



Risultati relativi alla prima fase di purificazione del litio circolante all'interno dell'impianto Lifus 6 e verifica ed ottimizzazione contestuale del suo funzionamento

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LIFUS 6 E VERIFICA ED OTTIMIZZAZIONE CONTESTUALE DEL SUO FUNZIONAMENTO

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Sommario

This report deals with the first months (~ 9) of Lifus 6 plant operations, after the filling of the plant with Lithium and the start of Lithium circulation. The plant was constructed during the previous years in ENEA Brasimone centre in order to perform erosion-corrosion resistance tests of specimens materials, when exposed to flowing liquid Lithium at 350°C. Before starting the corrosion tests, is anyway mandatory to reduce the concentration of non metallic impurities dissolved in Lithium, since these impurities strongly enhance the corrosive behaviour of the alkaline metal. Therefore, during these first months of operations the investigated specimens were not inserted inside the plant and the circulation was aimed only at the reduction of Carbon, Oxygen and Hydrogen concentration in Lithium, thanks to the employment of the Cold Trap, installed along the purification loop of the plant.

Although the Cold Trap functioning was proved to be efficient and led to the required degree of purification, some unexpected issues and troubles emerged during the period, particularly: the formation of solid aggregates in the lower part of the Test Section; the appearance of flow turbulence in the ingress of the Test Section; the malfunctioning of the Lithium mini-flowmeter installed in the purification loop; the difficult regulation of the automatic valve required to split Lithium flow rate between the main (~99%) and the purification loop (~1%). All these troubles have been studied and then solved by ENEA staff, sometimes stopping for a while the Lithium circulation to implement the required hardware modifications and introducing also optimizations in the plant control software.

At the end of this period therefore, the first phase of Lithium purification ascribed to the Cold Trap has been surely accomplished; at the same time, the plant has been improved and reached a full ripeness. Since the beginning of September, the second (and last) phase of Lithium purification has hence started, aimed at reducing the Nitrogen impurities content in Lithium, thanks to specifically conceived Hot Trap. After the completion of this phase, scheduled by the end of October, it will be possible to insert the specimens in the plant and start the real corrosion tests.

1 Introduzione

In the framework of the EVEDA phase of the IFMIF Project, which is part of the Broader Approach, ENEA has been assigned by F4E to carry out the Procurement Arrangements (PAs) LF03 and LF04-EU. The final purpose of the ENEA experimentation is to measure the erosion/corrosion resistance of Eurofer 97 and F82H, which are Reduced Activation Ferritic Martensitic Steels (RAFM) considered as candidate structural materials for the IFMIF facility, when exposed for many thousands hours to purified liquid Lithium flowing at 15 m/s at 350°C.

These tasks hence entail: from one side to design and construct a dedicated flowing Lithium loop, integrating a test section where the materials specimens can be inserted and undergo the requested corrosion test (specific goal of the LF03 PA); from the other side, to implement in the same plant a purification system, aimed at reducing non metallic concentrations in Lithium below fixed values and monitoring them (which is the specific goal of the LF04-EU PA), since these impurities strongly enhance the corrosive behavior of the liquid metal [1,2].

The fulfillment of these task has led, during the previous years, to the realization in ENEA Brasimone Centre of the Lifus 6 plant, as described in Rds/2013/124 [3] and Rds/2013/208 [4], and to the implementation of specific procedures for the purification of Lithium and the quantification of Nitrogen concentration in Lithium, as described in Rds/2013/125 [5], Rds/2013/126 [6] and Rds/2013/209 [7]. Figure 1 shows a 3D-view of the final design of Lifus 6 plant, at the end of the commissioning phase with Argon.

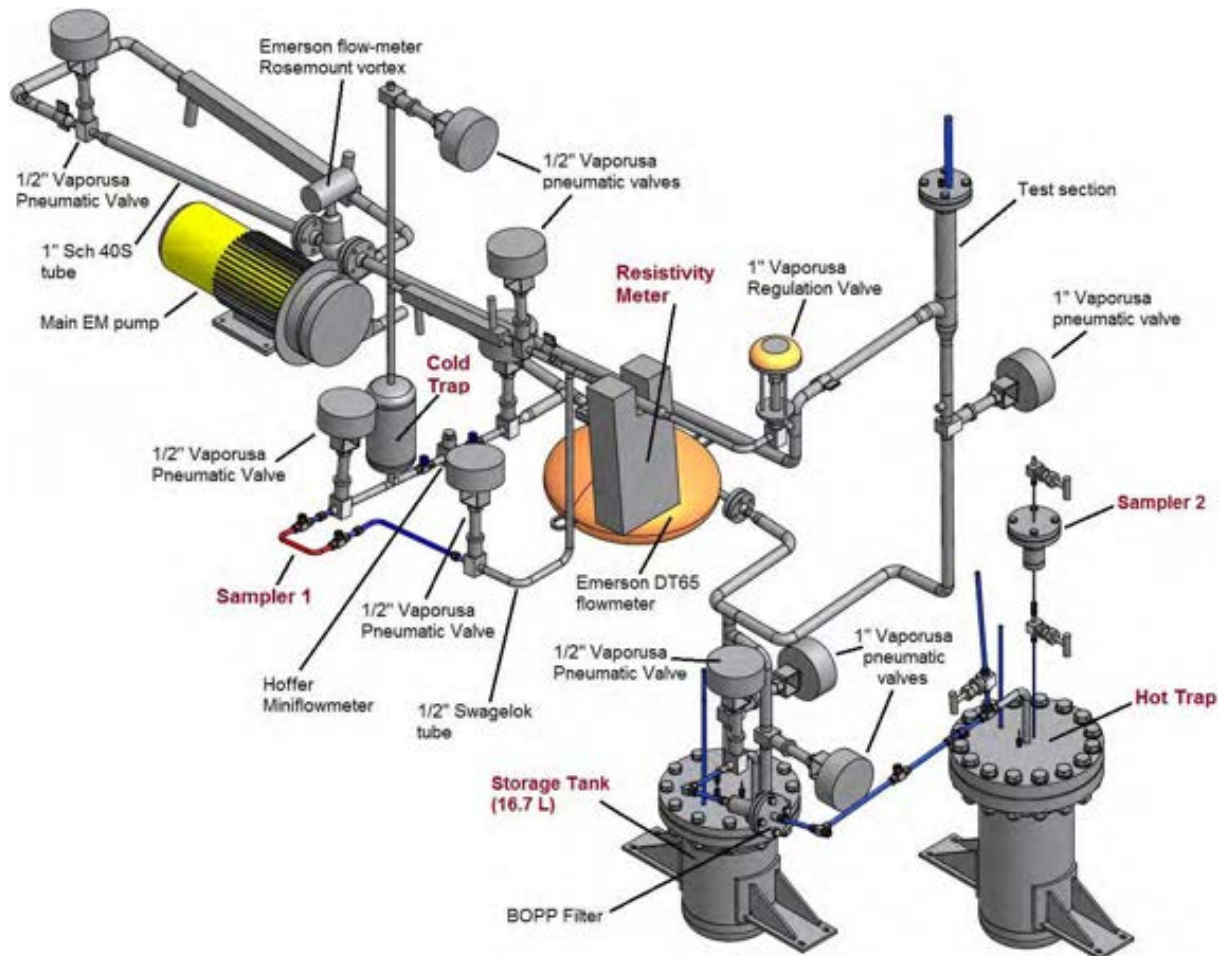


Figure 1: 3D view of the Design of Lifus 6 plant at the end of the commissioning phase with Argon

