

Causes of Failure of Experimental Molten Salt Research Device

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Abstract: In today's practice, molten salts are used in thermal energy storage technologies, solar tower technology, and the development of Generation IV nuclear reactors. The paper deals with the construction of an original experimental device for the research of the physical and chemical properties of molten salts, the behavior of molten salts, and corrosion effects of molten salts on various construction materials built in the Research Centre Řež. Due to experience with the first device construction, the risk-based design was chosen for the final device. The procedure of risk-based design is described. Based on this analysis, the critical points of the device were identified, the geometric modifications were made to the structure, and, with the help of the CFD tools, three modes of operation were established: normal, abnormal, critical in the event of a power outage. Resulting changes together with precisely defined operating regulations ensure safe construction, operation, and maintenance of the experimental device.

Keywords: Molten salt, device failure, risk-based design, risk-based operation, CFD tool, checklist, reconstruction, molten salt loop.

1. Introduction

In today's practice, molten salts are used in thermal energy storage technologies, solar tower technology (Dunn et al., 2012), and in the development of Generation IV nuclear reactors (Serp et al. 2014). In the nuclear industry, the most significant advantages of liquid salts undoubtedly include the possibility of achieving relatively high temperatures at atmospheric pressure, which leads to higher efficiency and safety of power plants using molten salts. Liquid salts have a high boiling temperature and a high density compared to water.

In nuclear power plants, molten salts might be used as coolant as well as fuel carrier. A combination of solid fuel and molten salt coolant has different advantages compared to molten salt fuel. Therefore, depending on a particular design, the following additional advantages might be associated with the use of molten salts:

- the elimination of the need for fuel fabrication,
- the possibility of continuous refueling,
- the possibility of online fission products removing.

Despite these indisputable advantages, some aspects of molten salts (e.g., corrosion effects, melting, and solidification) are little explored. Therefore, the behavior of molten salts and the technology of their use in practice is an important research subject. An experimental Molten Salt Loop (MSL) device was built at the Research Centre Řež (CVŘ) for the purpose of studying the material properties of selected salts (Pisko 2015).

During the MSL loop commissioning, several minor or more significant failures and defects occurred. Analysis of the causes of these defects showed that their origin was in both the device structure's incorrect design and the operating procedures (Cihlár & Mareček 2020). Based on this

analysis, the critical points of the device were identified, and geometric modifications were made to the structure. Moreover, with the help of the CFD (Computational fluid dynamics) tools, three modes of operation were established: normal; abnormal; critical in the event of a power outage.

Therefore, the risk-based design is used in the construction of the new equipment. Two procedures are used to do this. The aim of both procedures is to ensure process safety by reducing criticality. The first procedure is based on an analysis of the failure of the original installation – its critical points are identified based on technical principles and considering the possible phenomena that may affect the device operation, including the human factor; i.e., a checklist and scale for its assessment have been drawn up for assessment. The second procedure is theoretical and applied to the CFD models.

2. Benefits of Molten Salts for Nuclear Energy

Molten salts are considered as one of the promising coolants of future Generation IV nuclear power reactors. Compared to the most common working medium of human activities – water, the molten salts possess some specific properties, which include higher density, high values of dynamic viscosity, and higher thermal conductivity coefficient (Janz 1967). In practice, nitrate salts such as $\text{NaNO}_3\text{-KNO}_3$ for concentration solar power plants or carbonate salts (e.g., $\text{Li}_2\text{CO}_3\text{-Na}_2\text{CO}_3$) for high-temperature heat storage are currently used (Tian & Zhao 2013).

In the nuclear industry, fluoride salts FLiBe (LiF-BeF_2), FLiNaK (LiF-NaF-KF), or NaF-NaBF₄ are most often considered for use and research. Compared to the currently commercially used pressure reactors and water as a cooling medium, molten salts and salt reactors have, according to (Serp et al. 2014), the following advantages: high boiling temperature; high coolant output temperature; low pressure in the primary loop; elimination of the fuel fabrication; potential continuous refueling; potential continuous fissile products removing; and a possibility to work in breeding mode.

