Chapter 13

SAFETY GUIDELINES*

Lead is a highly toxic substance [EPA] [CIS-ILO]. Exposure to lead can cause irreversible health effects before signs and symptoms are seen or felt [Lewis, 1985]. Rules and regulations, safety controls and practices have been developed to mitigate the lead hazards over the years. Consequently, many occupational uses of lead now pose little risk of exposure if one takes precautions and follows procedures. However, when lead is used in a manner that is different from routine operations, the risk of exposure may be increased. For researchers developing lead-alloy technologies, it is imperative that safety principles are closely observed, proper protective measures taken and formal procedures followed.

In the context of lead-alloy coolant technology, lead oxide hazards may exist as well. Lead oxide exists in one of the two forms: red to reddish-yellow tetragonal crystals stable at room temperature, and yellow orthorhombic crystals stable at above 489°C. Lead oxide presents severe health hazards, especially in the form of lead fumes or dusts, and should be treated with the same caution as with lead.

On the other hand, bismuth does not present severe hazards except under several special circumstances. For example, bismuth dust or powder can be flammable or even explosive at high concentrations and exposed to heat or flame. Generally bismuth may cause mild eye, skin and gastrointestinal irritation. Due to its low hazard ratings, the measures taken to mitigate lead hazards should be sufficient when bismuth is used together with lead. Hence bismuth hazards and controls will not be discussed here.

It is worth noting that the safety practices are tightly linked to good and meaningful outcomes of experiments and tests because they ensure proper control of the environment. Some practical implementation of the coolant technology, on the other hand, can produce unique hazards if not done correctly.

This chapter outlines the effects of lead on human health and environment, the general rules and regulations governing the safe use and disposal of lead in many OECD countries [OSHA], some common safety measures and procedures, and recommendations for routine R&D operations illustrated with several typical experiments. It should be used as a starting point to specific information available in the references. The scope is based on a balance between brevity (ease of use) and completeness (usefulness), and is much more extensive than that of the Materials Safety Date Sheets (MSDS). The main text is based on the training manual for lead awareness at the Los Alamos National Laboratory [Grogin, 2001], with references on country-specific rules and regulations recommended by the expert working group members. A very comprehensive discussion on air-borne lead hazards can also be found in a position paper by a Working Group on Lead, Commission of EU [CEC, 1997]. Many other reports have detailed information and analyses on different aspects of lead, waste and environment (e.g. [Holm, 2002], [WHO, 1989]).

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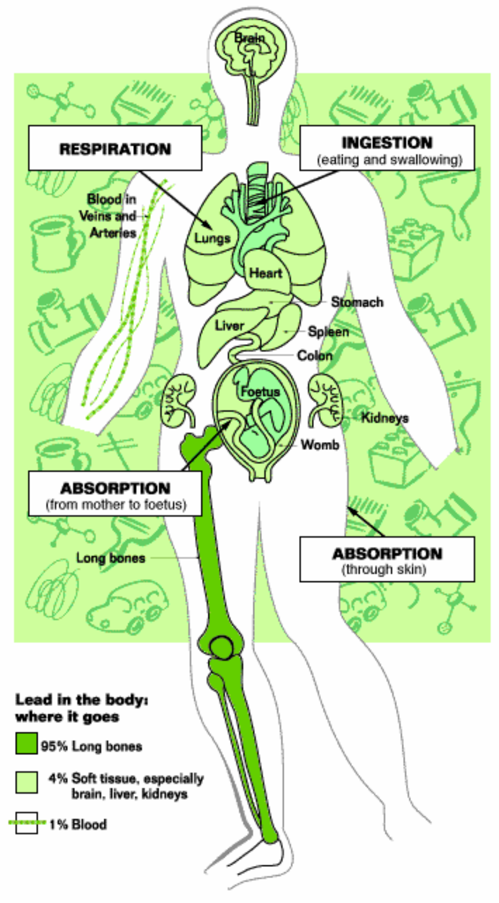
It should be pointed out that under proton and neutron irradiation, lead and lead-alloys all present varied, sometimes severe, radioactive hazards. These hazards and the conditions leading to their occurrence are complex and often system or alloy specific, e.g. ^{210}Po from irradiated bismuth [Cullen, 2003]. They are not covered in this chapter. However, this is an extremely important topic for lead-alloy nuclear coolant application and should warrant a separate and detailed review in the future.

13.1 Effects of lead on human health and environment

Lead can enter human body in three different ways: inhalation (breathing), ingestion (eating or drinking), or absorption (through skin).

