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Ricerca di Sistema elettrico

# Rapporto sulla validazione del sistema di purificazione del litio all'interno di Lifus 6 e del sistema di monitoring online di tali impurezze

*Paolo Favuzza*

Report RdS/2013/126

# RAPPORTO SULLA VALIDAZIONE DEL SISTEMA DI PURIFICAZIONE DEL LITIO ALL'INTERNO DI LIFUS 6 E DEL SISTEMA DI MONITORING ONLINE DI TALI IMPUREZZE

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Report Ricerca di Sistema Elettrico

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Area: Produzione di energia elettrica e protezione dell'ambiente
Progetto: Attività di fisica della fusione complementari a ITER
Obiettivo: *Fornitura ed implementazioni comuni per sperimentazioni di corrosione ed erosione per IFMIF – Fornitura ed implementazioni comuni per la qualifica del sistema di purificazione del litio per IFMIF*
Responsabile del Progetto: Ing. Aldo Pizzuto, ENEA

# Indice

# Sommario

This document deals with the purification system employed within Lifus 6, a plant devoted to the assessment of the corrosion-erosion resistance of metallic materials exposed to flowing liquid Lithium at 350°C. The purpose of the purification is to reduce as much as possible the concentration of Carbon, Oxygen, Hydrogen and especially Nitrogen anions solved in Lithium, making them approach the target IFMIF value of 10 wppm.

The first part of the report highlights the changes and upgrades in the purification system of the plant, introduced during last year, both in terms of design and functioning, in order to improve its efficiency and handling. The document then briefly summarizes the purpose, the operative conditions and the requirements of all the elements and devices aimed at the purification of Lithium and at the evaluation of the impurities concentration. On the basis of these preliminary information, the procedure for the validation of the purification traps (Cold and Hot Traps) and the functional calibration of the Resistivity Meter, able to online monitor the total impurity amount inside Lithium, is detailed.

This procedure, despite some intrinsic limit of the system, like the absence of instruments or devices (not existing in the market, nor as research prototypes) for the online measure of the specific concentration of each impurity, like the possibility of measuring only the Nitrogen concentration through an offline chemical analysis, and like the lack of large literature data on the trap efficiency, permits anyway: to achieve the maximum purification of Lithium feasible with the traps; to know the corresponding minimum Nitrogen concentration value and its related value of Lithium Resistance; to find out a functional relationship linking the measured Resistance value to the Nitrogen concentration.

This procedure will be employed for the initial purification of Lithium charged into Lifus 6 plant, before starting the corrosion tests (scheduled by December 2013), and will provide useful information to verify also the keeping of the desired purity conditions, during the experimental campaign. The activities to set-up the purification system of a Lifus6 have been performed during the PAR2012.

# 1 Introduzione

Lifus 6 plant, the revised version of Lifus 3, is a plant aimed at the evaluation of materials resistance to the erosion-corrosion mechanism exerted by flowing liquid Lithium. This experimental investigation constitutes one of the validation activities underlying the future IFMIF facility (EVEDA phase of the IFMIF project, which is part of the Broader Approach). ENEA is in charge of the design and construction of Lifus 6 plant, as well as the execution of the erosion-corrosion tests, in its research centre of Brasimone.

One of the fundamental requisites of these corrosion tests is the high purity degree of the flowing liquid Lithium: the presence of non metals impurities in fact (Carbon, Oxygen, Hydrogen and particularly Nitrogen [1,2]), in the form of anions solved by Lithium, greatly enhances the corrosion behavior of the liquid metal.

For this reason, Lifus 6 plant must also be equipped with solutions and devices able to perform the purification of the liquid Lithium, so to reduce non metals concentration as close as possible to IFMIF requirement ($\leq$ 10 wppm).

Most Lifus 6 elements devoted to the purification purpose are integrated inside a dedicated ‘purification loop’, which is a secondary loop working in parallel with the main one and characterized by a Lithium flow rate about 1% of the total. These are: the Cold Trap, the Resistivity Meter and a sampler aimed at taking a small Lithium volume for the offline chemical analysis. The others elements (not in the dedicated ‘purification loop’) are the Hot Trap and a second, different sampler, directly linked to the Hot Trap. All these elements are both to perform an initial purification of the Lithium, after charging it into the plant and before starting the corrosion tests, and to continue to guarantee the desired purity level all over the duration of the tests.

This report explains the purpose of each of these elements and details their validation procedure. This procedure will be performed just before starting the corrosion tests, after Lifus 6 plant complete realization, check of all its electrical, thermal, hydraulic, control and safety systems, and charging of the Lithium. The report outlines the philosophy of the procedure and details the step to follow, particularly highlighting the importance of their right sequence, as well as the strict interconnection between the online and the offline measurement results.

# 2 Descrizione delle attività svolte e risultati

## *2.1 Lifus 6 purification elements upgrade*

Before entering the details of the validation procedure, it's useful to have an overall look at Lifus 6 plant, particularly focusing on its devices and elements devoted to the purification of Lithium. Many of the underlying specifications are not here reported, since already present in the previous report Rds/2012/261 [3].
A 3D-view of the final Lifus 6 design is shown in Figure 1; a more detailed view of the purification branch alone, housing the Resistivity Meter, the Cold Trap and the Sampler (in red) is instead shown in Figure 2.