The use of liquid salts may also entail some unacceptable consequences caused by, e.g., high corrosion aggressivity (Ignatiev & Surenkov 2013, Kondo et al. 2009, Muránsky et al. 2019) towards structural materials (IAEA 2020); high melting temperature (Williams 2006); toxicity of salt compounds (Stefaniak 2011, Strupp 2011a, Strupp 2011b); inappropriate neutron properties; underdevelopment of the measurement devices and techniques (Gill et al. 2014, Sabharwall et al. 2010); and lack of operational experience. All the advantages and disadvantages are dependent on a particular design, and not all of them are included in every design.

In the Czech Republic, long-term experience with liquid salts is mainly in CVŘ and ÚJV Řež (Uhlíř & Mareček 2005, Uhlíř 2008). Therefore, an experimental device, "experimental FLiBe loop (MSL – Molten Salt Loop)," was built in CVŘ designed to study the behavior of molten salts and corrosion effects on various structural materials.

3. Safety and Reliability of Results of Experiment

In particular, we need factual data to solve the task of monitoring the properties of molten salts in our case. To obtain them by measurement, we need a high-quality method of measuring data from experiments, which are processed by an appropriate mathematical method. For the data obtained to be credible, the measurement process must be:

- sufficiently flexible,
- transparent,
- repeatable,
- accurate in the sense that it ensures the same results when repeated,
- correct in the sense that both types of uncertainties, the random and the knowledge, are evaluated.

To meet these requirements, it is necessary to have a safe measuring apparatus, a safe measuring procedure, and the ability to perform the measurement in a safe environment (Procházková 2011).

The measuring apparatus consists of a number of more or less complex elements, components, and systems. By the end of the 1980s, there was talk of a quality spiral in the creation of technical systems. Today we use safety management (Procházková 2017). The safety of each technical device is determined by many factors. At the design stage, it is about determining the correct specifications, which must respect the characteristics of the place in which the technical equipment is placed. Furthermore, these are measures built into the project that will facilitate the management of safety in operation under normal, abnormal, and critical conditions of different kinds (Procházková, Procházka, Lukavský, Beran, Šindlerová 2019).

In line with current knowledge, it should be ensured that existing norms and standards are applied because, without standards and legislation, experts and the professional public would be condemned to repeat the mistakes of the past and to add adjustments based on an evaluation of possible risks, which have origin in changes in time and the aging of materials that are the causes of knowledge uncertainties (Procházková 2017, Procházková, Procházka, Lukavský, Beran, Šindlerová 2019).

The experimental device "experimental FLiBe loop MSL" is a unique experimental device built in CVŘ and the first similar device designed by the manufacturers ExPS and TARPO. It is designed to study molten salts' behavior, the acquisition of technology for preparation and handling of molten salts, and material research. Due to the lack of operational experience and not the ideal design of the MSL, several minor failures occurred during the initial operation.

Faults and defects that occurred: flange leaks; unreliable detection of molten salt level; malfunction of the horizontal test reservoir; melt leakage; rupture of the flange joint; and a torn weld. The most damage occurred during the night operation when the operation was interrupted, the molten salt froze, and uneven melting of the frozen salt during restart happened. This uneven salt melting caused a large crack due to the thermal expansion of some already molten salt pushing on some still frozen salt bulks. The pressure caused the flange on the perpendicular part of the interconnecting pipe to rupture (Cihlár & Mareček 2020).

The fact stopped further use of the experimental loop, so we decided to make a major modernization, at which we consider all the risks. From the point of view of contemporary knowledge, there are used for experimental device design and operation the principles:

- a risk-based design (Procházková, Procházka, Lukavský, Beran, Šindlerová 2019),
- risk-based operation (Procházková, Procházka, Lukavský, Dostál, Procházka, Ouhrabka 2019).

4. Data

In the monitored case, the experimental device is in a closed hall (Fig. 1), in which temperature and humidity control are carried out, and requirements of the norm (ČSN 33-2000-1 ed.2 2009) are met.