Just to give a very concise recall of the plant structure, we can see that it is basically constituted by:

- a primary loop, integrating the main Electromagnetic (EM) Pump for the Lithium circulation and the Test Section for the arrangement of the investigated specimens;
- a secondary (purification) loop, integrating the Cold Trap for the impurities precipitation and the Resistivity Meter;
- the Storage Tank and the Hot Trap, for the Nitrogen removal, both set outside the loop.

The features of the most important elements of the plant are reported in table 1:

Table 1: Lifus 6 elements features

Element	Design Pressure [MPa]	Design Temperature [°C]	Max operative Temperature [°C]	Operative flow rate [L/min]	Volume [L]
Main loop	0.4	400	350	28	5.8
Secondary loop	0.4	400	350	0.3	3.8
Cold Trap	0.4	400	200	0.3	3.0
Hot Trap	0.4	750	650	stagnant	30

This report describes the experimental activities performed with Lifus 6 plant after the completion of the commissioning with Argon, corresponding to the filling with Lithium and the first months of Lithium circulation. As soon as Lithium circulation is started, Lithium purification is activated too, particularly its purification from Carbon, Oxygen and Hydrogen, whose compounds with Lithium can be precipitated as solid grains and kept confined inside the Cold Trap [8]. At the same time, the presence of Lithium inside the plant creates some new issues and dictates to perform new additional tests, for the control of Lithium temperature and flow rate in each part of the plant, the internal pressure and its drops, the filling and the draining procedures and the output of the Resistivity Meter, called to give an online answer about the total Lithium purity. The setup and optimization of all these aspects are described in the report, together with the solutions successfully employed to solve the encountered troubles.

The Nitrogen concentration in Lithium at the beginning of this experimental phase is reported too: it defines the starting point for the next purification phase, entailing the employment of the Hot Trap for the Nitrogen impurities removal.

2 Descrizione delle attività svolte e risultati

2.1 Check of the empty Resistivity Meter

The Resistivity Meter (RM) is a device installed inside Lifus 6 plant which is able to online measure the electrical resistance of the flowing Lithium. Actually, the RM is operated in parallel with the pipeline of Lifus 6 purification loop, just taking a trivial fraction of Lithium from it and measuring the resistance along its internal capillary. As already reported [5-8], the usefulness of this device relies on the knowledge that Lithium electrical resistance shall decrease the more is the purity of the metal (reaching its minimum value for a 100% pure Lithium). It must be considered, anyway, that the measured resistance (R_{tot}) doesn't correspond to Lithium alone resistance (R_{Li}), but includes also the contribution from the external capillary tube containing the metal (R_{cell}). In other words, the straight steel capillary and the Lithium inside act as two resistances in parallel, therefore to know the value of R_{Li} it is necessary to already know the value of the the resistance of the capillary when empty (R_{cell}) and employ the following expression:

$$R_{Li} = (R_{cell} \times R_{tot}) / (R_{cell} - R_{tot}) \quad (1)$$

The measure of the value of R_{cell} (at any desired temperature) must anyway be done before the first filling of the plant with Lithium, because after that we cannot be sure the internal capillary has been completely emptied. This value will be useful during the whole experimentation.

To get useful values of Resistance, it is anyway mandatory to dispose of a very precise control of the temperature, since we know that a change of even 0.1°C around 350°C translates into a resistance variation of about $0.2 \mu\Omega$. For this reason, the Resistivity Meter is monitored during time through the acquisition of 4 different temperature signals, each one related to a different Pt-100 sensor, installed along the RM capillary tube. The four temperature signals are indicated as TR1, TR2, TR3 and TR4, as shown in Figure 2, which is a detailed view of Lifus 6 P&ID in the RM region.

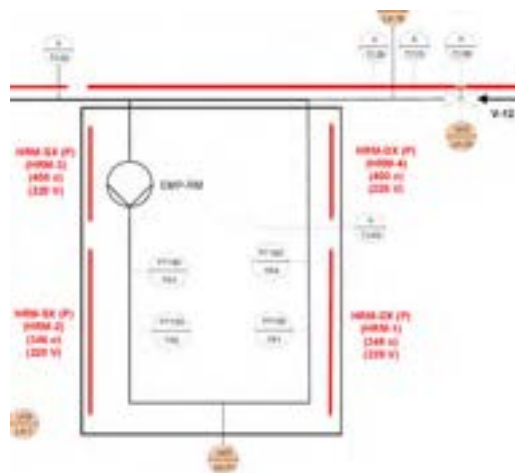


Figure 2: P&ID of the Resistivity Meter region (Lithium flows from the right)

The first test has been done at room temperature, with all the heating elements of the plant off (see Figure 3). The figure shows that in these conditions the outputs of the 4 Pt-100 sensors are very similar (they differ each other only of few hundredths of degree) and that each signal is very stable, with no drift or electrical noise at all. In fact, the stability of each signal is kept during time (about 3.5 minutes, with an acquisition

frequency of 1 Hz) with a maximum variation in the order of 0.001°C. The average temperature value (“Average TR”) is even more stable.