Figure 13.1.1. Absorption and storage of lead in human body [NSW]



Inhalation of airborne lead is the most common means by which lead can enter human body in the workplace. Inhalation can occur when lead is scattered in the air as a dust, fume, or mist. Inhaling small particles of lead allows the material to travel into lungs, where it can be absorbed into the bloodstream.

The second most common way is by ingestion (swallowing). Ingestion of lead in the workplace is nearly always the result of poor hygiene practices. Lead can be ingested by handling food, using cigarettes or chewing tobacco, or applying cosmetics when hands are contaminated with lead.

Absorption of lead through the skin is rare, but it can occur if skin is exposed to certain organic forms of lead or if certain chemicals (such as dimethyl sulfoxide) capable of transporting heavy metals through the skin are present.

All of the lead that deposits in lungs and 10-15% of the lead ingested enters bloodstream. In pregnant women, the amount of lead absorbed into the bloodstream through ingestion can increase to 50%. Once in the bloodstream, lead circulates throughout the body and is excreted at a steady rate. As exposure to lead continues, the amount stored in the body will increase if absorption is more than excretion. Of the total amount of lead stored in the body, about 90% is found in the bones and teeth. If no further uptake occurs, accumulated lead is slowly released from the blood and soft tissues over a period of months, but is only released from the bones and teeth over a period of decades. If uptake continues, stored lead will eventually reach toxic levels, a condition known as lead intoxication. Accumulated lead can cause irreversible body damage before showing any signs or symptoms.

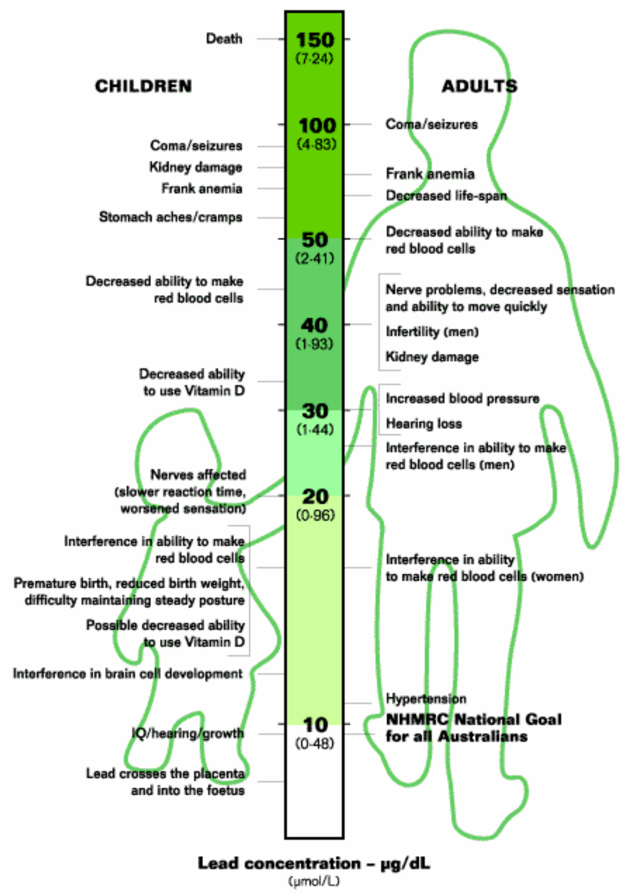
Exposure to lead through inhalation or ingestion can cause serious health effects in a variety of body systems. Such health effects may arise from acute (short-term) or chronic (long-term) exposures. The severity of an exposure depends upon the amount, or dose, of lead that enters human body. A short-term, high-dose lead exposure can cause kidney, nerve, and brain damage that may lead to seizures, coma, and death within a matter of days. Workplace exposures of this type are extremely rare but not impossible. Signs and symptoms of acute lead exposure are similar to many common ailments and, as a result, may not be immediately recognised. Signs and symptoms of acute lead exposure may include: blood in the urine or stool, paralysis, coughing, restlessness, digestive disorders, skin or eye irritation, disorientation, sleeplessness, drooling, sweating, fever and chills, thirst, frequent urination, tingling sensation, headache, vomiting, loss of memory, weakness, metallic taste, yellowing of the skin and eyes (jaundice), and muscle pain.

