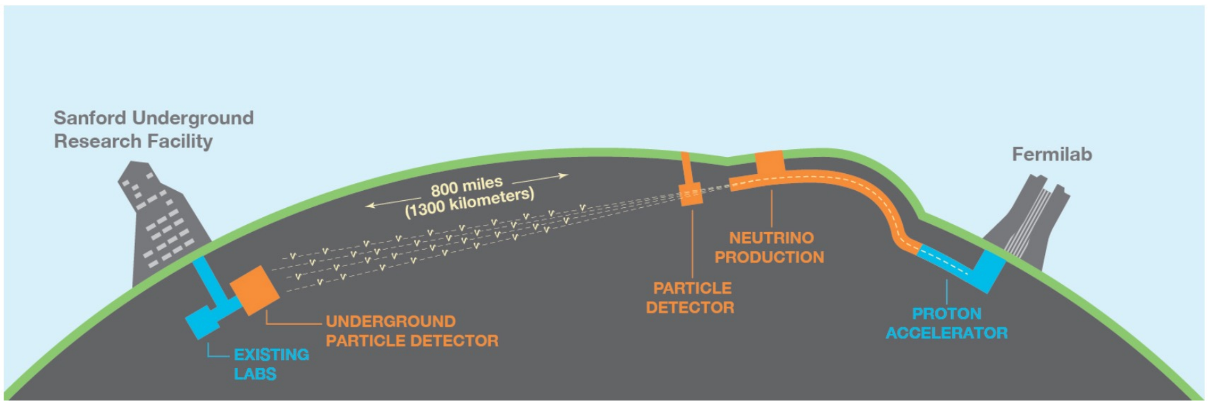
Optimization of Argon Purification, Regeneration and Condensation procedures for Large Scale Facilities for DUNE experiment.



Cris Adriano
on behalf of the Brazilian group

July, 13 2021

IFGW Instituto de Física Gleb Wataghin

UNICAMP

Fermilab

FAPESP

Equatorial Sistemas

akaer group

Collaborators and Partners

FAPESP
2020/01609-2

Fermilab

Equatorial Sistemas

akaer

UNICAMP

IFGW
Instituto de Física Gleb Wataghin

FACULDADE DE ENGENHARIA QUÍMICA
UNICAMP

PQGe

CBPF
Centro Brasileiro de Pesquisas Físicas

USP
são carlos

USP

ufscar

DUNE
DEEP UNDERGROUND NEUTRINO EXPERIMENT

Collaborators and Partners

Fermilab

Roza Doubnik, Mark Adamowski, David Montanari,

UNICAMP

IFGW Instituto de Física Gleb Wataghin

Pascoal Pagliuso, Thiago Alegre, Cris Adriano, Magda Fontes, Ana Machado, Ettore Segreto

akaer group Equatorial Sistemas

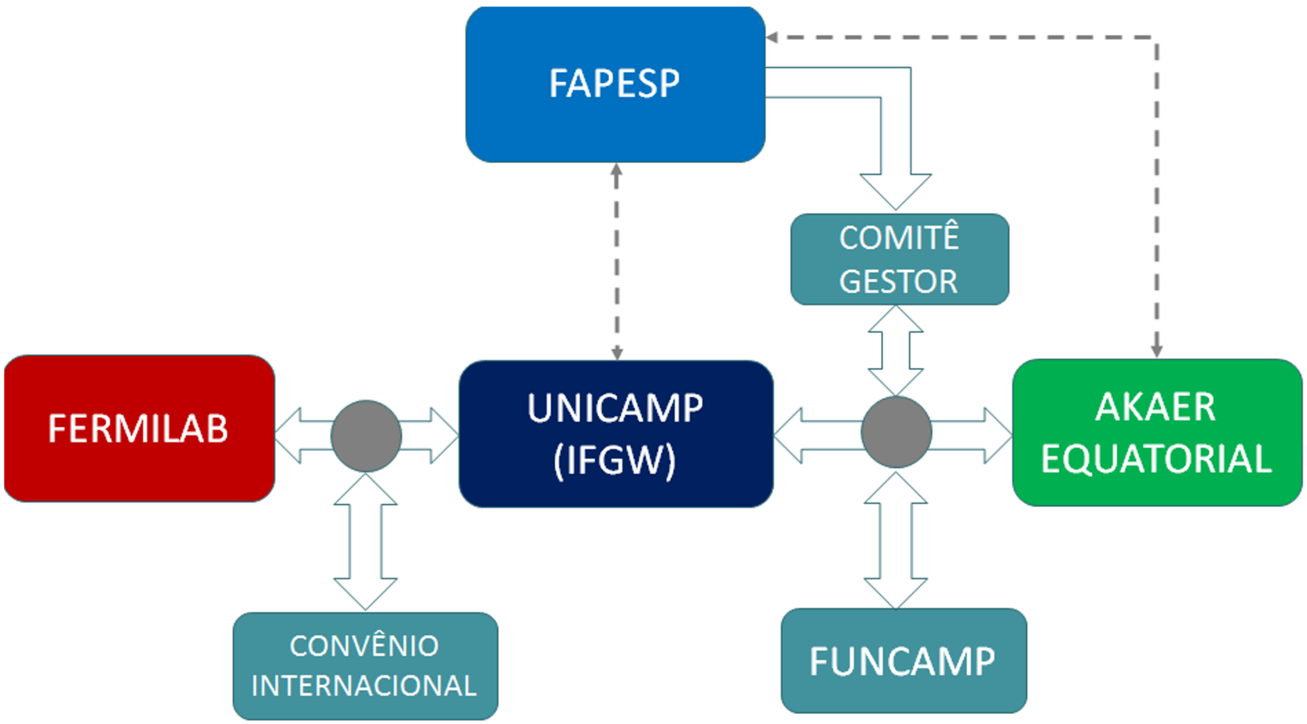
Dr. Cesar Celeste Ghizoni – Executive Director

Eng. Carlos Alberto Nogueira Carreira - System Engineer

MSc. Fernando C. Ferraz - Operational Vice President

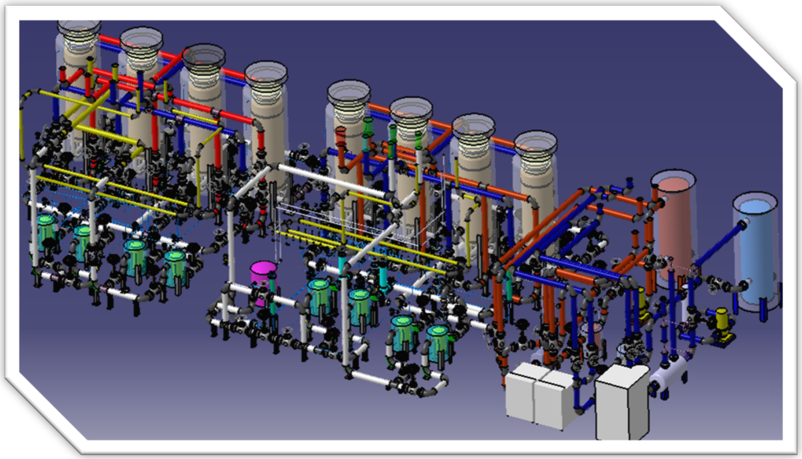
MSc. Joselito Rodrigues Henriques - Diretor - R&D and Innovation