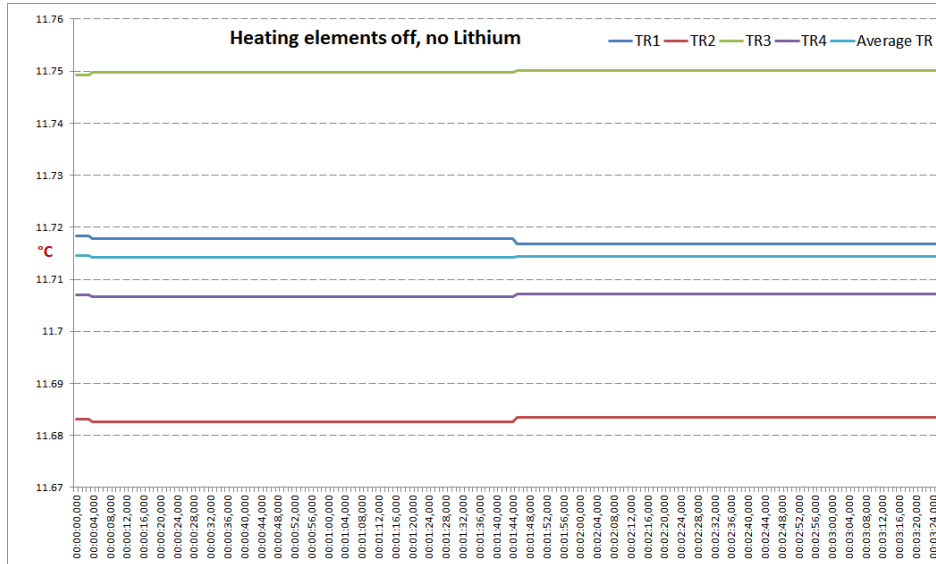


Figure 3: RM temperature signals variation with time at Brasimone room temperature (x axis= time, expressed in hh:mm:ss)

Figure 4 shows instead the temperature signals, when the RM is filled by Argon and operated at 300°C. Also now each signal is very stable, with a maximum oscillation contained in about 0.1°C, which is still a very good value considering that now the heating elements are continuously operating in order to maintain the proper thermal condition and that the investigated time is also very large (about 6 hours).

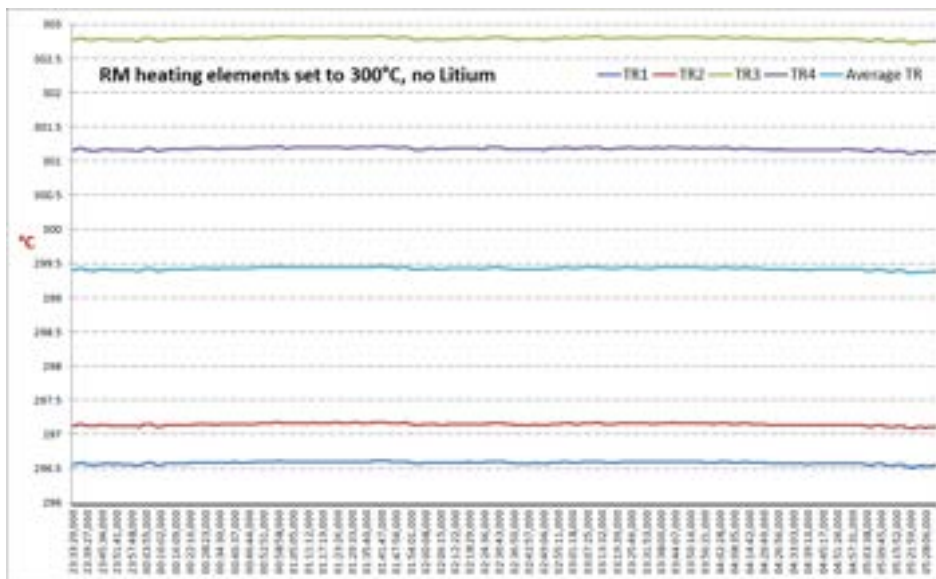


Figure 4: RM temperature signals variation with time when set at 300°C (x axis= time, expressed in hh:mm:ss)

When the RM was operated at Brasimone room temperature ($\langle T \rangle = 11.714^\circ\text{C}$; Figure 3 conditions), the acquired value of electrical resistance resulted $5.326 \text{ m}\Omega$ (very stable). Taking into account the theoretical 316L steel resistivity at this temperature ($0.764 \text{ }\mu\Omega \text{ m}$, as extrapolated from Table 4 of [9]) and the geometry of the RM capillary (as presented in [8]: length = 150 mm; area: $2 \cdot \pi \cdot (2.4^2 - 1.5^2) = 22.05 \text{ mm}^2$), the theoretical resistance should have been about $5.20 \text{ m}\Omega$. The agreement of the measured/theoretical values is very good and indicates the RM is giving values which make sense.

The trend of the electrical Resistance of the empty capillary (without Lithium) with temperature is instead shown in Figure 5. It is possible to see that the Resistance is perfectly fitted by a parabolic curve, which takes into account not only the resistivity increase, but also the small change of the capillary geometry due to the steel deformation. This fitting curve allows to calculate with a very good confidence the Resistance of the steel alone at each desired temperature. At 350°C , which is the selected operative Temperature of the plant and the Resistivity Meter during the erosion-corrosion test, the electrical Resistance results: $7.694 \text{ m}\Omega$.

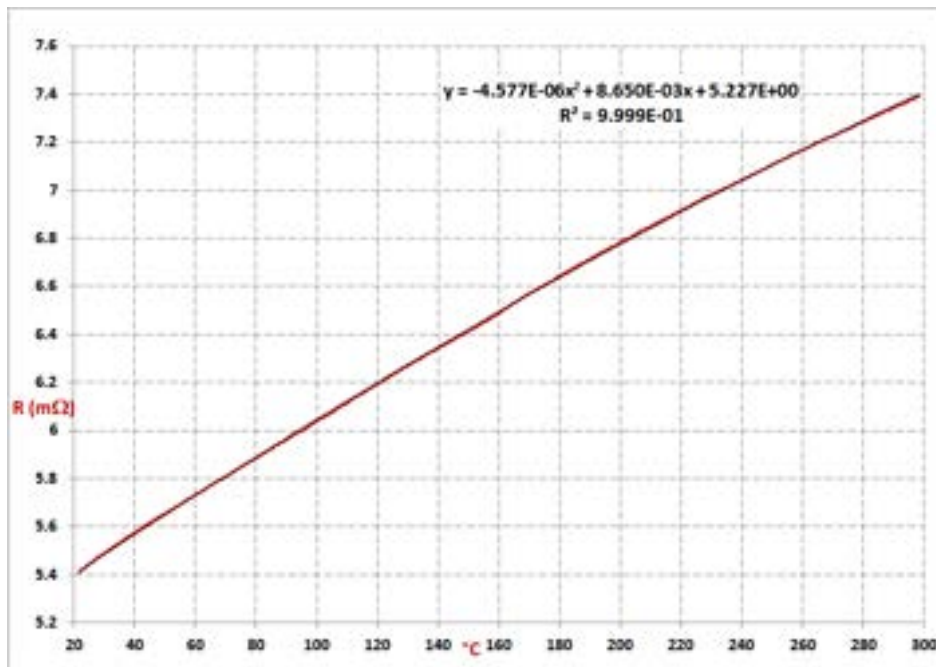


Figure 5: empty RM electrical resistance vs temperature. Red plot: experimental points; black plot: fitting curve

2.2 Filling of the plant with Lithium

Once all the commissioning test of Lifus 6 plant in Argon have been completed and the empty RM Resistance investigated, the plant has been filled with Lithium ($\sim 16.7 \text{ L}$). It is important to say that this Lithium is not fresh one, but comes from the old Lifus 3 plant, in ENEA Brasimone centre, where it was already employed for a previous experimentation [10] and then stored in solid phase at room temperature for many years. It must be also emphasized that in this test phase the Test Section doesn't hold yet the real specimens which will be subjected to the corrosion tests, but dummies specimens, having anyway the same geometry, to create the same identical Lithium flow condition. Real specimens will substitute dummies only after the conclusion of the Lithium purification phase, just before the start of the corrosion tests.