The experimental device is located on a supporting steel structure and is powered by a steel-and-steel switchboard. Inlet cables and inert gas pipes are located under the floor. The experimental device (Fig. 2) shall have:

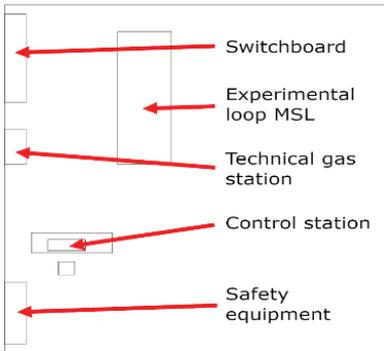


Fig. 1. The layout of the laboratory room with experimental loop MSL. Safety equipment includes but is not limited to fire extinguishing equipment, personal protective equipment, operating regulations, and safety data sheets.

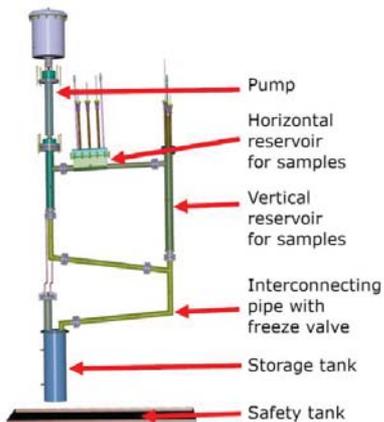


Fig. 2. Diagram of experimental loop MSL with its main components.

- eight components (technological units): handling storage tank with heating; heated pipe with freeze valve connecting the storage tank and the loop; the source of technical gases; experimental loop MSL; reservoirs for inserting samples; (pump); safety tank; and a forced ventilation and vacuuming system,
- heating elements that ensure the working temperature of the loop. The working temperature here is set to allow trouble-free operation and to ensure that the working medium remains liquid under all operating modes.

As the main construction material for the original design, the nickel-chromium alloy Inconel 718 was chosen. The working volume of the MSL loop is about 5.4 dm³. The heating is provided by heating modules consisting of metallic heating elements and vacuum-formed ceramic fiber insulation with a total power of about 23 kW.

Two sites of MSL are designated for insertion of the tested samples. During experiments, the loop itself is disconnected from the storage tank by a freeze valve in the interconnecting pipe. The freeze valve is a purposely frozen salt.

For research, it is necessary so the experimental loop may work:

- in two basic modes, namely with a natural flow and forced salt flow through the loop. The flow of the melt with natural convection is ensured by the difference of temperatures in different parts of the loop. This difference is achieved by regulating the heating elements. In forced flow mode, the circulation is ensured by a pump,
- both in short-term cycles where there will be frequent start-ups, shutdowns, cooling, etc., as well as in long-term steady-state cycles of hundreds of hours,
- and that short- and long-term operating cycles could take place in both operating modes, with both natural and forced flow.

5. Methods used to prepare risk-based design

When creating a risk-based design, the following methods are used: checklist and CFD. The checklist is used to assess component risks, link components, and the entire device. The CFD method is used to assess the operation modes.

5.1. Methodology for Compiling a Checklist for Risk-Based Design of Measuring Equipment

In accordance with the technique (Procházková, 2011), the following steps shall be followed when compiling checklists:

- specify the items to consider in the area to be monitored,
- create an order of items based on their severity for the area being tracked,
- identification of critical sites that are a source of risk (non-compliance means a severe risk),
- compiling checklist questions,
- determine how the checklist is to be assessed.

Based on data, and the model of the measurement process, a risk assessment checklist is drawn up in Table 1.

Table 1. Checklist for the risk-based design of the experimental device.

