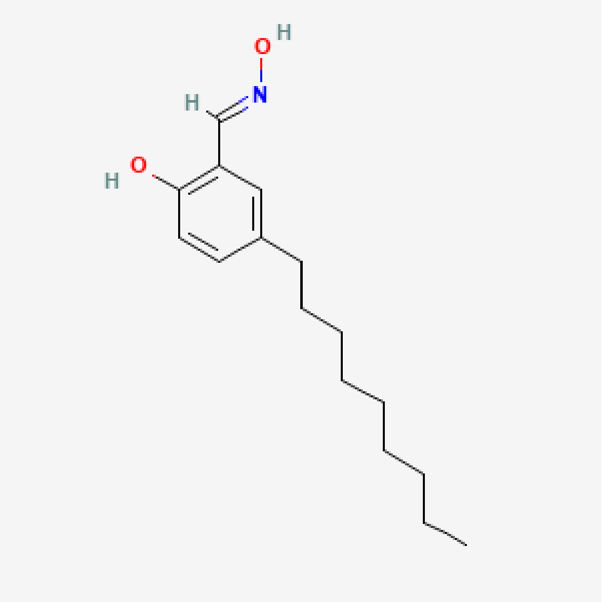
Figure 5. Flow -sheet for the recovery of copper, nickel and zinc from a leach liquor containing 0.150 g/L copper, 0.140 g/L nickel and 0.163 g/L zinc

Conclusion

LIX 984N was effectively used for the extractive separation of copper, nickel and zinc. Counter- current simulation extraction of these metals showed almost quantitative loading in two stages. The extraction of the metals depends on the equilibrium pH of the aqueous phase. Therefore, it was possible to separate the metals from one another based on their extraction behaviour at different equilibrium pH. Thus the recovery of these metals by counter-current stripping simulation was quantitatively accomplished.

https://www.researchgate.net/publication/258378620_Recovery_of_Copper_Nickel_and_Zinc_from_Sulfate_Solutions_by_Solvent_Extraction_Using_LIX_984N

Synthetic method of 5-nonyl salicylaldoxime



The invention discloses a synthetic method of 5-nonyl salicylaldoxime. The method comprises the following steps:

1. mixing 4-nonyl phenol and a formaldehyde solution, adding an alkali as a catalyst, and reacting for 2 hours under the thermal insulation condition
2. standing the solution for layering, taking an organic layer and adding chloroform and a Dess-Martin reagent for reaction for 1-3 hours
3. sequentially washing the solution obtained after reaction in the step b) at room temperature by using a saturated sodium carbonate aqueous solution, a sodium thiosulfate aqueous solution and deionized water and standing for layering, then, adding alkali as a catalyst into the organic layer, dropwise adding a hydroxyl amine salt solution, and reacting for 3 hours under the thermal insulation condition
4. adding sulfuric acid into the solution, standing for layering, washing the organic layer by using deionized water to be neutral, and removing the solvent to obtain 5-nonyl salicylaldoxime.

The method provided by the invention is simple in operation step, mild in reaction condition, easy to control and safe, convenient to operate and free of flammable and combustible exhaust gas. Based on 4-nonyl phenol, the yield of products can reach over 98% and the purity can reach over 95%.

Description

A kind of synthetic method of 5-nonyl salicyl aldooxime

Technical field

The invention belongs to chemical field, particularly a kind of synthetic method of copper extractant 5-nonyl salicyl aldooxime.

Background technology

Copper is a kind of important and have the non-ferrous metal of extensive industrial use, Present Domestic mainly adopts the technology of hydrometallurgy (leaching-Extraction electrodeposition) to produce copper outward, in this technique, extraction is the committed step realizing leach liquor metal separation, can say that the performance of extraction agent directly affects the efficiency of copper extraction, in the copper extractant developed at present, mainly be divided into oximes (ketoxime, aldoxime), beta-diketon class, ternary amine and composite class etc. are several, wherein, oximes extraction agent, particularly to have capacity large for aromatics aldoxime extractants (as 5-nonyl salicyl aldooxime), extraction ability is good, the characteristic that percentage extraction is high, it is the most widely used extraction agent in current hydrometallurgy Copper Processing Industry.

US Patent No. 5260487A discloses a kind of preparation method of 5-nonyl salicyl aldooxime, and the method take magnesium methylate as initiator, through salt-forming reaction, formylation reaction, the synthesis of oximation reaction three step, reaction process fierceness is wayward, and have a large amount of hydrogen to produce, have larger potential safety hazard, and solvent load is large, reclaim difficulty, long reaction time, temperature of reaction is high, and side reaction is many, yield (in 4-nonyl phenol) only has 86%, and content only has 85%;

Patent CN101326148A discloses the new catalyst mixture catalysis formylation reaction process of a kind of use, obtain higher yield 95%(in nonyl phenol), higher purity 95%, but the method adopts short distance distillation SPDU to distill out intermediate 5-nonyl salicylic aldehyde, higher to equipment requirements, requires also very high to temperature (160 ~ 240 DEG C) and vacuum tightness (1mmHg), energy consumption is very large, solvent loss is comparatively large, and cost is high, is unfavorable for large-scale commercial production;

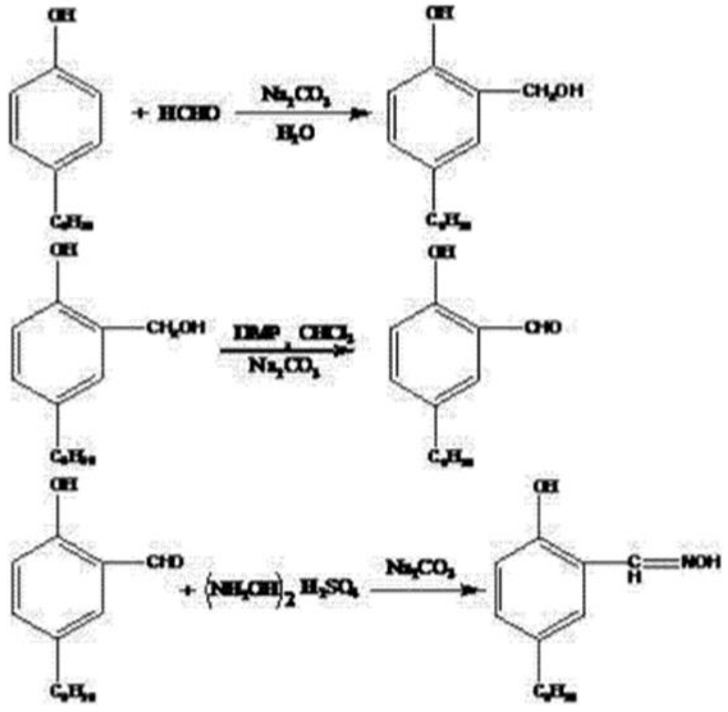
Patent CN101497576A also discloses a kind of preparation method of 5-nonyl salicyl aldooxime, and the method adopts and the technique similar with US Patent No. 5260487A, has identical shortcoming. Therefore, develop a kind of reaction temperature and, 5-nonyl salicyl aldooxime synthetic method that operational safety, feed stock conversion are high, be this area technical problem urgently to be resolved hurrily always.

Summary of the invention

For the problems referred to above, provide a kind of reaction temperature and, the synthetic method of the 5-nonyl salicyl aldooxime of operational safety, the method synthesis material transformation efficiency is high, and by product is few, and the present invention is achieved in that

A synthetic method for 5-nonyl salicyl aldooxime, step is as follows:

1. 4-nonyl phenol and formalin are dropped in reactor, add alkali as catalyzer, stir and be warming up to 50-70 DEG C, insulation reaction 2h; Wherein, add 4-nonyl phenol and formaldehyde mass ratio be 1:0.2-0.27;
2. by the solution left standstill layering that step a obtains, get organic layer and add chloroform, then add Dess-Martin reagent, at 25 ~ 30 DEG C, react 1 ~ 3h;
3. Wherein, Dess-Martin reagent is added and 4-nonyl phenol mass ratio is 0.77-0.81:1;
4. reacted for step b solution is at room temperature used successively stratification after saturated aqueous sodium carbonate, sodium thiosulfate solution, deionized water wash, alkali is added as catalyzer again in organic layer, hydroxylammonium salt solution is instilled, in 40 ~ 60 DEG C of insulation reaction 3h in 1h;
5. in step c solution, adding sulfuric acid, to be neutralized to pH value of solution be 6 ~ 7, and by solution left standstill layering, get organic layer with deionized water wash to neutral, finally distillation is except desolventizing, namely obtains 5-nonyl salicyl aldooxime.



Preferably, in the present invention, alkali described in step a is one or more in sodium carbonate, sodium hydroxide, sodium bicarbonate, sodium methylate, triethylamine, pyridine.

Preferably, in the present invention, alkali described in step a is sodium carbonate, add sodium carbonate quality be the 5-8% of 4-nonyl phenol quality.

Preferably, in the present invention, alkali described in step c is one or more in sodium carbonate, sodium hydroxide, sodium bicarbonate, sodium methylate, triethylamine, pyridine.

Preferably, in the present invention, alkali described in step c is sodium carbonate, add the 28.9-33.7% that sodium carbonate quality is 4-nonyl phenol quality.

Preferably, in the present invention, hydroxylammonium salt described in step c is oxammonium hydrochloride or oxammonium sulfate.

Preferably, in the present invention, hydroxylammonium salt described in step c is oxammonium sulfate, and the oxammonium sulfate added accounts for the 41-44.7% of 4-nonyl phenol quality.

Reaction principle of the present invention is, the first step, 4-nonyl phenol and formalin is reacted in the basic conditions and generates 2-methylol-4-nonylphenol; 2-methylol-4-nonylphenol Dess-Martin reagent oxidation is 5-nonyl salicylic aldehyde by second step; Finally, 5-nonyl salicylic aldehyde and hydroxylammonium salt generation oximation reaction are generated 5-nonyl salicyl aldooxime.

Using sodium carbonate as alkaline catalysts, oxammonium sulfate is example as hydroxylammonium salt, and reaction formula involved in the present invention is:

Operation steps of the present invention is simple, and replace metal (Pt etc.) conventional at present as oxygenant using Dess-Martin reagent, oxidation is directed high, reaction conditions is gentle, be easy to control, safe convenient operation, produces without inflammable and explosive waste gas, not high to equipment requirements, energy consumption is low, reaction efficiency is high, and feed stock conversion is high, and by product is few, can reach more than 98% in the product yield of 4-nonyl phenol, purity can reach more than 95%.

Embodiment

In order to make better elaboration to the present invention, make further detailed description by following examples to this, embodiment is not to Composition of contents restriction of the present invention.

Embodiment 1

The formalin 200g of 220g4-nonyl phenol and 30% mass concentration is dropped in 1L four-hole boiling flask, adds 18g sodium carbonate, stir and be warming up to 70 DEG C, stratification after insulation reaction 2h, discard water layer, organic layer to be proceeded in 1L four-hole boiling flask and to add 200ml chloroform, add 178g Dess-Martin reagent (production of Wei Meng Chemical Co., Ltd. of Weifang City) again at 25 DEG C of reaction 2h, react complete and at room temperature use 200g saturated aqueous sodium carbonate successively, 200g sodium thiosulfate solution, stratification after 200g deionized water wash, discard water layer, organic layer is proceeded to 1L four-hole boiling flask, add the aqueous sodium carbonate 300g of 25% mass concentration, 30% mass concentration hydroxylamine sulfate solution 328g is instilled in 1h, dropwise at 60 DEG C of insulation reaction 3h, the aqueous sulfuric acid 60g adding 20% mass concentration neutralizes (pH is 6-7), stratification, get organic layer 200g deionized water and be washed till neutrality, finally distillation is except desolventizing, obtain finished product 5-nonyl salicyl aldooxime 261g, in 4-nonyl phenol, yield 99.24%, purity 98.30%.

In specific operation process, the sodium carbonate in a kind of or several replacement embodiment in sodium hydroxide, sodium bicarbonate, sodium methylate, triethylamine, pyridine also can be used to use.

Embodiment 2

The formalin 150g of 220g4-nonyl phenol and 30% mass concentration is dropped in 1L four-hole boiling flask, add 10g sodium hydroxide, stir and be warming up to 50 DEG C, stratification after insulation reaction 2h, discard water layer, organic layer to be proceeded in 1L four-hole boiling flask and to add 200ml chloroform, add 170g Dess-Martin reagent (production of Wei Meng Chemical Co., Ltd. of Weifang City) at 30 DEG C of reaction 2h, react complete and at room temperature use 200g saturated aqueous sodium carbonate successively, 200g sodium thiosulfate solution, stratification after 200g deionized water wash, discard water layer, organic layer proceeds to 1L four-hole boiling flask, add the aqueous sodium hydroxide solution 175g of 25% mass concentration, 30% mass concentration hydroxylamine sulfate solution 300g is instilled in 1h, dropwise at 50 DEG C of insulation reaction 3h, the aqueous sulfuric acid 10g adding 20% mass concentration neutralizes (pH is 6-7), stratification, get organic layer 200g deionized water and be washed till neutrality, finally distillation is except desolventizing, obtain finished product 5-nonyl salicyl aldooxime 258g, in 4-nonyl phenol, the rate of recovery 98.10%, purity 95.66%.

Each embodiment is not limit the present invention above; during concrete enforcement; according to the basic general knowledge of this area; according to the disclosure of claim; can make the appropriate adjustments above-described embodiment content; if the sodium carbonate in a kind of or several replacement embodiment in sodium hydroxide, sodium bicarbonate, sodium methylate, triethylamine, pyridine is as required as catalyzer; make the appropriate adjustments according to the add-on of practical situation to catalyzer; and replace oxammonium sulfate to participate in reaction etc. as azanol with oxammonium hydrochloride, all belong to protection scope of the present invention.

1. a synthetic method for 5-nonyl salicyl aldooxime, is characterized in that, step is as follows:

1. A) by 4-nonyl phenol and formalin mixing, add alkali as catalyzer, stir and be warming up to 50-70 DEG C, insulation reaction 2h;

Wherein, add 4-nonyl phenol and formaldehyde mass ratio be 1:0.2-0.27;

1. B) by the solution left standstill layering that step a obtains, get organic layer and add chloroform, then add Dess-Martin reagent, at 25 ~ 30 DEG C, react 1 ~ 3h;

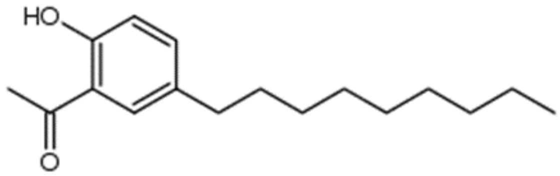
Wherein, Dess-Martin reagent is added and 4-nonyl phenol mass ratio is 0.77-0.81:1;

1. C) reacted for step b solution is at room temperature used successively stratification after saturated aqueous sodium carbonate, sodium thiosulfate solution, deionized water wash, alkali is added as catalyzer again in organic layer, hydroxylammonium salt solution is instilled, in 40 ~ 60 DEG C of insulation reaction 3h in 1h;
2. D) in step c solution, adding sulfuric acid, to be neutralized to pH value of solution be 6 ~ 7, and by solution left standstill layering, get organic layer with deionized water wash to neutral, finally distillation is except desolventizing, namely obtains 5-nonyl salicyl aldooxime.

2. the synthetic method of 5-nonyl salicyl aldooxime according to claim 1, it is characterized in that, alkali described in step a is one or more in sodium carbonate, sodium hydroxide, sodium bicarbonate, sodium methylate, triethylamine, pyridine.
3. the synthetic method of 5-nonyl salicyl aldooxime according to claim 2, it is characterized in that, alkali described in step a is sodium carbonate, add sodium carbonate quality be the 5-8% of 4-nonyl phenol quality.
4. the synthetic method of 5-nonyl salicyl aldooxime according to claim 1, it is characterized in that, alkali described in step c is one or more in sodium carbonate, sodium hydroxide, sodium bicarbonate, sodium methylate, triethylamine, pyridine.
5. the synthetic method of 5-nonyl salicyl aldooxime according to claim 4, it is characterized in that, alkali described in step c is sodium carbonate, add the 28.9-33.7% that sodium carbonate quality is 4-nonyl phenol quality.
6. according to the synthetic method of the arbitrary described 5-nonyl salicyl aldooxime of claim 1-5, it is characterized in that, hydroxylammonium salt described in step c is oxammonium hydrochloride or oxammonium sulfate.
7. the synthetic method of 5-nonyl salicyl aldooxime according to claim 6, it is characterized in that, hydroxylammonium salt described in step c is oxammonium sulfate, and the oxammonium sulfate added accounts for the 41-44.7% of 4-nonyl phenol quality.

source: https://patents.google.com/patent/CN104356025A/en

Synthesis process of 2-hydroxy-5-nonylacetophenone



Abstract

The invention discloses a synthesis process of 2-hydroxy-5-nonylacetophenone, which comprises adding 4-nonylphenol and anhydrous acetonitrile into an organic solvent, introducing dry hydrogen chloride, adding a catalyst at the same time to perform a reaction for forming nonylphenol ketimine, filtering off nonylphenol ketimine, adding hot water, refluxing to obtain 2-hydroxy-5-nonylacetophenone, extracting the 2-hydroxy-5-nonylacetophenone by using an organic solvent, distilling under reduced pressure to obtain high-purity 2-hydroxy-5-nonylacetophenone. In the process, the synthesis route is short, the 2-hydroxy-5-nonylacetophenone is formed by a one-step reaction instead of the original two-step reaction, the synthesis route is simplified, and the reaction time is short; and the total yield of the process is higher than that of the prior art, the total yield of the synthesis route is over 80 percent, and the purity of the obtained product is more than or equal to 85 percent.

Description

The synthesis technique of 2-hydroxyl-5-nonylacetophenone

Technical field

The invention belongs to chemical synthesis process, specifically, relate to the technology of a kind of chemosynthesis 2-hydroxyl-5-nonylacetophenone.

Background technology

2-hydroxyl-5-nonylacetophenone, English name: 2-hydroxy-5-nonylacetophenone(HNA), 2-hydroxyl-5-nonylacetophenone prepares metallic copper extraction agent 2-hydroxyl-5-nonylacetophenone oxime (HNAO) by oximation reaction, HNAO is a kind of efficient copper extractant, in hydrometallurgy, have widely and use, mainly from waste printed circuit board, copper mine and electroplating effluent, reclaim copper.

2-hydroxyl-5-nonylacetophenone has synthetic route now:

2-hydroxyl-5-nonylacetophenone synthesis technique is mainly at present:

1, is raw material with the 4-nonyl phenol, in the presence of aluminum trichloride (anhydrous), generates 2-hydroxyl-5-nonylacetophenone with excess acetyl chloride.

2, be raw material with the 4-nonyl phenol, generate magnesium salts, generate 2-hydroxyl-5-nonylacetophenone with the paraldehyde reaction again with the magnesium methylate reaction.

3, be raw material with the 4-nonyl phenol, generate ester with excess acetyl chloride earlier, in the presence of aluminum chloride, carry out Fries then and reset, can generate 2-hydroxyl-5-nonylacetophenone.

Article one, the route reaction by product is more, the bad control of reaction conditions, and yield is lower, and there is big difficulty in the purification aspect; The second route generates gel easily in reaction process, bad processing is bigger to the reaction influence, is not easy to suitability for industrialized production; Article three, use Acetyl Chloride 98Min. when route needs, production operation is relatively more dangerous, generates some by products during rearrangement easily, and difficulty is more during purification.

Summary of the invention

For solving above technical problem, the object of the present invention is to provide a kind of to need single step reaction, and the synthesis technique of 2-hydroxyl-5-nonylacetophenone of being improved significantly of yield.