There is a condition specific to exposure to lead fumes called metal fume fever. It is an influenza-like illness caused by inhalation of freshly formed metal oxide particles with sizes below 1.5 microns, and usually between 0.02-0.05 microns. Symptoms may be delayed 4-12 hours and begin with a sudden onset of thirst, and a sweet, metallic or foul taste in the mouth. Other symptoms may include upper respiratory tract irritation accompanied by coughing and a dryness of the mucous membranes, lassitude and a generalised feeling of malaise. Fever, chills, muscular pain, mild to severe headache, nausea, occasional vomiting, exaggerated mental activity, profuse sweating, excessive urination, diarrhoea and prostration may also occur. Tolerance to fumes develops rapidly, but is quickly lost. All symptoms usually subside within 24-36 hours. Resistance to the condition develops after a few days of exposure, but is quickly lost in 1 or 2 days.

Long-term exposures to lead can damage the blood-forming system, impairing the replenishment of blood cells and the blood’s ability to carry oxygen; the central nervous system and the brain; the kidneys, to the extent that kidney dialysis may be needed; and reproductive systems in both men and women. Signs and symptoms of chronic lead exposure can include: anaemia, lack of co-ordination, blue-black lines on the gums, twitching, high blood pressure, visual disturbances, loss of appetite, and weight loss.

Chronic, high-dose exposures can impair the reproductive system of both men and women. Effects include decreased sex drive, impotence, and sterility in men and decreased fertility and abnormal menstrual cycles in women. There is an increased risk of miscarriage and stillborn children in women whose husbands were exposed to lead or who were exposed to lead themselves. There is also an increased risk of birth defects, mental retardation, behavioural disorders, or death during the first year in children born to such parents.

Figure 13.1.2. Effects of lead on human health [NSW]



Lead may enter the environment during its usage and disposal [EPA]. The initial means of entry is via the atmosphere. Lead will be retained in the upper 2-5 cm of soil, especially soils with at least 5% organic matter or a pH 5 or above. It is expected to slowly undergo speciation to the more insoluble sulphate, sulphide, oxide, and phosphate salts.

Lead enters water from atmospheric fallout, runoff or wastewater. Metallic lead is attacked by pure water in the presence of oxygen, but if the water contains carbonates and silicates, protective films are formed preventing further attack. That which dissolves tends to form ligands. Lead is effectively removed from the water column to the sediment by adsorption to organic matter and clay minerals, precipitation as insoluble salt, and reaction with hydrous iron and manganese oxide. Under most circumstances, adsorption predominates.

Lake sediment micro-organisms are able to directly methylate certain inorganic lead compounds. Under appropriate conditions, dissolution due to anaerobic microbial action may be significant in subsurface environments. The mean percentage removal of lead during the activated sludge process was 82% and was almost entirely due to the removal of the insoluble fraction by adsorption onto the sludge and to a much lesser extent, precipitation.

The most stable form of lead in natural water is a function of the ions present, the pH, and the redox potential. In oxidising systems, the least soluble common forms are probably the carbonate, hydroxide, and hydroxycarbonate. In reduced systems where sulfur is present, PbS is the stable solid.

The solubility of Pb is 10 ppb above pH 8, while near pH 6.5 the solubility can approach or exceed 100 ppb. Pb(0) and Pb(+2) can be oxidatively methylated by naturally occurring compounds such as methyl iodide and glycine betaine. This can result in the dissolution of lead already bound to sediment or particulate matter.

Figure 13.1.3. Sources and pathways of lead in the environment [NSW]

Elevated levels of lead in the water can cause reproductive damage in some aquatic life and cause blood and neurological changes in fish and other animals that live there.

Lead does not appear to bioconcentrate significantly in fish but does in some shellfish such as mussels. Evidence suggests that lead uptake in fish is localised in the mucous on the epidermis, the dermis, and scales so that the availability in edible portions do not pose a human health danger.

Wild and domestic animals can ingest lead while grazing. They experience the same kind of effects as people who are exposed to lead. Low concentrations of lead can slow down vegetation growth near industrial facilities.

13.2 Rules and regulations

Lead is a technologically important metal throughout the history. It is widely used in many industries and applications. Because of this wide presence and its toxic nature, lead work is also one the most regulated fields of labour. There are many rules and regulations in all industrial countries governing the use of lead.

Since regulations are country-specific and compliance is strictly required, it is beyond the scope of this handbook to present a comprehensive review of this topic. A compilation of the most significant standards and requirements is presented in the following table. Researchers should consult safety authorities within own organisations/countries to determine specific requirements for compliance.

However, there are several common features worth highlighting. First, since inhalation of airborne lead is the most common pathway for lead to enter human bodies, all regulations place a maximal allowable concentration (for an 8 hour shift, it is variably termed PEL – permissible emission level, or MPC – maximum permissible concentration, or MAK-value – maximum allowable concentration in workstation at any time). PEL/MPC varies from 50-100 μg/m^3, while MAK-value ranges from 100-150 μg/m^3.