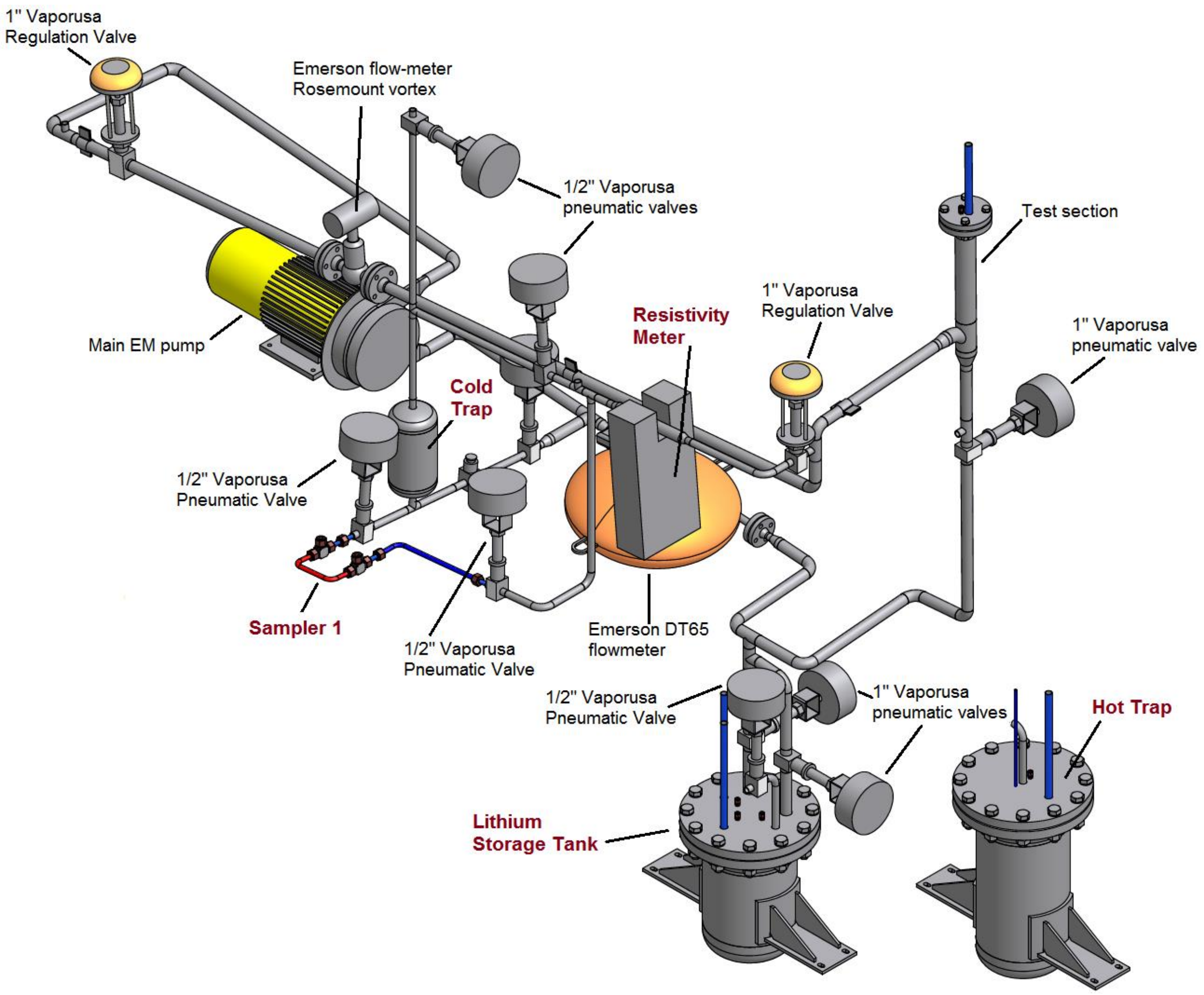
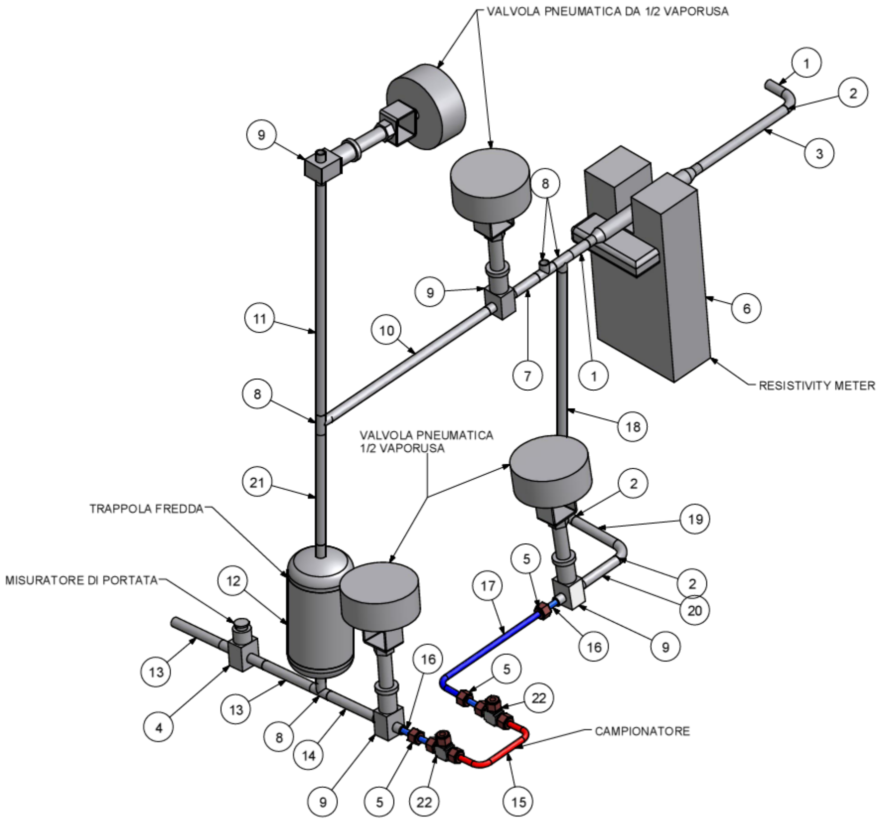