The present invention seeks to realize like this:

4-nonyl phenol and excessive anhydrous acetonitrile are joined in the organic solvent, feeding that the exsiccant hydrogen chloride gas adds simultaneously is that one or more of aluminum chloride, boron trichloride, zinc chloride generate the nonyl phenol ketoimine as catalyst reaction, filter out that the nonyl phenol ketoimine adds hot water, refluxing obtains 2-hydroxyl-5-nonylacetophenone, 2-hydroxyl-5-nonylacetophenone is by organic solvent extraction, organic molten highly purified 2-hydroxyl-5 nonylacetophenone that promptly gets of pressure reducing and steaming.

Reaction scheme is as follows:

Specifically carry out as follows:

- organic solvent is added in the exsiccant container, add 4-nonyl phenol and anhydrous acetonitrile then, add catalyzer again, feed the exsiccant hydrogen chloride gas, described catalyzer is aluminum chloride, boron trichloride, zinc chloride or one or more;
- step (1) reaction solution is heated to 30～100 ℃, reacted 5～24 hours, reaction solution is cooled to 0～10 ℃ then, separates out the nonyl phenol ketoimine, filters, and uses organic solvent washing;
- with the solid transfer after step (2) washing to container, add hot water, reflux, filter, use activated carbon decolorizing;
- step (3) filtrate is added in the entry, use water-fast organic solvent extraction, the pressure reducing and steaming organic solvent promptly gets 2-hydroxyl-5 nonylacetophenone.

Organic solvent is one or more in ether, tetrahydrofuran (THF), toluene, the chlorobenzene in the above-mentioned steps (1).

Organic solvent is one or more in toluene, trichloromethane, the methylene dichloride in the above-mentioned steps (4).

Above-mentioned technology filters out optimised process by orthogonal test, and proves through replication experiment, and the total recovery of Synthetic 2-hydroxyl-5 nonylacetophenone is stable, all reaches more than 80%, and product purity detects by HPLC, all reaches more than 85%.

Beneficial effect: (1) technology synthetic thread of the present invention is short out, and the present invention has simplified synthetic route with the synthetic step preparation of original two-step reaction 2-hydroxyl-5-nonylacetophenone, and the reaction times shortens.

(2) technology total recovery of the present invention is higher than prior art, and synthetic route total recovery of the present invention reaches more than 80%, the product purity that obtains 〉=85%.

Embodiment

Embodiment 1

In exsiccant 250ml round-bottomed flask, add 4-nonyl phenol 35.2g (0.16mol), 13g (0.32mol) anhydrous acetonitrile, 80ml anhydrous tetrahydro furan, the 4g Zinc Chloride Anhydrous feeds the exsiccant hydrogen chloride gas then, is warming up to 40 ℃ of about 4h of reaction, being cooled to 0 ℃ then and leaving standstill 3h, separate out the safran precipitation, promptly is the nonyl phenol ketoimine, filter, filter cake washs with tetrahydrofuran (THF), filter cake is changed in the there-necked flask of 1L, adds 0.6L hot water, backflow 1h, system becomes yellow solution, and cooling is filtered, filtrate is used decolorizing with activated carbon, product extracts with toluene, and underpressure distillation goes out solvent, gets product 2-hydroxyl-5-nonylacetophenone, yield 82%, purity 87%.

Embodiment 2

In exsiccant 250ml round-bottomed flask, add 4-nonyl phenol 0.2mol, 0.3mol anhydrous acetonitrile, 100ml ether, the 6g aluminum chloride feeds the exsiccant hydrogen chloride gas then, is warming up to 30 ℃ of about 24h of reaction, being cooled to 10 ℃ then and leaving standstill, separate out the safran precipitation, promptly is the nonyl phenol ketoimine, filter, filter cake washs with ether, filter cake is changed in the there-necked flask of 1L, adds 0.7L hot water, backflow 1h, system becomes yellow solution, and cooling is filtered, filtrate is used decolorizing with activated carbon, product extracts with toluene, and underpressure distillation goes out solvent, gets product 2-hydroxyl-5-nonylacetophenone, yield 83%, purity 86%.

Embodiment 3

In exsiccant 250ml round-bottomed flask, add 4-nonyl phenol 0.25mol, 0.32mol anhydrous acetonitrile, the mixed organic solvents of 120ml dry toluene and chlorobenzene, the mixture of 12g aluminum chloride and boron trichloride feeds the exsiccant hydrogen chloride gas then, be warming up to 100 ℃ of about 5h of reaction, being cooled to 5 ℃ then and leaving standstill, separate out the safran precipitation, promptly is the nonyl phenol ketoimine, filter, the filter cake toluene wash changes filter cake in the there-necked flask of 1L, adds 0.7L hot water, backflow 1h, system becomes yellow solution, and cooling is filtered, filtrate is used decolorizing with activated carbon, the product dichloromethane extraction, underpressure distillation goes out solvent, gets product 2-hydroxyl-5-nonylacetophenone, yield 84%, purity 87%.

https://patents.google.com/patent/CN102206148A/en

Chemical substance	Quantity	Availability	Price
Formaldehyde	1Kg	NO	
4-nonyl phenol	1Kg	NO	
Sodium carbonate	1Kg	**YES**	
Chloroform		NO	
Dess-Martin reagent	1Kg	NO We can get it from sigma aldrich	
Sodium thiosulfate	1Kg	NO	
Hydroxylamine sulfate	1Kg	NO	
Sulfuric acid		**YES**	
Distilled water			

4.2.4 synthesis of extractants[7]

purpose: synthesis of LIX 984N

LIX 984N is a 1:1 vol mixture of 2-hydroxy-5-nonylacetophenone ketoxime (LIX 84-I) and 5-nonylsalicylaldoxime (LIX 860-I)

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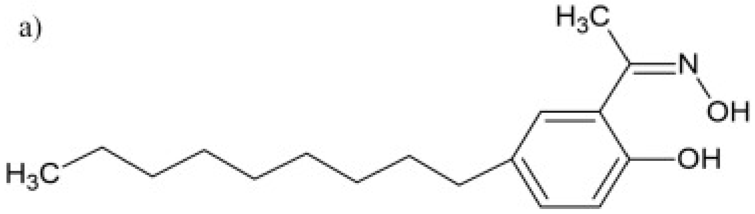
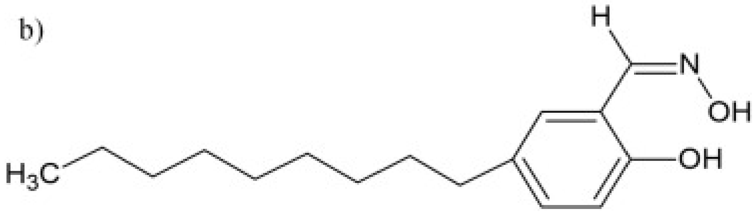
[7] Ref : https://www.aecenar.com/index.php/institutes/icpt/icpt-ashes-recycling/synthesis-of-extractants

a)



b)



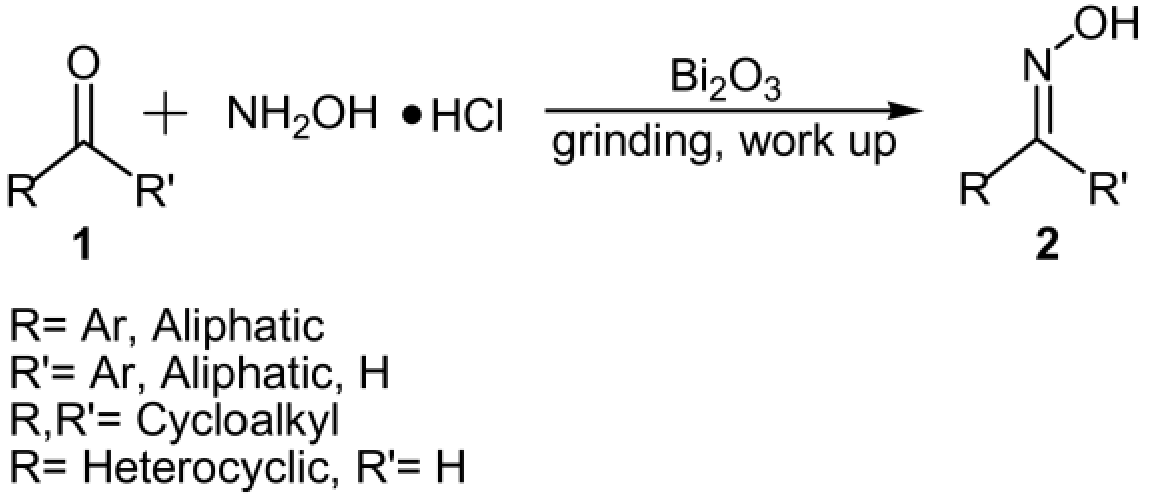
1. Represents the chemical structure of LIX 84-I
2. Represents the chemical structure of LIX 860-I

A- Preparation of oxime:

Classically, oximes are prepared [2] by refluxing an alcoholic solution of a carbonyl compound with hydroxylamine hydrochloride and pyridine. The method has multiple drawbacks such as low yields, long reaction time, toxicity of pyridine, and effluent pollution caused by the use of organic solvent.

However,

Carbonyl compounds (aliphatic, heterocyclic, and aromatic) were converted into the corresponding oximes in excellent yields by simply grinding the reactants at room temperature without using any solvent in the presence of Bi_2O_3. Most importantly, this method minimizes waste disposal problems, provides a simple yet efficient example of unconventional methodology and requires short time.

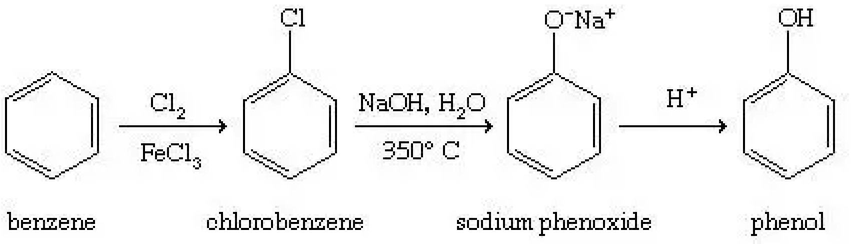


Typical procedure for the formation of oxime 2

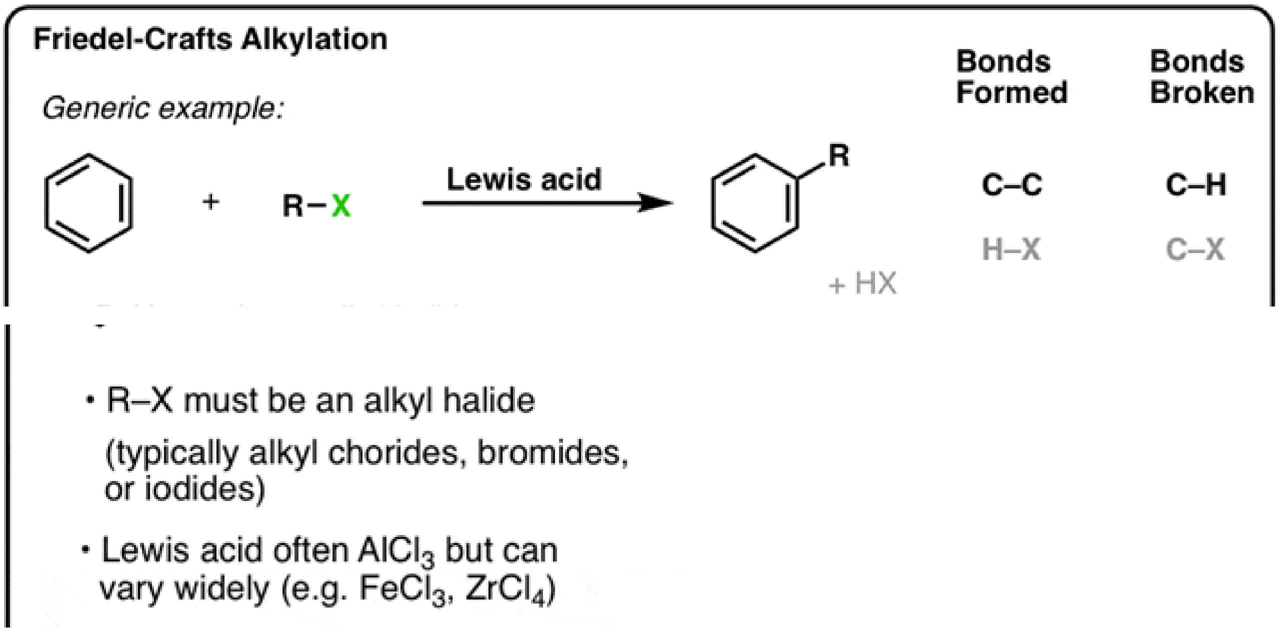
A mixture of aldehyde/ketone **1** (1 mmol), hydroxylamine hydrochloride (1.2 mmol), and Bi_2O_3 (0.6 mmol) was grounded in a mortar with a pestle for the required period of time. On completion of the reaction as monitored by TLC, ethyl acetate (2 × 10 mL) was added to the reaction mixture and filtered to separate the Bi_2O_3. The filtrate was concentrated down to approx. 6 mL, then added water to it when product precipitated out from the solution. The precipitate was filtered out and dried in high vacuum to furnish the pure oxime **2** in 60-98% yield.

B- substitution of the groups on benzene:

1- phenol preparation:



2- Friedel craft alkylation:



- R–X must be an alkyl halide (typically alkyl chorides, bromides, or iodides)
- Lewis acid often $AlCl_3$ but can vary widely (e.g. $FeCl_3$, $ZrCl_4$)

Synthesis process of 2-hydroxy-5-nonylacetophenone

embodiment 1:

In exsiccant 250ml round-bottomed flask, add 4-nonyl phenol 35.2g (0.16mol), 13g (0.32mol) anhydrous acetonitrile, 80ml anhydrous tetrahydro furan, the 4g Zinc Chloride Anhydrous feeds the exsiccant hydrogen chloride gas then, is warming up to 40 ℃ of about 4h of reaction, being cooled to 0 ℃ then and leaving standstill 3h, separate out the safran precipitation, promptly is the nonyl phenol ketoimine, filter, filter cake washs with tetrahydrofuran (THF), filter cake is changed in the there-necked flask of 1L, adds 0.6L hot water, backflow 1h, system becomes yellow solution, and cooling is filtered, filtrate is used decolorizing with activated carbon, product extracts with toluene, and underpressure distillation goes out solvent, gets product 2-hydroxyl-5-nonylacetophenone, yield 82%, purity 87%.

Compound	Quantity (moles)	Quantity (grams)	Quantity (ml)	CAS no	Cost (average)	Cost quantity	Notes

4-nonyl phenol	0.16	35.2		68081-86-7			
anhydrous acetonitrile	0.32	13		75-05-8			
anhydrous tetrahydro furan			80	109-99-9	21.9 $	250 ml	
Zinc Chloride Anhydrous		4		7646-85-7	27 $	25 G	
tetrahydrofuran (THF)				109-99-9	21.9 $	250 ml	Filtration(washing)
activated carbon							decolorizing
toluene				108-88-3			Product extraction

embodiment 2:

In exsiccant 250ml round-bottomed flask, add 4-nonyl phenol 0.2mol, 0.3mol anhydrous acetonitrile, 100ml ether, the 6g aluminum chloride feeds the exsiccant hydrogen chloride gas then, is warming up to 30 ℃ of about 24h of reaction, being cooled to 10 ℃ then and leaving standstill, separate out the safran precipitation, promptly is the nonyl phenol ketoimine, filter, filter cake washs with ether, filter cake is changed in the there-necked flask of 1L, adds 0.7L hot water, backflow 1h, system becomes yellow solution, and cooling is filtered, filtrate is used decolorizing with activated carbon, product extracts with toluene, and underpressure distillation goes out solvent, gets product 2-hydroxyl-5-nonylacetophenone, yield 83%, purity 86%.

Compound	Quantity (moles)	Quantity (grams)	Quantity (ml)	CAS no	Cost (average)	Cost quantity	Notes
4-nonyl phenol	0.2			68081-86-7			
anhydrous acetonitrile	0.3			75-05-8			

ether			100	60-29-7 (diethylether)			
aluminum chloride		6		7446-70-0	28$	100 g	
hydrogen chloride				7647-01-0			Gaz
ether				60-29-7 (diethylether)			Washing
activated carbon							Decolorizing
toluene				108-88-3			extraction

embodiment 3:

In exsiccant 250ml round-bottomed flask, add 4-nonyl phenol 0.25mol, 0.32mol anhydrous acetonitrile, the mixed organic solvents of 120ml dry toluene and chlorobenzene, the mixture of 12g aluminum chloride and boron trichloride feeds the exsiccant hydrogen chloride gas then, be warming up to 100 ℃ of about 5h of reaction, being cooled to 5 ℃ then and leaving standstill, separate out the safran precipitation, promptly is the nonyl phenol ketoimine, filter, the filter cake toluene wash changes filter cake in the there-necked flask of 1L, adds 0.7L hot water, backflow 1h, system becomes yellow solution, and cooling is filtered, filtrate is used decolorizing with activated carbon, the product dichloromethane extraction, underpressure distillation goes out solvent, gets product 2-hydroxyl-5-nonylacetophenone, yield 84%, purity 87%.

Compound	Quantity (grams)	Quantity (moles)	Quantity (ml)	CAS no	Cost (average)	Cost quantity	Notes
4-nonyl phenol		0.25		68081-86-7			
anhydrous acetonitrile		0.32		75-05-8			
Dry toluene +			120	Toluene: 108-88-3	Toluene:	Toluene:	Organic solvents

chlorobenzene		Chlorobenzene: 108-90-7	Chlorobenzene: 57.2 $ / 16 $ (resp.)	Chlorobenzene: 1 L / 500 g (resp.)	
aluminum chloride + boron trichloride mixture	12	Aluminum chloride: 7446-70-0	Aluminum chloride: 28$	100 g	
		Boron trichloride: 10294-34-5	Boron trichloride: 60$	100 ml	
hydrogen chloride		7647-01-0			gas
activated carbon					For decolorizing
toluene		108-88-3			Filtration (washing)

Preparation of 5-nonylsalicylaldoxime

The formalin 200g of 220g4-nonyl phenol and 30% mass concentration is dropped in 1L four-hole boiling flask, adds 18g sodium carbonate, stir and be warming up to 70 DEG C, stratification after insulation reaction 2h, discard water layer, organic layer to be proceeded in 1L four-hole boiling flask and to add 200ml chloroform, add 178g Dess-Martin reagent (production of Wei Meng Chemical Co., Ltd. of Weifang City) again at 25 DEG C of reaction 2h, react complete and at room temperature use 200g saturated aqueous sodium carbonate successively, 200g sodium thiosulfate solution, stratification after 200g deionized water wash, discard water layer, organic layer is proceeded to 1L four-hole boiling flask, add the aqueous sodium carbonate 300g of 25% mass concentration, 30% mass concentration hydroxylamine sulfate solution 328g is instilled in 1h, dropwise at 60 DEG C of insulation reaction 3h, the aqueous sulfuric acid 60g adding 20% mass concentration neutralizes (pH is 6-7), stratification, get organic layer 200g deionized water and be washed till neutrality, finally distillation is except desolventizing, obtain finished product 5-nonyl salicyl aldooxime 261g, in 4-nonyl phenol, yield 99.24%, purity 98.30%.