In practice, if we use the lead and LBE vapour pressures from the literature [Lyon, 1952][Morita, 2004], the 50-100 μg/m^3 PEL corresponds to 530-550°C for LBE and 500-520°C for Pb. Special care and controls must be taken to conduct experiments with exposed LBE and Pb above these temperatures. For airborne lead levels below PEL, the allowable work duration is lengthened proportionally so the total exposure equals to 8 hr times PEL.

Second, due to enhanced health threat (impairment of reproductive system and damages to children), the standards for young persons and pregnant women are more stringent. The Pb in blood (PbB) levels that require continued monitoring and work stoppage are lower for workers with heightened risks.

The monitoring frequency for airborne lead and PbB varies depending on severity from pervious measurement or whether changes in procedures and controls take place. It varies from 3 to 12 months, or longer when no changes occur. There are generally requirements on how long the monitoring and medical records should be kept for the workers.

There are rules and regulations governing the limit value and monitoring of lead in ambient air and in wastes.

National Ambient Air Quality Standard (NAAQS) is set by US EPA for pollutants that are considered to be harmful to public health and the environment; the NAAQS for lead is 1.5 μg/m^3, maximum arithmetic mean averaged over a calendar quarter [EPA].

Commission of EU established a limit value for Pb in ambient air of 2 μg Pb/m^3 [EU]. Some countries have reduced the limit to 0.5 μg/m^3. The sampling method is to collect atmospheric particles on a filter for subsequent determination of lead content. The reference method of analysis is atomic absorption spectrometry.

US EPA regulation sets the limit of Pb in solid waste to be 5 mg/L beyond which the waste is considered hazardous (EPA HW No. D008; CAS No. 2: 7439-92-1) [CFR]. This puts most of the lead contaminated materials in coolant R&D in the category of hazardous wastes, including residues from polishing corrosion test specimens with small amount of Pb adhered. EU has been moving toward lead-free solder so the requirements might become even more stringent. The concentration limit in consideration is 0.1 wt.% for EU and Japan [SolderTec, 2003].

For surface contamination, the following wipe sample clearance guidelines are recommended by US HUD [LLNL, 2005]:

- <100 μg/ft^2 (0.108 μg/cm^2) on uncarpeted floors;
- <500 μg/ft^2 (0.538 μg/cm^2) on interior window sills (stools);
- <800 μg/ft^2 (0.861 μg/cm^2) on window troughs (where the sash sits when closed);
- <800 μg/ft^2 (0.861 μg/cm^2) on exterior concrete.

These clearance guidelines are based on hazards to children, and are extremely low. So in R&D work, higher limits may be acceptable at the discretion of responsible industrial hygienist. Although in common laboratory settings, Pb vapour presence is minimal, long-term exposure without proper periodic clean-up could lead to high levels of Pb surface contamination.

Irradiated Pb and LBE may be considered low-level radioactive wastes. The disposal of such wastes is more difficult and their disposition sites very limited and country dependent. It is beyond the scope of this chapter and should be treated in the future in the context of decommissioning and decontamination (D&D).

13.3 Common safety controls and practices

General controls can be used to reduce the potential for occupational exposure to lead. They can be categorised as engineering controls and administrative controls. Additionally there may be specific controls unique to the job. Industrial hygienists or other relevant safety specialists should be consulted about these controls, and approvals sought for operations involving lead.

Engineering controls are mechanical or structural systems used to minimise hazards. They include ventilation systems (e.g. fume hoods), isolation systems (e.g. glove boxes), adhesive mats at doorways, hygiene facilities (e.g. hand-washing basins), etc.

Administrative controls are used to direct, restrict, or modify behaviour to prevent or minimise exposure to hazards. Specific requirements vary, but they generally include plans, procedures and permits, safe work practices, housekeeping, postings, access controls, and training.

Plans, procedures and permits define the operating conditions that one may encounter and special precautionary measures to minimise workplace exposure to lead. When there is a potential for lead to become airborne in a manner that potentially increases risk of exposure, the activity hazard analysis or hazard control plan should identify the hazard and specify controls.

There are several general safe work practices and housekeeping rules to minimise or prevent exposure to lead, including:

- Wash hands and face before eating, drinking, or applying cosmetics.
- Use only high-efficiency particulate air (HEPA) filter vacuums.
- Remove work clothes before entering eating areas, or use HEPA vacuums to clean dust off the clothes.
- Maintain all surfaces as free as practicable of accumulation of lead dust;
- Do not dry-sweep areas where lead may be present.
- Do not wear work clothes beyond the work area to prevent contamination.