**Figure 1: 3D view of Lifus 6 Design (the pipe linking the Lithium Storage Tank to the Hot Trap is not shown in the figure)**

It's worth here only highlighting the main differences respect to the previous Lifus 6 purification system, as reported in [3]. These changes are the outcome of a deep Design Review of the whole plant performed during last year by ENEA, and have been adopted in order to improve the functioning and the handling of the plant.




Elenco parti

| POS. | QTÀ | DESCRIZIONE | MATERIALE |
|---|---|---|---|
| 22 | 2 | T Swagelok 1/2" | Acciaio inox 316L |
| 21 | 1 | Tubo 1/2" Sch. 40S L= 287MM | A312-TP 316L |
| 20 | 1 | Tubo 1/2" Sch. 40S L= 105MM | A312-TP 316L |
| 19 | 1 | Tubo 1/2" Sch. 40S L= 125MM | A312-TP 316L |
| 18 | 1 | Tubo 1/2" Sch. 40S L= 598MM | A312-TP 316L |
| 17 | 1 | Tubo Swagelok 1/2" | Acciaio inox 316L |
| 16 | 2 | Giunti Swagelok saldati a valvola | Acciaio inox 316L |
| 15 | 1 | Campionatore da tubo Swagelok 1/2" | Acciaio inox 316L |
| 14 | 1 | Tubo 1/2" Sch. 40S L= 140MM | A312-TP 316L |
| 13 | 2 | Tubo 1/2" Sch. 40S L= 144,5MM | A312-TP 316L |
| 12 | 1 | Trappola fredda | Acciaio inox 316L |
| 11 | 1 | Tubo 1/2" Sch. 40S L= 600MM | A312-TP 316L |
| 10 | 1 | Tubo 1/2" Sch. 40S L= 478MM | A312-TP 316L |
| 9 | 4 | Valvolo pneumatica da 1/2" Vaporusa | Acciaio inox 316L |
| 8 | 4 | T 1/2" Sch 40S Asme B16.9 | A312-TP 316L |
| 7 | 4 | Tubo 1/2" Sch. 40S L= 70MM | Acciaio inox 316L |
| 6 | 1 | Resistivity meter | |
| 5 | 3 | Attacco Swagelok 1/2" | Acciaio inox 316L |
| 4 | 1 | Misuratore di portata | |
| 3 | 1 | Tubo 1/2" Sch. 40S L= 260MM | A312-TP 316L |
| 2 | 3 | Curva 1/2" Sch 40S Asme B16.9 | A312-TP 316L |
| 1 | 2 | Tubo 1/2" Sch. 40S L= 50MM | A312-TP 316L |

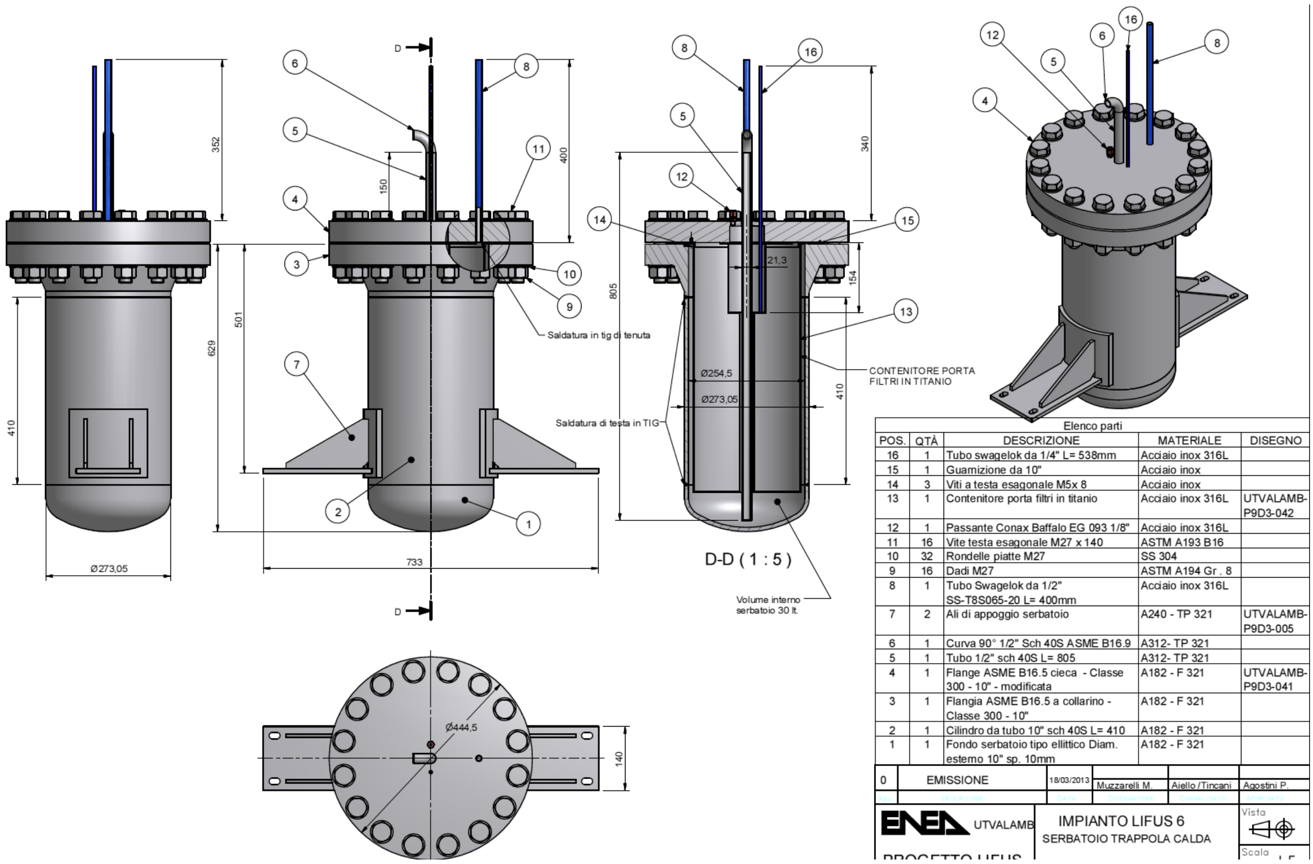
| Rev. | Descrizione | Data | Disegnatore | |
|---|---|---|---|---|
| 0 | EMISSIONE | 12/04/2013 | Muzzarelli M. | Aie |

