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Gallium Safety in the Laboratory

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Abstract

A university laboratory experiment for the US Department of Energy magnetic fusion research program required a simulant for liquid lithium. The simulant choices were narrowed to liquid gallium and galinstan (Ga-In-Sn) alloy. Safety information on liquid gallium and galinstan were compiled, and the choice was made to use galinstan. A laboratory safety walkthrough was performed in the fall of 2002 to support the galinstan experiment. The experiment has been operating successfully since early 2002.

Introduction

The US Department of Energy magnetic fusion research program has been investigating ideas for improving the longevity and operational availability of fusion experiments, and potentially, fusion power plants. One of the suggestions is to use flowing liquid walls to protect the vacuum vessel rather than solid armor tiles that have been used in the past. The liquid walls have the advantages of being "self-renewing" under radiation and thermal damage, they transfer heat well, and they shield the vessel walls well.[1] Experiments have been initiated to test liquid walls in fusion conditions.[2] The Magnetic Toroidal Liquid Metal Flow Loop (MTOR) experiment[3] at the University of California-Los Angeles (UCLA) was designed to test and develop models for the flow properties of unirradiated liquid metal as it traverses a magnetic field similar to the magnetic fields used for ion confinement in a fusion experiment. While liquid lithium and lithium-tin are the leading candidates for the liquid wall material, and would be the best fluids to test in the MTOR experiment, the university lab staff were reluctant to handle large quantities (50 liters and more) of 200°C lithium. Alternatives were sought to determine if any other liquid metals would behave similarly to lithium or Li-Sn so that experiment results could be scaled to give general results for lithium. After surveying the likely candidate metals, gallium was the obvious choice. Gallium is much less chemically reactive than lithium or other alkali metals, it melts at a lower temperature than other light metals, and is less costly than pure alkali metals. Gallium properties and industrial uses were investigated to determine if there were any unique or special hazards associated with gallium. The gallium alloy, galinstan, was also investigated to compare to gallium metal. This paper discusses the safety issues with using gallium and galinstan, the choice of galinstan alloy, and overview results of the safety walkthrough.

Gallium and Galinstan Hazards

Gallium is a metal (atomic weight 69.72) with a very low melting point (29.9°C) and a high boiling point (1983°C).[4] The density of gallium is approximately 6 g/cm^3 at 33°C. Gallium is electrically conductive and also paramagnetic. The gallium alloy, galinstan, is a mixture of gallium-indium-tin. Galinstan has several attractive properties. One of these is that it is liquid at room temperature, so no heat tracing or pipe insulation is needed to maintain the piping at elevated temperature. Pipe stresses from elevated temperature operation and the danger of "freeze-plugging" a pipe section are also avoided. Galinstan freezes at about –20°C and boils at about 2300°C, so it has a very wide temperature range where the liquid can be used. The vapor pressure is quite low, essentially zero at 20°C. Each of the elements in galinstan have very low vapor pressures, on the order of 10^{-4} Pa at over 540ºC,[5] and much lower at room temperature (~0 Pa at 21°C). Since each constituent has very low vapor pressure, and given that these metals are in eutectic alloy form, it is expected that a spill of galinstan at room temperature (~20°C) would not evolve any constituent metal vapor.

The chemical reactivity and toxicity of gallium and galinstan were investigated by literature search and computer program. The HSC computer code[6] was used to predict possible gallium and galinstan reactions when spilling in air at 20°C. The results predicted that low levels of oxides would form. The reaction rate at 20°C is not expected to proceed to the extent that significant quantities of Ga_2O_3 are produced from gallium. Very little gallium, indium, or tin oxide will be produced from galinstan, and only trace amounts of gallium nitride (GaN) are expected to be produced.

The HSC code was also used to investigate Ga-In-Sn reactions with some common materials in the laboratory, including an assumed copper grounding strap and two types of commonly used electrical insulation, polyethylene and polyvinyl chloride.[7] Those types of electrical insulation are most often used for electrical power and data or instrumentation cables. The HSC code results did not show any appreciable reactions with any of these materials. However, the discussion by Burton[8] indicated that galinstan does react with copper, even at low temperatures. Liquid gallium at ~30°C also slowly reacts with copper to leave the copper surface pitted.[9] This chemical reaction with copper is a concern if gallium or galinstan spills in the laboratory. Both gallium and galinstan are electrically conducting; thus the staff must verify that the spill is not in contact with any electricity before approaching to clean up the spill. Fortunately, it appears that neither gallium nor galinstan will react with electrical insulation. Since these materials are not an obvious hazard to approach (i.e., not at elevated temperature, no vapor over the liquid spill pool, etc.), lab personnel must verify that the spilled metal is not inadvertently conducting electricity. The easiest means to accomplish this verification is to de-energize the experiment before beginning spill cleanup. Certainly, the experiment procedure would be to secure the electromagnetic pump that circulates the liquid metal (to limit inventory loss), so de-energizing the remainder of the equipment should not pose a problem during spill cleanup.