For controls such as washing facilities, clean changing areas, separate clothes lockers, and showers to remain effective, workers must use them routinely and correctly.

Postings and access controls must be used in each work area where the permissible level is exceeded or as a precautionary measure where lead is present. Some areas may need access control to prevent unqualified personnel from entering.

Table 13.2.1. Regulations governing the safe use of lead in selected countries

PEL – Permissible Exposure Level, AL – Action Level)

Country/ region	Regulations	In air, max permissible exposure level for 8-hr shift (PEL, microgram/m^3)	In air, action level (AL, microgram/m^3)	Monitoring frequency (month)			Record keeping (yr)	Max Pb in blood (PbB, microgram/deciliter)	
				> PEL	< PEL & >= AL	< AL		Regular worker	Worker planning children
US	OSHA standards (www.osha.gov), 29 CFR, 1910.1025 – all occupational exposure to lead, and 1926.62 – all construction work where an employee may be occupationally exposed to lead	50 (100 MAK-value)	30	3	6	Only when new risks are introduced through change of work, control	The longer of 40 or duration of employment+20 (30 for construction worker)	40	30
Switzerland	Safety at Work is regulated by Federal Law (http://www.seco.admin.ch/..); Classification of products is in the competence of the Federal Government (http://www.bag.admin.ch/..); Surveillance that regulations are applied is in the competence of SUVA (http:	100 (workers < 18 years of age, pregnant women, anemic persons excluded from working in areas > PEL)		2-6 depending on severity of exposure to be agreed upon with surveillance body	12	12		70 (male, female > 45 years of age)	30 (female < 45 years of age)
EU	Exposure to metallic lead and its ionic compounds (http://europa.eu.int/scadplus/leg/en/cha/c11133.htm)	150	75	3	3			70	
France	EU Diretives								
Germany	EU Diretives								
Sweden	Swedish Standards for Lead, AFS 1992:17, Swedish Standards for Air Control, AFS 1988:3, AFS 2000:3 (www.av.se)	50 (100 MAK-value)	40	1st 6 month, inspection required; afterwards, if Pb in air<1/5 PEL, no more inspection is needed; if 1/5 PEL<Pb in air <1/2 PEL, the authority decides the monitoring frequency; if 1/2 PEL<Pb in air < PEL, inspection is required every one year				PbB, micromol/litre, or 20.7 microgram/deciliter, value in () for women under 50: < 1.0 (0.8): no medical control; 1.0-1.5 (0.8-1.2): six months; 1.5-2.0 (1.2-1.5): 3 months; > 2.0 (1.2): measures taken to reduce PbB; > 2.5 (1.5): stop work	
UK	The Control of Lead at Work Regulations 2002 (http://www.hmso.gov.uk/si/si2002/20022676.htm)	150 (100 lead alkyls)		3 mon. if exposure from lead alkyls, or previous measurement > 100 microgram/m^3		12 mon. max if: exposure not from lead alkyls; no change; 2 previous consecutive tests show < 100 microgram/m^3	5 from date made	50 (young); 60 (others) (action levels: 40/50)	30 (action level: 25)
Japan	Safety at lead work is regulated by Japanese law and ordinance (http://www.chemlaw.co.jp/LaborLink/namari_1.htm); English version (http://www.jicosh.gr.jp/Japanese/oshlaw/LeadPoisoning/index.html)	100				12	min. 5	40	
S. Korea		50							
Russian Federation		10							
Belgium	Expsure limits (http://meta.fgov.be/pdf/pk/nlkfg03.pdf)	150		6	6	6	10	70	40

Training is required for all personnel subject to lead exposure. Typically it consists of two components: a general lead awareness training covering the topics outlined here, and a facility or task specific training provided by supervisors or facilities.

Personal protective equipment (PPE) is secondary protection that is used when other control measures are not feasible or not sufficiently effective at reducing exposures to lead. PPE can include coveralls, gloves, a face shield or vented goggles, booties, and respirators. In case respiratory protection is needed, proper training will be necessary.

When working with molten lead and lead-bismuth, toxic lead fumes are usually present, and the appropriate respirator should be selected if the concentration of fumes is above the permissible limit (PEL). Table 13.3.1 is based on US OSHA standards and represents the minimal respiratory protection at each action level [OSHA].