Firstly, Lithium has been transferred from the old Lifus 3 plant to Lifus 6 Storage tank, through a temporary branch in the Hot Trap to Storage Tank connecting pipe (blue in Figure 1); then it has been transferred from the Storage Tank to the above loop, by pressurizing the Tank with Argon gas. Since Lithium temperature

inside the Storage Tank is 250°C, while the empty loop is already set at 350°C, it is possible to follow the upward movement of Lithium during the filling, by observing the small momentary decrease of the measured local temperature caused by the incoming Lithium.

The arrival of the Lithium in the top of the Test Section is anyway better indicated by the signal coming from the related sensor level: this corresponds to the completion of the filling procedure and activates the closing of the Lithium exiting valve from the Storage Tank. Finally the main electromagnetic pump for the Lithium movement throughout the plant is switched on, at low rpm, and then raised to reach the desired flow rate.

2.3 Purification exerted by the Cold Trap

Once Lithium circulation started, the first phase of Lithium purification started too, since, during the circulation the metal crosses the Cold Trap, which is able to significantly reduce Carbon, Oxygen and Hydrogen concentration. A quantification of the content of these impurities during time is not possible, but it is possible anyway to perform a theoretical evaluation to have an idea of the time required for the complete purification.

The Cold Trap was dimensioned in order to set a defined value of Lithium residence time. The precipitation equilibria occurring inside the trap, in fact, require that the residence time is greater than a minimum value, in order to assure a good trap efficiency. Due to the lack in literature of specific studies about Lithium Cold Traps, ENEA decided to trust the experimental observations and results of Sodium Cold Traps [11-14]. Particularly, Grundy [11] reported, for a Sodium trap packed with a stainless steel mesh, that the efficiency ε depends on the residence time t according to the relation:

$$\varepsilon = \frac{1}{1 + 122t^{-3.4}} \quad (2)$$

where ε is defined as:
$$\frac{C - C_{out}}{C - C_e} \quad (3)$$

C is the concentration of each impurity in Lithium entering the trap;

C_{out} is the concentration at the exit of the trap;

C_e is the equilibrium theoretical concentration (minimum value achievable with the trap).

From the expression for the efficiency above reported, it's possible to calculate that, setting a residence time of 5-6 min, the efficiency results about 0.7-0.8, which is already an appreciable value: this means that the liquid metal will exit the Cold Trap containing about 20-30% of the total removable impurities. In Lifus 6 case, where the flowing metal is Lithium, we decided to roughly double the value of the residence time, leading it to about 10 minutes: should the precipitation equilibria be slower than for Sodium, this raised value could be enough to guarantee that similar purification efficiency are anyway achieved.

Even if the trap efficiency is lower than 1, the purification of Lithium from impurities can approach the value of 100%, considering that the metal does not pass through the Cold Trap only once, but continuously during the plant operations. Although the Lithium flow rate in the purification loop is only 1% of the main loop

flow rate, taking into account that the purified Lithium exiting the Cold Trap mixes continuously with the unpurified one at the exit of the purification loop, it follows that the totality of the circulating metal will be subjected, through iterative steps, to the purification. A rough estimate of the overall time required for the completion of this procedure is possible.

Let's consider that the impurity concentration is constant throughout Lifus 6 plant: in this case we may speak in terms of a 'medium' concentration along the main loop and we can monitor how does it change with time. From the conservation of mass, we can impose that the decrease of the impurity in the main loop must, in any time, be balanced by the mass of the impurity retained by the Cold Trap:

$$-dM_m(t) = M_i(t) - M_{out}(t) \quad (4)$$

where: M_m = mass of the impurity in the main loop

M_i = mass of the impurity entering the Cold Trap

M_{out} = mass of the impurity at the exit of the Cold Trap

Speaking in terms of concentrations and considering that the concentration at the entrance of the Cold Trap is equal to the concentration inside the main loop we obtain:

$$-\frac{dC(t)}{dt}W = \phi[C(t) - C_{out}(t)] \quad (5)$$

where: W = total Lithium mass inside the main loop

ϕ = Lithium mass flow rate inside the Cold Trap

C = mass concentration inside the loop

C_{out} = mass concentration at the exit of the Cold Trap

t = circulation time

Integrating the above equation and remembering the definition of ε in equation (3), we obtain:

$$\ln \frac{C - C_e}{C_o - C_e} = -\frac{\varepsilon\phi}{W}t \quad (6)$$

where C_o is the starting concentration value.

Assuming the value of 0.052 min^{-1} for (ϕ/W) ($= 0.3/5.8$; our configuration: see Table 1) and the ending point for the complete purification corresponding to: $(C - C_e)/(C_o - C_e) \leq 0.01$, it is possible to roughly estimate the time required for the purification as a function of ε ; the results are shown in Table 2. Even with a Trap efficiency equal to 0.5, about 3 hours can be considered enough to reach the ending point of the purification of all the circulating Lithium.

Table 2: Estimated time required to reduce the residual impurities relative content to 1% of all the removable impurities, according to equation (6)

Supposed Efficiency (ϵ)	1	0.9	0.8	0.7	0.6	0.5
Required Time (min) ^{1,2}	88	98	111	126	147	177

¹a 10 minutes (= CT residence time) startup should be add to these values

²in absence of other impurities entering the plant during the purification time

Not all the Lithium is actually circulating, because the loop volume is about 50% of all the Lithium inventory, while the remaining Lithium is kept in the Storage Tank. Anyway, during the long phase of Lithium circulation (many months), Lithium was often discharged in the Storage Tank and then, after a variable time, transferred back in the plant and circulated again (this happens also each time the plant was necessarily stopped to solve emerged issues of the plant and implement different solutions/optimizations – see section 2.5). This gave the chance each time to mix purified (circulated) Lithium with unpurified one (static Lithium in the Storage Tank).