UTVALAMB
PROGETTO LIFUS
IMPIANTO LIFUS 6
ASSIEME IMPIANTO PURIFICAZIONE E CAMPIC
Ricavato dal: Tipo Distr. N° UTVALAMB-

**Figure 2: 3D-Design of Lifus 6 dedicated 'purification loop'**

Basically, in the old configuration the Lithium storage tank, from which the liquid metal is charged/drained to/from the main Lifus 6 loop, had been conceived also as the Hot Trap: for this reason, it should have been filled with Titanium sponge getter and activated the purification from Nitrogen in static conditions after the raise of Temperature to 600°C. Nevertheless, with this configuration, the substantial risk was produced of not being able to perform a fast draining of all the Lithium from the loop, as a consequence of the viscosity of the liquid metal and the hydraulic resistance exerted from the Titanium sponge in pellets, tightly packed inside the tank. Moreover, if in this situation Lithium wasn't able to freely flows amid the pellets, metal squirts might be produced, dirtying the gas lines and the other instrumentations.
For this reason, it has been decided to realize a new component, i.e. a new tank, which is devoted exclusively to the purification of Lithium from Nitrogen. In other words, the storage tank and the Hot Trap become two separate units. The new Hot Trap, having an inner volume of about 30 L, will be again filled with the Titanium sponge getter, but the getter will be enclosed inside a confined volume (delimited by a narrow grid steel structure) allowing an easy removal of the whole getter as an unique element. The design of the new Hot Trap is shown in Figure 3.
An other advantage of the new Hot Trap is that it will be realized in a stainless steel certified up to the working temperature of 650°C (321 Stainless Steel). Operating the purification at this higher temperature, it will be possible to enhance the kinetic mechanism responsible for the adsorption of Nitrogen from Lithium solution (N diffusion inside Ti getter and TiN formation) and to reduce the time required for the purification.

Lithium storage tank will remain the same as before, only a minor modification will be introduced, lengthening the external Lithium inlet pipe and transforming into an inlet/outlet pipe, to permit the transfer of Lithium forward and backward to the Hot Trap.




| POS. | QTÀ | DESCRIZIONE | MATERIALE | DISEGNO |
|---|---|---|---|---|
| 16 | 1 | Tubo swagelok da 1/4" L= 538mm | Acciaio inox 316L | |
| 15 | 1 | Guarnizione da 10" | Acciaio inox | |
| 14 | 3 | Viti a testa esagonale M5x 8 | Acciaio inox | |
| 13 | 1 | Contenitore porta filtri in titanio | Acciaio inox 316L | UTVALAMB-P9D3-042 |
| 12 | 1 | Passante Conax Baffalo EG 093 1/8" | Acciaio inox 316L | |
| 11 | 16 | Vite testa esagonale M27 x 140 | ASTM A193 B16 | |
| 10 | 32 | Rondelle piatte M27 | SS 304 | |
| 9 | 16 | Dadi M27 | ASTM A194 Gr . 8 | |
| 8 | 1 | Tubo Swagelok da 1/2" SS-T8S065-20 L= 400mm | Acciaio inox 316L | |
| 7 | 2 | Ali di appoggio serbatoio | A240 - TP 321 | UTVALAMB-P9D3-005 |
| 6 | 1 | Curva 90° 1/2" Sch 40S ASME B16.9 | A312- TP 321 | |
| 5 | 1 | Tubo 1/2" sch 40S L= 805 | A312- TP 321 | |
| 4 | 1 | Flange ASME B16.5 cieca - Classe 300 - 10" - modificata | A182 - F 321 | UTVALAMB-P9D3-041 |
| 3 | 1 | Flangia ASME B16.5 a collarino - Classe 300 - 10" | A182 - F 321 | |
| 2 | 1 | Cilindro da tubo 10" sch 40S L= 410 | A182 - F 321 | |
| 1 | 1 | Fondo serbatoio tipo ellittico Diam. esterno 10" sp. 10mm | A182 - F 321 | |