Table 13.3.1. Respiratory protection for lead aerosols [OSHA]

Airborne concentration of lead or condition of use	Required respirator
Not in excess of 0.5 mg/m^3 (10X PEL).	Half-mask, air-purifying respirator equipped with high efficiency filters.[2,3]
Not in excess of 2.5 mg/m^3 (50X PEL).	Full face-piece, air-purifying respirator with high efficiency filters.[3]
Not in excess of 50 mg/m^3 (1000X PEL).	(1) Any powered, air-purifying respirator with high efficiency filters[3]; or (2) Half-mask supplied-air respirator operated in positive-pressure mode.[2]
Not in excess of 100 mg/m^3 (2000X PEL).	Supplied-air respirators with full face-piece, hood, helmet, or suit, operated in positive pressure mode.
Greater than 100 mg/m^3, unknown concentration or fire fighting.	Full face-piece, self-contained breathing apparatus operated in positive-pressure mode.

[1] Respirators specified for high concentrations can be used at lower concentrations of lead.
[2] Full face-piece is required if the lead aerosols cause eye or skin irritation at the use concentrations.
[3] A high efficiency particulate filter means 99.97% efficient against 0.3 micron size particles.

13.4 Safe operations in HLM R&D

Using lead in heavy liquid metal coolant technology R&D has many aspects of operations that can be hazardous if proper controls are absent and procedures are not followed. The hazards are multiplied many times in radiation environment, when alpha-emitting polonium-210, radioactive mercury isotopes and others are produced (see Chapter 8). Establishing the safety envelop and operating procedures, and getting approvals from safety authorities can be very labour intensive and time consuming, as the MEGAPIE project has demonstrated. This section, however, will not cover that but instead use several typical experimental operations with no irradiation to illustrate some safety practices in the more routine use of lead and LBE for HLM R&D. These are from observations and practices of many experiments and test facility operations around the world.

Many laboratories use small containers of LBE or lead to conduct experiments in the study of corrosion, wetting, embrittlement, oxygen control, etc. The generic operation steps may include setting up apparatus, preparation, melting, filling, insertion, sealing, heating, gas injection, cooling, opening, extraction, cleaning, and freezing. Transferring, draining, circulating, etc. are typically used in larger test loops and facilities, so covered separately later.

Setting up apparatus: The first consideration should be the location of the experiment and its environment. It should be in a dedicated laboratory or area separate from offices and eating areas with good ventilation and nearby washing facilities. The area should have non-porous, clean and uncluttered surfaces and walls to facilitate cleanup. Since leakage and spillage can occur, it is advisable to have metal drain pans underneath the apparatus and handling operations. If hoods or other active ventilation systems are used, HEPA filters should be installed.

The access should be controlled with proper warnings and contact information posted at prominent locations, including entrances, exits and next to the experiments. If large quantities of HLM are used, sticky mats on the floor at the exits may be used to limit contamination.

Periodic cleaning should be performed to eliminate deposited lead or its oxides. Use wet wipes for surfaces, and HEPA-filtered vacuum for floors. If a laboratory has been used for long time without frequent cleaning, surface sampling should be conducted to determine levels of contamination before cleaning is commenced.

Preparation: Solid LBE or lead ingots are typically used. Small amount of surface oxides can usually be dissolved into the melt (sometimes desirable if higher oxygen concentrations are needed), or cleaned up with mechanical means from the melt (e.g. picked up by a wire mesh scoop). If there is too much oxide, e.g. from long-term exposure to the environment, they should be discarded. Cleaning oxides in solid form may produce dangerous air-borne lead dust, and should be avoided.

Melting: Melting ingots into a crucible or a vessel is straightforward. However, melting solidified LBE or lead in a container requires some caution and well-planned procedures. There can be significant differential volume expansion during heat-up of HLM and container, leading to stresses that in some situations cause crack or rupture. HLM should be melted from free surfaces into more confined volumes. For large containers, separate heating zones with correct heating sequences should be used. Some design features, e.g. a few degrees of slant in a melting tank wall (expanding toward the top), are better suited than the others (e.g. a straight cylindrical tank is not as accommodating). A horizontal cylindrical melting tank more than half filled is not a good configuration. Due to such considerations, HLM is usually drained from test apparatus with complex internal structured for freezing and melting.

Filling: If a large amount of LBE or lead is used for the first time, a pre-melting and transfer device, draining from the bottom into the experimental apparatus, is recommended. Many suspended impurities, including sludge of oxides, can be removed from the free surface by mechanical means. Such operations should be carried out at temperatures as low as possible, i.e. just high enough to prevent accidental freezing and plugging. The specific temperatures depend on the setup, e.g. heating of the transfer line and the receiving vessel, but usually should be 150-180°C or slightly higher for LBE, and 380-400°C for lead. Such tasks can be carried out in open vessels, but the operators should wear respirators if more than a few kg of HLM is involved, and should in principle have air sampling done as well. While the vapour pressures at these temperatures are below permissible emission levels (PEL), the handling may create high levels of dusts, including lead oxides. Cleaning the surrounding afterwards is necessary to prevent the spread of contaminations. Vacuuming with a HEPA-filtered vacuum cleaner for loose dusts and wet wiping of exposed surfaces are recommended.

