Arsenic Exposure in Gallium Arsenide Device Manufacturing: An Evaluation of Control Methods during Molecular Beam Epitaxy (MBE) Maintenance

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Abstract: Special hazards exist during maintenance of Molecular Beam Epitaxy (MBE) systems used for GaAs device manufacturing when the reactor chamber must be accessed. This paper summarizes an arsenic exposure evaluation conducted during a maintenance task on a MBE system on the University of California at Berkeley campus and describes some exposure control methods that are used in industry and academic settings. Ten recommendations to reduce arsenic exposure during MBE maintenance tasks are provided that can be used in R&D and academic settings, such as the UC Berkeley Integrated Materials Laboratory where exposure monitoring was conducted.

Keywords: arsenic, GaAs, compound semiconductor manufacturing, control methods, glove bag, local exhaust ventilation, maintenance operations, MBE, Molecular Beam Epitaxy, surface wipe samples.

Background

Molecular beam epitaxy (MBE) uses advanced thin-film growth techniques to grow semiconductor heterostructure layers by combining group III and group V elements. MBE technology is forecasted to expand because it produces compound semiconductor materials with high precision and purity that are used in cellular phones, fiber-optics, satellites, radar systems, and display devices (Veeco-Applied Epi, 2003). Performed at very low pressures inside a stainless steel MBE chamber, ultra-pure elements are delivered as a beam of gas to the substrate wafer, resulting in high precision products (Veeco-Applied Epi, 2003). Gallium Arsenide (GaAs) device manufacturing using MBE is the focus of this paper in evaluating the potential health hazards of worker exposure to arsenic, specifically during maintenance activities. Typical maintenance tasks that require access to the internal MBE chamber include source replenishment, broken wafer retrieval, and chamber cleaning (Jones, 2001). This paper will review arsenic health hazards, summarize an arsenic exposure evaluation conducted during a maintenance task on a MBE system on the University of California Berkeley (UCB) campus, and summarize exposure control methods that are used in industry settings and another academic setting. The objective of this paper is to recommend effective control methods during MBE maintenance tasks that can be used in R&D and academic settings, such as the UCB Integrated Materials Laboratory where the exposure monitoring was conducted.

Health hazards and regulations

Gallium arsenide typically contains 48.2% gallium and 52.8% arsenic. Gallium arsenide toxicity is primarily due to the inorganic arsenic component, not the gallium component. Adverse health effects may be caused by exposure to inorganic arsenic via inhalation, ingestion or dermal contact. Acute effects due to high levels of arsenic exposure include poor appetite, nausea, vomiting, and muscle cramps. Acute effects due to low level exposure include nose and throat irritation and a rash. Eye contact can cause redness, irritation, and watery eyes. Chronic effects of arsenic exposure include liver damage and decreased ability to make red blood cells. Repeated skin contact can cause thickened skin and/or patchy areas of darkening and loss of pigment. Some people develop white lines on their finger and toenails. High or repeated exposure can cause nerve damage, resulting in feelings of "pins and needles", burning, numbness, and weakness of the arms and legs. Arsenic is also a known human lung and skin carcinogen regulated by Federal OSHA and Cal/OSHA. The inhalation and absorption of inorganic arsenic increases the risk of lung cancer and tumors of the bladder, kidney, and lung. Upon contact with water vapor, arsenic particulates can produce arsine (AsH_3), a potent hemolytic agent (Klaasen, 1996). It is theorized that the presence of aluminum may catalyze this reaction (Asom *et al*, 1991). Acute symptoms of arsine gas exposure include nausea, vomiting, shortness of breath, and headache; persistent exposure can cause jaundice and anemia (Klaasen, 1996).