In specific operation process, the sodium carbonate in a kind of or several replacement embodiment in sodium hydroxide, sodium bicarbonate, sodium methylate, triethylamine, pyridine also can be used to use.

compound	Quantity (moles)	Quantity (grams)	Quantity (ml)	[Mass] %	CAS no	cost	Cost quantity	Company's name	Notes
formalin		200			50-00-0	27.7$	25 ml	Sigma-aldrich	
4-nonylphenol		220			104-40-5	49.8$	1g	Alfa aesar	
Sodium carbonate		18			497-19-8	14$	1 kg	Chem-impex international	Saturated aqueous/aqueous
chloroform			200		67-66-3				
Dess-martin reagent		178			87413-09-0	10 $	1 G	Chem-impex international	1,1,1-triacetoxy-1,1-dihydro-1,2-benziodoxol-3(1H)-one, 1
Sodium carbonate		200 + 300(25 %)			497-19-8	14 $	1 kg	Chem-impex	
sodium thiosulfate		200			7772-98-7	35$	1 kg	Chem-impex international (united states)	
hydroxylamine sulfate		328		30	10039-54-0	25,9$	1 kg	Chem-impex international, inc (united states)	solution

sulfuric acid	60	20	7664 -93-9	aqueous

Solvent extractant preparation

A- Synthesis process of 2-hydroxy-5-nonylacetophenone

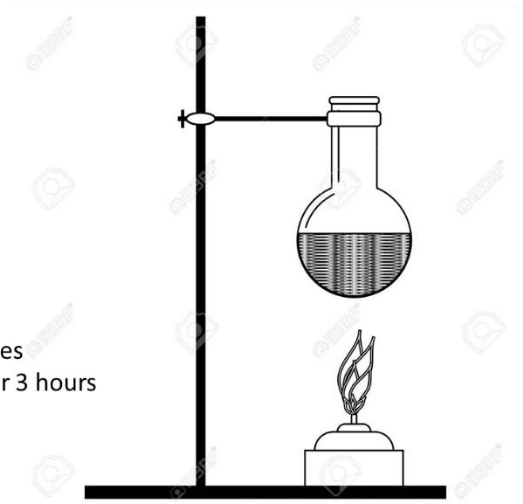
Reation method 1:
step 1:

4-nonyl phenol + anhydrous acetonitrile+
anhydrous tetrahydro furan + Zinc
Chloride Anhydrous (catalyst)(we may
also use aluminum chloride,
boron trichloride) this catalyst is going
To feed the system with HCl gaz

Reation method 1:

Step 2:
Warming up the set-up
Temperature : 40 degrees Celsius
Duration: 4 hours

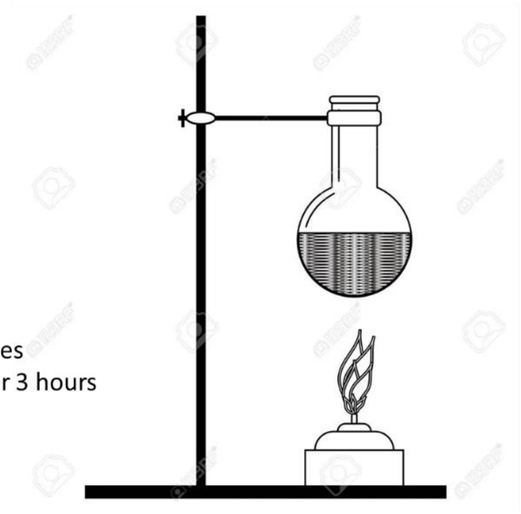
Step 3:
We cool the set-up down at 0 degrees
And we leave it standstill for another 3 hours



Reation method 1:

Step 2:
Warming up the set-up
Temperature : 40 degrees Celsius
Duration: 4 hours

Step 3:
We cool the set-up down at 0 degrees
And we leave it standstill for another 3 hours



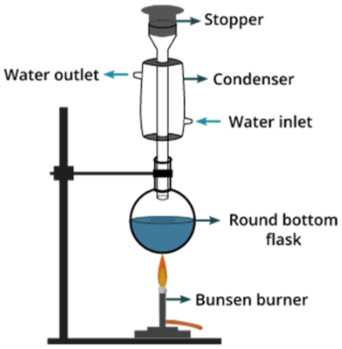
Reation method 1:

Step 5:

Reflux

Add the filtrate
Add 0.6 L of hot water
Reflux for 1 hour

➡ We should get a yellow solution



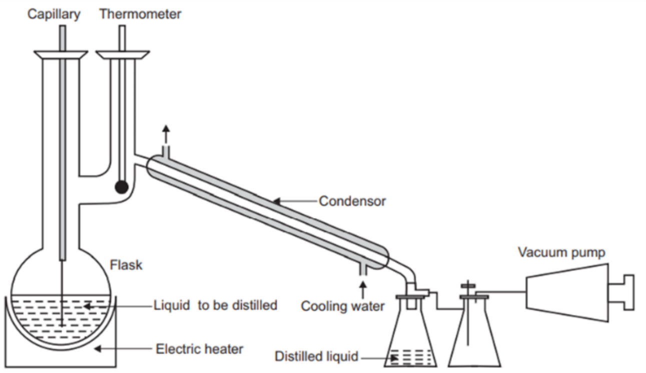
Heating under Reflux

Step 6:
Cool down
Filter:
using activated Carbon for decolorising
washing agent used is toluene
Step 7: underpressure distillation to remove the solvent

Reation method 1:

Step 7: underpressure distillation to remove the solvent

yield 82%, purity 87%.



B- Preparation of 5-nonylsalicylaldoxime

Step 1 : add the reagents
Step 2 : stirring + boiling at 70 degrees
Step 3 : insulation then stratification
Duration of step 3: 2 hours
Separation of watery content from organic content
Step 4: Take the organic layer and add it to a boiling round bottomed flask also with 4 holes
Step 5: add chloroform
Step 6: at 25 degrees add Dess-Martin reagent
Let the reaction proceed for 2 hours
Step 7: add also at room temperature saturated aqueous sodium carbonate + sodium thiosulfate
Step 8: wash with deionized water
Step 9: stratification into watery and organic layers

Step 10: take the organic layer, proceed in a 4 holes boiling round bottom flask

Step 11: add the aqueous sodium carbonate

Step 12: add dropwise hydroxylamine sulfate solution (which occur for 1 hour).

Step 13: at a 60 degrees for 3 hours reaction, add dropwise the aqueous sulfuric acid (neutralization of ph)

Step 14: stratification

Step 15: wash the organic layer with ionized water.

Step 16: distillation

	Reaction method 1	Reaction method 2	Reaction method 3
total cost (price of each reagent*quantity needed)			
heating	40(4 hours) Cooling at 0 degrees (3 hours) Backflow (1h)	30 degrees(24 hours) Cooling to 10 Backflow 1 hour	100 degrees(5 hours) Cooling into 5 degrees Backflow 1 hour

4.2.5 The cost and of the different extractents[8]

Chamical campounds	CAS No	costs	provider
Cyanex 272	83411-71-6	250$/1kg	alibaba
Cyanex 301	107667-02-7	≈900$/5g	A2B Chem
		/	Jinhua huayi chemical co. LDT
		/	Merck
Cyanex302	132767-86-3	100 ML/61,80€	Merck
		100ML/55$	Toronto Rescarch chemical

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[8] Ref : https://www.aecenar.com/index.php/institutes/icpt/icpt-ashes-recycling/the-cost-and-of-the-different-extractents

5 Energy Storage: Electrolyser, Ammonia Production, Fuel Cell

5.1 Electrolyze System Requirements

System requirement

- The system shall be able to produce hydrogen and oxygen, separately.

Physical requirements

- The electrodes shall be able to withstand the electrolysis temperature.

- The pipe system shall be able to withstand a temperature up to 100°C.

- The valves shall be able to resist the temperature and the pressure.

- The condensers shall be able to condense the vapor leaving the cell, with water.

Chemical requirements

- The electrodes shall be able to withstand the corrosion with KOH.

- The membrane shall be able to allows the ions to pass through so that electrolysis takes place when the current is connected.

- The membrane shall be able to insulate the two half-cells, chemically.

- The membrane shall be made of Nafion.

- The pipe system used shall be able to withstand the corrosion with KOH.

- The valves shall be able to withstand the corrosion with KOH.

- The metal of condenser used shall be able to withstand the corrosion with KOH.

Mechanical requirements

- The electrodes shall be thick enough to withstand the pressing (the pressing of the screw rods).

- The electrodes shall be thick enough to press the gaskets so that no gas can exit.

- The gaskets shall be able to prevent the leakage of gases and liquids from the cells.

- The gaskets shall be made of thermal caoutchouc.

- The pipes connections shall be able to resist the gas pressure without let gas exit through.

- The pressure of the pump shall be sufficient to fill the cells and not too high for the pipes system.

- The valves shall be able to close completely.

- The valves shall be able to open or close with independent pressure.

Electrical requirements

- For power supply, DC the current that pass through one cell shall be about 140 A DC and we have to test how much voltage shall be needed to make that.

- The power supply shall be able to let the electrolyze generates enough hydrogen so that we can burn it.

- The valves shall be able to be controlled from the GUI.

Safety requirement

- The hydrogen burner shall be able to burn the produced hydrogen gas to avoid the risk of its explosion.

5.2 Electrolyser System Test Specification

WE_Automation_Process

Step 1: Camera connections

1- Connect the camera Adapter

2- Connect the camera with PC

On PC, open “Internet explorer”, then enter the IP address “192.168.1.64“(written on the camera box), password: a1111111

Step 2: PLC

1- Connect wire of PLC.

2- Turn UP (Turn On) the PLC breaker.

GUI_Operation

1- Open file named “***Electrolysis GUI***” placed on the desktop of “*Mediston Laptop”.*

2- Through the GUI, we enter the solution.

3- When all valves are closed, turn on the power supply until burner is on.

If we find the voltage is up to 16V, the voltage must be reduced. *(Working on updates)*

5.2.1 System_test_cases

00001: The lack shall be detected when it exists

Step	Step Description	Expected Result
Precondition	System is off	
Switch ON the system	Turn on the air compressor manually	Air enters to the whole system
Lack is detected	Air exit from the pie system	Lack position shall be detected with a marker
Switch OFF the system	Turn off the air compressor manually	The air stops enter to the pipe system
Postcondition	System is OFF	

00002: WHOLE SYSTEM TEST

Step	Step Description	Expected Result
Precondition	System is off	
Open the valves V_1 and V_2	Open the valves V_1 and V_2 from the GUI	The valve V_1 and V_2 are open and enable to let the nitrogen gas pass

WASH THE SYSTEM WITH NITROGEN	Wash the system with Nitrogen for few minutes BY TURNING ON THE GLOBLE VALVE on the nitrogen tank	One can see that there is nitrogen exit from the gas outlets
Switch ON the system	Turn Off the globle nitrogen valve Turn Up (Turn On) the breaker (behind the Kelvinator refrigerator) Turn Up (Turn On) the two breakers (red & blue breakers in PLC box) Turn ON the system from the GUI	THE SYSTEM IS GENERATING hydrogen and oxygen
Burn the hydrogen	Turn On the transformator	The Hydrogen is burning
Switch Off the system	Switch Off the system from the GUI Switch Off the transformator	The system goes down
WASH THE SYSTEM WITH NITROGEN	Wash the system with Nitrogen for few minutes BY TURNING ON THE VALVE on the nitrogen tank	All the hydrogen existing in the pipe system exit
Postcondition	Turn off the system	System is off

00003: KOH pipe system test

The lack have to be marked with a marker and fixed

Step	**Step Description**	**Expected Result**
Precondition	System is off (No LIN signal)	

Switch on the KOH system	Open the KOH valves Turn on the KOH pump	The water passes through the KOH pump to the cell
Lack is detected	Look at the KOH pump system and look if there is exiting of water from the system	There is entering of water from the pipe system
Switch off the system.	Switch off the the pump and then the 2 KOH vlves	The system goes down.
Postcondition	System is off	

00004: WHOLE SYSTEM TEST WITH ANOTHER POWER SUPPLY

Step	**Step Description**	**Expected Result**
Precondition	System is off	
THE CHANGING F THE POWER SUPPLY WIT A POWER SUPPLY OF HIGHER VOLTAGE	Replacing the power supply with the welding machine	More hydrogen is generated
Open the valves V_1 and V_2	**Open the valves V_1 and V_2 from the GUI**	The valve V_1 and V_2 are open and enable to let the nitrogen gas pass
WASH THE SYSTEM WITH NITROGENE	Wash the system with Nitrogen for few minutes BY TURNING ON THE VALVE on the nitrogen tank	One can see that there is nitrogen exit from the gas outlets
Switch on the system	Turn off the nitrogen Turn on the system from the GUI	THE SYSTEM IS GENERATING hydrogen and oxygen
Burn the hydrogen	Turn on the transformator	The Hydrogen is burning

Switch off the system.	Switch off the system from the GUI Switch off the transformator	The system goes down.
WASH THE SYSTEM WITH NITROGENE	Wash the system with Nitrogen for few minutes BY TURNING ON THE VALVE on the nitrogen tank	All the hydrogen existing in the pipe system exit
Postcondition	Turn off the system	System is off

00005: WHOLE SYSTEM TEST WITH ONLY ONE CELL CONNECTED

Step	**Step Description**	**Expected Result**
Precondition	System is off	
CONNECTING THE COMPLETE VOLTAGE TO ONLY ONE CELL	DISCONNECT THE POWER SUPPLY FROM TWO CELLS AND CONNECT IT TO ONLY ONE CELL.	MORE CURRENT WILL DRIVE THROUGH THE CELL
Open the valves V_1 and V_2	**Open the valves V_1 and V_2 from the GUI**	The valve V_1 and V_2 are open and enable to let the nitrogen gas pass
WASH THE SYSTEM WITH NITROGEN	Wash the system with Nitrogen for few minutes BY TURNING ON THE VALVE on the nitrogen tank	One can see that there is nitrogen exit from the gas outlets
Switch on the system	Turn off the nitrogen Turn on the system from the GUI	THE SYSTEM IS GENERATING hydrogen and oxygen
Burn the hydrogen	Turn on the transformator	The Hydrogen is burning
Switch off the system	Switch off the system from the GUI Switch off the transformator	The system goes down.
WASH THE SYSTEM WITH NITROGEN	Wash the system with Nitrogen for few minutes BY TURNING ON THE VALVE on the nitrogen tank	All the hydrogen existing in the pipe system exit

Postcondition	Turn off the system	System is off

00006: WHOLE SYSTEM TEST after increasing of the KOH concentration

Step	**Step Description**	**Expected Result**
Precondition	System is off	
Emptying the cells	Placing a container under the two emptying valves and open the two emptying valves so that the containers are filled with the solution of the cells.	The solution flows into the containers.
Closing of the emptying valves.	closing the emptying valves after the whole solution flowed from the cells into the containers.	The emptying valves are closed
Increase the KOH concentration.	For the solution from the cells into the KOH tank and increase the KOH concentration, by adding new KOH.	The KOH concentration increase
Open the valves V_1 and V_2	Open the valves V_1 and V_2 from the GUI	The valve V_1 and V_2 are open and enable to let the nitrogen gas pass
WASH THE SYSTEM WITH NITROGEN	Wash the system with Nitrogen for few minutes BY TURNING ON THE VALVE on the nitrogen tank	One can see that there is nitrogen exit from the gas outlets
Switch on the system	Turn off the nitrogen Turn on the system from the GUI	THE SYSTEM IS GENERATING hydrogen and oxygen
Burn the hydrogen	Turn on the transformator	The Hydrogen is burning
Switch off the system.	Switch off the system from the GUI Switch off the transformator	The system goes down.
WASH THE SYSTEM WITH NITROGEN	Wash the system with Nitrogen for few minutes BY TURNING ON THE VALVE on the nitrogen tank	All the hydrogen existing in the pipe system exit

Postcondition	Turn off the system	System is off

00007: Test whether the membrane is ruptured

If there are air bubbles from the hydrogen half cells set the membrane have to be changed.

Step	**Step Description**	**Expected Result**
Precondition	System is off	
Emptying the cells	Placing a container under the two emptying valves and open the two emptying valves so that the containers are filled with the solution of the cells .	The solution flows into the containers.
Closing of the emptying valves.	Closing the emptying valves after the whole solution flowed from the cells into the containers.	The emptying valves are closed
Let the air enter to the half-cell set of oxygen.	Connect the air compressor to one set of the half cell sets and tur on the compressor.	Air bubbles are seen only in the one set on which the air bubbles are connected
Stop the air	Turn off the compressor	The compressor is off
Postcondition	Turn off the system	System is off

00008: A simple test of an Electrolysis system -Case add water manually-

Steps	**Steps description**	**Excepted result**
Precondition	System is Off	
	The cells are partially filled with NaOH solution	

Run pump to fill the cells with the NaOH solution	Open the valves (V_3) & (V_4) from the GUI	(V_3) & (V_4) are opened and enable to let the electrolyte solution pass
	Click "Start" on the Electrolyte bottom from the GUI	The cells enable to filled with electrolyte solution
	Wait 5 seconds, the pump (P_1) run automatically	The pump (P_1) is turned On
Turn Off the pump when the cells are filled with NaOH solution	When (L_3) & (L_4) go from "Low" to "High", turn Off the pump from the GUI	The cells filled with electrolyte solution
		Pump (P_1) turned Off
Close the valves (V_3) & (V_4)	Turn Off the valves (V_3) & (V_4) from the GUI	The valves (V_3) & (V_4) are closed
Wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass
	Close the blue ball valve of the water tank manually	The blue ball valve is closed
	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The Nitrogen gas pass through the system
		The air bubbles appear (generate) in the Gas AutoSafety
	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed
		The air bubbles disappear (doesn't generate) in the Gas AutoSafety
	Open the blue ball valve of water tank manually	The blue ball valve is opened

	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed
Turn ON the burner to burn the gas	Click "Start" on Fuel Burner from the GUI	The transformer is turned On
		The burner is turned On
		Redness of the metal strip of the burner
Connect the Power Supply on the system	Ensure that the pump (P_1) is turned Off	Pump (P_1) is closed
	Click "Start" on the Power Supply bottom from the GUI	The Power Supply is On
		The system is generating Hydrogen and Oxygen
		5 minutes after turning On the burner, a flame appears
Add water to the system	Click "Start" on Water from the GUI	
	Turn On the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the water pass through the cells
	Turn On the pump (P_2)	Pump (P_2) is turned On
	After few minutes, Click "Stop" on the valves (V_1) & (V_2) with the Command OFF "CMD OFF" from the GUI	The valves (V_1) & (V_2) are closed
	When (L_3) & (L_4) go from "Low" to "High", turn Off the pump (P_2) from the GUI	The pump (P_2) is turned Off

	When (L_3) & (L_4) go from "Low" to "High", Turn Off the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed
Disconnect the Power Supply on the system	Click "Stop" on Power Supply bottom from the GUI	The electricity is turned Off from the system
		The generation of Hydrogen and Oxygen are stopped
Turn Off the fuel burner	Click "Stop" on the Fuel Burner bottom from the GUI	Burner (Transformer) is Off
	Click "Stop" on the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed
		Flame disappears (is Off)
Re-wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass
	Close the blue ball valve of the water tank manually	The blue ball valve is closed
	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The Nitrogen gas pass through the system
		The air bubbles appear (generate) in the Gas AutoSafety
	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed
		The air bubbles disappear (doesn't generate) in the Gas AutoSafety
	Open the blue ball valve of water tank manually	The blue ball valve is opened

	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed
Post condition	Turn Off the system	System is Off
	The cells are partially filled with NaOH solution	
	The cells are filled totally with NaOH solution	

00009: A simple test of an Electrolysis system -Case add water automatically-

Steps	**Steps description**	**Excepted result**
Precondition	System is Off	
	The cells are partially filled with NaOH solution	
Run pump to fill the cells with the NaOH solution	Open the valves (V_3) & (V_4) from the GUI	(V_3) & (V_4) are opened and enable to let the electrolyte solution pass
	Click "Start" on the Electrolyte bottom from the GUI	The cells enable to filled with electrolyte solution
	Wait 5 seconds, the pump (P_1) run automatically	The pump (P_1) is turned On
Turn Off the pump when the cells are filled with NaOH solution	When (L_3) & (L_4) go from "Low" to "High", turn Off the pump from the GUI	The cells filled with electrolyte solution
		Pump (P_1) turned Off