The vessels and pipes to be filled should be properly pre-heated to temperatures not too different from the melt. Difference of a few tens of degrees is acceptable, but 50°C or more may produce thermal shocks that could damage the apparatus or sensors. There can be mitigation measures (e.g. use of bellows for joints) and procedural control (e.g. slow fill when accidental freezing can be excluded) deployed to reduce such effects. If there are areas not very accessible to pre-heating (e.g. pipes inside a vessel), caution must be taken to prevent accidental freezing (e.g. use a higher melt temperature for filling).

Insertion: Sensors, test samples and gas lines are often inserted after the filling. This should be performed at the low temperatures. Since HLM is heavier than most materials, there will be some buoyancy so the fixtures should be sturdy and can prevent upward turning. Hanging is not an option here. At this stage, the free surfaces of HLM should have been cleaned up already so the insertion does not contaminate the undersides with oxides and other floating impurities.

For all the above operations involving open vessels, temperature should be kept low both for safety reasons, and for avoiding excessive oxidation. While using an inert heavy cover gas (e.g. Ar) to reduce oxidation may be desirable, it is not necessary.

Sealing: Due to the needs for safety and good coolant chemistry control, tight seals are essential for experiments and operations. External rotating seals should be avoided. Flanges with gaskets in contact with high temperature HLM should be avoided as well, although some have worked reasonably well (graphite or spiral wound steel gaskets). Since even small ingress of air will contaminate the HLM, a slightly positive cover gas pressure should be maintained during normal operations.

Due to relaxation of bolts from thermal cycles, flanges will loosen over time. There should be a periodic retightening scheduled and performed. If leakage does occur, the experiment should be stopped, HLM drained into a melt tank, and apparatus cooled down before repair and cleanup is performed. Liquid metal leakage detection can be based on simple conductivity probes in areas surrounding flanges or joints or likely collection basins, or inferred from loss of volume through level sensors, and loss of pressure.

Heating: Heating up the HLM in the molten state is quite simple but may have consequences beyond the obvious. Very rapid heat-up may be detrimental to some components, especially ceramic parts (such as oxygen sensors). There is no definitive rules reported, but a less than 50°C/hr heating rate is recommended for some Russian-made oxygen sensors. We have used much more rapid heating rates in small experiments with simple configurations. For large facilities, this should be determined carefully with analyses, simulations and testing.

Due to rapidly increasing solubility of elements in HLM with temperature, surface slags, especially lead oxides, will decrease and disappear. Oxidation of materials will also consume oxygen and clean up HLM surfaces. If no deliberate oxygen addition or air ingress takes place, the HLM at high temperatures usually has oxygen concentrations far below solubility or the so-called oxygen controlled range for corrosion mitigation. If no protective coatings or pre-oxidation has been applied to apparatus built with steels, this could lead to rapid dissolution attack at high temperatures (above 500-550°C for 316L type austenitic steels, or T91 and HT-9 type of ferritic and martensitic steels (for the compositions of these steels, see Chapter 6). Such dissolution attacks may be even accelerated if oxygen is added while the dissolution already started by removing dissolved elements through oxidation in HLM. It is highly recommended that heating be carried out through planned stages during the initial start-up, or after insertion of fresh test specimens, so that protective oxidation can be initiated and established. Since many test apparatuses and loops are built with steels, such precaution is essential in maintaining integrity of containment and safety.

If in static tests at high temperatures the oxygen concentration is not first adjusted to sufficient levels, due to the slow diffusion of oxygen from cover gas into the depths of stagnant HLM and rapid local consumption of oxygen by oxidation, unprotected fresh test specimens made of steels may start to dissolve, setting up a competition of mass transfer processes between oxygen diffusion to the steels and dissolution products migrating toward high oxygen areas (e.g. free surface in contact with cover gases). This could render test results highly dependent upon heating rate, initial oxygen concentrations in HLM and the effectiveness of the mass exchange from gas to liquid. It may even be dependent upon placement of specimens, size of surfaces and other system specific conditions, which are usually not accounted for or reported.

Such scenarios can also arise in flow systems where gas exchange for oxygen control is conducted through a stagnant volume. In extreme cases, it could lead to much more rapid corrosion than expected and have severe safety consequences.