Order	Question	Answer 0 - 1
<i>Locality</i>		
1	Is the reliable function of air conditioning (temperature, humidity, ventilation) in the hall ensured?	
2	Is the condition of the switchboard in accordance with the safe operation?	
3	Is the condition of the technical gas station in accordance with safe operation standards?	
...		
10	Do the staff have knowledge and competencies in accordance with safe operation standards?	
...		
<i>Components of the experimental device</i>		
15	Is the pump (material, size, power) in accordance with safe operation standards?	
16	Is the storage tank heater - bottom (material, type, size, power) in accordance with safe operation standards?	
...		
19	Is the heater of pipe from/to the storage tank - section 1 (material, type, size, power) in accordance with safe operation standards?	
...		
34	Are the storage tanks (material, type, size) in accordance with safe operation standards?	
35	Are the individual pipe parts (material, type, size) in accordance with safe operation standards?	
36	Are the sensors (material, type, size, performance) in accordance with safe operation standards?	
...	...	
<i>Proposal of the design procedure and verification of the quality of the measuring equipment</i>		
39	Is the appropriate principle of inherent safety used in the design of the experimental device?	
40	Are passive safety systems used in the proposal?	
41	Are active safety systems used in the proposal?	
42	Is welding (material, type) of individual pipe parts, storage tanks, and others in accordance with safe operation standards?	
43	Is the pump assembly in accordance with safe operation standards?	
...		
53	Is the quality of loose and tight joints verified by appropriate non-destructive tests in harmony with safe operation standards?	
<i>Linked Parts</i>		
54	Was it verified that the supporting structure of the loop is robust enough to withstand the supercritical conditions caused by the explosion?	
55	Was it verified that the supporting structure of the loop is robust enough to withstand the supercritical conditions caused by the fire?	
56	Was it verified that the supporting structure of the loop is robust enough to withstand the supercritical conditions caused by a power failure?	
57	Was it verified that the supporting structure of the loop is robust enough to withstand the supercritical conditions caused by the impact?	
58	Was it verified that the supporting construction of the loop is robust enough to withstand the supercritical conditions caused by high temperature, i.e., a temperature exceeding the expected maximum operating temperature?	
...		
61	Was it verified that the set of reservoir, pipe and storage tank set is robust enough to withstand the supercritical conditions caused by fire?	

Table 1 (continued).

66	Was it verified that the set of reservoirs, pipe, storage tank, and pump is robust enough to withstand the supercritical conditions caused by fire?
	...
66	Was it verified that the set of reservoirs, pipe, storage tank, and pump is robust enough to withstand the supercritical conditions caused by fire?
	...
96	Was it verified that the hall, staff, and the whole loop are safe under supercritical conditions caused by fire?

The partial risks' assessment n_i shall be carried out in such a way that the question under assessment is assigned a value of 0 in the event of a negative answer or 1 in the case of a positive answer. Exceptionally, a value between 0 and 1 can also be assigned in the case of partially met conditions (Procházková 2018). First of all, the number of risk items in a given case should be determined for the assessment of the selected part or all risks, i.e., N ; n is the total sum of n_i in the followed part. The level of risk in the case under assessment R is equal to n/N as a percentage where N is 100 %. Its assessment is carried out according to the scale used in technical standards of the Czech Republic since the 1980s, Table 2.

Table 2. Value scale for determining the level of risk.

Risk rate	n/N [%]
Negligible – 0	More than 95 %
Low – 1	70–95 %
Medium – 2	45–70 %
High – 3	25–45 %
Very high – 4	5–25 %
Extremely high – 5	Less than 5 %

To build the risk-based design of the experimental device, in accordance with the findings at work (Procházková 2018), we will use the reference procedure to adjust according to the size of the risk level of the items listed in Table 3.

We then break down the risk according to the size of the risk rate in Table 3 into acceptable, conditionally acceptable, or unacceptable. The usual method of dividing according to knowledge summarized in (Procházková 2018) is following:

- if the level of integrated risk for the items in Table 3, calculated in accordance with Table 2, is negligible or low, the risk is acceptable,
- if the level of integrated risk calculated according to the items in Table 3 of Table 2 is medium or high, the risk is conditionally acceptable (ALARA),
- if the level of integrated risk calculated for the items in Table 3 according to Table 2 is very high or extremely high, the risk is not acceptable.

Table 3. Items of considered integrated risk for design modifications to the experimental device model and its location in the hall.