At the end of this long phase, even if many stops of the circulation occurred, we can hence calculate that the effective circulation time is of the order of some weeks and that the Lithium composition has begun homogeneous. According to table 2 values, the Cold Trap disposed of sufficiently large time to purify all the Lithium inventory.

2.4 Check of the Air-cooler upstream of the Cold Trap

As shown in Table 1, the Cold Trap requires to be operated at 200°C to reduce the solubility of Lithium impurities. Since the Temperature of the rest of the plant is 350°C (the corrosion test are requested by LF03 PA to be performed at 350°C), an Air-cooler (AC-2) was installed upstream of the Cold Trap to lower the incoming Lithium temperature to 200°C. The Air-cooler, dimensioned to remove 1.65 kW of thermal content from the flowing Lithium, consists of an external stainless steel concentric jacket (square section) around the Lithium pipe; the jacket is swept by a controlled flow of air, which is the real responsible for the heat removal. Figure 6 is an enlarged view of Lifus 6 P&ID in the Cold Trap Air-cooler region, while Figure 7 is a screenshot of the Lifus 6 control software window for the Air cooler control.

The software is able to automatically manage the air flow rate in the external jacket of the Air-cooler to get the target Lithium output temperature. The upper part of the window (AC2-SCADA) is for setting the desired target values; the lower part (AC2-RT) is for real time reading the functioning parameters.

After a very short setup period, it has been possible to verify the proper functioning of the Air-cooler and the stability of the Temperature inside the Cold Trap. Figure 8 shows the contemporary (real time) acquisition of the Temperature signals related to both the bottom of the Test Section of the plant (TC10) and the inside of the Cold Trap (TC38: lower Cold Trap; TC39: upper Cold Trap). It is possible to see that for all the time shown in the x-axis (more than 12 hours), each temperature signal is constant and fixed on its desired value.

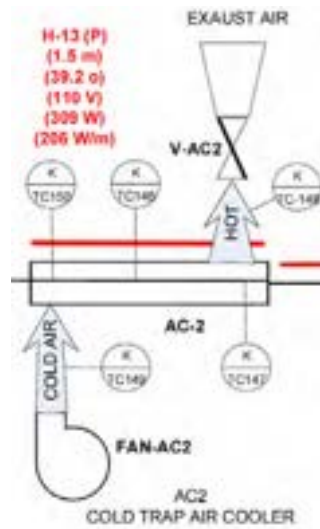


Figure 6: P&ID of Lifus 6 in the Cold Trap Air-cooler region (AC-2).

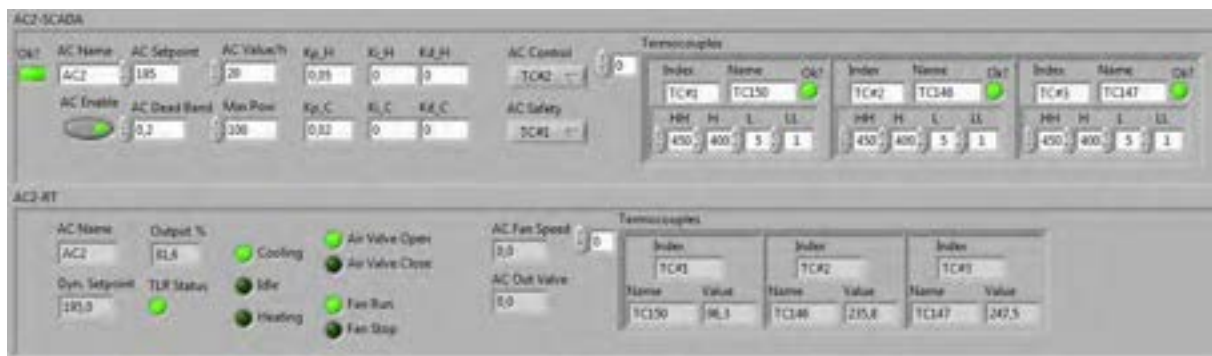


Figure 7: screenshot of the software window for the control of the Cold Trap Air-cooler (AC-2)

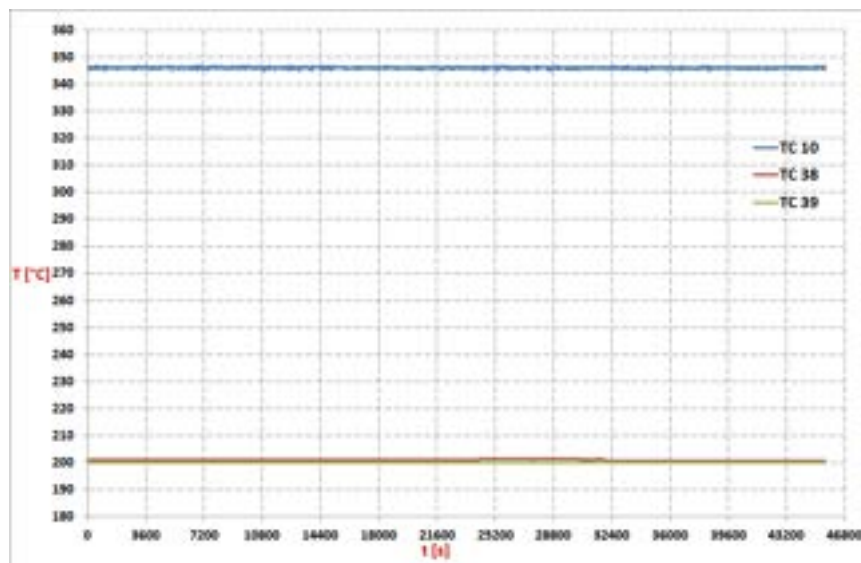


Figure 8: Measured Temperatures versus acquisition time. TC10: bottom of Test Section; TC38: lower Cold Trap; TC39: upper Cold Trap.

Considering the constancy of the Cold Trap temperature and the constancy of the Lithium flow rate through it (300 mL/min), as revealed by the mini-flowmeter downstream of the trap, it is possible to exclude Lithium solidification inside the Trap and plugging problems. We can conclude the Cold Trap worked exactly in the requested thermo-hydraulic conditions.