Gas injection: This is often done to adjust coolant chemistry, either by adding oxygen or using hydrogen to reduce oxygen, or using gas mixtures (e.g. steam and hydrogen, or CO and CO_2) to maintain oxygen levels. Sometimes, to create circulation (e.g. gas lift or stirrer), a gas injection of high flow rate is applied.

It should be noted that even the highest purity supply gases usually contains ppm level of oxygen, which is too high for HLM if continuously applied over long periods of time. A getter system is needed to strip incoming gases of oxygen, unless adding oxygen is the goal. It is also not advisable to inject gases directly into HLM melt, since small amount of residual oxygen will lead to slag formation and plugging of the injection line, and the transport of slags to other parts may lead to flow restriction or blockage. This is true even for hydrogen injection unless an oxygen getter system, pre-heating to complete reaction with hydrogen, or entrainment by flow for prolonged hydrogen retention, is used.

From the safety perspective, injecting gases with lead oxide formation may create aerosols in the cover gas space, leading to potentially very hazardous conditions when opening and repair are conducted (see below on opening). It is therefore emphasised that cautions be applied when gas injection into liquid metal is deployed in experiments and operations.

Cooling: In the molten state, cooling should observe the same caution with heating. Rapid quench is not advisable.

The effects of cooling should be carefully considered when establishing procedures. Because solubility decreases with decreasing temperature, undesirable precipitation and deposition may occur in systems at hard to reach or technologically important sections. Once the solids deposit to walls, merely heating back up will not remove them since they either have low solubility or high melting temperatures. This could lead to reduced performance, restriction of flow and eventual blockage. It may also create unsafe conditions for maintenance and repair. The cleanup and restoration process can be very time consuming.

When temperature of HLM is lowered substantially from the operating conditions, oxygen concentration can reach saturation and precipitation will occur. Heating up quickly can lead to deficiency in oxygen for corrosion protection. For example, the typical oxygen control point is 10^{-6} wt.%, or 10 ppb of oxygen in LBE. This corresponds to approximately saturation at 200°C. So cooling below that for an extended period will lead to significantly lower oxygen concentrations, and addition of oxygen will be necessary on re-heating if the precipitated oxides are somehow removed of left out (e.g. cooling is in the melt tank).

Opening: As in all open vessel operations, this should be conducted at temperatures as low as possible. Lower temperatures suppress oxidation and vapour emission.

The top concern upon opening vessels or pipes is the potential presence and release of fine oxides or aerosols. While good ventilation is necessary, strong air perturbation at the openings should be avoided. When possible, a small enclosure of suitable sheets may be used to contain any releases. Operators should wear respirators if opening is carried out through cutting or other means of strong disturbances.

Extraction: It should be done at low temperatures. Very often, reheating is needed to extract specimens or sensors after solidifying HLM in place, or being “welded” to holders or sheaths by the residual HLM.

Cleaning: Cleaning of specimens is a difficult and delicate task. There are a number of methods reported for removing the adhered LBE from the specimens, including simple swiping, boiling in glycerine, silicon oil, or acid, or dipping in sodium. The safety precaution for each is obvious and will not be discussed here. One should keep in mind that hazardous waste is usually generated, as is also the case for polishing, and disposal needs to comply with applicable rules and regulations. Therefore, minimising the use of supplies and recycling of some media should be attempted.

It is also a good practice to clean up the surrounding areas after each opening or certain time, to reduce surface contamination and spread beyond the laboratory.

Freezing: While it is desirable to keep the HLM molten at all time, especially in test facilities, HLM will often be allowed to freeze. Due to volume changes upon solidification, and continued changes during recrystallisation in LBE, caution and proper procedures must be applied. Freezing should start at more confined space toward free surfaces, and should be avoided in regions where complex and/or delicate components lie.

An important consequence of the freezing is the reduction of oxygen in HLM. Upon melting, certain amount of time should be allowed for re-oxygenation before transferring and raising the temperature high.

For large test facilities, there are several additional steps or functions. The generic forms are transferring, priming, circulation, active cooling, draining, etc. The actual procedures will be facility specific. However, similar safety cautions are required. In particular, because of the stored kinetic and thermal energy in the circulating liquid metals, local containment in the forms of pipe cladding, loop enclosure, drainage collection floor pan, with ventilation, will be necessary.

The danger posed by lead to human health and environment is very real and serious, and should not be overlooked in the research and development of lead-alloy coolant technology. It is also demonstrated in the worldwide use of lead throughout the history that when rules and regulations are properly observed, and safe work practices and controls are employed and effectively utilised, lead hazards are manageable. It is imperative for this field of R&D and future applications that the safety guidelines are followed, and the workers and the environment are not harmed.

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