Project's Actions Flow Chart



Project

PHASE 1

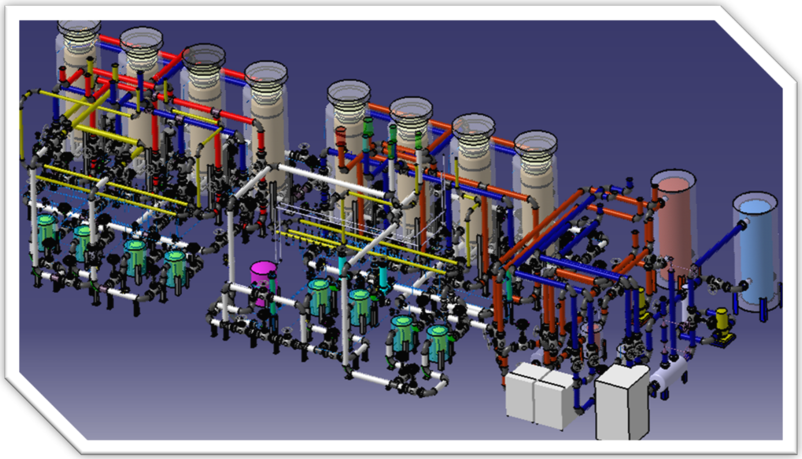


Budget FAPESP for Phase 1:
Temático IFGW-UNICAMP: ~ 4 mi BRL
2 PIPES Grupo Akaer: ~ 2 mi BRL each project

PHASE 2

Project goals

PHASE 1



To structure a team of scientists, engineers and technicians from the IFGW/UNICAMP and from Equatorial/AKAER, assisted by the team of Fermilab's cryogenics (LBNF), to carry out R&D, testing and prototype construction for the optimization of argon purification, regeneration and condensation procedures in large-scale installations, with the aim of generating the scientific and technological base necessary to enable the IFGW/Equatorial, supported by FAPESP, take the lead in the construction of the entire cryogenic system of DUNE detectors near and far, which includes all tanks for filtration / regeneration, storage, etc.

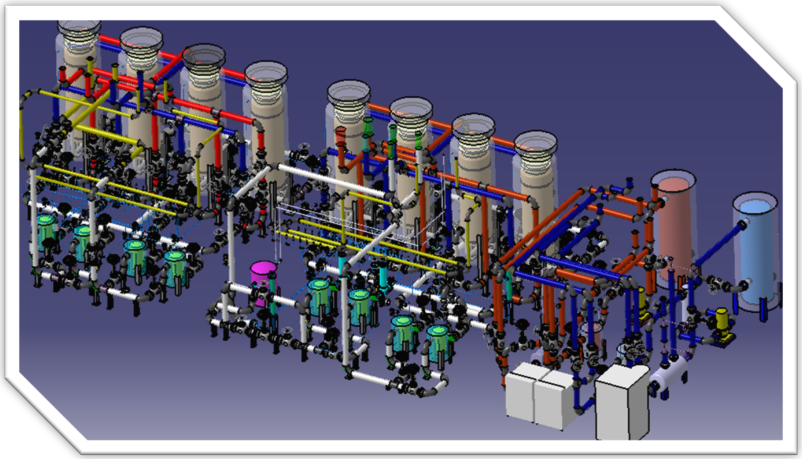
Project goals

PHASE 2 refers to the detailing and supply of the entire large-scale argon purification and regeneration system. This phase will involve different Brazilian companies in different segments such as cryogenics, fine chemicals (at the molecular level), control and automation, logistics, among others.

PHASE 2

Schedule

PHASE 1



03/2020 – ICRADA Signing in Brasilia;
05/2020 – Fapesp R&D opportunities Projects approval
– Agreement Unicamp/Equatorial Signing;
06/2020 – Teams started to work;
New deadline for the end of 1st phase: December, 2021;

Schedule

Budget estimative: 2021
Financial projects: 2022
Industrialization phase: 2022/2023
Potential Suppliers Evaluated: 2023/2024
Acceptance at FermiLab: TBD
Transport, logistic and installation: TBD

PHASE 2

Workshop da RENAFAE
12-14 July 2021

Brazilian Organization – R&D at the Universities

Pascoal Pagliuso
IFGW

Consultants:

Ney Oliveira
(USP)
Luis Ghivelder (UFRJ)

Wilson Ortiz (UFSCAR)
Claudio Magon
(USP-São Carlos)

Thiago Alegre (Simulation)

Gustavo Wiederhecker (IFGW)
Dirceu Noriler (FEQ)
Renato Soccol (FEQ)

Cris Adriano (Media)

Pascoal Pagliuso (IFGW)
José Mansur (UFSCAR)
Dilson Cardoso (UFSCAR)
Elisabete Assaf (USP-SC)
Cristhiane Reis (UFSJ)

Magda Fontes (Cryo-Setup)

Krystal Oliveira (CBPF)
Ettore Segreto (IFGW)

Ana Machado (Purity Monitor)

Frederico Demolin
Heriques Frandini

Brazilian Organization – R&D at the Universities

Tasks of our group

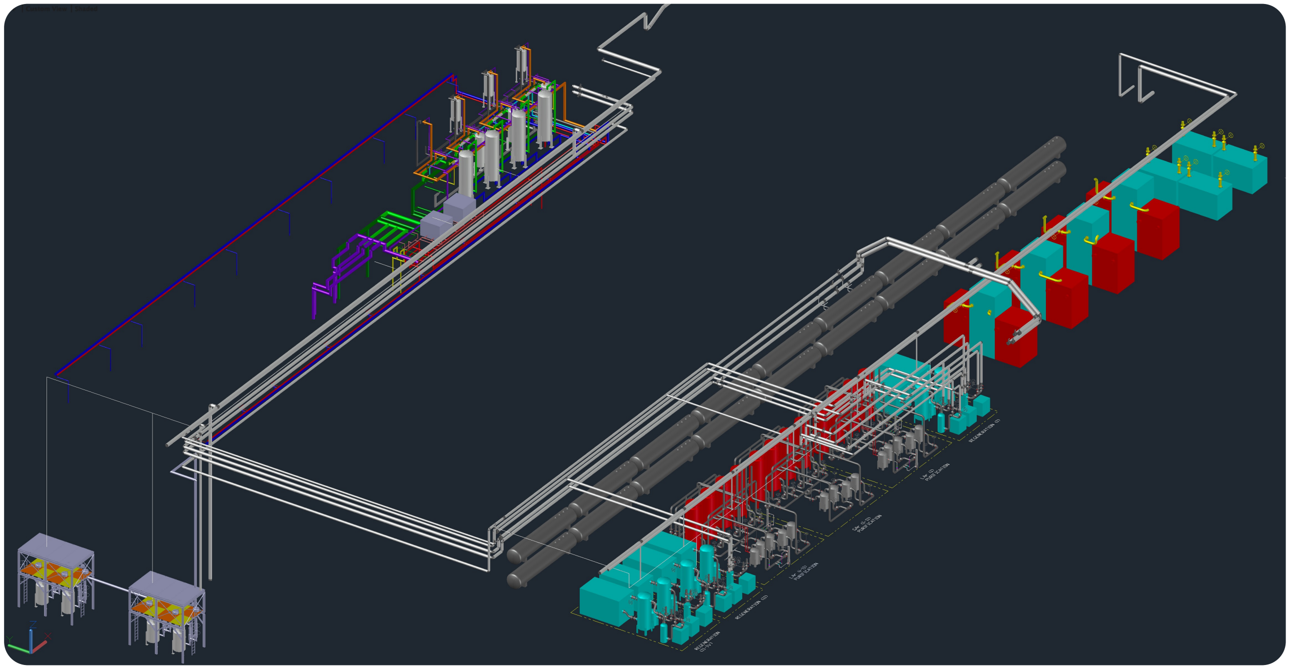
- Performing calculation, simulations and project conceptual design of the Fluid-dynamic LAr circulation with two pumps.
- Proposing, testing and validating the temperature monitoring of purification systems.
- Synthesis, characterization and production (or purchasing) of candidate materials to be used as purification media.
- Proposing, testing and validating the media to be used in the purification systems.
- Production and test of small scale prototypes
- Testing materials, pumps and piping for gas/liquid circulation and storage in reduced-size model systems.
- Production and construction of purity monitors and monitoring system. Contribution to slow control development