In 2000, American Xtal, a supplier of GaAs wafer substrates, was cited by Cal/OSHA for over $300,000 in penalties due to health and safety violations. The citations alleged the exposure of workers to arsenic levels greater than the Cal/OSHA PEL of 10 $\mu g/m^3$ (DIR, 2000). The high dollar amounts of the penalties were due to a recently enacted state law, AB 1127, that became effective in California on January 1, 2000. The law was enacted to increase the effectiveness of Cal/OSHA's enforcement efforts through higher citation penalties. Historically, arsenic exposure of workers has been a significant occupational health concern in most semiconductor facilities. Most modern fab facilities have done a good job of controlling significant occupational exposures, yet there is still concern over the effects of low level exposure in the industry. A recently published study of semiconductor workers in Taiwan found a significant decrease in white blood cell count compared to a control group of workers. The researchers found that arsenic was a listed potential source toxin in this plant, along with glycol ethers (Luo *et al*, 2002). Although there is not a regulated surface contamination standard for inorganic arsenic, hazardous surface dust may be dislodged, and become a fugitive emission that can be inhaled. Additionally, the dust may be ingested via hand-to-mouth contact, eating or smoking, or be tracked out of the laboratory/maintenance area on shoes, clothing, skin surfaces or hair. A study of low level arsenic exposure suggested that arsenic intake via ingestion rather than inhalation might play a significant role in observed elevated urinary arsenic levels among workers because measured air concentrations were very low, but high arsenic levels were found in wipe samples (Hwang, 2000). This may indicate that improved control methods should be implemented to reduce surface arsenic contamination and that maintaining air concentrations below OSHA standards is not totally adequate in controlling arsenic exposure.

Exposure Monitoring

The author worked as an intern for the UCB Office of Environment, Health and Safety (EH&S). In that role she evaluated arsenic exposure during maintenance work on the Molecular Beam Epitaxy (MBE) equipment in the Integrated Materials Laboratory (IML) on the UCB campus. The MBE machine in the IML is an Intevac Mod-Gen II MBE chamber that has been operational at UCB since January 1996. It is currently being used for the growth of non-stoichiometric Gallium Arsenide layers. Solid source dopant materials include beryllium and silicon. Because beryllium is known to cause respiratory disease and there may be no safe level of exposure to some individuals that have become sensitized, air and wipe samples were analyzed for both arsenic and beryllium. On the day monitoring was conducted, the internal substrate heater was being replaced and other components within the MBE that get deposited/crusted with arsenic residues were removed, scraped clean within a laboratory-type fume hood, and replaced in the MBE. When maintenance is performed inside the MBE, there is a much higher potential for contaminant exposure that is not present during normal MBE use. The methodologies of air and wipe sampling are detailed in the sampling methods section that follows. Two personal air samples and one area air sample were collected to quantify airborne particulate arsenic and beryllium levels to which employees might be exposed during maintenance of the MBE. The area air sample was collected just below the MBE chamber opening (see photo 1) where the heater and components were removed. Three area surface contamination wipe samples were collected at the beginning of the day and again after the maintenance was completed. The wipe samples were collected on the bench top directly below the opening to the MBE (see photo 1), on the floor below the MBE (see photo 2), and on the desktop in front of the keyboard in a clean area. In addition, one wipe sample was collected in the hallway outside of room 157 after the maintenance and cleanup had been performed (see attached Diagram 1 for room layout).

Photo 1. Main access port of MBE and bench top where a sample wipe was collected

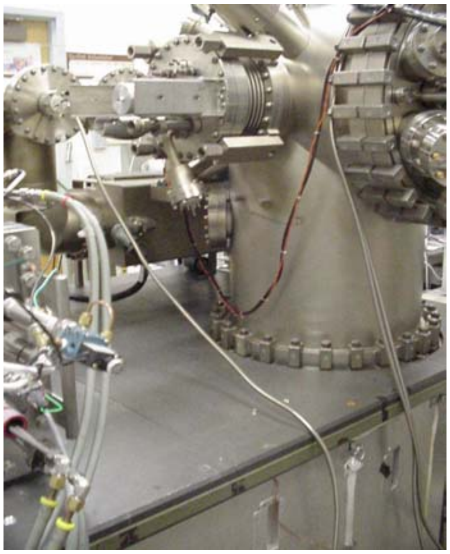
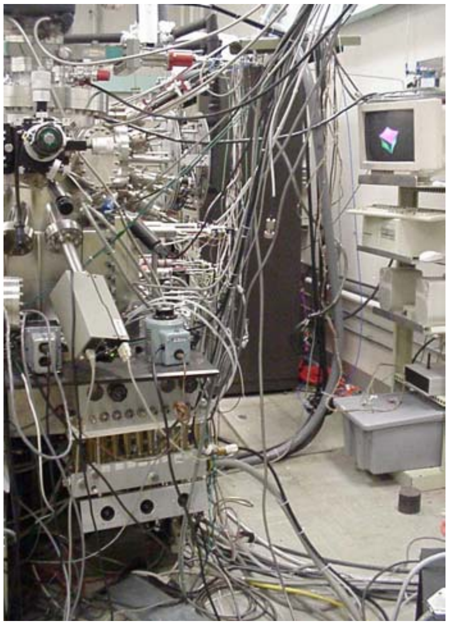


Photo 2. Area around MBE - bench top and main access port is located behind the wires



Ventilation

A laboratory-type fume hood near the MBE was used to store and work on contaminated components of the MBE. On the day of monitoring, it was functioning correctly with an average face velocity of 128 linear feet per minute. Face velocity readings were measured using a calibrated TSI VelociCalc anemometer. General exhaust was provided by the room's mechanical ventilation system.