**Figure 3: new Hot Trap design**

Another significant improvement of Lifus 6 purification system is the presence of a second (additional) sampler for taking a small amount of Lithium from the plant, to be employed for the offline chemical analysis of its Nitrogen concentration. The design of this new sampler is illustrated in Figure 4. The sampler, initially hosting a known Argon pressure (the top pipe is linked to a gas line), is then filled by Lithium from the bottom pipe by raising the external pressure. The subsequent decrease of the external pressure produces the reverse effect, emptying the sampler, a part from the Lithium that remains trapped in the annular volume around the bottom pipe. Once closed the valves upstream of both the two pipes, it is possible to detach the whole sampler: this way, no Lithium will remain exposed to atmosphere, nor in contact with the external valves. The Lithium is then removed from the sampler, inside a glove box, by simply opening the flange; it’s then melted again and transferred, in liquid phase, to the glass vessel for the chemical analysis.

While the first sampler (Sampler 1 in Figure 1) is installed in a bypass along the purification loop, for checking the Nitrogen concentration in Lithium during plant operations, this second sampler (Sampler 2) will be instead directly connected to the Hot Trap, so to check the outcome of a just performed purification, without having to refill the plant with Lithium, and possibly repeat the purification step, until the Nitrogen content matches the desired value.

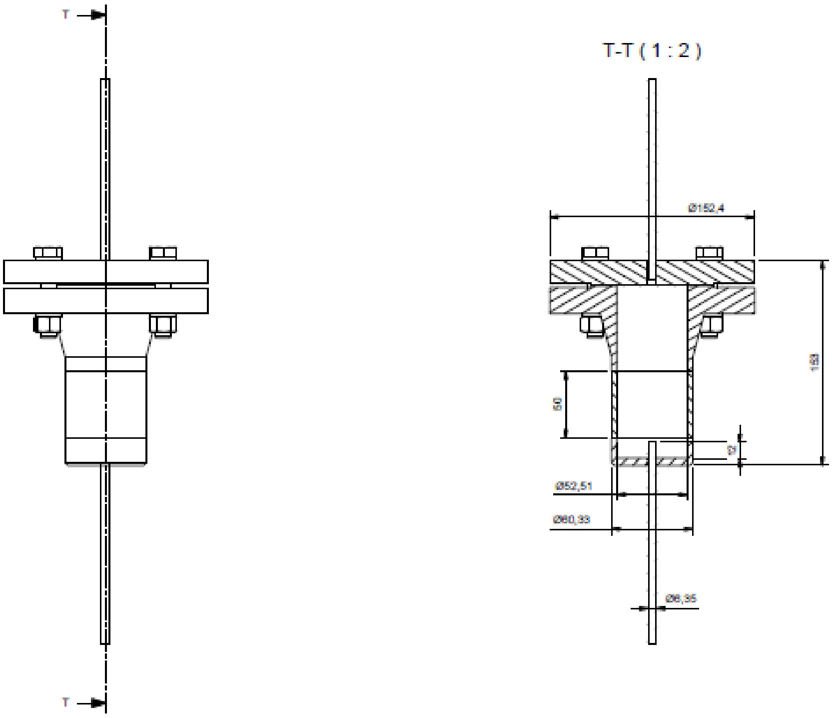



**Figure 4: design of the new (additional) sampler for taking Lithium from Lifus 6**

## *2.2 Purification elements: summary of functions and requirements*

Lifus 6 elements which actually produce the purification of Lithium are the Cold Trap and the Hot Trap; the elements for checking its purity degree are instead the Resistivity Meter and the samplers for the offline chemical analysis.

As already reported [3], the purpose of the Cold Trap is to reduce non metallic impurities concentration, exploiting the precipitation phenomenon of their compounds with Lithium. Solid Lithium Carbides, Hydrides, Nitrides and Oxides are expected to accumulate inside the Cold Trap, until the remaining content in Lithium solution will match the solubility value at the trap temperature. Since the Cold Trap is operated at 200°C, the maximum impurities residual content is 2 wppm for Carbon, 7 wppm for Oxygen, 63 wppm for Hydrogen and 1461 wppm for Nitrogen [4]; therefore the Cold Trap results particularly efficient for the removal of Carbon and Oxygen.

The purpose of the Hot Trap is to further reduce Nitrogen concentration in Lithium below the value guaranteed by the Cold Trap alone. For this reason, the Hot Trap is filled with a Titanium getter (in the form of sponge pellets), which adsorbs Nitrogen from the liquid metal, leading to the formation of a layer of Titanium Nitride. This way, operating the Hot Trap at 650°C, residual Nitrogen concentration may be reduced $\leq 10$ wppm [5].