Computacional Fluid Dynamics (CFD) Simulations

Thiago Alegre (Simulation)

Gustavo Wiederhecker (IFGW)
Dirceu Noriler (FEQ)
Renato Soccol (FEQ)

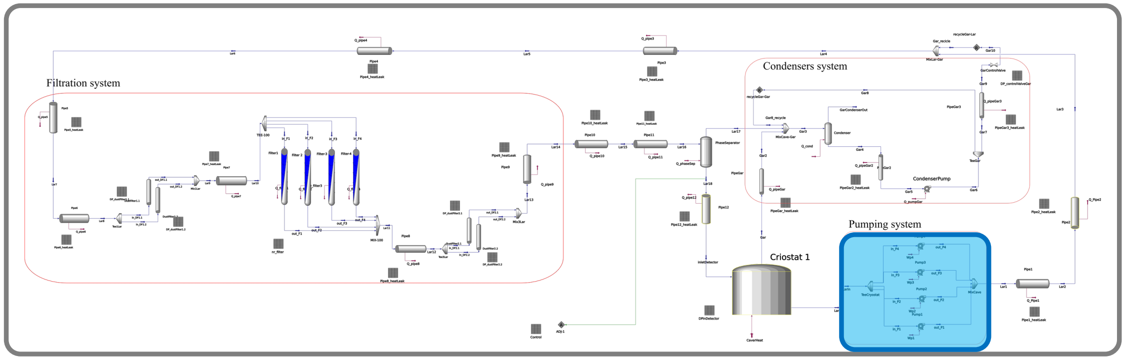
3D-Piping Modeling



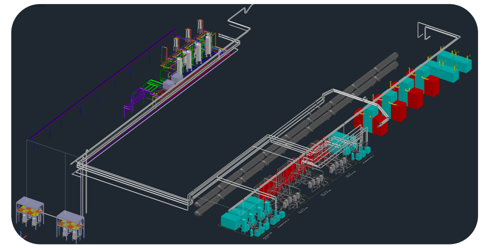
Source: https://edms.cern.ch/file/2254565/1/3D_model_in_AutoCAD._Cryogenics_in_CUC_and_DC.dwg

Energy Mass Balance Simulations

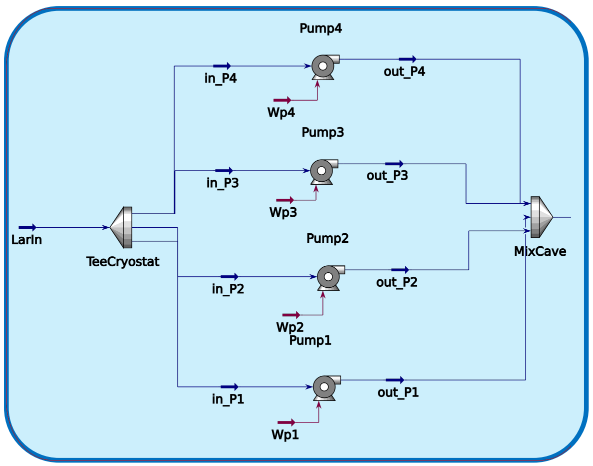
Process Simulation



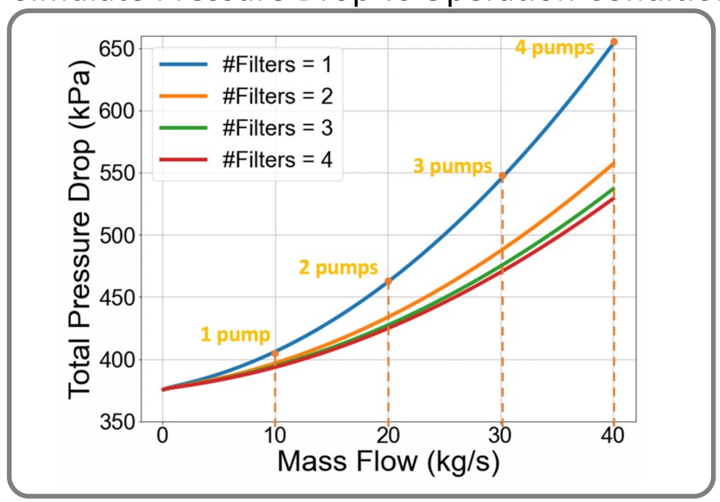
3D-Piping Modeling



Pumping System



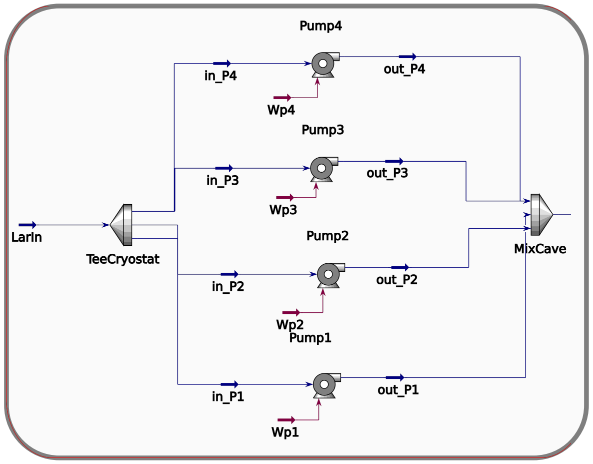
Simulate Pressure Drop vs Operation Condition