Domain	Items	Risk rate
Indoor Environment	1 – 12	
Components	13 – 38	
Construction procedure	39 – 53	
Gradually connected parts	54 – 58	
	59 – 63	
	64 – 68	
	69 – 73	
	74 – 78	
	84 – 88	
Whole loop	13 – 93	
Complex measuring equipment and environment	1 – 98	

Concerning the knowledge summary in (Procházková 2018, Procházková, Procházka, Lukavský, Beran, Šindlerová 2019, Procházková, Procházka, Lukavský, Dostál, Procházka, Ouhřabka 2019), where the risk is:

- acceptable, so there is no need to take further measures since the level of safety is at an acceptable level,
- conditionally acceptable, when technically and financially possible, technical measures (material, technical principle, construction procedure, barriers against the impact of critical phenomena, component backups) should reduce the rates of both partial and integrated risks and, if this is not technically and financially possible, enable the implementation of a response by means of additional technical equipment and organizational measures to enable the performance of the measuring equipment and the acceptable level of safety of the reference after the repair,
- unacceptable, it is necessary to implement essential technical measures in the field of material, technical principles, construction procedures, barriers against the impact of critical phenomena, component backups) in order to reduce the rates of partial, integrated risks and integral (overall) risk.

5.2. CFD Method

In our case, the CFD analysis (ANSYS 2017a,b) is used to define temperature limits and thermal stress limits for operation modes and transition modes of MSL. Among potentially critical modes belong some operating conditions (start-up, shut down, switching to a different temperature level) and the abnormal conditions (heating/cooling of the loop in the event of a power failure or a decrease in the ability to deflect heat from the loop space due to malfunctioning ventilation).

The application of CFD methods aims to define procedures based on simulations of selected transition states that ensure the safe operation of the loop during these states. It is the determination of limit heating rates or cooling of the loop as a whole. In practice, this means that both the maximum permitted power to individual pipe heaters and the maximum permissible power differences between individual heaters are determined on the basis of the operating condition. Simulated states include processes associated with the melting and solidification of molten salts.

6. Method of Creating a Risk-Based Design of Experimental Device

To build a risk-based design, we first compiled a safety manual containing the individual steps, their correct execution, and order. Description of the process of risk management used in the risk-based design procedure is given in (Prochazkova, Prochazka 2021); its scheme is in Figure 3.

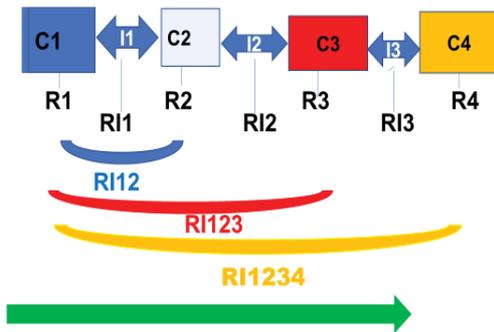


Fig. 3. Scheme of risk-based design.

Figure 3 shows the procedure of formation of risk-based design:

- we propose components (C1, C2, C3, C4) and their interfaces (I1, I2, I3) according to norms,
- according to Table 1 we determine risks of components (R1, R2, R3, R4) and interfaces (RI1, RI2, RI3) connected with disasters in the site and judge them according to Table 2 and if necessary, we make corrections of components or interfaces (material, construction procedure etc.),
- according to Table 1 we determine the integrated risk of set RI12 connected with disasters in the site and judge them according to Table 2 and if necessary, we

make corrections of components or interfaces (material, construction procedure etc.),

- according to Table 1 we determine the integrated risk of set RI123 connected with disasters in the site and judge them according to Table 2 and if necessary, we make corrections of components or interfaces (material, construction procedure etc.),
- according to Table 1 we determine the integrated risk of set RI1234 connected with disasters in the site and judge them according to Table 2 and if necessary, we make corrections of components or interfaces (material, construction procedure etc.).

We do not consider the responsibility and management way of the experiment by specialists and other service personnel in measuring, i.e., the causes associated with the human factor.

The real results of the method described in paragraph 5.1 are obtained by five specialists who independently judge the checklist. The median values of their judgments are given in Table 4.

Table 4. Risk rates (median values) for the experimental device model and its location in the hall.

Domain	Items	Risk rate [%]
Indoor Environment	1 – 12	0.83
Components	13 – 38	0.97
Construction procedure	39 – 53	0.65
Gradually connected parts	54 – 58	0.92
	59 – 63	0.96
	64 – 68	0.64
	69 – 73	0.64
	74 – 78	0.64
	84 – 88	0.56
Whole loop	13 – 93	0.79
Complex measuring equipment and environment	1 – 98	0.77

From Table 4 it follows that some risks are conditionally unacceptable (ALARA). The highest risks identified represent the supercritical conditions caused by the fire and caused by the high temperature, i.e., a temperature exceeding the expected maximum operating temperature.