The different philosophy of employment of the Cold and the Hot Trap is illustrated in the simplified block-scheme of Lifus 6 in Figure 5. Lithium flows continuously through both the main loop (on the left in the figure) and the purification loop (on the right) during operations (the flow in the purification loop is about 1% of the total Lithium flow). Therefore, since the Cold Trap is part of the purification loop, it exerts the purification continuously, while Lithium flows through it. On the contrary, since the Hot Trap is not part of the loop but it's only linked to the Storage Tank, it performs Lithium purification at discrete time steps, in static conditions, on the whole Lithium mass previously transferred inside it. Actually, the purification from Nitrogen in the Hot Trap will be performed at the beginning (before starting the corrosion tests) and each time the Nitrogen concentration in Lithium will reach a too high value, as revealed by the impurities control procedures (see later): in these cases, it is necessary to stop the circulation of Lithium inside Lifus 6 loop, discharge it in the storage tank, transfer it from here to the Hot Trap and give it time to react again with the Titanium getter. The reason for a static purification in the case of the Hot Trap is that the reaction between Nitrogen and Titanium getter is so slow that a very high residence time is required in order to approach the equilibrium value.

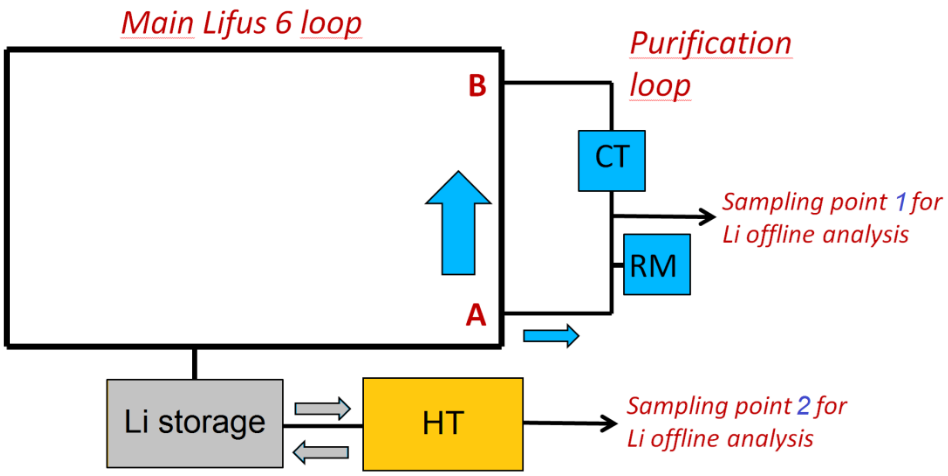



**Figure 5: block diagram of Lifus 6 purification elements. CT = Cold Trap; HT = Hot Trap; RM = Resistivity Meter. Arrows: Lithium flow**

For what concerns the evaluation of the impurities concentrations in Lithium (hence the estimation of the efficiency of the performed purification), it is possible to rely both on the output of the Resistivity Meter (RM), providing an online value, and on the result of the chemical analysis of Lithium samples taken discontinuously from the plant, providing an offline value.
It is important to remember that, since the Resistivity Meter measures the total resistance of the flowing Lithium, it is not chemically specific respect to each non metal, but gives an overall indication of the total impurities amount: the higher the impurities total concentration, the higher the resistivity value [6-8]. In order to compare the relative effect of these elements on the resistivity (ρ), it's possible to observe the value experimentally calculated at 480°C by Creffield [8]:

$\Delta\rho = 2.1 \times 10^{-8}\ \Omega m\ (\text{atom \% } \mathbf{O})^{-1}$
$\Delta\rho = 3.8 \times 10^{-8}\ \Omega m\ (\text{atom \% } \mathbf{H})^{-1}$
$\Delta\rho = 7.3 \times 10^{-8}\ \Omega m\ (\text{atom \% } \mathbf{N})^{-1}$

It appears that that the resistivity is primarily sensitive to Nitrogen, then to Hydrogen and Oxygen. Anyway, considering the molar mass and the relative concentration of the various non metals inside Lithium, it is possible to almost neglect the contribution of Oxygen (and Carbon) to the resistivity, limiting the effect to Nitrogen and Hydrogen atoms. Clearly, at 350°C, which is the temperature of Lithium inside the RM, those coefficient are slower (for instance, $\Delta\rho = 6.8 \times 10^{-8}\ \Omega m\ (\text{atom \% } \mathbf{N})^{-1}$) but the general trend is the same. In any case, the RM sensitivity is not very high, therefore, at 350°C it will not possible to detect a concentration variation lower than about 50 wppm of Nitrogen [3].

In order to overcome the limits of the Resistivity Meter, an offline chemical analysis is employed too, already described in [3] and subject of another dedicated report ("Rapporto sulla procedura offline di

analisi chimica della concentrazione di Azoto in Litio", RdS/2013). It is important here only to point out that this analysis is, on the opposite, very chemically specific and provides as result the concentration of Nitrogen alone in the sampled Lithium. With this technique, it is possible to measure down to few ppm of Nitrogen concentration, with a relative precision of some %; the disadvantage is that it is quite time and work consuming, and there is a delay in the instrumental answer respect to the sampling time. Two different sampling points are present inside Lifus 6 plant, the first one for sampling from the purification loop and checking during operations, the second for sampling directly from the Hot Trap, after a just performed purification.
Table 1 summarizes the main aspects related to the purification elements of Lifus 6.