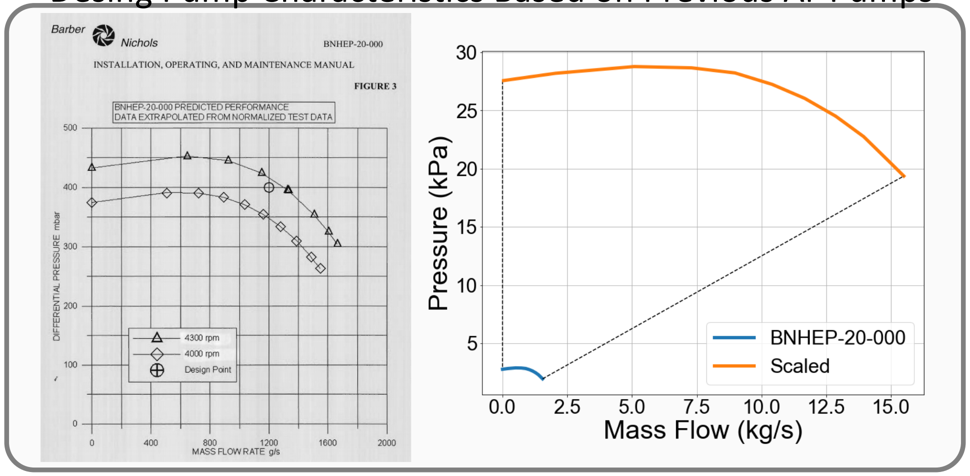
Workshop da RENAFAE
12-14 July 2021

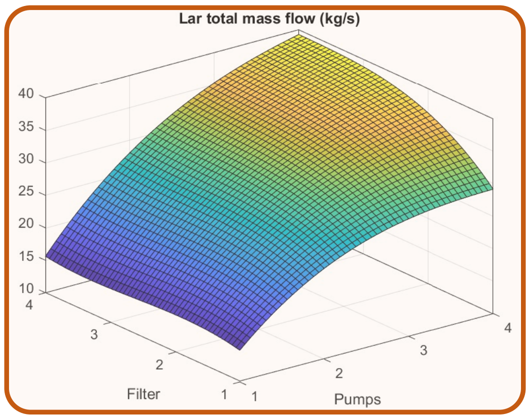
Energy Mass Balance Simulations

Pumping System



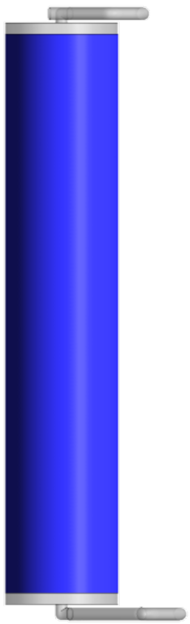
Desing Pump Characteristics Based on Previous Ar-Pumps





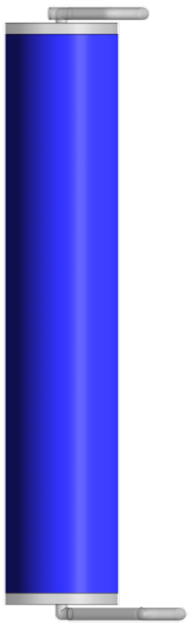
Process simulations allowed for ***designing*** the Liquid Argon (LAr) pump for cryo-system. These pumps bare similar characteristics then previously fabricated pumps (proto-DUNE). On the left the predicted LAr mass-flow (kg/s) for different combinations of the number of pumps and the number of LAr filters.

CFD Simulation: Lab Scale LAr Filter



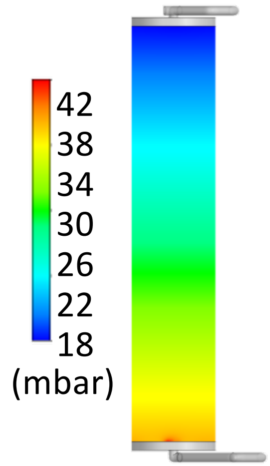
Porous Domain (in Blue):
Particle size: 1mm
Length = 480 mm
Diameter = 95.5 mm

CFD Simulation: Lab Scale LAr Filter



Porous Domain (in Blue):
Particle size: 1mm
Length = 480 mm
Diameter = 95.5 mm

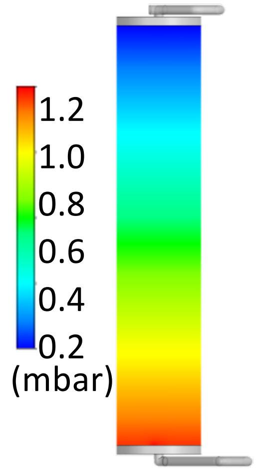
Commercial Pump



Flow: 4.3 liters/min.

Barber Nichols

Bellows Pump



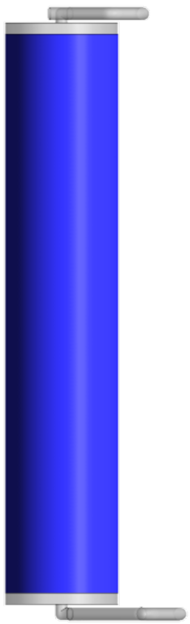
Flow: 0.4 liters/min.

K Mavrokoridis et al., JINST **6** (2011)

Exploring two different scenarios: using a commercial pump from Barber & Nichols (with high flow rate) or using a in-house fabricated pump based on bellows tubing (with low flow rate).

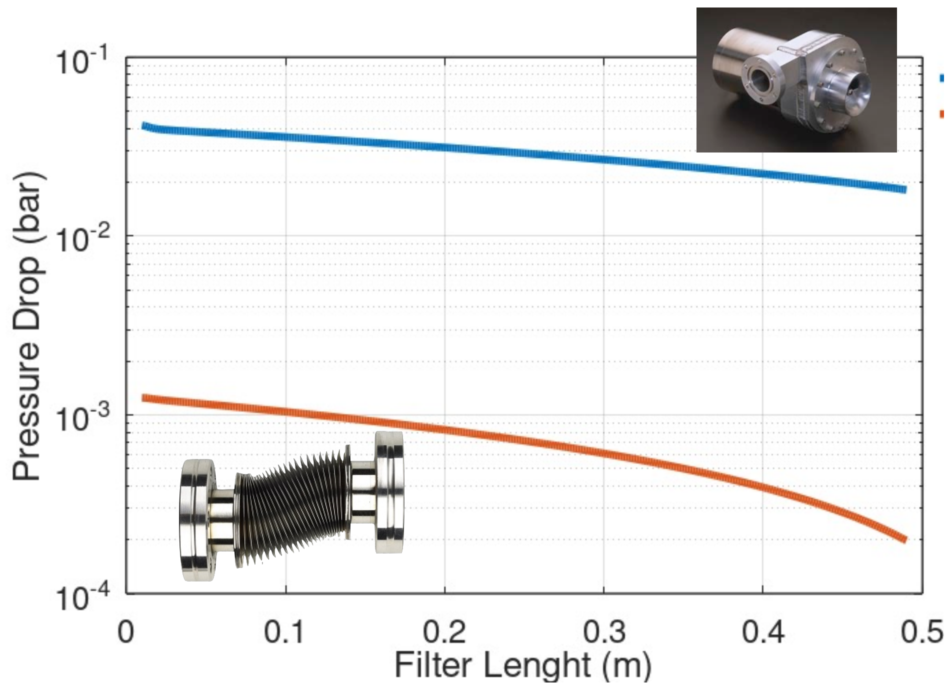
Workshop da RENAFAE
12-14 July 2021

CFD Simulation: Lab Scale LAr Filter

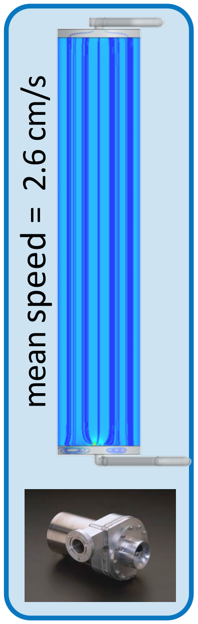


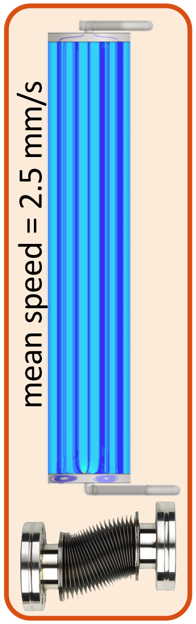
Porous Domain (in Blue):
Particle size: 1mm
Length = 480 mm
Diameter = 95.5 mm