Close the valves (V_3) & (V_4)	Turn Off the valves (V_3) & (V_4) from the GUI	The valves (V_3) & (V_4) are closed
Wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass
	Close the blue ball valve of the water tank manually	The blue ball valve is closed
	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The Nitrogen gas pass through the system
		The air bubbles appear (generate) in the Gas AutoSafety
	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed
		The air bubbles disappear (doesn't generate) in the Gas AutoSafety
	Open the blue ball valve of water tank manually	The blue ball valve is opened
	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed
Turn ON the burner to burn the gas	Click "Start" on Fuel Burner from the GUI	The transformer is turned On
		The burner is turned On
		Redness of the metal strip of the burner
	Ensure that the pump (P_1) is turned Off	Pump (P_1) is closed

Connect the Power Supply on the system	Click "Start" on the Power Supply bottom from the GUI	The Power Supply is On
		The system is generating Hydrogen and Oxygen
		5 minutes after turning On the burner, a flame appears
Add water to the system	Click "Start" on Water from the GUI	
	Turn On the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the water pass through the cells
	Turn On the pump (P_2)	Pump (P_2) is turned On
	When (L_3) & (L_4) go from "Low" to "High", the pump (P_2) is turned Off automatically from the GUI	Pump (P_2) is turned Off
	When (L_3) & (L_4) go from "Low" to "High", the valves (V_1) & (V_2) are turned Off automatically from the GUI	The valves (V_1) & (V_2) are closed
	Click "Stop" on Water from the GUI	
Disconnect the Power Supply on the system	Click "Stop" on Power Supply bottom from the GUI	The electricity is turned Off from the system
		The generation of Hydrogen and Oxygen are stopped
Turn Off the fuel burner	Click "Stop" on the Fuel Burner bottom from the GUI	Burner (Transformer) is Off

	Click "Stop" on the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed Flame disappears (is Off)
Re-wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass
	Close the blue ball valve of the water tank manually	The blue ball valve is closed
	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The Nitrogen gas pass through the system The air bubbles appear (generate) in the Gas AutoSafety
	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed The air bubbles disappear (doesn't generate) in the Gas AutoSafety
	Open the blue ball valve of water tank manually	The blue ball valve is opened
	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed
Post condition	Turn Off the system	System is Off
	The cells are partially filled with NaOH solution	
	The cells are filled totally with NaOH solution	

00010: A simple test of an Electrolysis system -Case without add water-

Steps	Steps description	Excepted result
Precondition	System is Off	
	The cells are partially filled with NaOH solution	
Run pump to fill the cells with the NaOH solution	Open the valves (V_3) & (V_4) from the GUI	(V_3) & (V_4) are opened and enable to let the electrolyte solution pass
	Click "Start" on the Electrolyte bottom from the GUI	The cells enable to filled with electrolyte solution
	Wait 5 seconds, the pump (P_1) run automatically	The pump (P_1) is turned On
Turn Off the pump when the cells are filled with NaOH solution	When (L_3) & (L_4) go from "Low" to "High", turn Off the pump from the GUI	The cells filled with electrolyte solution
		Pump (P_1) turned Off
Close the valves (V_3) & (V_4)	Turn Off the valves (V_3) & (V_4) from the GUI	The valves (V_3) & (V_4) are closed
Wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass
	Close the blue ball valve of the water tank manually	The blue ball valve is closed
	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The Nitrogen gas pass through the system

		The air bubbles appear (generate) in the Gas AutoSafety
	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed
		The air bubbles disappear (doesn't generate) in the Gas AutoSafety
	Open the blue ball valve of water tank manually	The blue ball valve is opened
	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed
Turn ON the burner to burn the gas	Click "Start" on Fuel Burner from the GUI	The transformer is turned On
		The burner is turned On
		Redness of the metal strip of the burner
Connect the Power Supply on the system	Ensure that the pump (P_1) is turned Off	Pump (P_1) is closed
	Click "Start" on the Power Supply bottom from the GUI	The Power Supply is On
		The system is generating Hydrogen and Oxygen
		5 minutes after turning On the burner, a flame appears
Disconnect the Power Supply on the system	Click "Stop" on Power Supply bottom from the GUI	The electricity is turned Off from the system
		The generation of Hydrogen and Oxygen are stopped

Turn Off the fuel burner	Click "Stop" on the Fuel Burner bottom from the GUI	Burner (Transformer) is Off
	Click "Stop" on the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed
		Flame disappears (is Off)
Re-wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass
	Close the blue ball valve of the water tank manually	The blue ball valve is closed
	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The Nitrogen gas pass through the system
		The air bubbles appear (generate) in the Gas AutoSafety
	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed
		The air bubbles disappear (doesn't generate) in the Gas AutoSafety
	Open the blue ball valve of water tank manually	The blue ball valve is opened
	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed
Post condition	Turn Off the system	System is Off
	The cells are partially filled with NaOH solution	

	The cells are filled totally with NaOH solution

5.3 Electrolyzer tests

5.3.1 Electrolyzer test 5.5.2023

Test whether the pressure is equilibrium 05.05.2023

If the water level sensors are in the parallel level throughout the test period, we have come to a solution to the problem of pressure difference within a single cell. Now we can replace the membrane with Nafion.
But if the water level sensors aren't in the parallel level throughout the test period, this means that there is a problem with suffocation (closed) in one of the condenser tubes.

Goal:

In the previous test, we had a problem with the pressure balance between the two half-cells, and to resolve this problem, we suggested placing flashback arrestor at the oxygen gas outlet. The aim of this test is to ensure the correctness of the pressure balance between the two half-cells.

Expected result:

In case of equilibrium between the two half-cells, the level sensor pointer for each half-cell (water level parallels between the cathodic half-cell and the anodic half-cell).

If there is a pressure difference between the two-level sensors, we should notice a difference in the level of the solution at the two sensors.

Operation Steps:

00008: A simple test of an Electrolysis system -Case add water manually-

Steps	Steps description	Excepted result	Result
Precondition	System is Off		✓

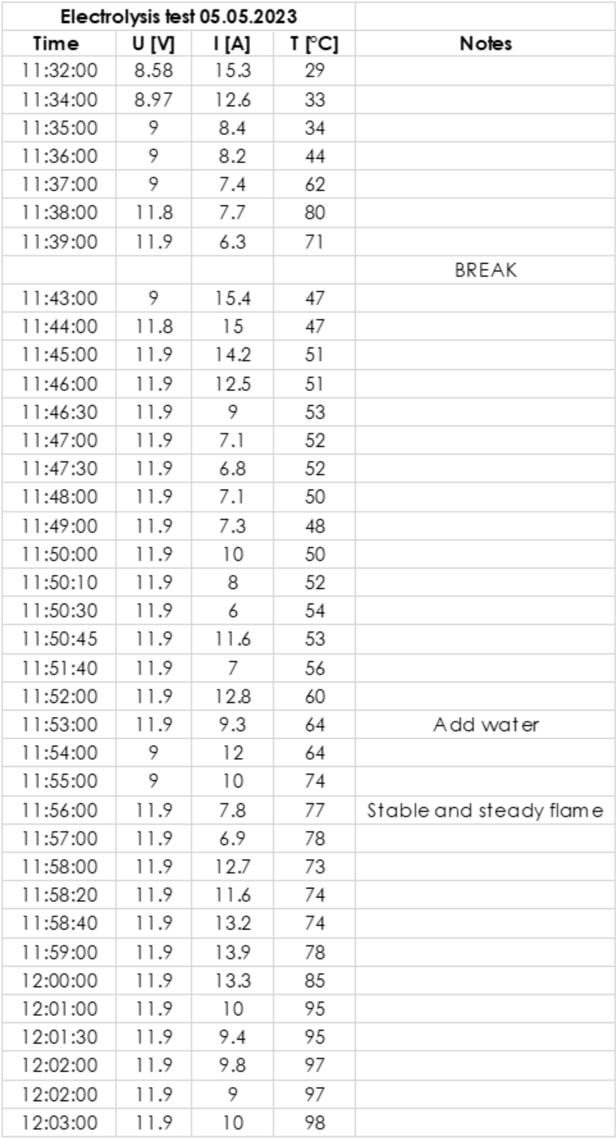
		The cells are partially filled with NaOH solution	✓
Run pump to fill the cells with the NaOH solution	Open the valves (V_3) & (V_4) from the GUI	(V_3) & (V_4) are opened and enable to let the electrolyte solution pass	✓
	Click "Start" on the Electrolyte bottom from the GUI	The cells enable to filled with electrolyte solution	✓
	Wait 5 seconds, the pump (P_1) run automatically	The pump (P_1) is turned On	✓
Turn Off the pump when the cells are filled with NaOH solution	When (L_3) & (L_4) go from "Low" to "High", turn Off the pump from the GUI	The cells filled with electrolyte solution	✓
		Pump (P_1) turned Off	✓
Close the valves (V_3) & (V_4)	Turn Off the valves (V_3) & (V_4) from the GUI	The valves (V_3) & (V_4) are closed	✓
Wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass	✓
	Close the blue ball valve of the water tank manually	The blue ball valve is closed	✓
	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The Nitrogen gas pass through the system	✓
		The air bubbles appear (generate) in the Gas AutoSafety	✓

	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed	✓
		The air bubbles disappear (doesn't generate) in the Gas AutoSafety	✓
	Open the blue ball valve of water tank manually	The blue ball valve is opened	✓
	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed	✓
Turn ON the burner to burn the gas	Click "Start" on Fuel Burner from the GUI	The transformer is turned On	✓
		The burner is turned On	✓
		Redness of the metal strip of the burner	✓
Connect the Power Supply on the system	Ensure that the pump (P_1) is turned Off	Pump (P_1) is closed	✓
	Click "Start" on the Power Supply bottom from the GUI	The Power Supply is On	✓
		The system is generating Hydrogen and Oxygen	✓
		5 minutes after turning On the burner, a flame appears	✓
Add water to the system	Click "Start" on Water from the GUI		✓
	Turn On the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the water pass through the cells	✓

	Turn On the pump (P_2)	Pump (P_2) is turned On	✓
	After few minutes, Click "Stop" on the valves (V_1) & (V_2) with the Command OFF "CMD OFF" from the GUI	The valves (V_1) & (V_2) are closed	✓
	When (L_3) & (L_4) go from "Low" to "High", turn Off the pump (P_2) from the GUI	The pump (P_2) is turned Off	✗
	When (L_3) & (L_4) go from "Low" to "High", Turn Off the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed	✗
Disconnect the Power Supply on the system	Click "Stop" on Power Supply bottom from the GUI	The electricity is turned Off from the system	✓
		The generation of Hydrogen and Oxygen are stopped	✓
Turn Off the fuel burner	Click "Stop" on the Fuel Burner bottom from the GUI	Burner (Transformer) is Off	✓
	Click "Stop" on the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed	✓
		Flame disappears (is Off)	✓
Re-wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass	✓
	Close the blue ball valve of the water tank manually	The blue ball valve is closed	✓

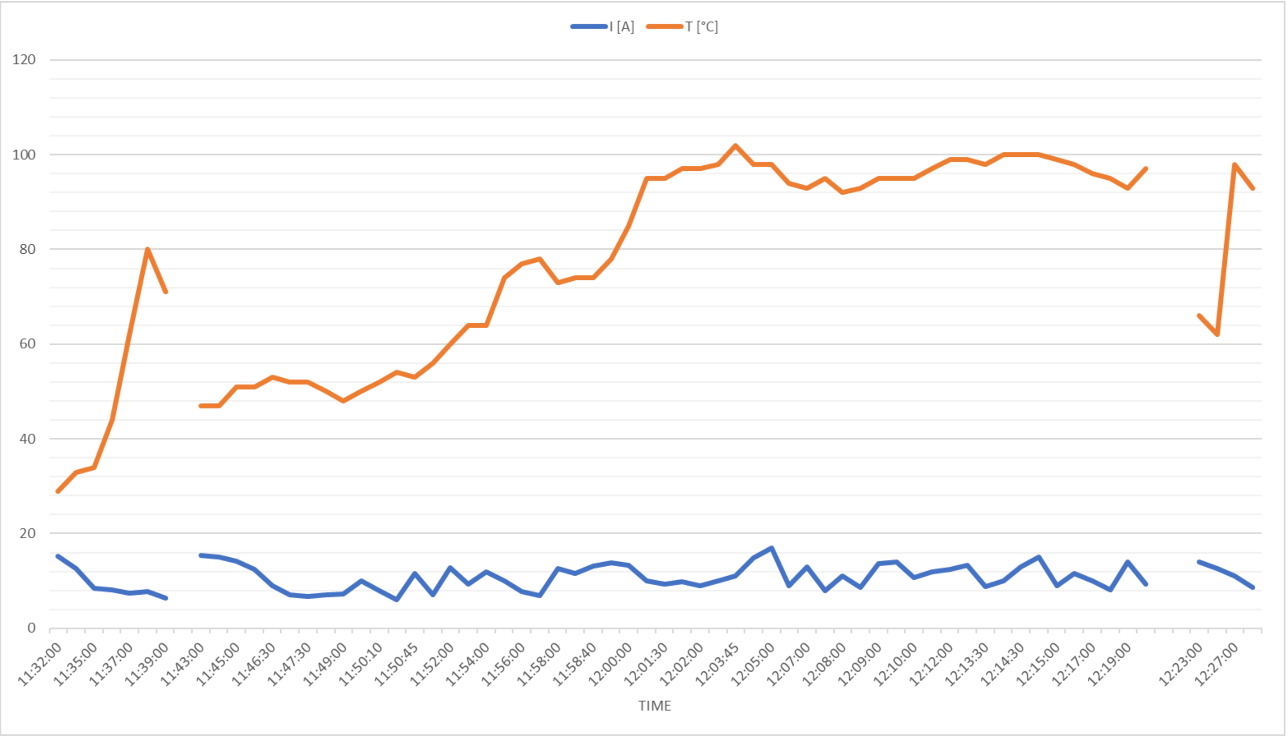
	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The Nitrogen gas pass through the system	✓
		The air bubbles appear (generate) in the Gas AutoSafety	✓
	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed	✓
		The air bubbles disappear (doesn't generate) in the Gas AutoSafety	✓
	Open the blue ball valve of water tank manually	The blue ball valve is opened	✓
	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed	✓
Post condition	Turn Off the system	System is Off	✓
	The cells are partially filled with NaOH solution		✓
	The cells are filled totally with NaOH solution		✗

Data collected during test:



Electrolysis test 05.05.2023				
Time	**U [V]**	**I [A]**	**T [°C]**	**Notes**
11:32:00	8.58	15.3	29	
11:34:00	8.97	12.6	33	
11:35:00	9	8.4	34	
11:36:00	9	8.2	44	
11:37:00	9	7.4	62	
11:38:00	11.8	7.7	80	
11:39:00	11.9	6.3	71	
				BREAK
11:43:00	9	15.4	47	
11:44:00	11.8	15	47	
11:45:00	11.9	14.2	51	
11:46:00	11.9	12.5	51	
11:46:30	11.9	9	53	
11:47:00	11.9	7.1	52	
11:47:30	11.9	6.8	52	
11:48:00	11.9	7.1	50	
11:49:00	11.9	7.3	48	
11:50:00	11.9	10	50	
11:50:10	11.9	8	52	
11:50:30	11.9	6	54	
11:50:45	11.9	11.6	53	
11:51:40	11.9	7	56	
11:52:00	11.9	12.8	60	
11:53:00	11.9	9.3	64	Add water
11:54:00	9	12	64	
11:55:00	9	10	74	
11:56:00	11.9	7.8	77	Stable and steady flame
11:57:00	11.9	6.9	78	
11:58:00	11.9	12.7	73	
11:58:20	11.9	11.6	74	
11:58:40	11.9	13.2	74	
11:59:00	11.9	13.9	78	
12:00:00	11.9	13.3	85	
12:01:00	11.9	10	95	
12:01:30	11.9	9.4	95	
12:02:00	11.9	9.8	97	
12:02:00	11.9	9	97	
12:03:00	11.9	10	98	

12:03:45	11.9	11	102	
12:04:00	11.9	14.9	98	
12:05:00	11	17	98	
12:06:00	11	9	94	
12:07:00	11	13	93	
12:07:30	11.9	8	95	
12:08:00	11.9	11	92	
12:08:45	11.9	8.7	93	
12:09:00	11.9	13.6	95	
12:09:30	11.9	14	95	
12:10:00	11.9	10.8	95	
12:11:00	11.9	12	97	
12:12:00	11.9	12.5	99	
12:13:00	11.9	13.3	99	
12:13:30	11.9	8.8	98	
12:14:00	11.9	10	100	
12:14:30	11.9	13	100	
12:14:40	11.9	15	100	
12:15:00	11.9	9	99	
12:16:00	11.9	11.5	98	
12:17:00	11.9	10.1	96	
12:18:00	11.9	8.1	95	
12:19:00	11.9	14	93	
12:19:30	11.9	9.3	97	
				BREAK
				O2 condenser is more warm than H2 condenser
12:23:00	9.93	14	66	
12:25:00	9.93	12.7	62	
12:27:00	9.93	11	98	
12:29:00	9.93	8.7	93	



⚠ **Note:**

1. he intensity (I) measures the AC, while the voltage (V) measures the DC.

2. The inlet of O_2 condenser is too warm than inlet of H_2 condenser.

Result view:

Level sensors: on the left side, the level sensor placed on the anodic half-cell. On the right side, the level sensor placed on cathodic half-cell.

Conclusion:

At the end of the test, we found that the water level in the sensor was almost identical. Which means that the pressure differential problem has been preliminarily resolved.

5.3.2 Electrolyzer test 28.06.2023

For the purpose of measuring the DC intensity of the cells system, an intensity sensor (Ammeter) with a capacity of 200 A has been installed.

Goal:

This test aims to collect the DC intensity data of the system cells during operation, in order to know the amount of hydrogen and oxygen gas generated.

Expected result:

Obtaining the required DC intensity data to balance it with the amount of Hydrogen and Oxygen gas generated.