**Table 1: main aspects of Lifus 6 purification elements ($C_X$ = weight concentration of X element in Li)**

| Purification Element | Function | Note |
|---|---|---|
| Cold Trap (CT) | Reduces non metals impurities precipitating them as solid Lithium compounds. | It works continuously during operations. At 200°C: $C_C \leq 2$ wppm; $C_0 \leq 7$ wppm; $C_H \leq 63$ wppm; $C_N \leq 1461$ wppm |
| Hot Trap (HT) | Further reduces N concentration through the reaction with Ti-getter. | It works in static condition, whenever necessary. At 650°C: $C_N \leq 10$ wppm |
| Resistivity Meter (RM) | Continuously (online output) indicates total impurities concentration variation. | Particularly sensitive to H e N atoms, but little sensitive in absolute terms (~ 50 wppm of N at 350°C). |
| Sampler 1 | Takes a Lithium sample from the purification loop, during operations. | For the Nitrogen evaluation through the offline chemical analysis.<br>Range: 5 wppm < $C_N$ < 1500 wppm;<br>Precision: some % |
| Sampler 2 | Takes a Lithium sample from the Hot Trap, just after a purification step. | As above. |

## *2.3 Procedure for the estimation of the purification progress and the validation of the Resistivity Meter*

Before starting the corrosion/erosion experimental tests, it's necessary to perform an initial purification of all the Lithium flowing inside Lifus 6, which is the same Lithium previously employed in the old Lifus 3 plant [9] and here-hence transferred. In order to guarantee that the desired purity degree has been achieved, it is necessary to find out a procedure to assess the efficiency of the purification exerted by the Cold and Hot Trap; on the same time, in order to significantly employ the Resistivity Meter, a functional calibration is required. The procedure to fulfill, as best as possible, these targets, has been conceived taking into account all the considerations previously reported in this report, summarized in Table 1, and the related following points:

A. Unfortunately, we do not dispose of a Lithium standard, characterized by a known value of impurities concentration. Only a commercial Lithium is available on the market, for which the supplier declares a Nitrogen Concentration lower than 300 wppm (but not a precise value). It is hence not possible to perform a preliminary calibration of the Resistivity Meter or a tuning of the offline chemical analysis employing such a Lithium.

B. The resistivity value (ρ) of Lithium is affected by the totality of the impurities, being particularly sensitive to Hydrogen and Nitrogen concentration.
C. It is not possible to measure, with our devices, the concentration of Carbon, Oxygen and Hydrogen.
D. It is possible to measure, through the offline batch analysis of a Lithium sample, its Nitrogen concentration.

Since our experimental outputs are the Resistance of Lithium, from one side, and the Nitrogen concentration, on the other, it is necessary to relate them such a way that, each time a Nitrogen concentration value (**C**) is obtained of a sample taken from the plant, it can be linked to the Resistance value (**R**) (provided by the RM) at the time that sampling was executed. In other words, we have to generate the couples of points (**R, C**), and use them to follow the evolution of the system. The procedure is detailed in the steps below reported.

Phase 1: initial purification exerted by the Cold Trap

1) First of all, before filling Lifus 6 with Lithium, it is necessary to measure the electric Resistance of the RM empty capillary alone ($R_{cell}$), which is a constant (if temperature is constant). According to the expression for parallel resistances:

   $R_{Li}= (R_{cell} \times R_{tot}) / (R_{cell} – R_{tot})$

   it is so possible to use this constant ($R_{cell}$) to indirectly know, each time, the specific value of Lithium resistance alone ($R_{Li}$) from the measured value of the total resistance ($R_{tot}$: metallic capillary + Lithium).

2) Lifus 6 is filled with Lithium, which is expected to contains the maximum amount of impurities, since it has not been purified at all. After a short time (~ 10') to allow some circulation inside the plant, a first reading of the Resistance value is done; at the same time, a first sampling from the sampler 1 is executed, followed by the offline analysis for the Nitrogen determination. We have so created the first couple of values; let's indicate it as: ($\mathbf{R_0}$, $\mathbf{C_0}$), since this is point "0" of our procedure.

3) After about 6 hours of Lithium circulation inside the plant (hence of purification by the Cold Trap), a new reading of the Resistance (by the RM) is done, together with a sampling from the sampler 1 and a Nitrogen determination. Let's call this couple of values: ($\mathbf{R_{CT1}}$, $\mathbf{C_{CT1}}$)[1], where the value of $\mathbf{R_{CT1}}$ is expected to be $\mathbf{< R_0}$ (due to the decrease of impurities produced by the Cold Trap, especially of Hydrogen concentration), while the value of $\mathbf{C_{CT1}}$ should be similar to the value of $\mathbf{C_0}$, since the Cold Trap does not affect Nitrogen concentration.

4) After 1 day of Lithium circulation inside the plant, a new RM reading together with a parallel Nitrogen concentration of the sampled Lithium is done. The couple of point ($\mathbf{R_{CT2}}$, $\mathbf{C_{CT2}}$) is obtained, where, again, $\mathbf{R_{CT2}}$ is expected $\leq \mathbf{R_{CT1}}$ and $\mathbf{C_{CT2}}$ is expected $\sim \mathbf{C_{CT1}}$.

5) Step 4 is repeated until $\mathbf{R_{CTn} \sim R_{CTn-1}}$, in other words the Resistance value has been stabilized. We can now assume we have achieved the maximum purification of Lithium from Carbon, Hydrogen and Oxygen. The time lapse between each data acquisition may be adjusted according to the observed speed of change of the acquired values.

[1] CT1 stands for: Cold Trap 1. It means: the first point after the purification by the Cold Trap.