In both cases the evaluated pressure drop is negligeable

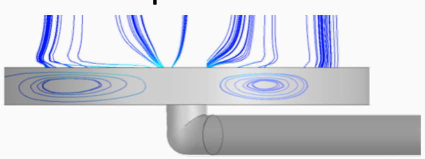


CFD Simulation: Lab Scale LAr Filter



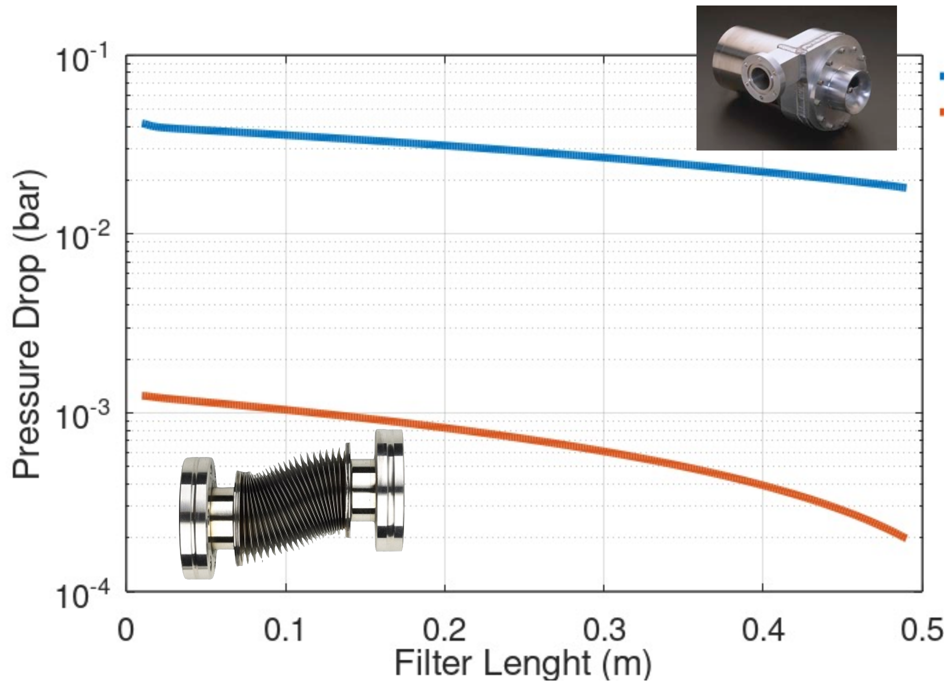


recirculation on gap before porous media



(flow distribution with no dead zones)

In both cases the evaluated pressure drop is negligeable



Media Research

Two small companies created to support the project

Cris Adriano
(Media)

Pascoal Pagliuso (IFGW)
José Mansur (UFSCAR)
Dilson Cardoso (UFSCAR)
Elisabete Assaf (USP-SC)
Cristhiane Reis (UFSJ)

Activated-copper-coated alumina granules research group:

- New routes to improve the dispersion of the active phase (Cu^0) to oxygen capture during the argon purification process;
- New materials such as CuO/CeO on Al_2O_3 granules;
- Thus, we propose the use of the impregnation of Cu on Al_2O_3 by the sol-gel method, using copper salt and poly (ethylene oxide) polymer (PEO) as a dispersing agent and co-precipitation of layered double hydroxide (LDH) as an intermediate structure to increase the dispersion of the active phase Cu^0;
- For comparison, BASF commercial copper material (Cu-02265) is used as a reference;

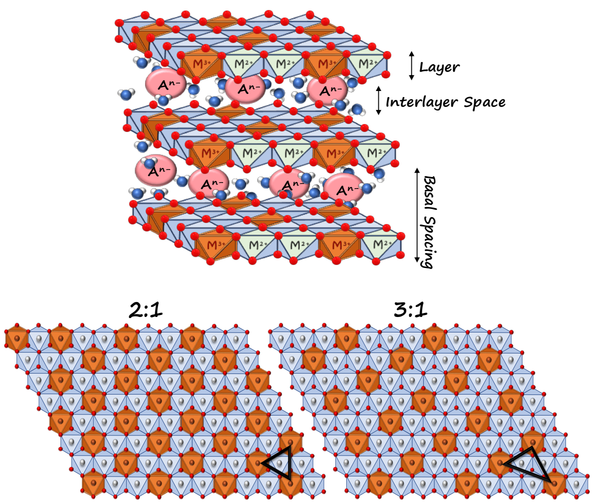
Molecular Sieve research group

- The objective is to study other types of molecular sieve grains containing Ca (5A LTA) and Li as compensation cation and Faujasite zeolite (FAU), to characterize and validate their properties for use in drying argon;
- The specific objective is to test modification int the zeolite structure to increase nitrogen adsorption that can contaminate LAr;
- For comparison, commercial LTA 4A is used as a reference;

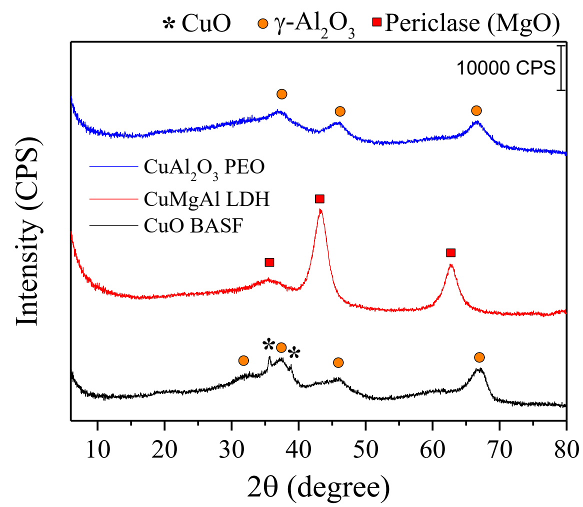
Media Research

Activated-copper-coated alumina granules research group

Layered Double Hydroxide (LDH):



The idealized crystal structure of LDH with different M^{2+}:M^{3+} molar.

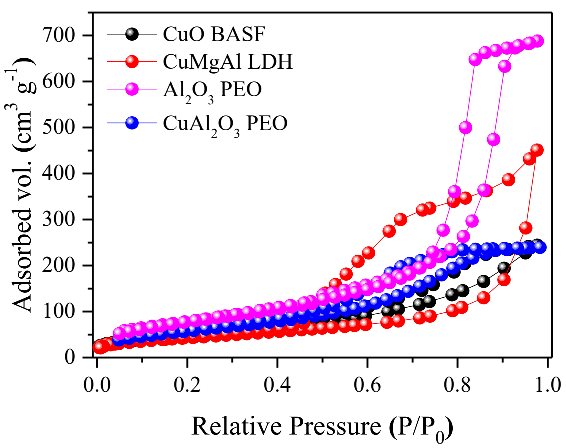


XRD patterns of CuO BASF, CuMgAl LDH and $CuAl_2O_3$ PEO.