If this is achieved, we can estimate the amount of hydrogen produced and thus select a suitable flowmeter for the hydrogen gas emitted

Test specifications:

00010: A simple test of an Electrolysis system -Case without add water-

Steps	Steps description	Excepted result	Result
Precondition	System is Off		✓
	The cells are partially filled with NaOH solution		✓
Run pump to fill the cells with the	Open the valves (V_3) & (V_4) from the GUI	(V_3) & (V_4) are opened and enable to let the electrolyte solution pass	✓

NaOH solution	Click "Start" on the Electrolyte bottom from the GUI	The cells enable to filled with electrolyte solution	✓
	Wait 5 seconds, the pump (P_1) run automatically	The pump (P_1) is turned On	✓
Turn Off the pump when the cells are filled with NaOH solution	When (L_3) & (L_4) go from "Low" to "High", turn Off the pump from the GUI	The cells filled with electrolyte solution	✓
		Pump (P_1) turned Off	✓
Close the valves (V_3) & (V_4)	Turn Off the valves (V_3) & (V_4) from the GUI	The valves (V_3) & (V_4) are closed	✓
Wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass	✓
	Close the blue ball valve of the water tank manually	The blue ball valve is closed	✓
	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The Nitrogen gas pass through the system	✓
		The air bubbles appear (generate) in the Gas AutoSafety	✓

	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed	✓
		The air bubbles disappear (doesn't generate) in the Gas AutoSafety	✓
	Open the blue ball valve of water tank manually	The blue ball valve is opened	✓
	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed	✓
Turn ON the burner to burn the gas	Click "Start" on Fuel Burner from the GUI	The transformer is turned On	✓
		The burner is turned On	✓
		Redness of the metal strip of the burner	✓
Connect the Power Supply on the system	Ensure that the pump (P_1) is turned Off	Pump (P_1) is closed	✓
	Click "Start" on the Power Supply bottom from the GUI	The Power Supply is On	✓
		The system is generating Hydrogen and Oxygen	✓

		5 minutes after turning On the burner, a flame appears	✓
Disconnect the Power Supply on the system	Click "Stop" on Power Supply bottom from the GUI	The electricity is turned Off from the system	✓
		The generation of Hydrogen and Oxygen are stopped	✓
Turn Off the fuel burner	Click "Stop" on the Fuel Burner bottom from the GUI	Burner (Transformer) is Off	✓
	Click "Stop" on the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed	✓
		Flame disappears (is Off)	✓
Re-wash the system with Nitrogen gas	Open the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are open and enable to let the nitrogen gas pass	✓
	Close the blue ball valve of the water tank manually	The blue ball valve is closed	✓
		The Nitrogen gas pass through the system	✓

	Open the bottle of Nitrogen gas 5 minutes @ 4 bars	The air bubbles appear (generate) in the Gas AutoSafety	✓
	Turn Off the Nitrogen bottle by its gate valve manually	The Nitrogen bottle is closed	✓
		The air bubbles disappear (doesn't generate) in the Gas AutoSafety	✓
	Open the blue ball valve of water tank manually	The blue ball valve is opened	✓
	Close the valves (V_1) & (V_2) from the GUI	The valves (V_1) & (V_2) are closed	✓
Post condition	Turn Off the system	System is Off	✓
	The cells are partially filled with NaOH solution		✓
	The cells are filled totally with NaOH solution		✗

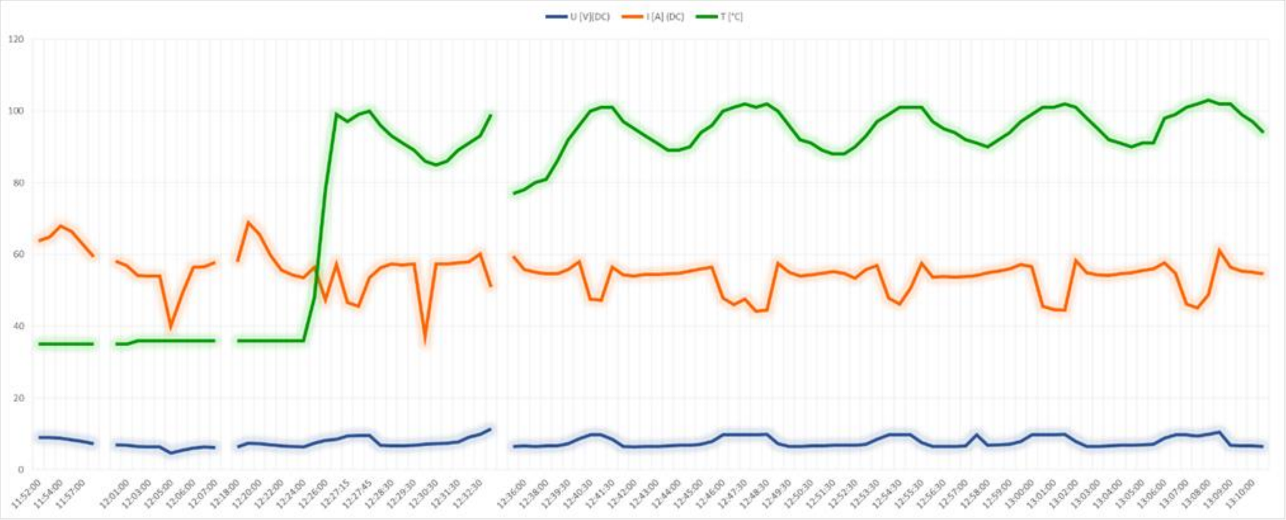
Data collected during test:

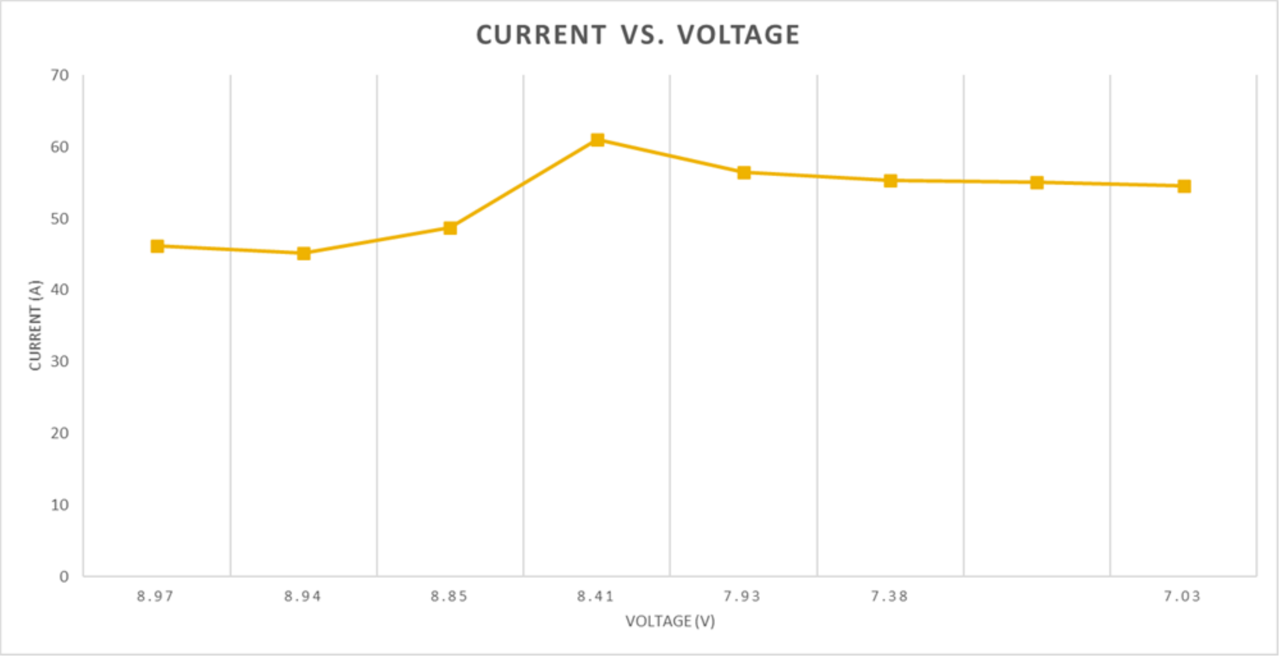
Time	U [V](DC)	I [A] (DC)	T [°C]	Notes
11:52:00	8.97	63.78	35	
11:53:00	8.94	64.8	35	
11:54:00	8.85	68	35	
11:56:00	8.41	66.47	35	
11:57:00	7.93	62.86	35	
11:58:00	7.38	59.43	35	
				BREAK ON/OFF
12:00:00	7.03	58.21	35	
12:01:00	6.8	56.89	35	
12:02:00	6.55	54.1	36	Appearance of Hydrogen gas
12:03:00	6.45	54.01	36	
12:04:00	6.32	54.08	36	
12:05:00	4.7	40.07	36	
12:05:30	5.44	49.15	36	
12:06:00	6.13	56.51	36	
12:06:30	6.36	56.6	36	
12:07:00	6.24	57.8	36	
				BREAK ON/OFF

12:18:00	6.41	58	36	
12:19:00	7.43	68.78	36	
12:20:00	7.36	65.57	36	
12:21:00	7.03	59.85	36	
12:22:00	6.7	55.74	36	
12:23:00	6.54	54.35	36	
12:24:00	6.39	53.53	36	
12:25:00	7.39	56.48	48	Stable and steady flame
12:26:00	8.19	47.44	78	
12:27:00	8.54	57.23	99	
12:27:15	9.51	46.7	97	
12:27:30	9.63	45.6	99	
12:27:45	9.55	53.55	100	
12:28:00	6.82	56.28	96	
12:28:30	6.7	57.35	93	
12:29:00	6.74	57	91	
12:29:30	6.9	57.35	89	
12:30:00	7.11	37.29	86	
12:30:30	7.23	57.42	85	
12:31:00	7.39	57.36	86	
12:31:30	7.81	57.67	89	
12:32:00	9.14	58	91	
12:32:30	9.95	60.06	93	
12:32:45	11.43	51	99	
				BREAK ON/OFF

12:35:00	6.5	59.56	77	
12:36:00	6.63	55.78	78	
12:37:00	6.6	55.08	80	
12:38:00	6.63	54.6	81	
12:39:00	6.73	54.6	86	
12:39:30	7.28	55.78	92	
12:40:00	8.62	58.01	96	
12:40:30	9.71	47.61	100	
12:41:00	9.77	47.22	101	
12:41:30	8.55	56.48	101	
12:41:45	6.53	54.3	97	
12:42:00	6.4	54.04	95	
12:42:30	6.49	54.41	93	
12:43:00	6.57	54.4	91	
12:43:30	6.67	54.57	89	
12:44:00	6.81	54.79	89	
12:44:30	6.91	55.37	90	
12:45:00	7.16	55.95	94	
12:45:30	7.94	56.53	96	
12:46:00	9.76	47.9	100	
12:47:00	9.8	46	101	
12:47:30	9.78	47.6	102	
12:48:00	9.82	44.23	101	
12:48:30	9.86	44.46	102	
12:49:00	7.23	57.55	100	
12:49:30	6.57	55.04	96	
12:50:00	6.6	54.04	92	
12:50:30	6.72	54.37	91	
12:51:00	6.73	54.81	89	
12:51:30	6.9	55.2	88	
12:52:00	6.83	54.71	88	flame
12:52:30	6.91	53.32	90	flame
12:53:00	7.17	55.77	93	flame
12:53:30	8.5	56.94	97	
12:54:00	9.72	47.87	99	
12:54:30	9.78	46.13	101	
12:55:00	9.81	50.7	101	
12:55:30	7.6	57.48	101	
12:56:00	6.5	53.63	97	
12:56:30	6.48	53.82	95	
12:56:45	6.5	53.74	94	
12:57:00	6.64	53.85	92	
12:57:30	9.75	54.2	91	
12:58:00	6.83	54.86	90	
12:58:30	7.02	55.45	92	

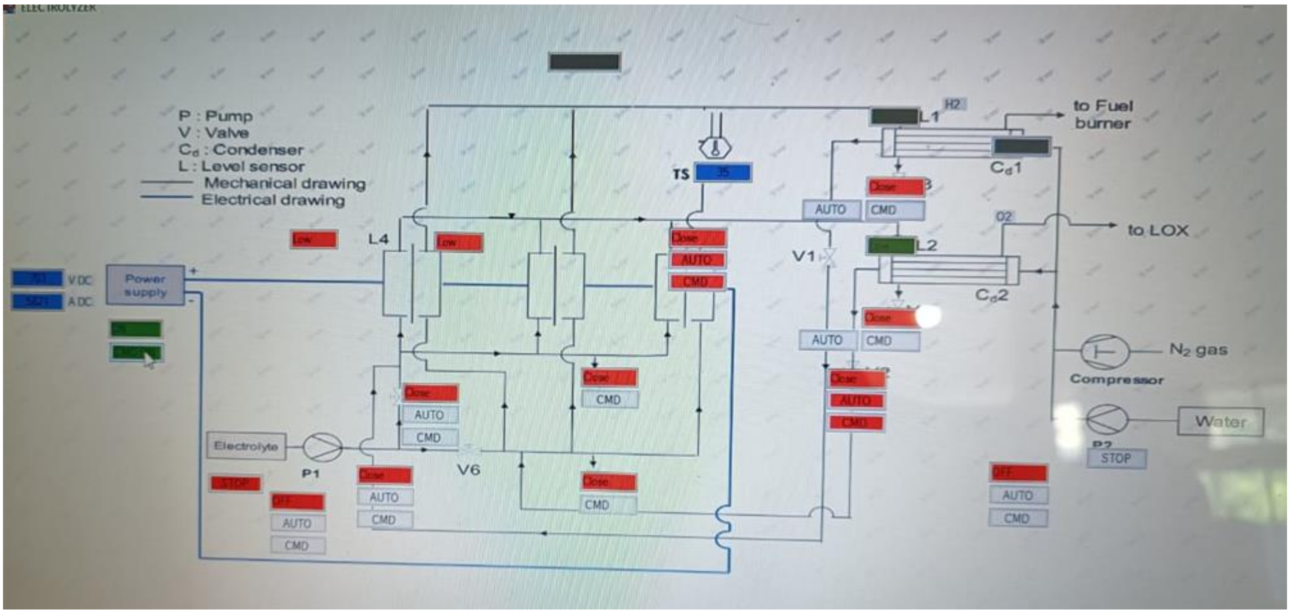
12:59:00	7.15	56.04	94	
12:59:30	7.87	57.25	97	Flame
13:00:00	9.76	56.6	99	
13:00:30	9.83	45.59	101	
13:01:00	9.83	44.68	101	
13:01:30	9.85	44.51	102	
13:02:00	7.92	58.23	101	
13:02:30	6.55	54.89	98	
13:03:00	6.59	54.24	95	
13:03:30	6.68	54.14	92	Flame
13:04:00	6.78	54.64	91	Flame
13:04:30	6.85	54.89	90	Flame
13:05:00	6.95	55.52	91	Flame
13:05:30	7.19	55.92	91	Flame
13:06:00	8.77	57.65	98	Flame
13:06:30	9.77	54.77	99	Flame
13:07:00	9.79	46.18	101	
13:07:30	9.48	45.12	102	
13:08:00	9.96	48.74	103	
13:08:30	10.59	61	102	
13:09:00	6.85	56.44	102	
13:09:30	6.64	55.39	99	
13:10:00	6.65	55.09	97	
13:10:30	6.61	54.59	94	




CURRENT VS. VOLTAGE
70
60
50
40
30
20
10
0
CURRENT (A)
8.97
8.94
8.85
8.41
7.93
7.38
7.03
VOLTAGE (V)

Result view:


Camera 01



Conclusion:

Test passed successfully. The next step is to install the produced hydrogen meter.

5.4 Multistage Electrolyzer[9]

5.4.1 Product requirements of the multistage electrolyse cell

The material of the electrolyze: stainless steel 304

The thickness of the electrodes: thick enough to withstand the pressing without change in shape

The dimensions of the electrodes: consider the current calculated

Taking into account the relation:

$I = 0{,}4$ x the surface that touches the solution$=0{,}4$ x $A=0{,}4\times3{,}14\times r^2$

The cell voltage is $U = 2{,}4$ Volt

The thickness of the gasket:

On the cathode side: 0,5 cm

On the anode side: 0,25 cm

The calculations must be done again taking into account the conductivity of the stainless steel 304

The resistivity of the stainless steel 304 is very small ($0{,}72*10^{-6}$ (ohm meter)$^{-1}$) so it can be neglect and the calculations above are correct.

The dimension of the endplates: a bit bigger in dimension then the electrodes and thin enough to withstand the stress of the screws

The dimension of the wholes for the screw in the endplates: asking a mechanist

The dimensions of the screws: asking a mechanist

The type of the membrane: search it in google available and to be searched which one is the best:

---

[9] Ref : https://www.aecenar.com/index.php/institutes/icpt/icpt-electrolyser/system-concept-system-design/mechanical-design

Nafion n117 price: 340USD/pcs for 30*30cm. or 136USD for 15*15cm.

Nafion n115 price: 136USD/pcs for 20*20cm.

NR212 price is 303USD/pcs, 61cm*30cm.

NR211 price is 114USD/pcs, 20*20cm

Nafion N115 price is 136USD/pcs for 20*20cm

All the named Nafion membranes are from Alibaba:

https://www.alibaba.com/product-detail/Ion-Exchange-Membrane-Manufacturer-In-China_1600326782762.html

The diameter of the wholes

Of the water inlet:

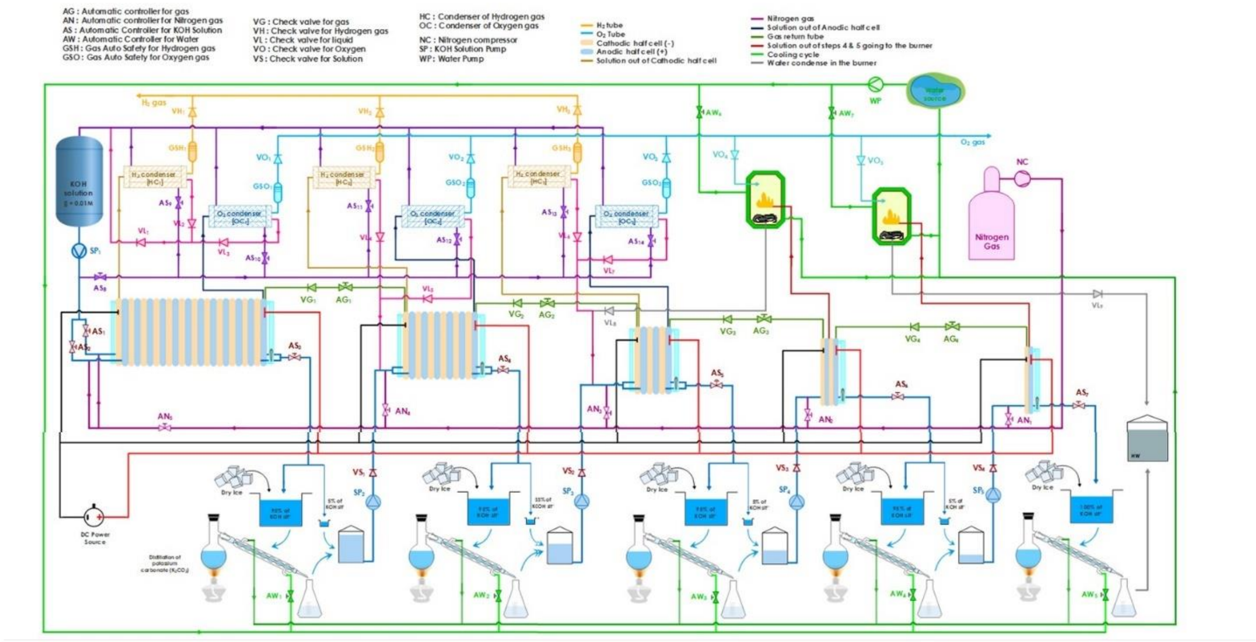
Of gas outlet:

The dimensions of the whole of the gasket based on the

The dimensions of the catalyzer of the upcoming water vapor based on the calculation of the temperature of the cells and the amount of the water vapor upcoming.