The biggest contribution to the risks is caused mainly by all the types of joints and by the pump. The weak places of joints are at welds, sealings and fittings. Therefore, many procedures were modified including the proper welding techniques, welding certifications and appropriate non-destructive tests.

Because, some risks change in time, it is necessary to manage some risks permanently, and therefore, in agreement with design procedure (Procházková, Procházka, Lukavský, Beran, Šindlerová, 2019) we used the approach based on the bow-tie diagram (Zio 2016), Figure 4. We distributed the risks which we cope in the design by preventive measures and risks which we cope by mitigating measures at operation.

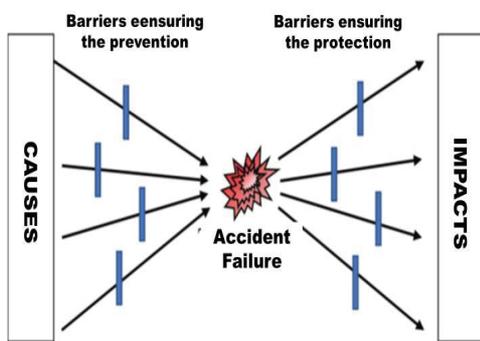


Fig. 4. Bow-tie diagram (Zio 2016).

Considering the results in Table 4 and just described principle we accept after the expert judgment of expenses, material accessibility and personnel knowledge and skill, the following measures for final construction:

- redesigned main construction suspension,
- redesigned horizontal sample reservoir,
- redesigned vertical sample reservoir,
- redesigned pump,
- main construction material is changed to stainless steel 316L,
- installation of experimental loop MSL monitoring,
- compilation of safety documentation and manual for operation,
- making a schedule for training
- plan for risk-based inspections according to principles summarized in (Procházková, Procházka, Lukavský, Dostál, Procházka, Ouhřabka 2019).

Redesign of the main construction suspension was done to eliminate the buildup of the forces caused by the thermal expansion of the construction and the loop itself during normal operation conditions as well as during transitional and critical modes of operation. The new suspension design utilizes two sets of bearings in each point of attachment to allow translation in both horizontal directions.

The horizontal reservoir for insertion of the tested samples was redesigned to ensure better and safer connection using standardized sealings. Moreover, the new cylindrical design reduces thermally induced stresses and results in better molten salt flow.

The vertical sample reservoir for insertion of the tested samples was again redesigned to ensure a better and safer connection, to prevent molten salt leakage and to reduce the possibility of molten salt freezing in the area of the inert gas inlet and outlet.

The reasons for choosing stainless steel 316L as the new main construction material are its lower price and, more importantly, easier workability. The easier workability, together with safer weld technology available, leads to risk reduction of the molten salt leakage, pipe rupture and weld defects. The expected shorter lifetime of the device should not affect its safety, which is also ensured by following established operation limits and conditions, training and extensive permanent monitoring.

All the measures listed and described above should reduce the risk rates to an acceptable value. However, it is important to acknowledge that the use of the risk-based design is an iterative process that is changing as the construction progress and it needs to be re-evaluated again and again.

7. Conclusion

The risk-based design was chosen as the approach for the new design of experimental molten salt loop MSL. As the primary tool of the risk-based design, an extensive checklist was compiled. Based on this analysis, the critical points of the device were identified, the geometric modifications are made to the structure, and, with the help of the CFD tools, three modes of operation are established: normal, abnormal, critical in the event of a power outage. All the findings, changes, and components are assessed during each design iteration stage, before and after the final assembly. Further improvements and changes of the next iterations are based on the results of this assessment.

This analysis ensures safe construction, operation, and maintenance of the MSL in the following years. Moreover, it should allow more precise experimental measurements due to well-defined modes of operation. Lastly, following the presented analysis results, the recurrence of failures and defects from the initial operation of the previous design should be minimized.

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