Media Research

Activated-copper-coated alumina granules research group

N_2 adsorption/desorption isotherms



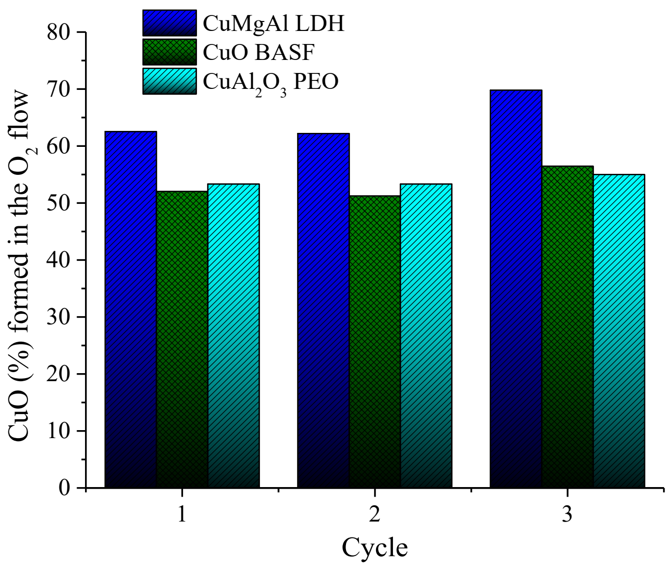
Surface area and porosity

Sample	Specific surface ($m^2 g^{-1}$)	Pore volume ($cm^3 g^{-1}$)	Average pore size (nm)
CuO BASF	166	0.39	3.5
CuMgAl LDH	152	0.92	3.8
Al_2O_3 PEO	286	1.12	10
$CuAl_2O_3$ PEO	200	0.38	5.4

Media Research

Activated-copper-coated alumina granules research group

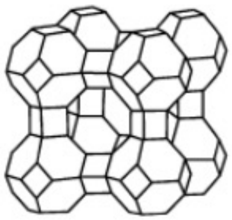
O_2 capture capacity through the oxidation of the Cu^0 species ($2Cu^0 + O_2 \rightarrow 2CuO$)



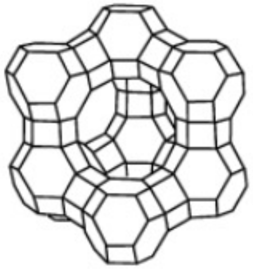
- The preliminary results obtained show that the CuMgAl LDH and $CuAl_2O_3$ PEO samples prepared by our group have excellent structural characteristics such as good dispersion of the CuO phase, high porosity, and high O_2 capture capacity.
- By comparison with the commercial CuO BASF it was observed that the sample $CuAl_2O_3$ PEO presented similar results regarding the capture of O_2 and that the CuMgAl LDH sample has the highest O_2 capture capacity among these evaluated materials.

Media Research

Molecular Sieve research group



ZEOLITE 5A



ZEOLITE FAU (X and Y)

- Normally they are synthesized in the sodium form 4A, when substituting sodium for calcium results in the form 5A.
- The effective pore diameter depend on the type of compensation cation. In this case, the diameter will be 5Å;
- There is still the option of synthesize using the cation Li, which result in diameter of about 6 Å;

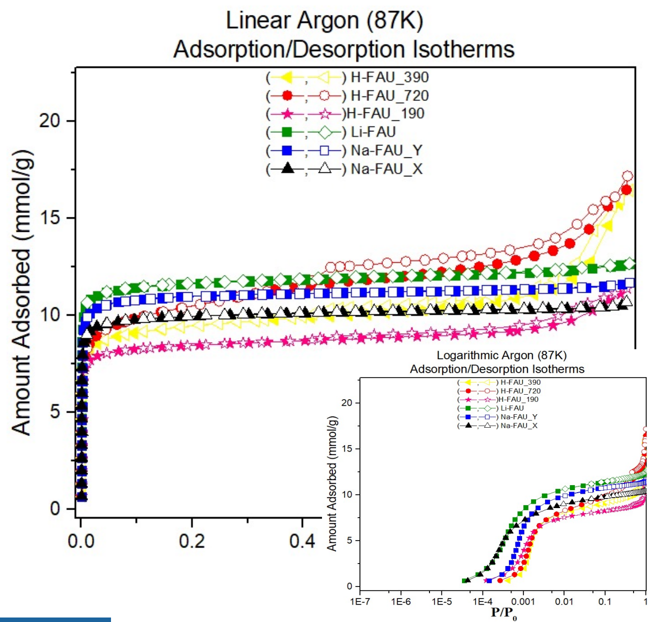
- Widely used in adsorption due to their stable crystalline structures and to have large pore volumes.
- A FAU structure comprises zeolites Y and X, which differentiate them as Si / Al ratios (X = 1.0 - 1.5 and Y above 2.5).

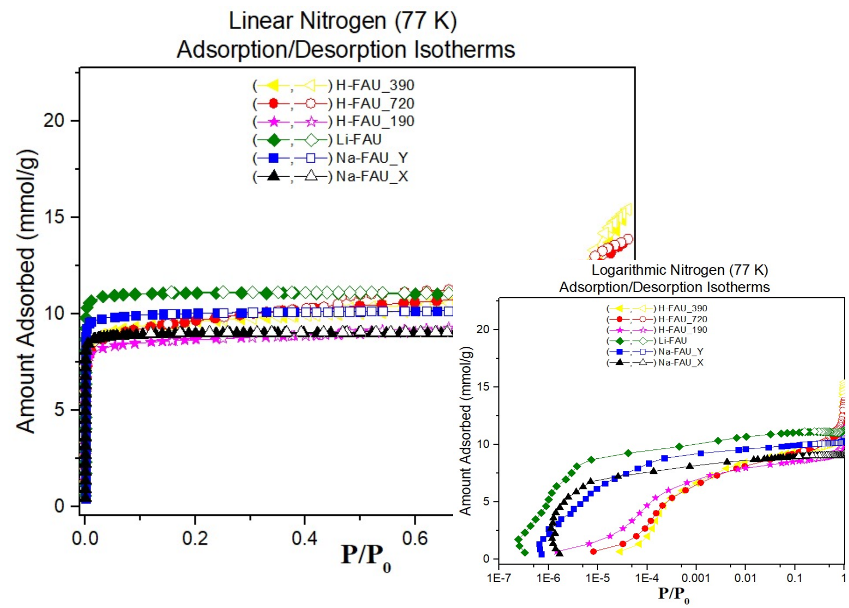
- In our research has been verified that the LTA zeolites structure neither nitrogen nor argon can be adsorbed by zeolite 4A (Na-LTA) at their cryogenic temperatures;
- The zeolite 5A (Ca-LTA) can adsorb both molecules because of the polar molecule of nitrogen with calcium ions present in zeolite 5A, in this sense adsorbent has a preference for nitrogen the adsorption in comparison to argon.

Media Research

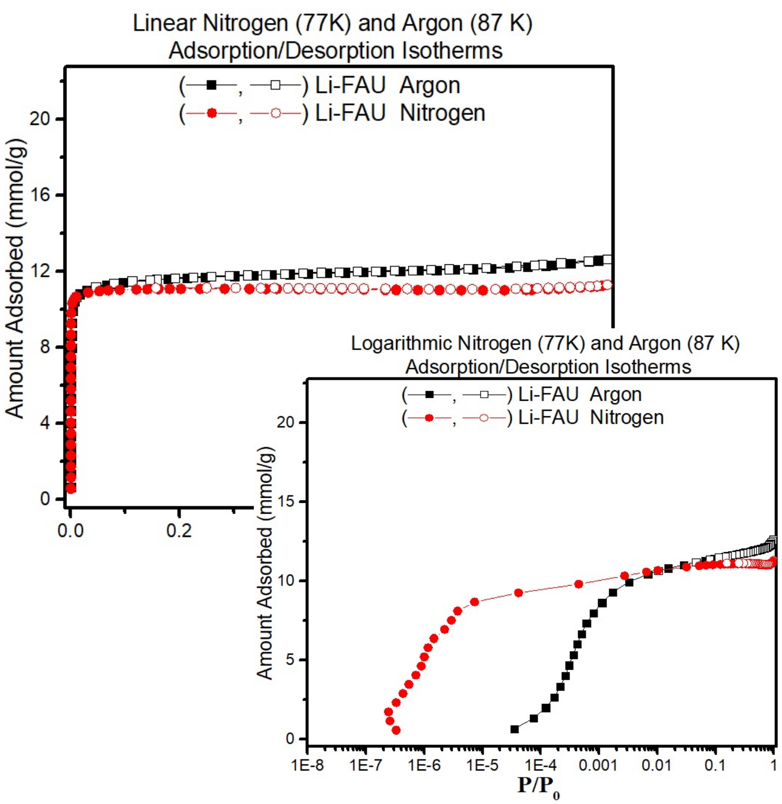
Molecular Sieve research group

- Although, zeolite 5A had been a promising candidate to purifying argon contaminated with nitrogen we verified that theFAU containing Li cations has showed better results for low temperature and low pressure regime:





Media Research



The best results were obtained with zeolite Li-FAU which can adsorb both Ar and N_2 besides, at low pressures, this zeolite has more affinity for N_2 than for Ar.