2.5 Problems encountered during the first months of Lithium circulation

2.5.1 Problems in the Test Section

After few days of Lithium circulation, a sudden increase of the pressure drop in the Test Section occurred. This fact prevented the Lithium to reach the reference velocity of 15 m/s. To solve the problem, the Test Section was opened and investigated with an endoscopic probe. As shown in Figure 9, clear flakes of unknown material were discovered on the bottom of the section. The SEM-EDS analysis of these aggregates indicated the main presence of Carbon and Oxygen, and a minor content of Iron, Chromium and Aluminum (Lithium cannot be detected by EDS). A Carbon-Oxygen-Lithium composition was then inferred, as originated by a locally high (at the beginning) concentration of Oxygen and Carbon in Lithium, the latter probably coming from remains of oil and lubricants on the wall of the pipes.

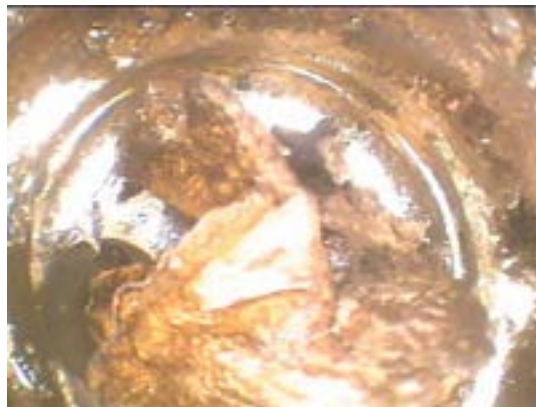


Figure 9: solid aggregates at the bottom of the Test Section

The Test Section was hence cleaned and assembled again, then Lithium circulation was restarted. Moreover it was decided to increase the operating pressure of the loop to 3 bar, to avoid flow cavitation in the Test Section, which is characterized by the highest Lithium linear velocity.

Another problem occurred anyway in the following weeks. It seemed that during the circulation Lithium reached a too high quote in the Test Section, far above the one desired and associated to the proper sensor level. This phenome, also related to the presence of not easily removable Argon bubbles in the plant, was accompanied by a low and unstable flow rate of Lithium, by turbulences at the inlet of the Test Section and by Lithium squirts on the top of it, contaminating at some extent the gas lines here interfaced. To solve the problem, ENEA has decided to add an expansion vessel (nothing but a cylindrical tank: the design is shown in Figure 10) on the top of the Section and to fill the entire Test Section with Lithium up to the connecting pipe to the new vessel. The reference sensor levels for Lithium are now installed directly inside the expansion vessel: if the highest one is contacted by the metal, it suddenly activates the draining procedure. The expansion vessel is not installed coaxially with the Test Section, but slightly sideways; this avoids blocking the access to the Test Section and impeding a prompt specimen insertion/removal.

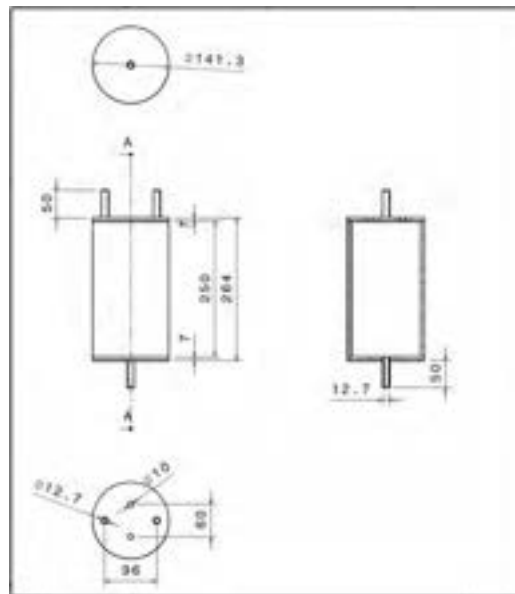


Figure 10: Design of the expansion vessel above the Test Section. Physical dimensions are expressed in mm.

2.5.2 Problem with the mini-flowmeter along the purification loop

While the Lithium flowmeters installed along the main Lifus 6 loop demonstrated to work properly, it was noted that the micro-turbine flowmeter located inside the purification loop downstream of the Cold Trap (commercial product: *Hoffer Flow Control Inc.* mod. MF1/2X40B-.02-.15-T-1HTM-1/2-TS s.n. 151293, equipped with Pick-up mod. PC28-13G-43 and pulse to 4-20 mA converter mod. CAT 1-1-7-D-CE, s.n. 151294) didn't indicate flow at all, constantly measuring flow rate = 0 (the detected current was constantly 4.00 mA, on a output scale 4-20 mA).

In order to understand the nature of this malfunctioning, the flowmeter was removed from the plant and cleaned with demineralized water at 80°C. After verifying the functioning of the Pick-up and the electrical signal from the meter to the acquisition software, with the approval of the supplier the flowmeter was completely disassembled (see Figure 11) using the procedure described in its own manual.

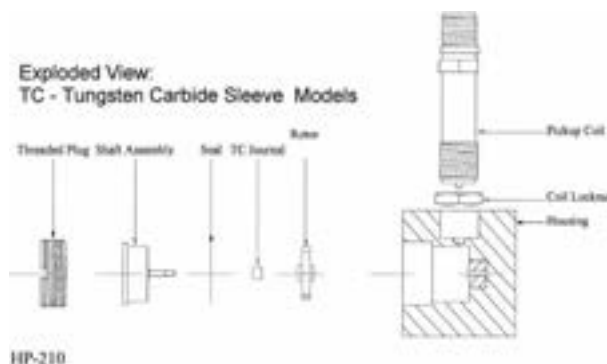


Figure 11: exploded view of the miniflowmeter mechanical drawing



Figure 12: “black mud” after shaft assembly removal

After removing the shaft assembly, the gasket/seal was not found in its seat; moreover several grams of a “black mud” were found under the shaft assembly and around the internal volume that hosts the rotor (Figure 12). The semi-quantitative analysis of this black mud reveals the presence of Carbon, Oxygen and Fluorine, suggesting it as the residual of the fluorine polymer (PTFE) seal after its reaction with liquid Lithium. This hypothesis is further corroborated by the fact that two mechanical filters both upstream and downstream of the mini-flowmeter exclude the ingress of solid particles. The maximum operating temperature of a PTFE seal is around 250°C and this material is not compatible (because is reactive) with alkaline metals. Considering that the housing of the flowmeter was isothermal during the operation and that the seal side not in contact with lithium appears still in good condition, it can be concluded that the seal was destroyed by the chemical action, not by the excessive temperature.

The flowmeter was then re-assembled with cleaned components. Since it was not possible to get a new (metallic) seal in reasonable time from the instrument supplier, ENEA decided to weld a metallic cover sheet of AISI 314 over the threaded plug, as shown in Fig. 13. This kind of sealing was checked using pressurized air at 8 bar. The flowmeter was immersed in demineralized water for ten minutes and no air bubble was detected. In the absence of any visually detectable leakage, the sealing quality of the welding has been qualified as good enough for the Lifus 6 application (max pressure = 4 bar).