Gallium has long been noted for its chemical corrosiveness at high temperatures.[10] A few recent studies have shown that the corrosion of gallium with engineering materials such as stainless steel occur at high temperature in the 300°C to 400°C range and higher range.[11,12,13] The corrosion rate for a room temperature application of gallium is quite low, so gallium or its alloys at room temperature should not pose any corrosion concerns for many years of use. Narh[12] also tested the gallium corrosion resistance of high density polyethylene, polypropylene, polystyrene, and polymethyl methacrylate (PMMA) resins. There was no evidence of adverse interaction between gallium and these plastics, even when the plastics were stored in contact with gallium for several days at temperatures close to the plastic softening or melting points. Such chemical inertness with plastics has allowed Tagawa[14] to use a plexiglas (i.e., a form of PMMA) container to test liquid gallium heat transfer under a static magnetic field. The MTOR experiment uses reinforced vinyl tubing for part of the Ga-In-Sn flow loop, and a clear plastic window on the test section of the flow loop to allow observation of the flow behavior.

Gallium toxicology was also researched. Gallium metal is insoluble in water; consequently gallium is not readily absorbed through the skin. Eye contact with, or inhalation of, gallium dust or powder may cause irritation. Subcutaneous implantation of gallium metal or alloy in guinea pigs caused necrosis *in situ*.[15] Therefore, care should be exercised to avoid injecting gallium through the skin (i.e., preclude puncture or incision wounds that leave gallium contamination in the wound). Gallium has temporary emergency exposure limits (TEELs): TEEL-0 is 10 mg/m^3, TEEL-1 is 30 mg/m^3, TEEL-2 is 50 mg/m^3, and TEEL-3 is 250 mg/m^3. Gallium oxide also has TEELs; they are the same as for gallium metal, except TEEL-3 is 500 mg/m^3.[16] Gallium does not pose any large toxicological hazard in use.

Galinstan toxicology was similarly researched. Galinstan is a eutectic mixture estimated to be 66.0% Ga, 20.5% In, and 13.5% Sn by weight.[17,18,19] Galinstan has been adopted as a replacement for mercury in oral thermometers,[20] and has been used successfully as a dental filling alloy.[21,22,23,24] Released gallium from a gallium alloy dental restorative material was moderately cytotoxic to in vitro Balb/c mouse fibroblasts after 8 h, and continued to increase in cytotoxicity thereafter, which correlated with a substantial and persistent release of gallium from this material.[15,25]

The only acute toxicology information found in the literature was a study of gallium nitrate and gallium sulfate ingestion.[26] The results showed that single large intragastric doses were only mildly toxic for rats and mice.

Gallium oxide (Ga_2O_3) poses a toxicity concern from both elemental gallium and galinstan. Ga_2O_3 chronic industrial inhalation exposure[27,28] has been examined. Hahn[27] concluded:

> "The results presented here indicate that 4-week exposures of rats to inhaled Ga_2O_3, at concentrations near time-weighted average threshold limit values (TLVs), can induce progressive lung damage. The severity of the damage and its fibrogenic nature appear to be comparable to those from inhalation of quartz. These observations suggest that exposures to Ga_2O_3 in the work place should be limited, and that the TLV for nuisance dust is probably not appropriate for this material. Additional long-term exposures to multiple concentrations of Ga_2O_3 would be helpful in defining a TLV for the work place."

Information obtained from the literature states that oxide formation on a free surface of gallium creates a thin, viscous film.[9,29,30] This information suggests that while modest oxide-producing reactions do occur at low temperatures, most of the produced oxide forms a layer rather than becoming airborne. However, mechanical processes (such as scooping) could release very small amounts of particulate into the air in the vicinity of the cleanup operation. These toxicity discussions have shown that oxide compounds formed from gallium or galinstan constituents can be toxic, at least in chronic exposures. Care should be taken when handling spills to minimize exposure. Overall, gallium and galinstan do not pose a large toxicity hazard.