Workshop da RENAFAE
12-14 July 2021

UNILArC - UNICAMP Liquid Argon Cryostat

Magda Fontes
(Cryo-Setup)

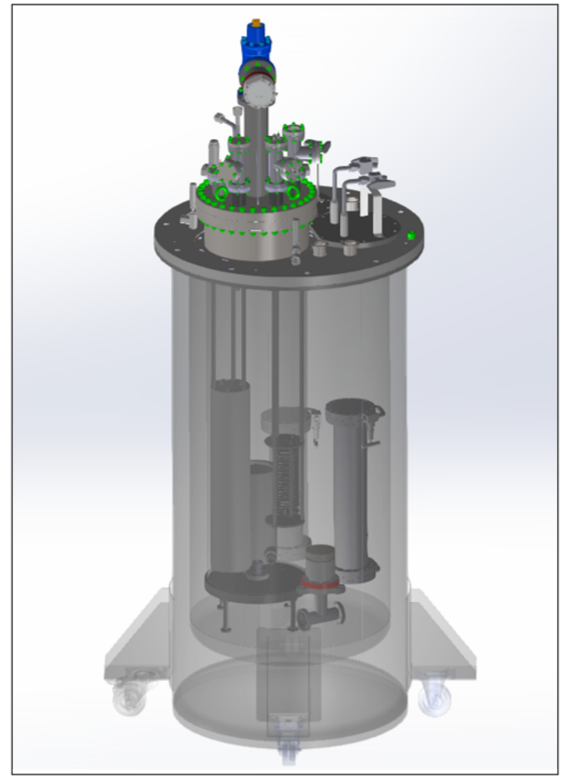
Krystal Brant (CBPF)
Ettore Segreto

Setup

- A cryostat was designed for testing filtering media for liquid argon purification.

- The design presented is a compact construction for LAr re-circulation composed of:

  - Bath dewar;
  - Vessel;
  - Filter set;
  - Purity Monitor;
  - Circulation pump;



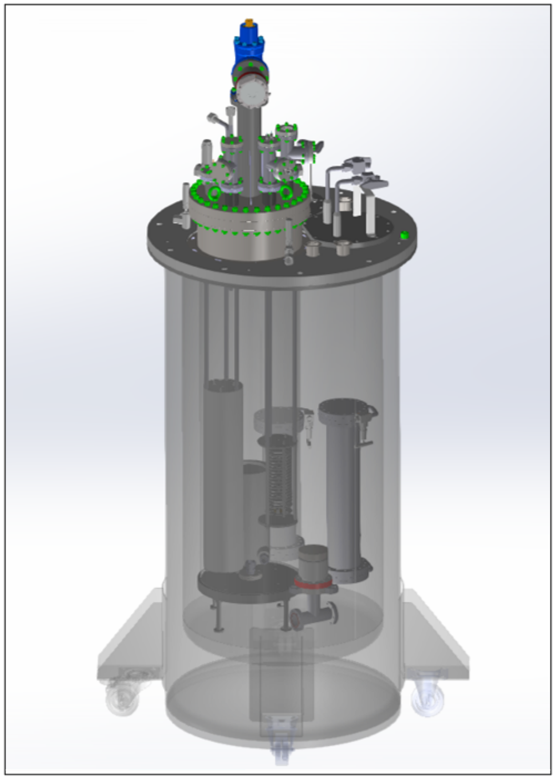
UNICAMP Liquid Argon Cryostat

- The bath dewar will contain all components, to keep them immersed in a thermal bath.

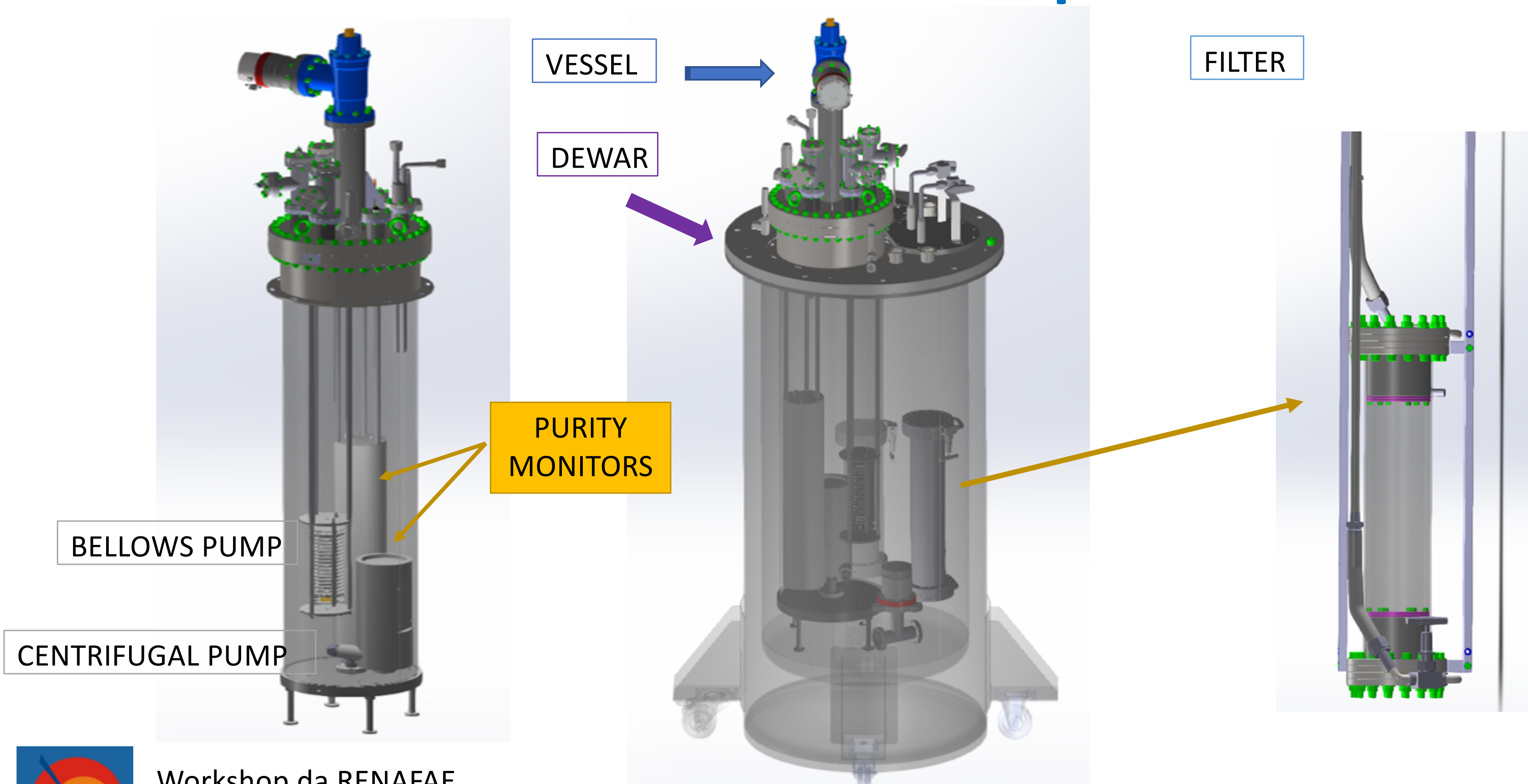
- The vessel will contain the purity monitors and a liquid level sensor, and later in the project a submerged pump. Its top flange holds a variety of feedthrough for signal and electrical wiring, and for liquid transfer purposes.