Figure 13: Flowmeter with welded cover sheet

Then the flowmeter calibration was re-checked in ENEA lab using a specifically arranged water loop (Figure 14), based on a voltage controlled pump (ZKSJ brushless immersion pump, supply voltage = 3-12 V, $\Delta P_{\max} = 0.5$ bar, $\text{flow}_{\max} = 125$ mL/s). The pump was connected with a plastic transparent tube to the inlet of the flowmeter, while its output went to a secondary tank. An electronic balance (± 1 g) was employed to measure the total volume of pumped water; a chronometer was used to measure a standard time (one or two minutes, ± 1 s accuracy) and so calculate the average flowrate. A digital oscilloscope measured the output signal from the pick-up; the same signal went to the pulse/current converter. A second 24V DC source PSU2 supplied the power for the pulse/current converter; a digital ammeter measured the output current (e.g. the flow signal from the flowmeter sensor under test). This way, the calibration chart reported in Table 3 was obtained, showing also the agreement with the original values from the supplier (*Hoffer*).

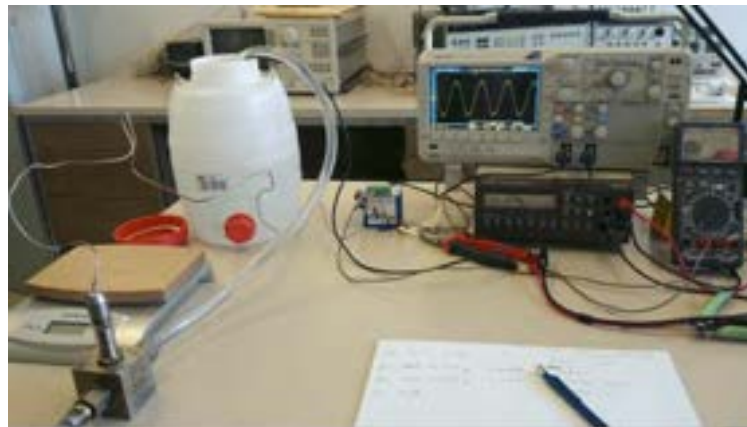


Figure 14: Water test loop (the immersion pump is inside the tank)

Table 3: F-3 Flowmeter calibration chart, after ENEA maintenance operation

Cal. Point	V_{pump} [Volt]	Freq. [Hz]	$V_{\text{pick-up}}$ [mV]	I_m [mA]	Vol. [mL]	Time (min)	Flow rate [L min ⁻¹]	Hoffer Cal. Table Flow rate [L min ⁻¹]	Deviation (%)
1	07.24	458	160	08.00	538	2	0.269	0.284	-5.3
2	17.50	987	248	20.00	539	1	0.539	0.568	-8.0

The deviation around the flow rate selected for Lifus 6 operation (0.3 L min^{-1}) is good ($\sim 5\%$) and surely acceptable, considering such a variation in Lithium residence time inside the Cold Trap would affect very poorly the trap efficiency.

2.5.3 Additional issues and checks

Some additional issues have emerged during the circulation of Lithium. One of them is related to V-12, the regulating valve at the entry of the purification loop. This valve was set to reduce the incoming Lithium flow rate from about 28 L min^{-1} (value in the main loop) to about 0.3 L min^{-1} , but it failed to satisfy the requirement, since its regulation was not fine enough (either it was fully closed or too much open). To overcome this problem, a new additional valve was installed during the first months of 2015 at the exit of the purification loop. This valve is a manual one, not remotely controlled by the software, but after a

lengthy setting procedure the desired Lithium flow rate was obtained. This setting will not be changed any more during the experimental campaign, unless specific operations or maintenance will require it.

Another check performed during this period studied the transfer procedure of Lithium from the Storage Tank to the Hot Trap and back (at low temperature, $\sim 250^\circ$), to prevent possible problems during the future purification phase by the Hot Trap ($550-650^\circ\text{C}$). The procedure worked but about 25% (4 L) of Lithium remained 'trapped' in the Hot Trap, i.e. in the bottom dead volume and in the internal structure of the Titanium sponge (getter). For this reason some Lithium was added into Lifus 6, transferring it from the old Lifus 3 plant, to retrieve the proper level of metal in the Storage Tank.

Finally, as a consequence of the above described changes to the plants, it has been necessary in many cases to modify the DACS software too, by introducing new additional control variables or changing some operative parameter.

2.6 Check of the Resistivity Meter with flowing Lithium

Once the Lithium circulation has started, it has been also possible to monitor the functioning of the Resistivity Meter in presence of flowing Lithium. After a first setup phase, aimed mostly at reducing the presence of parasitic currents and their effect on the signal noise, it has been possible to get the device operating in stable and meaningful conditions. Figure 15 shows the output of the 4 Pt-100 sensors (TR) set in contact with the RM capillary, during 4 hours time (14400 seconds). It is possible to see that each signal is very stable, with a maximum oscillation of about 0.05°C . The trend of the signals are perfectly identical and reflect to some extent the variation of the temperature of the Lithium upstream of the RM; the absolute difference is very small, a part from TR3, which is higher because in the upper box of the RM and moreover close to one of the heating elements. It is possible to select one of the 4 signals as indicative of the Lithium temperature inside the RM, provided the absolute differences between the 4 signals are kept constant, or the average of the 4 signals.

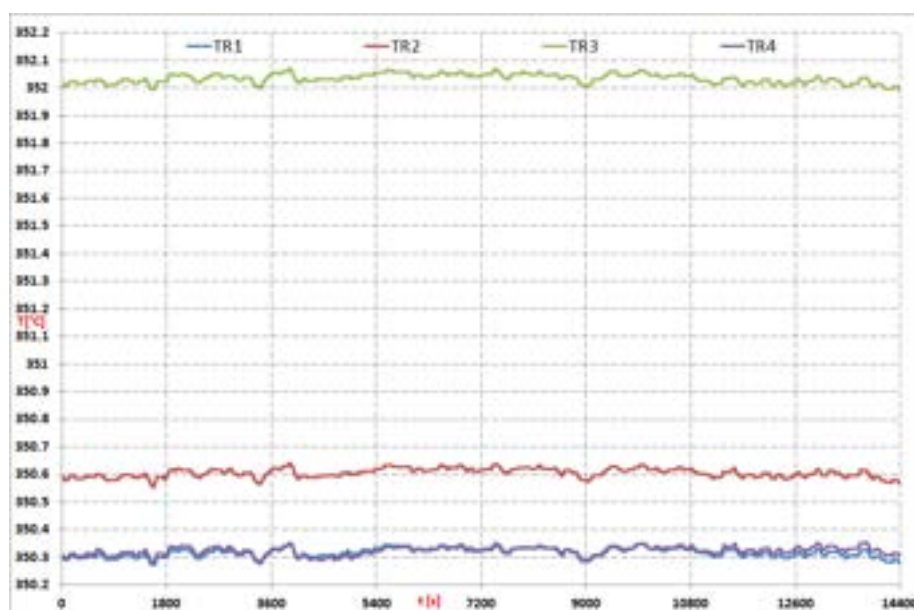


Figure 15: output of the 4 temperature sensors during Lithium operation (see Figure 2 for the location of each TR)