Phase 2: initial purification exerted by the Hot Trap

6) All the Lithium is discharged from Lifus 6 plant and transferred into the Hot Trap. A sampling from the sampler 2 is possible, followed by the offline chemical analysis for Nitrogen content determination. Lithium purification from Nitrogen is activated by raising the temperature of the Trap to 650°C. After 1 day of purification, the temperature of the Trap is lowered, so to deactivate it. A small amount of Lithium is again taken from sampler 2 and analyzed for its Nitrogen concentration. If no Nitrogen concentration decrease is evident, the Hot Trap has to be reactivated and execute the purification for a longer, additional time. In case a significant decrease has been instead observed, all the Lithium is recharged to Lifus 6 plant, and after some circulation inside it, the Resistance values is acquired, together with the corresponding Nitrogen concentration value (Lithium taken from sampler 1). The couple of points ($\mathbf{R_{HT1}}$, $\mathbf{C_{HT1}}$)[2] is created, where $\mathbf{R_{HT1}}$ is expected to be **<** $\mathbf{R_{CTn}}$ and $\mathbf{C_{HT1}}$ is expected to be **<** $\mathbf{C_{CTn}}$ (the Hot Trap reduces Nitrogen impurities, hence also the Resistance).

7) Step 6 is repeated many times, each time varying the purification time, if necessary, according to the speed of change of the acquired values. This way we get the sequence of couple of points: ($\mathbf{R_{HT2}}$, $\mathbf{C_{HT2}}$), ($\mathbf{R_{HT3}}$, $\mathbf{C_{HT3}}$),….. ($\mathbf{R_{HTn}}$, $\mathbf{C_{HTn}}$), characterized by a trend of decrease with time of both the measured Resistance and Nitrogen concentration. We repeat this step, until we approach a constant value of Resistance ($\mathbf{R_{HTn} = R_{HTn-1}}$), and, hopefully, the final Nitrogen concentration ($\mathbf{C_{HTn}}$) is $\leq$ 30 wppm (target of our experimental corrosion tests). Due to the time cost of this step, we also hope that 5-6 couples of values could be enough to reach the stationary condition.

At the end of all these steps, it is so possible to know the Resistance value corresponding to the purest Lithium achievable with the adopted traps system: $\mathbf{R_{HTn}}$. This information will be useful during the long corrosion/erosion tests, providing an online reference value to match all over the experimental time. Moreover, plotting each value of $R_{HT}$ versus the corresponding $C_{HT}$ one, it will be possible to construct an experimental graph, finding out a nearly linear relationship linking these two quantities. This relationship will be useful, in a qualitative way, to predict Nitrogen concentration in Lithium, on the basis of the read resistance values.
Finally, from the theoretical evaluation of the geometric parameter (A/l) of the RM capillary, where A is the section of the capillary and l is its length, and employing the relation:

$$\rho = R \times (A / l)$$

it will be possible to convert all the resistance values in resistivity values and compare them with the results obtained by Nottingham researchers [10] to see if they qualitatively agree.

[2] HT1 stands for: Hot Trap 1. It means: the first point after the purification by the Hot Trap.

# 3 Conclusioni

Due to the lack of specific devices or analytical techniques, it is not possible to measure Hydrogen, Carbon and Oxygen concentration in Lithium circulating inside Lifus 6. It's only possible to measure, with an offline chemical analysis, which is unfortunately time and work consuming, the value of Nitrogen concentration. Fortunately, Nitrogen is the most abundant and corrosion affecting elements, so it's concentration is surely the most important parameter to monitor during the experimental tests.
On the other hand, it is possible to have a qualitative image of the total impurity content of the Lithium thanks to the Resistivity Meter, which has the advantage of providing a real time (online) answer.
Integrating this answer with the result of the chemical analysis performed over a Lithium sample taken at a the same moment from the plant, it has been possible to define a procedure that:

- permits to achieve the maximum purification of Lithium from Carbon, Oxygen and Hydrogen feasible with the Cold Trap.
- permits to achieve the maximum purification of Lithium from Nitrogen, feasible with the Hot Trap
- permits to know the minimum achievable value of Nitrogen concentration in Lithium, as a reference value
- permits to know the minimum achievable value of Lithium Resistance, as a reference value
- permits to find out a functional relationship which, in our specific system, links the measured Resistance value with the Nitrogen concentration.
- permits to know at any time the Resistivity value of Lithium and compare it with literature data.

After the executions of all the steps of the procedure aimed at the initial purification of Lithium, it will be possible to start the corrosion tests (scheduled by December 2013). During this campaign, both the Resistivity Meter and the chemical offline analysis will be employed again, to verify the keeping of the desired purity conditions. Anyway, due to the limited sensitivity of the Resistivity Meter, the offline chemical analysis will be the reference procedure for the evaluation of Nitrogen concentration: some sampling will be therefore occasionally executed. The Resistivity Meter will be however useful, during the corrosion tests, to continuously monitor Lithium purity during the time between a sampling and its subsequent one, and eventually to induce to a new purification by the Hot Trap.

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# 5 Abbreviazioni ed acronimi

| | |
|---|---|
| A | Cross section (area) |
| C | Carbon |
| $C_X$ | Concentration of the X element |
| CT | Cold Trap |
| EVEDA | Engineering Validation and Engineering Design Activities |
| H | Hydrogen |
| HT | Hot Trap |
| IFMIF | International Fusion Materials Irradiation Facility |
| l | length |
| N | Nitrogen |
| O | Oxygen |
| R | Electric Resistance |
| RM | Resistivity Meter |
| wppm | weight part per million (=mg/Kg) |
| $\rho$ | Electric Resistivity |