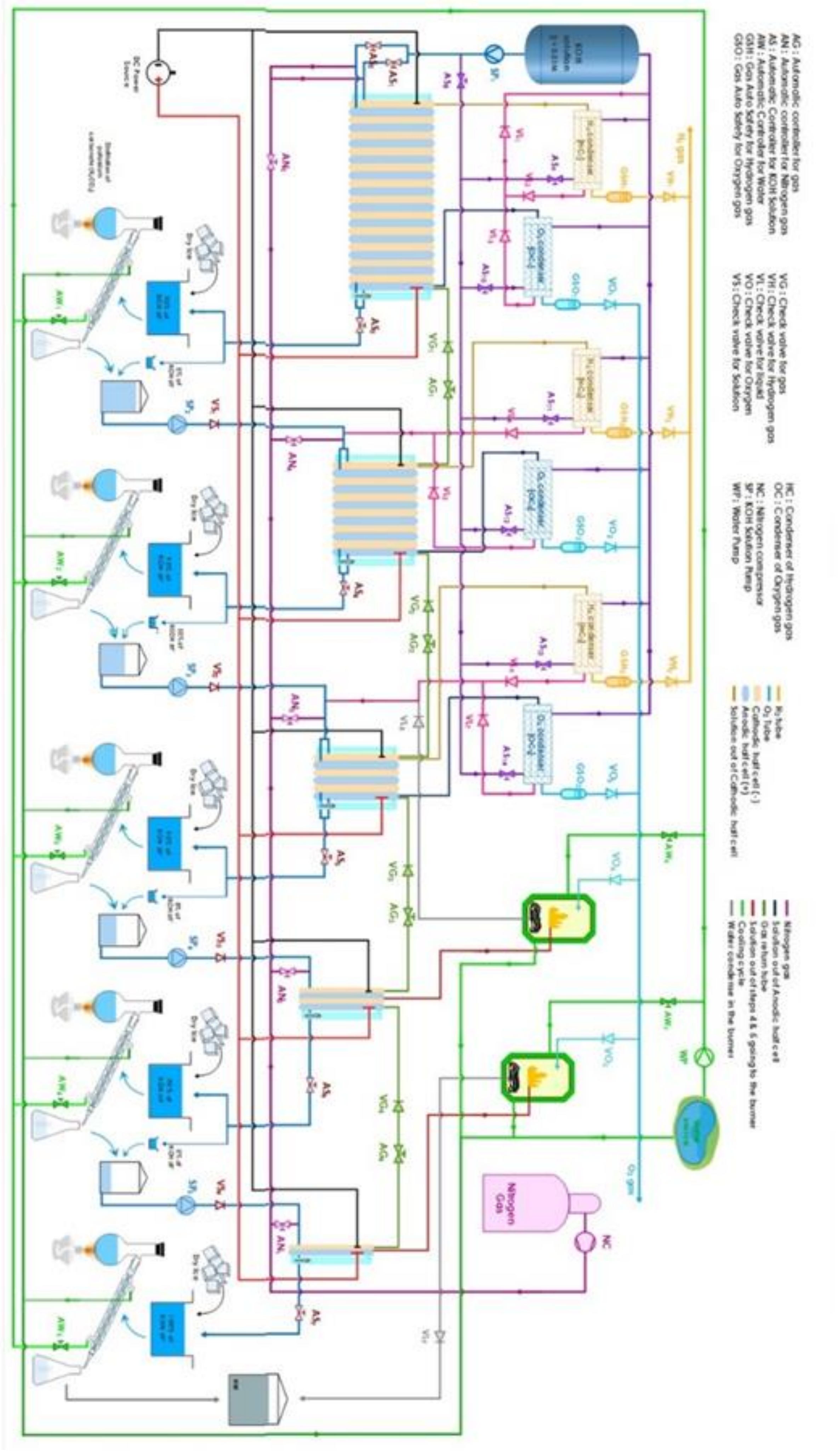
The current = I = 0,4 x the surface that touches the solution=0,4 x A=$0{,}4\times3{,}14\times r^2$.

5.4.2 Electrolysis multistage design overview



[Multistage design (27072023) _ Edraw file]

01082023_MSE flow
chart - with electricity.


AG : Automatic controller for gas
AN : Automatic controller for Nitrogen gas
AS : Automatic Controller for KOH Solution
AW : Automatic Controller for Water
GAH : Gas Auto Safety for Hydrogen gas
GAO : Gas Auto Safety for Oxygen gas
VG : Check valve for gas
VH : Check valve for Hydrogen gas
VL : Check valve for liquid
VO : Check valve for Oxygen
VS : Check valve for Solution
HC : Condenser of Hydrogen gas
OC : Condenser of Oxygen gas
NC : Nitrogen compressor
SP : KOH Solution Pump
WP : Water Pump
H₂ tube
O₂ Tube
Cathodic half cell (-)
Anodic half cell (+)
Solution out of Cathodic half cell
Nitrogen gas
Solution out of Anodic half cell
Gas return tube
Solution out of steps 4 & 6 going to the burner
Cooling cycle
Water condense in the burner
KOH solution
DC Power Source
H₂ gas
O₂ gas
Nitrogen Gas
Dry Ice
NC
WP

5.5 Fuel Cell

For immediate electricity production after Electrolysis.

5.5.1 Concept and Design of a Prototype Fuel Cell

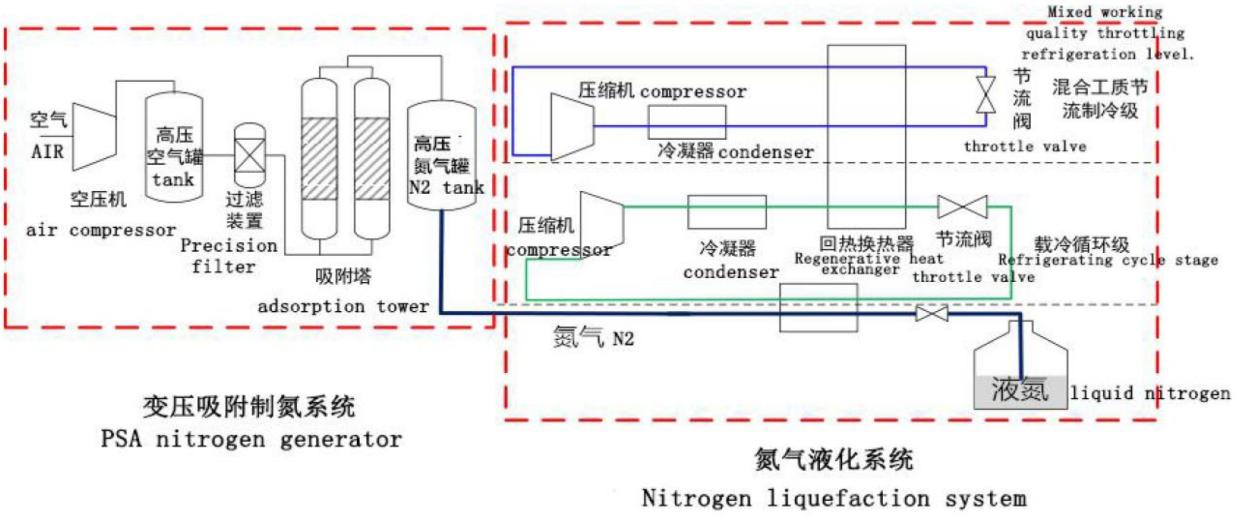
See ICPT Report 2021

5.6 Ammonia Production: For longtime storage of hydrogene

See ICPT Report 2022

5.7 LOX (Liquefaction of air and oxygene)

5.7.1 System Design Apr 2023 (based on Chinese supplier)[10]



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[10] Ref : https://aecenar.com/index.php/institutes/icpt/liquefication-of-air-and-oxygen/icpt-lox-system-concept-design

5.7.2 LOX Mechanical Realization

5.7.2.1 Prototype Location

	

5.7.2.2 Prototype Installation

		

5.7.2.3 Compressor:

Figure 4. LR25B Compressor

5.7.2.4 Cooler:

Figure 6. Cooler Tube Implementation

5.7.2.5 Evaporator:

Figure 7. Evaporator

5.7.2.6 Heat exchanger

5.7.3 Liquefication of oxygen System Test Specification[11]

5.7.3.1 First Experiment:

The first experiment (Expr #1) aims to:

1- Compressor operation test with nitrogen gas instead of R-134a,

2- Make sure that the Kelvinator refrigerator is running correctly

3- Ensure that the expansion valve is compatible with the design.

Therefore, the three heat exchangers (HX-N2/N2, HX-N2/O2 main, and HX-N2/O2 2nd) will be excluded from this experiment.

In this experiment, the oxygen will not be liquefied, but only the components will be tested and designed to ensure the proper functioning of the refrigeration cycle.

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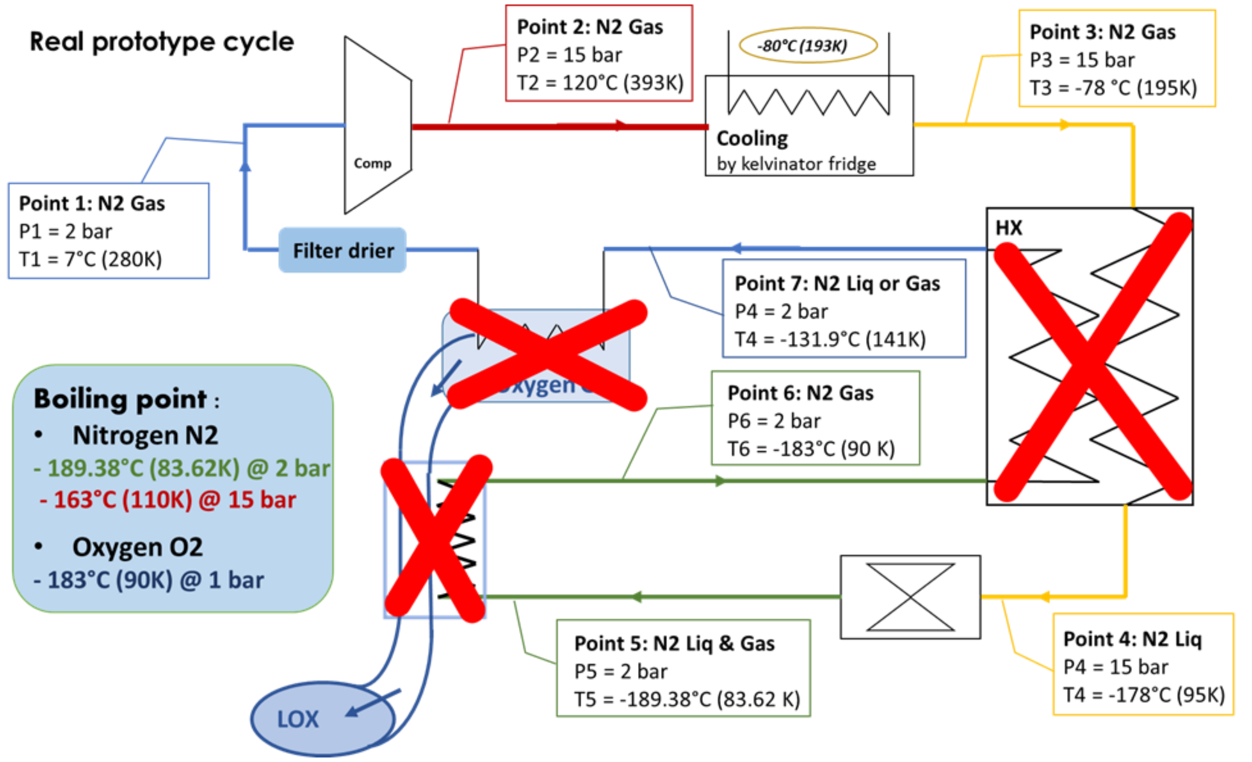
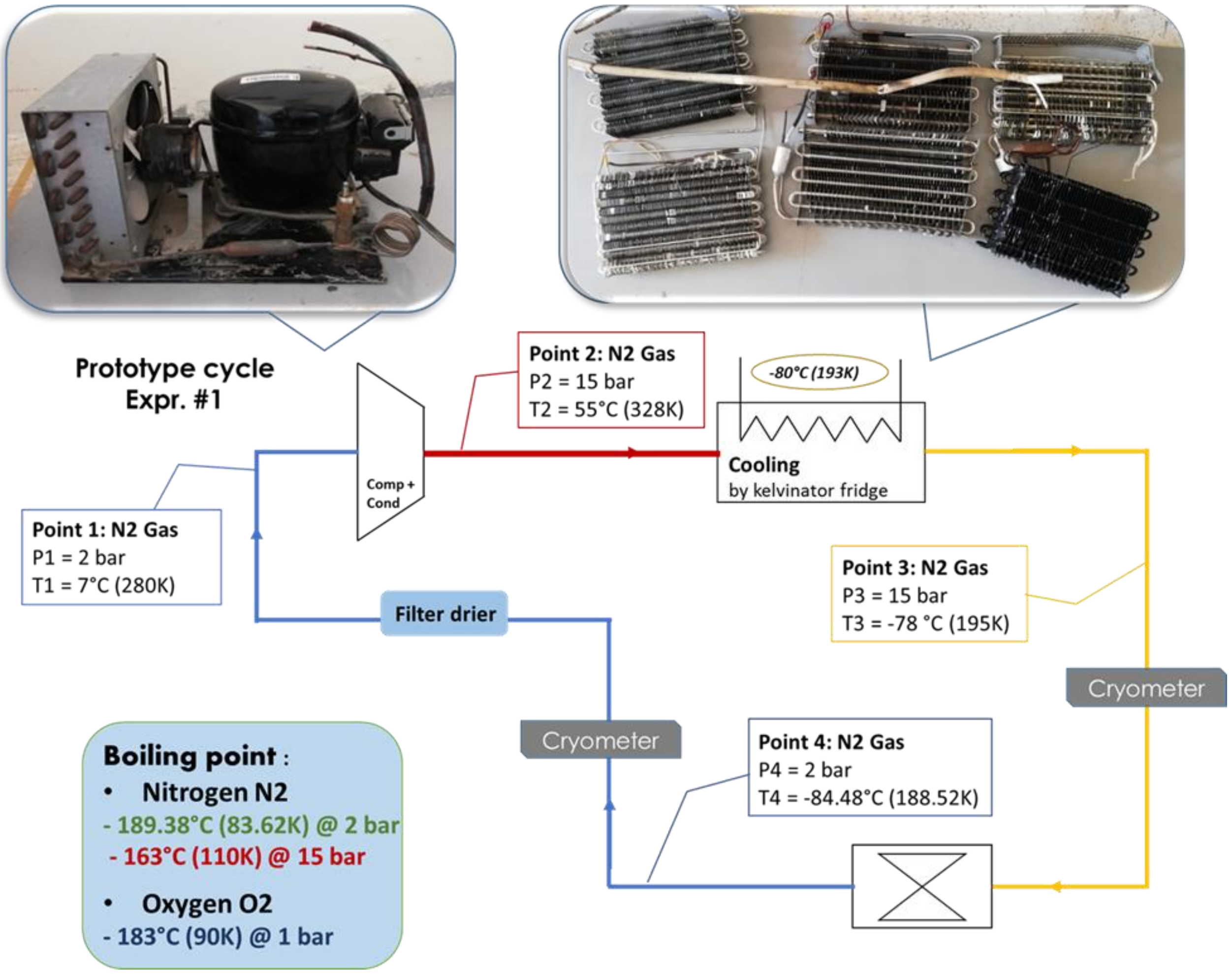
[11] Ref : https://aecenar.com/index.php/institutes/icpt/liquefication-of-air-and-oxygen/icpt-lox-system-test-specification



The first experiment consists of a simple cycle consisting of a compressor (LR25B Laboratory) with a condenser, cooling through a Kelvinator refrigerator, and an expansion valve (taken from an LR25B Laboratory refrigerator).



The components that must be provided to carry out this experiment:

- Filling the design with nitrogen gas, immediately before the pump.
- Covering the design with thermal insulation material to maintain the temperature of the refrigeration cycle,
- The presence of a thermometer (below -100˚C (173 K)) to measure the cooling outlet (an inlet of the expansion valve) and the outlet of the expansion valve,
- Also, a weather thermometer (from -10˚C to 20˚C (263 K to 293 K)) to measure the temperature of the compressor inlet.

For **the safety of the compressor**:

- In the **first hour of operation**, a 5-minute break must be taken for the compressor every 15 minutes of operation, in order to avoid an explosion of the compressor or one of the designed cables.

- Also, the compressor must **not run for more than two hours** in a row.

- Also, **cold nitrogen gas must be entered into the compressor** to reduce the speed of nitrogen flow, in addition to its role in cooling the compressor.

During this experiment (Expr. #1), the **amount of nitrogen gas filled** in the cycle, the **time of the experiment**, the **temperature reached** by the refrigeration cycle, and the **pressure during operation** will be calculated.

5.7.4 LOx Requirements

System requirement

- The lox system shall be able to liquefy oxygen.

Physical requirements

- The pipes shall be able to withstand the temperatures and pressures that exist at the points.

- Temperature that shall be withstood:
- Pressure that shall be withstood:

The different temperatures and pressures are listed in the figures below:

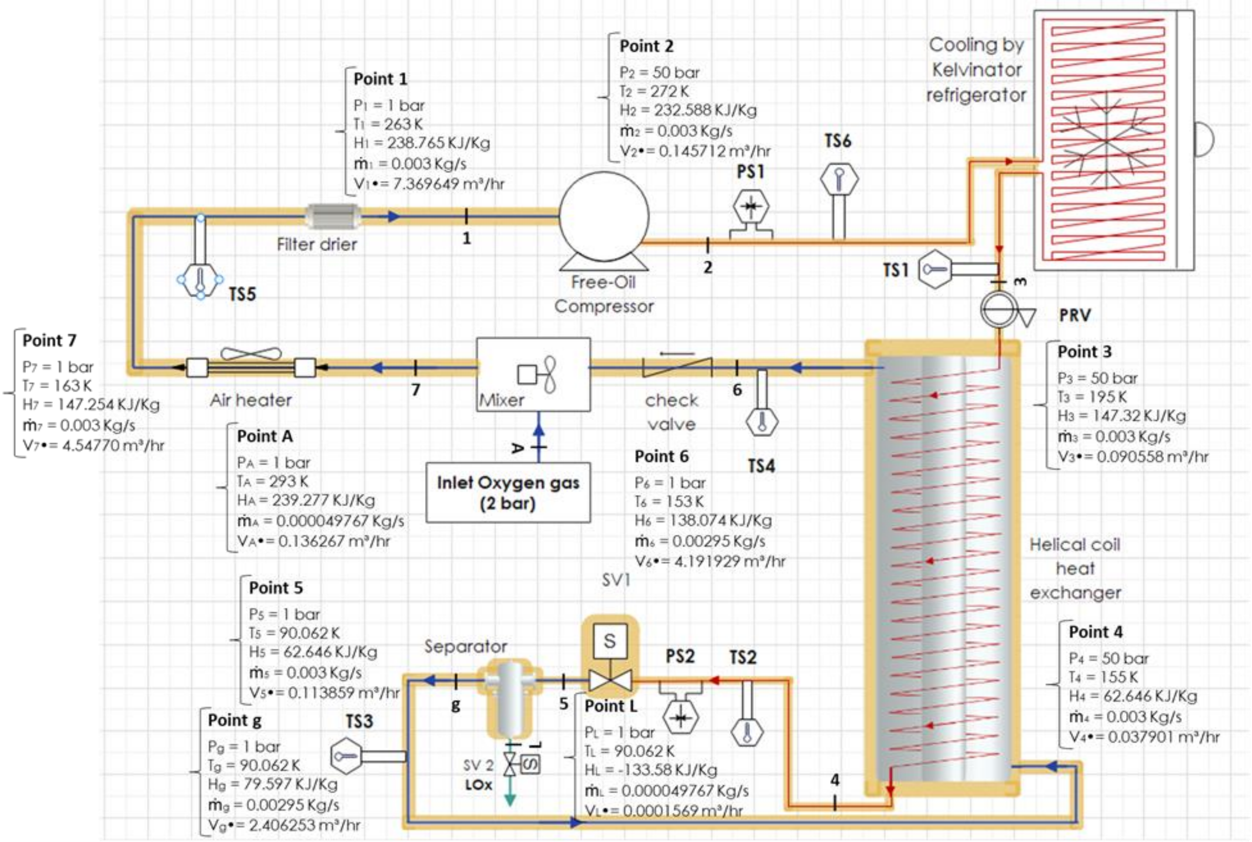


Fig-1-

Points	Pressure	Temperature	Enthapy	Mass flow		Density	Volumetric flow	
	P [bar]	T [°K / °C]	H [KJ/Kg]	m• [Kg/s]	m• [Kg/h]	D [Kg/m³]	V• [L/s]	V• [m³/hr]
Pt 1	1	263 / -10	238.765	0.003	10.8	1.46547	2.047124813	7.369649328
Pt 2	50	272 / + 10	232.588	0.003	10.8	74.1186	0.04047567	0.145712412
Pt 3	50	195 / -78	147.32	0.003	10.8	119.26	0.025155123	0.090558444
Pt 4	50	155 / -118	62.646	0.003	10.8	284.95	0.010528163	0.037901386
Pt 5	1	90.062 / -183	62.646	0.003	10.8	94.854	0.031627554	0.113859194
Pt g	1	90.062 / -183	79.597	0.00295	10.62	4.4135	0.668403761	2.40625354
Pt L	1	90.062 / -183	-133.58	0.000049767	0.1791612	1141.8	4.35864E-05	0.000156911
Pt 6	1	153 / -120	138.074	0.00295	10.62	2.53344	1.164424656	4.191928761
Pt 6'	1	263 / -10	238.765	0.00295	10.62	1.22598	2.406238275	8.662457789
Pt A'	1	263 / -10	238.765	0.000049767	0.1791612	1.22598	0.040593648	0.146137131
Pt A	1	293 / + 20	239.277	0.000049767	0.1791612	1.31478	0.03785196	0.136267056
Pt 7	1	163 / -110	147.254	0.003	10.8	2.3751	1.263104711	4.547176961
This table is based on thermodynamic properties tables of oxygen and formula of ideal gas law								

Table -1-

- The heat exchanger shall be able to decrease the temperature of the oxygen in order to become close to liquefaction temperature at the pressure present.