For what concerns the corresponding Resistance values, they are shown in Figure 16. Apparently the signal is characterized by a significant noise, but we have to consider how fine is the y-axis scale. Actually it can be seen that the signal noise is at maximum only $0.5 \mu\Omega$ around the central value; some low frequency oscillations can be somehow related also to the small temperature variations, since we know that theoretically a 0.1°C variation around 350°C should translate into a Resistance variation equal to $\sim 0.2 \mu\Omega$.

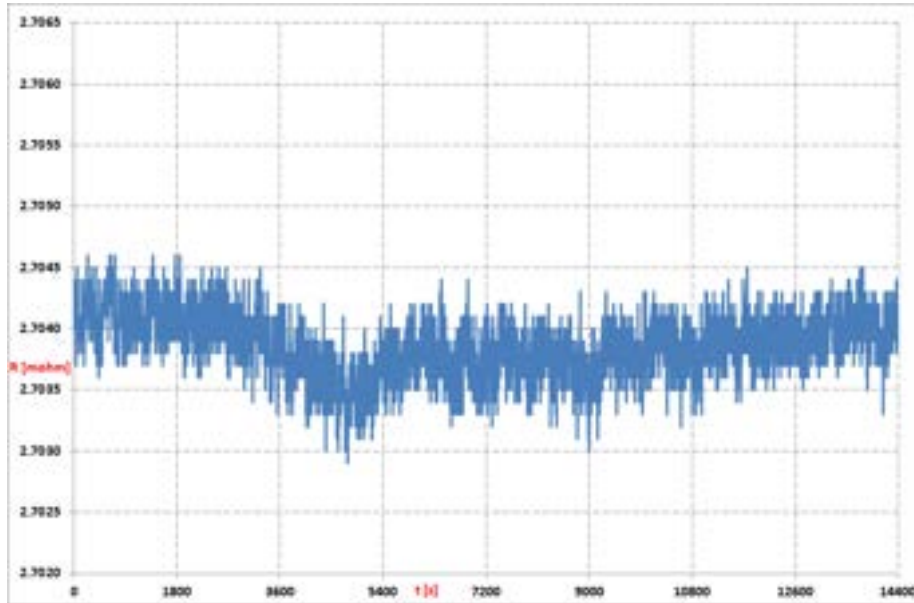


Figure 16: electrical resistance output of the RM during Lithium operation

In such conditions, the RM is able to provide a qualitative description of the status of the flowing Lithium, since we know [8] that a Resistance variation of $0.5 \mu\Omega$ (easily seen in the plot) corresponds to a Nitrogen concentration variation of only 30 wppm.

2.7 Chemical analysis of Lithium at the start of the purification phase

A reference starting value for the Nitrogen concentration in Lithium has been achieved by the off-line analysis of a sample of Lithium taken from the purification loop just after the first contact of Lithium with all the parts of the plant (included the Hot Trap) and the additional transfer of 4 Liters of Lithium to the plant. Operating inside the glovebox, the sampler was cut at two different locations. From each cut about 0.6 g of Lithium were taken for the specific chemical analysis. The details of the general procedure are reported in [5,7], while the details of this analysis are reported in Table 4. The result is that Nitrogen concentration in starting Lithium is: 70.4 ± 2.7 wppm.

This value is not significantly changed during the first phase of the purification, since the Cold Trap doesn't capture Nitrogen from Lithium. On the contrary, this value will decrease during the second phase of the purification, involving the Hot Trap and its Titanium getter. Therefore, during this second phase, which has just started at the beginning of September, frequent samplings and chemical analyses of Lithium will be performed, to monitor during time the efficiency of the procedure, hopefully finding an endorsement from the online output of the Resistivity Meter.

Table 4: Details of the analysis result of the first Lithium sampled from the Lifus 6 loop
*** after subtracting the blank value (4.0 µg)**

Analysis n°	Lithium mass analyzed [g]	Nitrogen mass found [mg]*	Nitrogen concentration in Lithium [wppm]
1	0.5991	0.0409	68.3
2	0.6700	0.0485	72.4
Average			70.4
Systematic error (2.5%)			1.76
Std. dev.			2.90
Random uncertainty			2.04
Total uncertainty			2.70

3 Conclusioni

Since the last quarter of 2014, Lithium circulation inside Lifus 6 plant has started. During all the following months, the first phase of Lithium purification has been operated, reducing, thanks to employment of the Cold Trap, the concentration of Carbon, Hydrogen and Oxygen impurities in Lithium. At the same time, many plant issues and troubles have emerged, forcing in many cases to stop the circulation for a while, until the proper solution or optimization has been found and implemented: all the problems have been anyway internally solved and the plant reached a full ripeness. The purification of Lithium has been hence operated discontinuously during time, but the overall integrated time can be surely considered higher than the requested one for an efficient purification, on the basis of the theoretical evaluations.

For what concerns the Nitrogen impurities solved in Lithium, they have not been affected by the first phase of the purification. The second phase, entailing the use of the Hot Trap at 550-650°C, will instead be able to reduce Nitrogen concentration from its starting value of about 70 wppm to at least 30 wppm, the value requested for the following corrosion tests. This second phase has just started at the beginning of September 2015 and will be concluded before the end of November.

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5 Abbreviazioni ed acronimi

AC	Air-cooler
C	Concentration
DC	Direct Current
EDS	Energy Dispersion Spectroscopy
EMP	Electromagnetic Pump
IFMIF	International Fusion Materials Irradiation Facility
M	Mass
PA	Procurement Arrangement
PTFE	Polytetrafluoroethylene
R	Electrical Resistance
RM	Resistivity Meter
SEM	Scanning Electron Microscopy
t	Time
TC	Thermocouple
TR	Thermo-resistance (Temperature monitor)
W	Total Lithium mass inside the Main Loop
wppm	Weight Parts per Million
ε	Efficiency
ϕ	Mass Flow Rate inside the Cold Trap