Flow Loop Operating Experiences

Some researchers have used pure liquid gallium flow loops. These experiences were sought to determine if there are any safety issues with these flow loops. Smither[31] discussed use of gallium as a coolant for some high heat flux components. No adverse experiences have been reported with usage of gallium. Smither[31] stated that the surface tension of gallium is much higher than that of water. Due to high surface tension, gallium is stated to be “immune” to the presence of small cracks in piping, or channels in an imperfect seal, that would result in a serious leak if water were the cooling fluid.

One long-term operating experience with Ga-In-Sn alloy is from a radiation loop that has been used for many years at the Latvian Academy of Sciences. This loop flowed gallium alloy into the reflector of a small fission reactor (the Institute of Physics 5 MW_{th} research reactor from 1961),[32] irradiating the indium to create In-116, then flowing the irradiated material to a working chamber where the gamma rays created by In-116 decay were used for studying effects of radiation. Dinduns[32] reported that this loop was operated for 7500 hours between 1977 and 1981 (note: the loop[33] has continued to operate since 1981), and did not experience any leaks. There were 750 startups in that time period, but no reported losses of pumping capability. After a gamma ray exposure of 1E+08 to 1E+10 rads, there was no change in the operating parameters of the loop equipment.

Baranov[34] stated that a Ga-In-Sn alloy flow experiment to simulate liquid metal free surface cooling used argon cover gas to prevent liquid metal oxidation. Baranov[34] mentioned an operational issue of concern - wetability of surfaces. Liquid metals tend to ‘bead up’ on surfaces rather than easily contact surfaces, and gallium has been noted to behave in that way.[35] After forcing a liquid metal to contact a surface, such as by pressure or by impact, the liquid metal generally wets and flows on that surface in the future. When the flow nozzle surfaces were not wetted, the flow did not properly fill the nozzle; the liquid metal film thickness was not uniform.

Gallium and Galinstan Spill Cleanup Procedures

Smither[36] indicated that his experience with gallium shows that it is not very chemically reactive with most materials. If gallium were to spill on a floor, the suggested practice is to pour very cold water (~a few degrees C) on the gallium so that the gallium freezes, then mechanically lift up the frozen puddle of gallium with spatulas or other tools. Since gallium is insoluble in water, this approach should not create any volume of contaminated water for disposal. Smither[36] stated that there is virtually no residue left on the floor surface after mechanical cleaning, since gallium does not easily wet surfaces. After retrieval, the “spheroid” or “frisbee” (depending on the wettability of the spill surface) of gallium can be repurified. Gloves are recommended; although the gallium should not be absorbed through the skin of a person's hands, the experimenters will not want to contaminate the gallium surfaces with oils from human skin, and gloves protect against puncture wounds. Eye protection is also suggested, since gallium or its oxides in the eyes can be harmful. While gallium oxide formation and mobilization seem to be small, a respirator is a suggested safety precaution in case any oxide is lofted into the air during the spill or cleanup. Further work may determine if a respirator was truly needed during cleanup.

Galinstan spill cleanup procedures were also sought from the RG Medical Diagnostics company in Southfield, Michigan. This company markets the galinstan thermometer in the US for the German company, Geratherm. The RG Medical Diagnostics representative stated that cleanup is by mechanical means (plastic scoop, spatula, etc.), and then soap and water or other commercial cleanser to clean the floor, countertop, or other spill surface.[37] Generally, a typical fever thermometer contains only about 0.01 g of galinstan, so their cleanup activities have been limited to very small quantities. The experimentalists at UCLA stated that the gallium oxide that had formed during a minor spill event appeared dark in color, and was easily cleaned up in the MTOR lab. Galinstan and any oxides cleaned up very well using Fantastik® brand spray cleaner.

Another safety suggestion is that if galinstan alloy spills at MTOR, the aluminum frame supporting MTOR magnets should be visually inspected to verify that none of the frame members have been chemically attacked. The magnets create force loading on the frame due to their weight and their electromagnetic force interactions. Therefore, the frame members must not be weakened by surface corrosion. Galinstan is an electrically conducting fluid, so power near a galinstan spill should be shut down before cleanup.