- The filter set was meant to be easily removable from the system dues allowing rapid exchange of the filtering media and to be able to undergo a regeneration process.

- The bellows pump will guarantee a flow rate close to 0.75 l/min and total separation between the LAr under test and the bath. In the next phase, it will be replaced by a centrifugal pump with a flow rate close to 95 l/min, thus reducing measurement time.



UNILArC - Setup

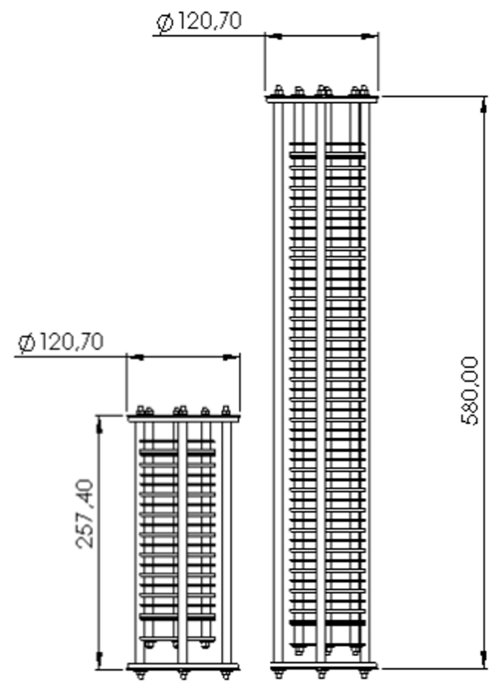


Purity Monitor

Ana Machado
(Purity Monitor)

Frederico Demolin
Heriques Frandini

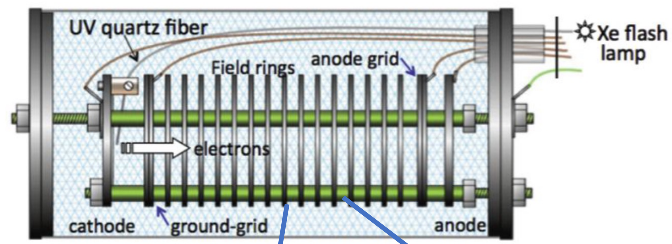
- A key component of the calibration of a LArTPCs is the measurement of the lifetime of drift electrons. It allows to correct for the charge attenuation caused by drift electrons being captured by impurities.
- A purity monitor is a miniature TPC that measures the lifetime of electrons generated from the photocathode via the photoelectric effect.



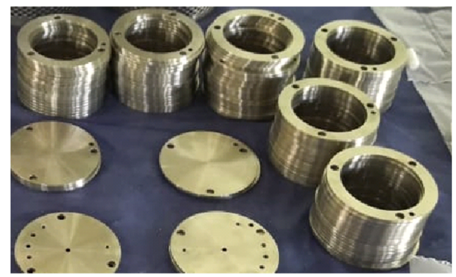
- **Measures the electronegative contamination level of the LAr, due typically to oxygen and water.**

- The cryogenic test setup for media purification will have 2 purity monitors for two different ranges of lifetimes, the long (~1ms) and short (~300ms) versions.

Purity Monitor



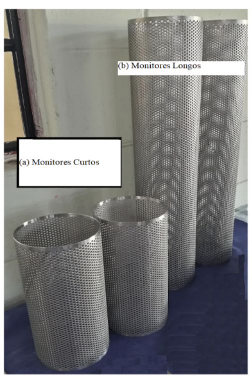
Rings



Spacers

The main components are:

- Photocathode: stainless steel disc, Al foils (0.8mm thickness) coated with 50Å Ti and 1000Å Au;
- Grid (Anode/Cathode) : Tungsten wires gold plated 25µm and 2mm pitch (SBND ref.);
- Xe lamp 225nm to photo-extract electrons from the cathode;
- Stainless steel Faraday cage;

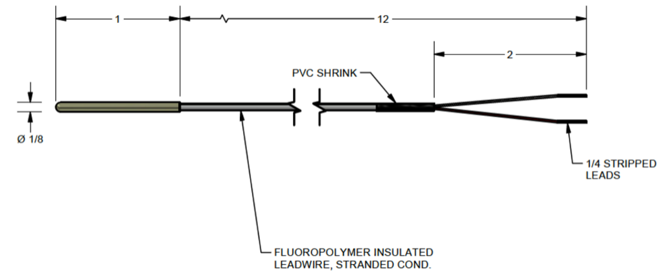


- All components are already produced by Equatorial company;
- Next month we will assemble the monitors at UNICAMP.

Sensor Level – Temperature Sensor

- In order to control the level of LAr inside the vessel we will install 8 temperature/level sensors;
- 4 inside the vessel at the extremities of the purity monitors and the upper level foreseen for liquid argon;
- 4 inside the external bath to monitor ithe LAr level.
- We will test 3 diferentes sensors:
  - Encapsulated Pt100 in magnesium oxide and SS (3cm)
  - Not encapsulated Pt100
  - Not encapsulated Pt1000

- During the tests we will use external controllers for the readout;
- The slow control of the cryogenic setup will be made with custom software;



Quotes for our project

Phase 2 – Potential Suppliers Evaluated

Supplier	Scope
INOXPLASMA	CRES welding and cutting, laser cutting
NEOLIDER – Tubos e Conexões de Aço	Tubes & connections in CRES
ZEOCHEM®	Molecular sieve
upagchem – performance in chemicals	Molecular sieve
BASF – We create chemistry	Molecular sieve
Röchling	Thermal insulation materials & Cryogenics
NITROTEC	Cryogenics equipment, vacuum tubing and vessels
Metal Cryo – Equipamentos Criogênicos	Cryogenic equipment
INOXCVA – Historically Futuristic	Cryogenics equipment, vacuum tubing and vessels
HEROSE	Cryogenic valves
Qualitec	Cryogenic valves and instruments
Parker Bestobell	Cryogenic valves
JN 九洲电子 Jiuzhou Electron	Electronic components
Lydall – specialty engineered products and materials	MLI – Thermal insulation
EMERSON	Electronic components & instruments
ABB	Electronic components & instruments
InstruTech®	Electronic components & instruments

Final Remarks

- Two phase project; Phase 1 ongoing, phase 2 to be approved;
- Temático FAPESP led by the IFGW-UNICAMP;
- Two PIPES-FAPEPS led by the Akaer group;
- In the Fapeps-temático: 4 teams carrying out research and development in the areas:
    - Fluid-dynamic simulation;
    - New media possibly with more efficient materials for filtering oxygen, water and nitrogen from the liquid argon stream;
    - Preparation of the cryostat for the test system and;
    - Construction of the purity monitor for small scale tests at UNICAMP;

Thank you for your attention!