The temperature that must be reached: -120°C (154 K) @50bar.

- The cooler (Kelvinator refrigerator) shall be able to supply a temperature that is low enough.

The temperature that shall be reached: -80°C (193K).

- The separator shall be sufficient in volume to allow the gas expansion.

- The separator shall be able to separate the oxygen gas from the liquid oxygen.

- The heater shall be able to warm the cryogenic gas, so that their temperature is suitable for entering the compressor.

Chemical requirements

- The compressor shall be free of oil (oil-free) to avoid its reaction with oxygen.

Mechanical requirements

- The material of the pipes shall be made a Copper (the ideal shall be made a stainless steel)

- The dimensions of the pipes that shall be:

Diameters are listed in the figure below:

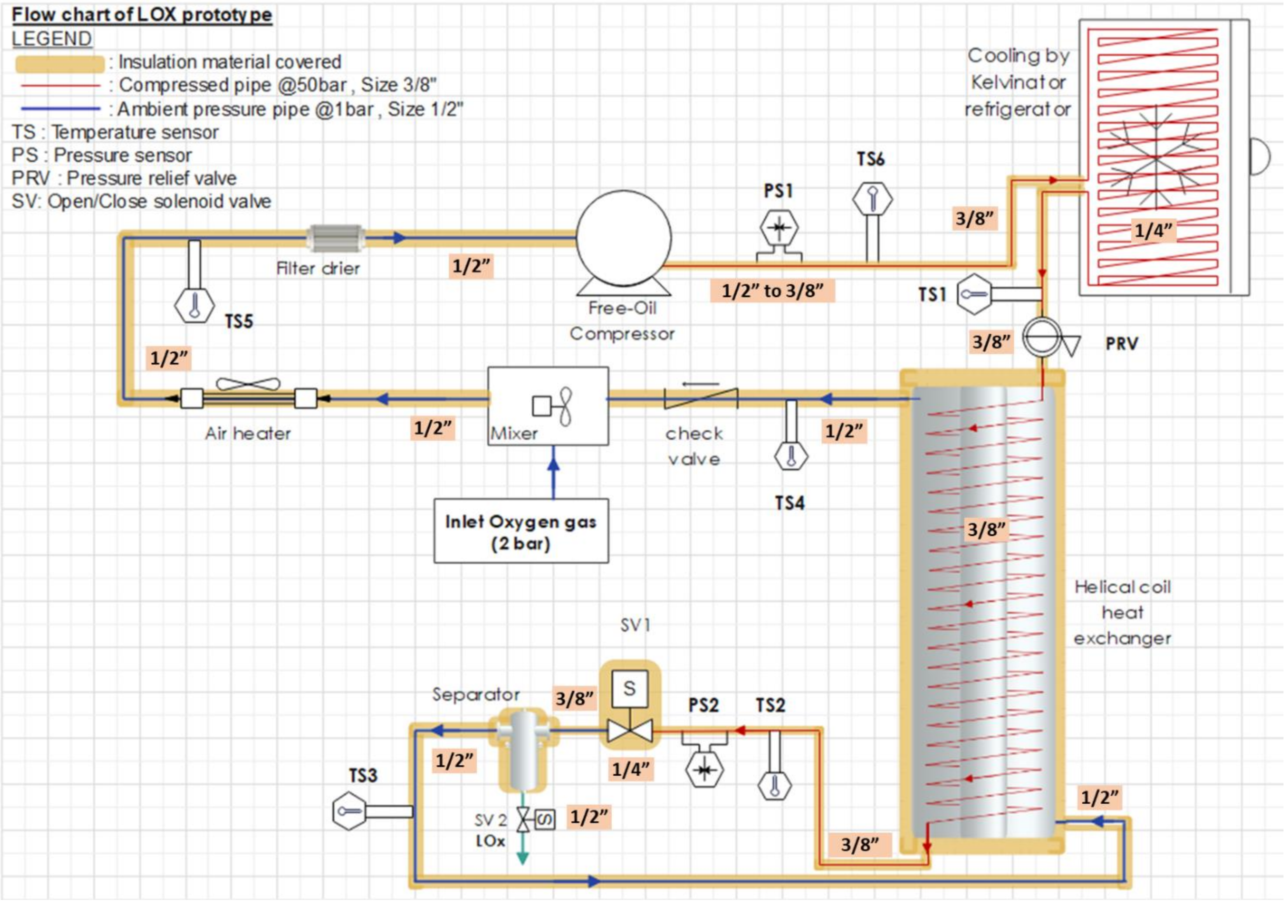


Fig-2-

- The compressor shall be able to increase the pressure of the gas to the required level, from -15°C (258K) @1bar (inlet temperature) - ambient temperature @50bar (outlet pressure).

- The sensors shall be able to measure in the temperature and pressure range, shows in Fig -1-.

- The expansion valve (solenoid valve with separator) shall be able to drop the pressure abruptly, from 50 bar to 1 bar.

- The mixer shall be able to introduce an appropriate amount of fresh oxygen gas into the system; the amount of fresh oxygen gas added shall be equal the amount of liquid oxygen, taking into account the effect of the temperature difference.

- The filter drier shall be able to dry the oxygen gas before entering the compressor.

- The thermal insulation material shall be able to isolate pipes and components from any heat leakage.

- The thermal insulation material shall be made a fiber glass (the ideal thermal insulation shall be made a Flexible EPDM).

- The separator shall be made of stainless steel.

- The LOx system shall be design according to " LOx Mechanical Design ".

Automation requirements

- The sensors shall be able to be controlled from the GUI.

- The valves shall be able to be controlled from the GUI.

5.7.5 ICPT LOX Compressor Development[12]

to be done:

- Specification of Oil Free Compressor for LOX Testrig (M. El Rez)

- Design of Oil Free Compressor for LOX Testrig (M. El Rez/J.Bachir)

- Manufactoring Oil Free Compressor for LOX Testrig (J. Bachir)

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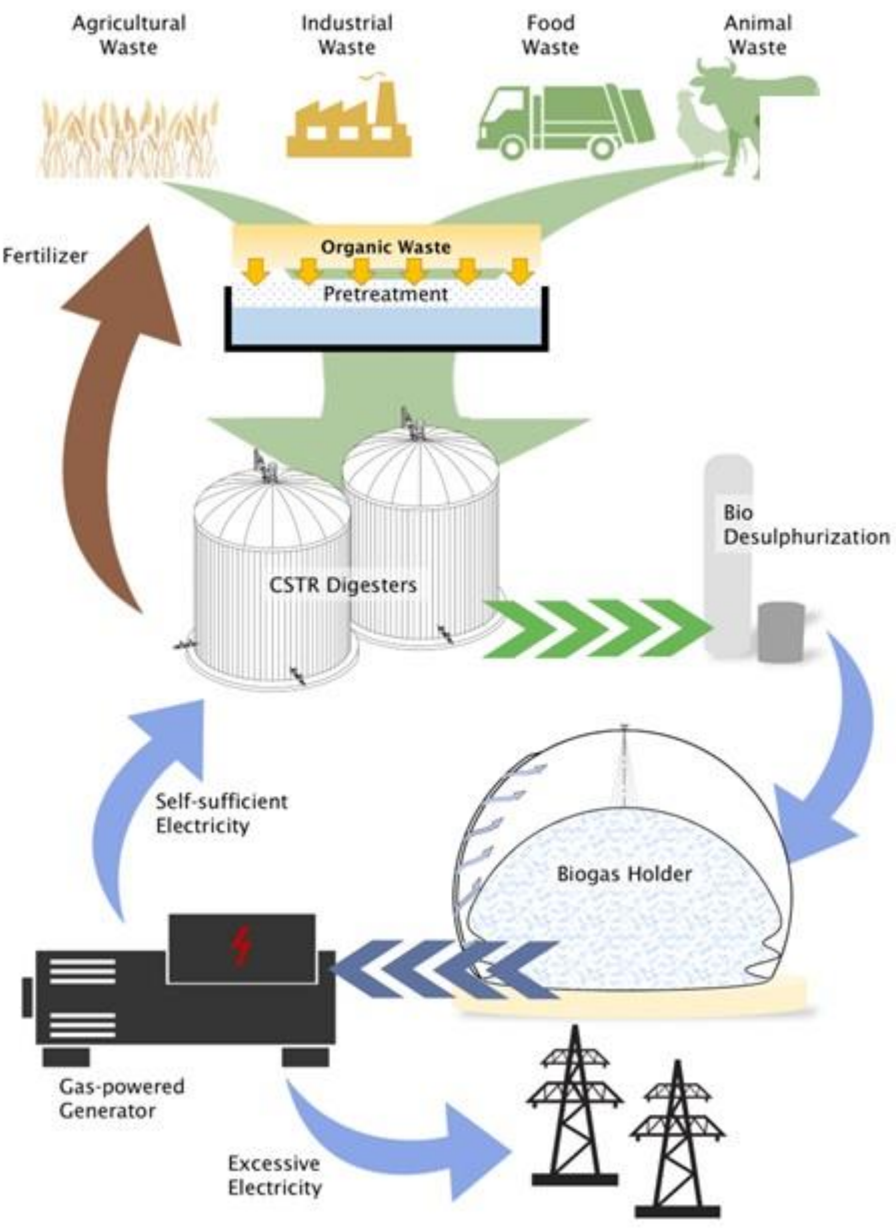
[12] Ref : https://aecenar.com/index.php/institutes/icpt/liquefication-of-air-and-oxygen/icpt-lox-compressor-development

6 Mobile Biogas Generation and Gas Turbine Testrig[13]

Biogas production from municipal waste



From Biogas Plant Development Handbook | BiogasWorld:

6.1 Wet Digestion Technology

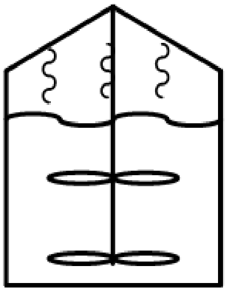
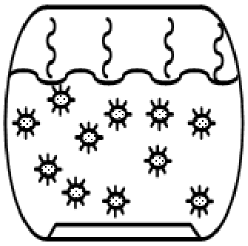
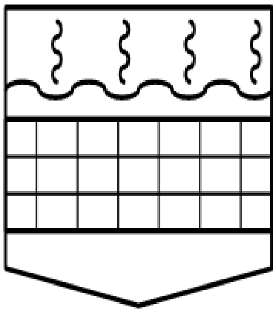
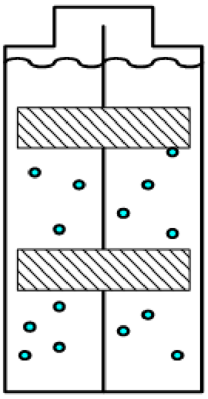
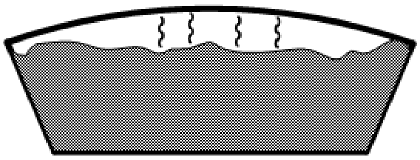
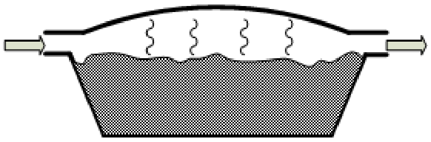
The process is considered wet digestion when the content of the digester is pumpable. That means that the material inside the digester has a consistency of approximately 10% dry matter or less (90% water).

---

[13] **Ref :** https://www.aecenar.com/index.php/institute-projects/nlap-wedc/mobile-biogas-generation-and-gas-turbine-testrig/biogas-production-from-municipal-waste

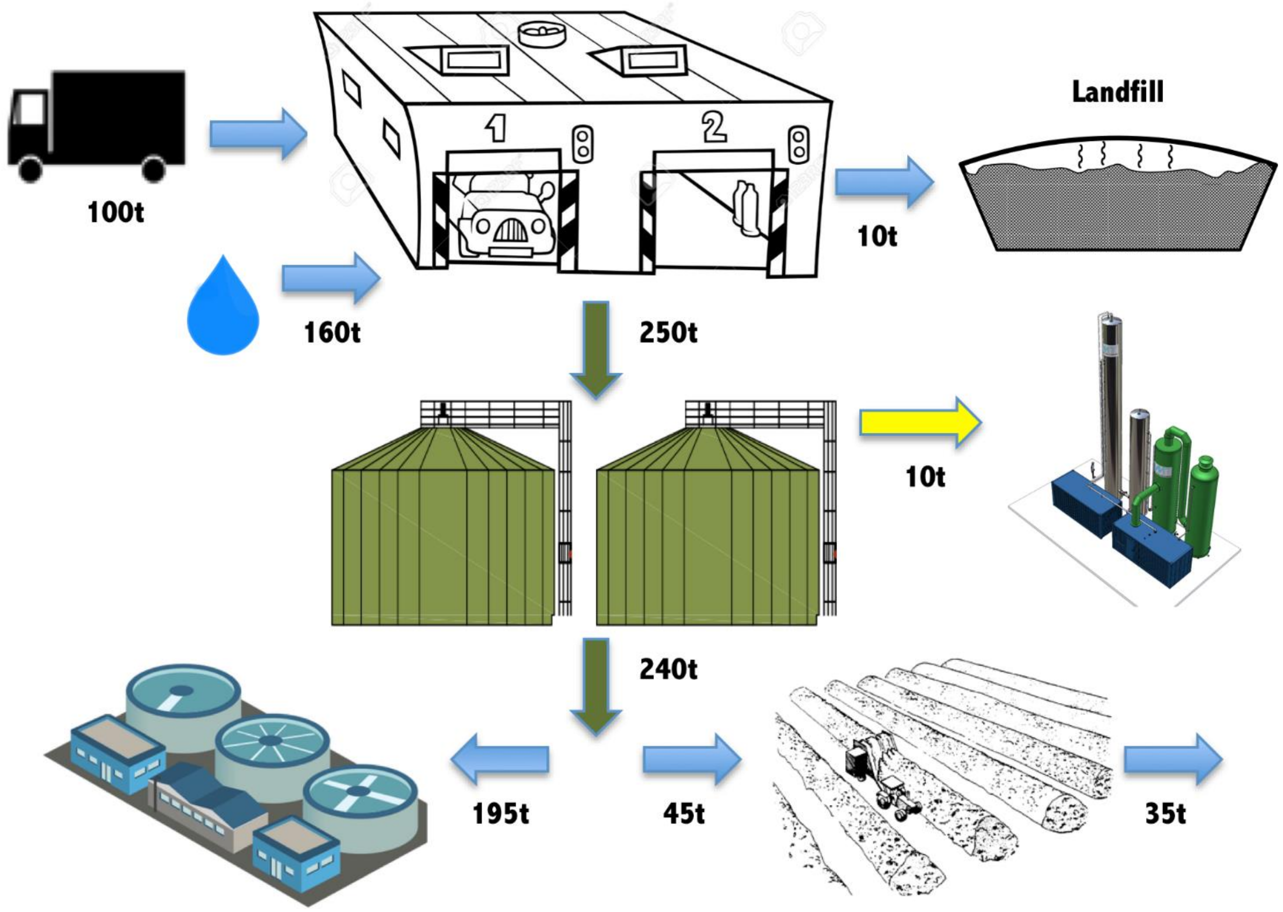
There exist many configurations of **wet digesters**:

- Complete mix or Completely stirred tank reactor (CSTR)
- Plug-flow
- Upflow Anaerobic Sludge Blanket (UASB)
- Fixed film reactor
- Floating films reactors
- Sludge bed reactors
- And more.

These configurations have been designed to optimize the process for various feedstock conditions and market applications.

The mass balance of a typical wet digestion process looks like this:



For example, 100 tonnes of solid municipal residential source separated organics (SSO) arrives at the biogas plant using wet digestion (complete mix). This feedstock needs to be pretreated to remove potential contaminants (plastics, metal, sands, etc.). Approximately 10 tonnes will be removed as contaminants and will probably be landfilled.

In order to be pumpable (10% TS), the feedstock will be diluted with water that may come from a fresh source or from a mixed of fresh and recycled water from the wastewater portion of the biogas plant. The liquid feedstock going to the digester will be approximately 250 tonnes.

In the digesters, the bacteria will consume the majority of the volatile solids in the feedstock and will convert them into biogas. Approximately 10 tonnes of gas will come out of the digesters. The more liquid digestate will represent approximately 240 tonnes.

At this point, the digestate may be applied to land directly. Please note that 100 tonnes of solid material turned into 240 tonnes of liquid and land applying the digestate in this form will present significant transportation costs.

The digestate may also be separated into a solid fraction (45 tonnes) to be land applied (or composted down to 35 tonnes) and a liquid fraction (195 tonnes) to be returned the sanitary sewage or directly back to nature.

One may be tempted to use the treated wastewater as dilution water for the input feedstock and limit the amount of water consumed and rejected by the process. It is possible only if the wastewater plant removes almost all nutrients (salts and ammonia/ammonium) in the water. Without this removal, there will be a rapid build up of nutrients in the water and this will inhibit and/or kill the anaerobic digestion process.

Requirements

No matter which design is chosen, the digester (fermentation tank) must meet the following requirements:

- **Water/gas-tightness** - watertightness in order to prevent seepage and the resultant threat to soil and groundwater quality; gas-tightness in order to ensure proper containment of the entire biogas yield and to prevent air entering into the digester (which could result in the formation of an explosive mixture).

- **Insulation** - if and to which extent depends on the required process temperature, the local climate and the financial means; heat loss should be minimized if outside temperatures are low, warming up of the digester should be facilitated when outside temperatures are high.

- **Minimum surface area** - keeps cost of construction to a minimum and reduces heat losses through the vessel walls. A spherical structure has the best ratio of volume and surface area. For practical construction, a hemispherical construction with a conical floor is close to the optimum.

- **Structural stability** - sufficient to withstand all static and dynamic loads, durable and resistant to corrosion.]