Galinstan Selection

After carefully considering gallium and galinstan properties, the experimentalists chose galinstan. Galinstan offered the advantage of being easily scaled to lithium properties, and did not require heating to liquefy the metal alloy, which precluded hazards associated with heat tracing the system piping. In addition, vinyl piping could be used with galinstan. Overall, galinstan is a good choice to study liquid metal flow without the safety concerns of high temperature operation, or the chemical reactivity issues of Na or Li. One potential drawback to use of galinstan is its cost. In the Sigma Aldrich chemical catalog (usually a premium cost for small quantities), Ga-In-Sn alloy is roughly \$4/g. The RG Medical Products company quoted a price of roughly \$1/g (as of September 2002). The MTOR staff stated that they had found a California metal alloys firm that would produce galinstan for ~ \$0.25/g. Thus, the cost issue was more manageable.

Safety Walkthrough Results

In October 2002, a safety walkthrough was performed on the MTOR experiment. The experiment uses magnetic fields, a set of 24 magnets that create a 0.6 Tesla field (1,800 amperes/coil at 350 Vdc, totaling 630 kW), the galinstan flow loop, and electrical energy for powering the equipment and the control and data acquisition systems. There is also a small overhead crane to lift and place the magnet coils. The experiment procedures are followed, and each experiment operation is attended by the lead staff researcher, two or more graduate students, and the lab safety officer. Due to noise levels created by the magnet power supply fans, the magnets are pulsed for short periods, 10 to 20 seconds. The flow loop operates continuously during an experiment runs (e.g., for a morning or an afternoon). The lab room was found to be typical of university lab settings. The good practices and safety suggestions from the walkthrough were presented to the MTOR researchers, and are listed below:

Good Practices at the MTOR lab:

1. Galinstan is a good substitution alloy in place of alkali metal coolants such as Li, LiSn, or LiPb. Galinstan has several safety attributes compared to Li or Na: low chemical reactivity, low temperature, and low toxicity. UCLA researchers have obtained the alloy at a low price.

2. Gallium and Ga-In-Sn flow loop operating experiences have been positive at several institutions in the US and abroad. The alloy is apparently easy to work with in the laboratory and the ability to use plastic piping simplifies flow loop plumbing.

3. The staff has shown intuitive understanding of the experiment hazards. For example, they posted hand-made warning signs for magnetic fields and use a rotating strobe light (with a yellow color panel) to indicate caution when the experiment is in operation.

4. The staff enforces the 'buddy system' laboratory safety rule. This is a commendable practice in a university, because graduate students are often known to perform their experiments alone.

Safety Suggestions for the MTOR lab:

1. The quantity, mobility, and toxicity of gallium oxide should be further investigated. The formed oxide appears to remain in a viscous crust at gallium-air interfaces.

2. There were no procedures noted for lab events, such as galinstan spill cleanup. Perhaps this is because senior lab personnel are always present when the experiment is operated.

3. If alloy spills, the staff should verify that it is not conducting electricity before cleaning it up. If spilled alloy touches the MTOR aluminum frame then the frame should be inspected for surface corrosion damage.

4. Warning signs for 5 gauss magnetic fields can be downloaded from the fnal.gov and other internet sites; such a sign would have a universal symbol that transcends language barriers.

5. Magnetic field strength contours should be remapped when the magnets are run at 1.2 Tesla. Warning signs may be needed in the public hallway near the experiment room.

6. Ferromagnetic objects (e.g., hand tools) were left near the magnet coils. The staff stated that they had not seen any magnetic field induced missiles when ramping up to, or operating at,

the 0.6 Tesla level. The staff should consider the possible effects of magnetic field-induced missiles when they double the field strength.

7. Older magnets could give reliability problems, such as overheating, or insulation breakdown that results in arcing. Reviewing fire-fighting and room evacuation procedures is prudent.

Conclusions

Galinstan safety concerns include skin injection toxicity, oxide exposure, and electrical safety; the gallium alloy spill would easily conduct electricity. The walkthrough showed that the MTOR experiment safety was consistent with other scholastic research laboratories. Lab funding is modest; there is a "make it work" attitude among the students and staff. They are accomplishing their tasks, performing very interesting research - often reusing equipment from other UCLA labs or building their own equipment. As noted at other schools, the UCLA student focus is on the successful completion of the experiment and analysis of results rather than on experiment safety. The staff must continue with diligence in laboratory and experiment safety. The MTOR staff reviewed the safety suggestions and agreed to implement those they deemed necessary and cost effective. The MTOR experiment has been operating safely since early 2002.

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