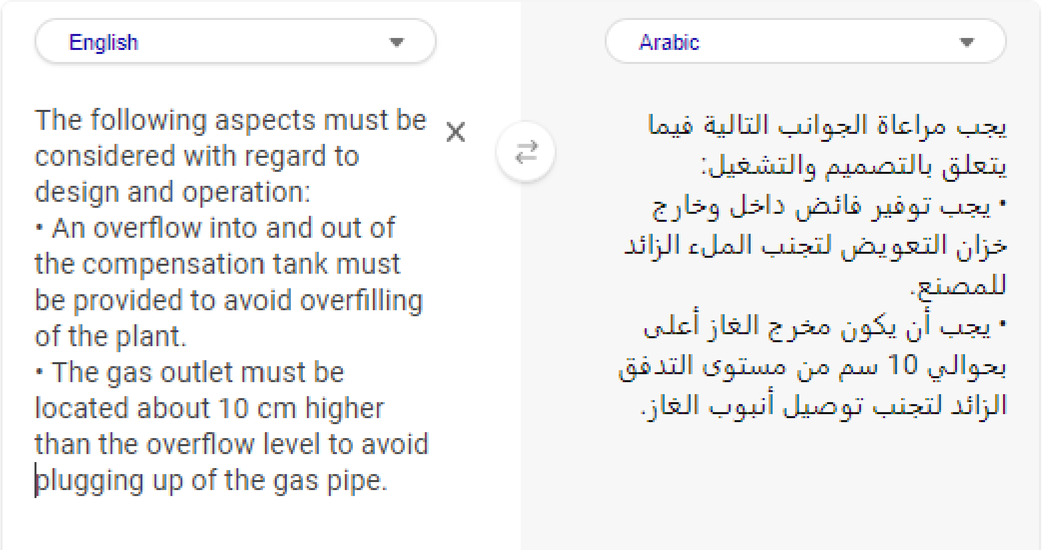
بغض النظر عن التصميم الذي يتم اختياره ، يجب أن يفي الهاضم (خزان التخمير) بالمتطلبات التالية:

- **المياه / النفاذية** - مقاومة الماء من أجل منع التسرب وما ينتج عن ذلك من تهديد لنوعية التربة والمياه الجوفية ؛ مقاومة النفاذية من أجل ضمان الاحتواء المناسب لكامل محصول الغاز الحيوي ولمنع دخول الهواء إلى الهاضم (مما قد يؤدي إلى تكوين خليط متفجر).

- **العزل** - إذا كان وإلى أي مدى يعتمد على درجة حرارة العملية المطلوبة ، والمناخ المحلي والوسائل المالية ؛ يجب تقليل فقد الحرارة إلى أدنى حد إذا كانت درجات الحرارة الخارجية منخفضة ، ويجب تسهيل تسخين الهاضم عندما تكون درجات الحرارة الخارجية مرتفعة.

- **الحد الأدنى من مساحة السطح** - يحافظ على تكلفة البناء إلى أدنى حد ويقلل من فقد الحرارة عبر جدران الوعاء. يحتوي الهيكل الكروي على أفضل نسبة من حيث الحجم ومساحة السطح. بالنسبة للبناء العملي ، فإن البناء النصف كروي بأرضية مخروطية يكون قريبًا من المستوى الأمثل.

- **الاستقرار الهيكلي** - كافٍ لتحمل جميع الأحمال الساكنة والديناميكية ، ودائم ومقاوم للتآكل.

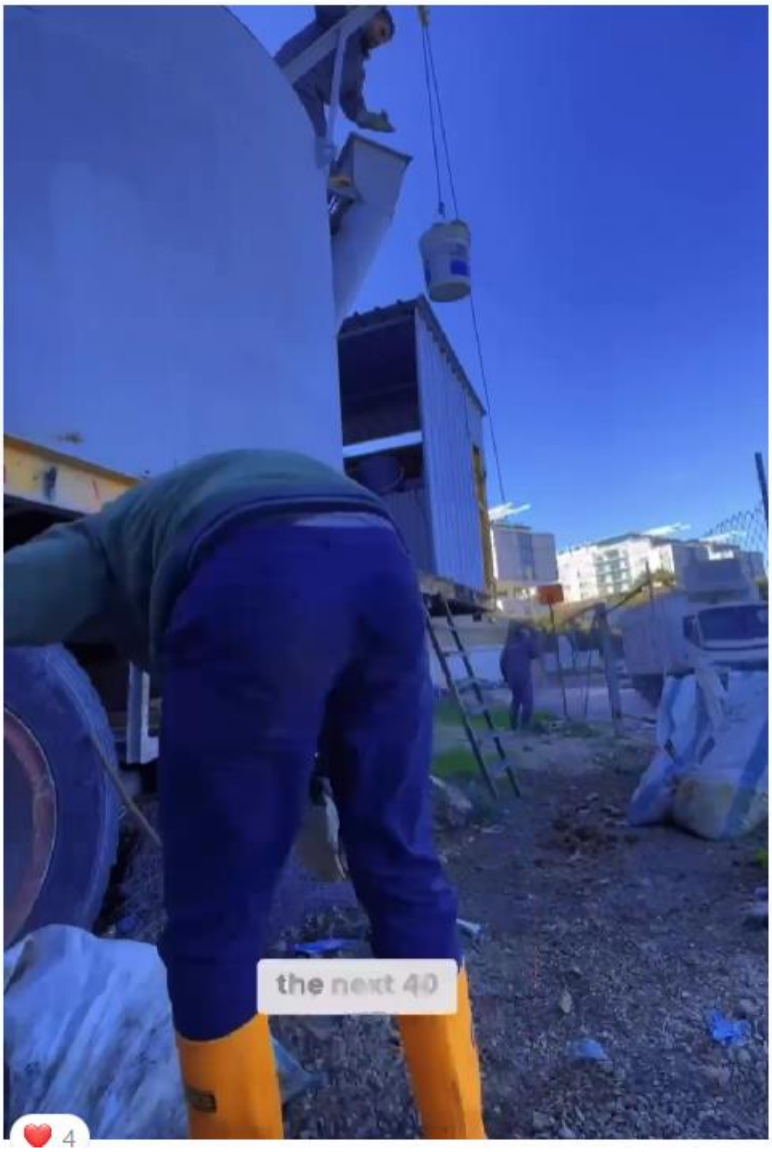


The following aspects must be considered with regard to design and operation:
- An overflow into and out of the compensation tank must be provided to avoid overfilling of the plant.
- The gas outlet must be located about 10 cm higher than the overflow level to avoid plugging up of the gas pipe.

يجب مراعاة الجوانب التالية فيما يتعلق بالتصميم والتشغيل:
- يجب توفير فائض داخل وخارج خزان التعويض لتجنب الملء الزائد للمصنع.
- يجب أن يكون مخرج الغاز أعلى بحوالي 10 سم من مستوى التدفق الزائد لتجنب توصيل أنبوب الغاز.

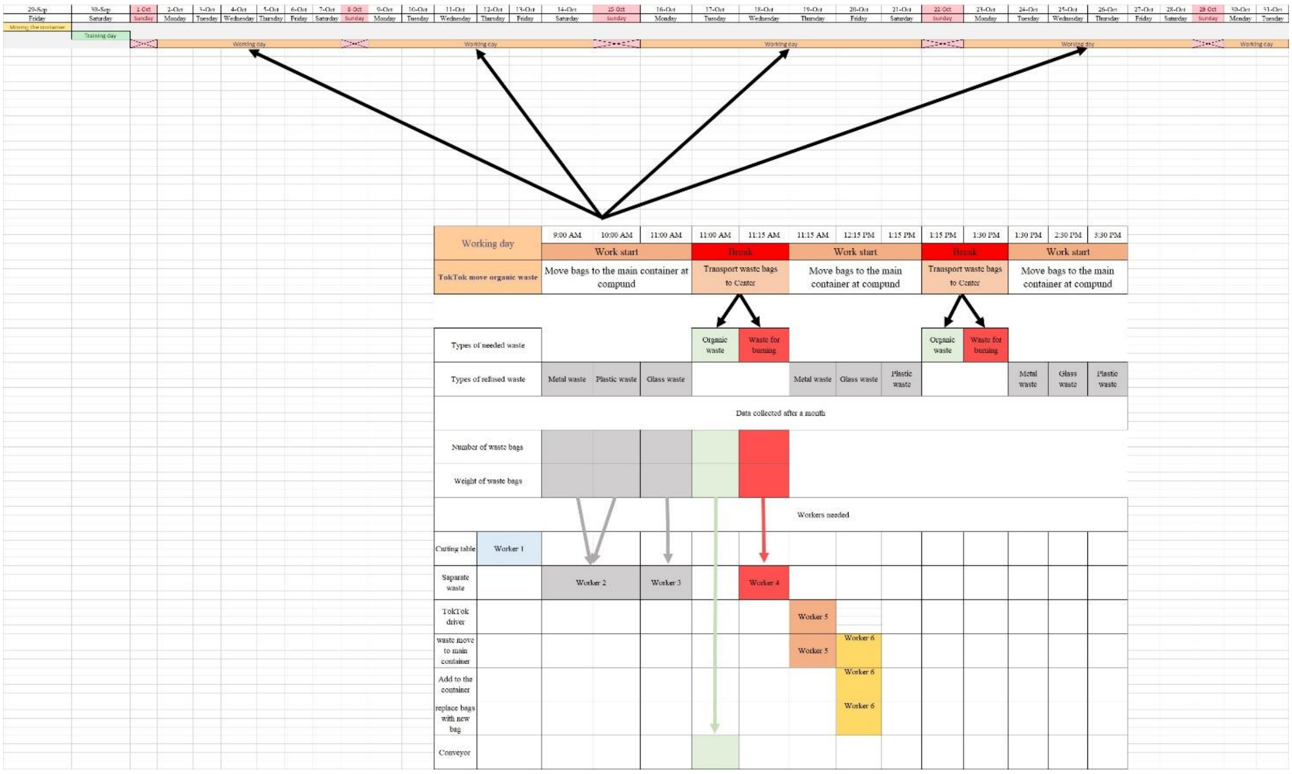
6.2 Biogas Generation Tests and Data

to be documented


the next 40
4

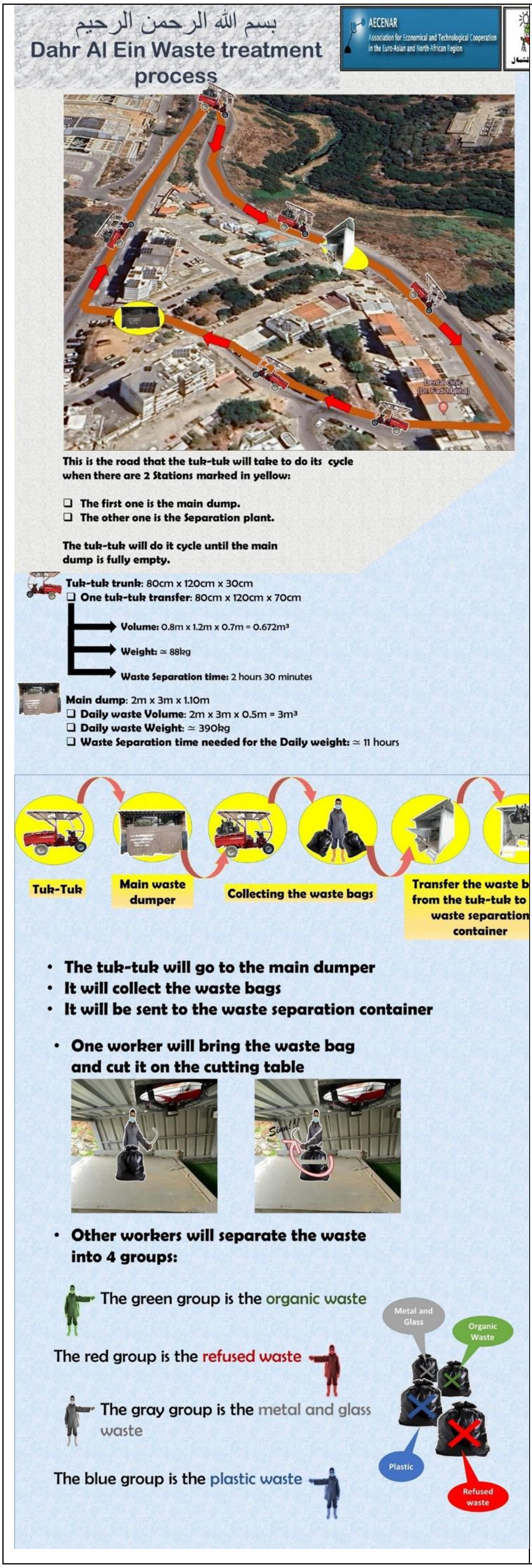
7 Waste Treatment at Ras Maska in October 2023

7.1 Planning



29-Sep	30-Sep	1 Oct	2-Oct	3-Oct	4-Oct	5-Oct	6-Oct	7-Oct	8 Oct	9-Oct	10-Oct	11-Oct	12-Oct	13-Oct	14-Oct	15 Oct	16-Oct	17-Oct	18-Oct	19-Oct	20-Oct	21-Oct	22 Oct	23-Oct	24-Oct	25-Oct	26-Oct	27-Oct	28-Oct	29 Oct	30-Oct	31-Oct
Friday	Saturday	Sunday	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday	Monday	Tuesday
Moving the container																																
	Training day																															
			Working day							Working day							Working day							Working day							Working day	

Working day		9:00 AM	10:00 AM	11:00 AM	11:00 AM	11:15 AM	11:15 AM	12:15 PM	1:15 PM	1:15 PM	1:30 PM	1:30 PM	2:30 PM	3:30 PM
		Work start			Break		Work start			Break		Work start		
TokTok move organic waste		Move bags to the main container at compund			Transport waste bags to Center		Move bags to the main container at compund			Transport waste bags to Center		Move bags to the main container at compund		
Types of needed waste					Organic waste	Waste for burning				Organic waste	Waste for burning			
Types of refused waste		Metal waste	Plastic waste	Glass waste			Metal waste	Glass waste	Plastic waste			Metal waste	Glass waste	Plastic waste
Data collected after a month														
Number of waste bags														
Weight of waste bags														
Workers needed														
Cutting table	Worker 1													
Saparate waste		Worker 2		Worker 3		Worker 4								
TokTok driver							Worker 5							
waste move to main container							Worker 5	Worker 6						
Add to the container								Worker 6						
replace bags with new bag								Worker 6						
Conveyor														



بسم الله الرحمن الرحيم

Dahr Al Ein Waste treatment process

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

This is the road that the tuk-tuk will take to do its cycle when there are 2 Stations marked in yellow:

- ☐ **The first one is the main dump.**
- ☐ **The other one is the Separation plant.**

The tuk-tuk will do it cycle until the main dump is fully empty.

Tuk-tuk trunk: 80cm x 120cm x 30cm
- ☐ **One tuk-tuk transfer**: 80cm x 120cm x 70cm
  - **Volume:** 0.8m x 1.2m x 0.7m = 0.672m³
  - **Weight:** ≃ 88kg
  - **Waste Separation time:** 2 hours 30 minutes

Main dump: 2m x 3m x 1.10m
- ☐ **Daily waste Volume**: 2m x 3m x 0.5m = 3m³
- ☐ **Daily waste Weight**: ≃ 390kg
- ☐ **Waste Separation time needed for the Daily weight:** ≃ 11 hours

- **The tuk-tuk will go to the main dumper**
- **It will collect the waste bags**
- **It will be sent to the waste separation container**

- **One worker will bring the waste bag and cut it on the cutting table**

- **Other workers will separate the waste into 4 groups:**

The green group is the organic waste

The red group is the refused waste

The gray group is the metal and glass waste

The blue group is the plastic waste

[Dahr Al Ein waste treatment process POSTER (pptx file)](http://aecenar.com/index.php/downloads/send/4-iep-institute/1421-dahr-al-ein-waste-treatment-process-poster-pptx)

http://aecenar.com/index.php/downloads/send/4-iep-institute/1421-dahr-al-ein-waste-treatment-process-poster-pptx

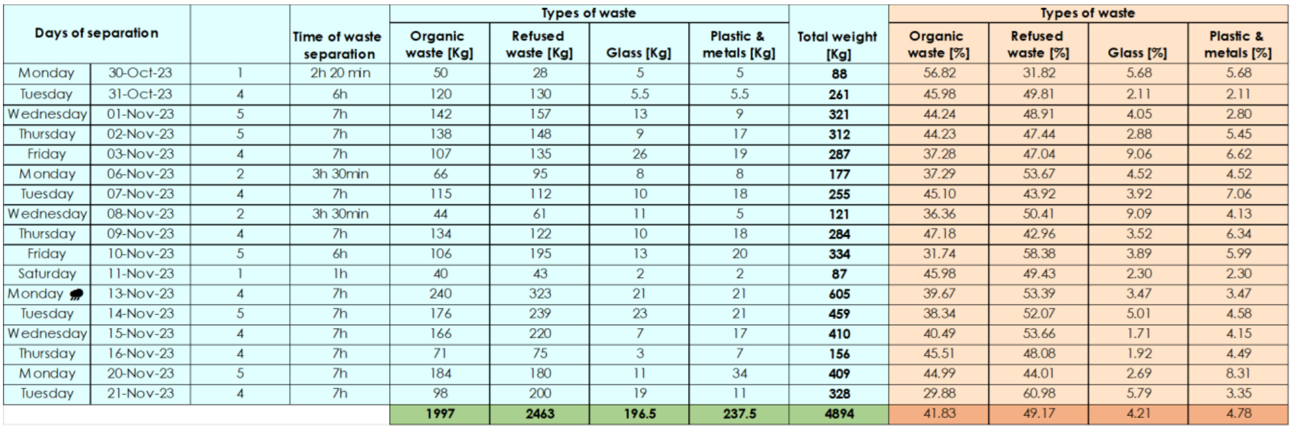
[Dahr Al Ein waste treatment process POSTER (pdf file)](http://aecenar.com/index.php/downloads/send/4-iep-institute/1422-dahr-al-ein-waste-treatment-process-poster-pdf)

http://aecenar.com/index.php/downloads/send/4-iep-institute/1422-dahr-al-ein-waste-treatment-process-poster-pdf

7.2 Results (Waste Collection and Separation Data)

http://aecenar.com/index.php/downloads/send/5-nlap/1444-nlap-waste-separation-data

Dahr Al Ein waste separation data (Excel Sheet)



Days of separation			**Time of waste separation**	**Types of waste**				**Total weight [Kg]**	**Types of waste**			
				Organic waste [Kg]	**Refused waste [Kg]**	**Glass [Kg]**	**Plastic & metals [Kg]**		**Organic waste [%]**	**Refused waste [%]**	**Glass [%]**	**Plastic & metals [%]**
Monday	30-Oct-23	1	2h 20 min	50	28	5	5	**88**	56.82	31.82	5.68	5.68
Tuesday	31-Oct-23	4	6h	120	130	5.5	5.5	**261**	45.98	49.81	2.11	2.11
Wednesday	01-Nov-23	5	7h	142	157	13	9	**321**	44.24	48.91	4.05	2.80
Thursday	02-Nov-23	5	7h	138	148	9	17	**312**	44.23	47.44	2.88	5.45
Friday	03-Nov-23	4	7h	107	135	26	19	**287**	37.28	47.04	9.06	6.62
Monday	06-Nov-23	2	3h 30min	66	95	8	8	**177**	37.29	53.67	4.52	4.52
Tuesday	07-Nov-23	4	7h	115	112	10	18	**255**	45.10	43.92	3.92	7.06
Wednesday	08-Nov-23	2	3h 30min	44	61	11	5	**121**	36.36	50.41	9.09	4.13
Thursday	09-Nov-23	4	7h	134	122	10	18	**284**	47.18	42.96	3.52	6.34
Friday	10-Nov-23	5	6h	106	195	13	20	**334**	31.74	58.38	3.89	5.99
Saturday	11-Nov-23	1	1h	40	43	2	2	**87**	45.98	49.43	2.30	2.30
Monday 🌧	13-Nov-23	4	7h	240	323	21	21	**605**	39.67	53.39	3.47	3.47
Tuesday	14-Nov-23	5	7h	176	239	23	21	**459**	38.34	52.07	5.01	4.58
Wednesday	15-Nov-23	4	7h	166	220	7	17	**410**	40.49	53.66	1.71	4.15
Thursday	16-Nov-23	4	7h	71	75	3	7	**156**	45.51	48.08	1.92	4.49
Monday	20-Nov-23	5	7h	184	180	11	34	**409**	44.99	44.01	2.69	8.31
Tuesday	21-Nov-23	4	7h	98	200	19	11	**328**	29.88	60.98	5.79	3.35
				1997	**2463**	**196.5**	**237.5**	**4894**	41.83	49.17	4.21	4.78

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8 References

- Project 2 _ Water electrolysis references

[1] Straight-Parallel Electrodes and Variable Gap for Hydrogen and Oxygen Evolution Reactions

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– Project 7_ Ashes recycling references

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