Personal Protective Equipment

All employees working on the MBE wore Tyvex coveralls, foot covers, a hair cap, and latex gloves during their work on the MBE, as well as full-face respirators with HEPA cartridges. Respirator-users had been fit-tested and medically approved prior to work involving hazardous materials.

Preparation and Clean-up Procedures

Before maintenance on the MBE began, work areas were cleared of clutter by the maintenance technicians. The inside of the laboratory-fume hood where internal MBE components were to be cleaned had been fully covered with disposable aluminum foil. Also, an equipment cart covered in disposable aluminum foil was used to move contaminated parts from the MBE to the laboratory-fume hood. At the end of the day, after the maintenance work was completed and the MBE had been closed, the laboratory supervisor cleaned all work areas using a HEPA vacuum with brush attachment. The lab supervisor also used ethanol to wet wipe tools and the work surfaces inside the fume hood where most of the spillage had occurred. Contaminated material was placed into a bag and left inside the laboratory-fume hood for later disposal as extremely hazardous waste.

Sampling Methods - Air

Sampling and analysis of airborne particulate arsenic and beryllium were conducted using National Institute for Occupational Safety and Health (NIOSH) method 7900. Pre-calibrated sampling pumps drew room air through a 0.8 micron-pore-sized mixed cellulose ester (MCE) filter at a rate of 2.5 liters per minute. The filters were analyzed using graphite furnace atomic absorption spectroscopy (GFAAS) by Forensic Analytical, an American Industrial Hygiene Association (AIHA) accredited laboratory in Hayward, CA. For personal air samples, the pump was worn on the belt connected with Tygon tubing to a filter cassette which was hung from the neckline of the laboratory engineer's Tyvek coveralls in order to sample in the worker's breathing zone (see Photo 3). The pump for the area air sample was set-up on a bench top with the 0.8 micron-pore-sized mixed cellulose ester (MCE) filter cassette hanging from wiring on the MBE approximately 12 inches below the chamber access port (see attached Diagram 1).

Photo 3. Engineer wearing an air sampling pump and filter cassette before performing maintenance work on the MBE

Sampling Methods - Surface Wipes

Surface contamination was measured by wiping a 6" by 6" area using an overlapping "S" pattern with a pre-moistened commercial towelette. The seven samples were analyzed for total inorganic arsenic content by Forensic Analytical of Hayward, CA, an AIHA accredited laboratory, following NIOSH method 7300 of Inductively Coupled Plasma (ICP) Spectroscopy. Two selected wipe samples were also analyzed for beryllium content by the same method. Wipe results, reported by the laboratory in mg/ft^2, were converted to $\mu g/100cm^2$ for comparison with industry standards.

Air Samples Results

All three air samples resulted in airborne inorganic arsenic concentrations below the Cal/OSHA PEL of 10 $\mu g/m^3$ (see Table 1). The supervisory engineer's personal air sample was the highest result with an 8-hour time weighted average of 2.8 $\mu g/m^3$, which is less than 30% of the permissible exposure limit (PEL). The area sample collected below the opening of the MBE had a detectable amount of arsenic with a time weighted average of 1.2 $\mu g/m^3$. The personal air sample of the lab technician did not have a detectable amount of inorganic arsenic. Based on the analytical laboratory's detection limit, his 8-hour time weighted average inorganic arsenic concentration was less than 0.5 $\mu g/m^3$. All three air samples had beryllium concentrations below the detection limit of the method and also well below the Cal/OSHA PEL (see Table 2).

Table 1. Arsenic Air Sample Results - UC Berkeley IML

Sample #	**Sample Location**	**Time (min)**	**Volume (Liters)**	**Average Conc. (μg/m^3)**	**8-Hour TWA (μg/m^3)**	**Cal/OSHA PEL (μg/m^3)**
SA080702-6	Personal Sample/ Supervisor	448	1231	**3.0**	2.8	10
SA080702-7	Area Sample/ bench top under MBE opening	447	1224	**1.2**	1.1	10
SA080702-8	Personal Sample/ Lab Tech.	316	846	**ND, < 0.8**	ND, < 0.5	10
SA080702-16	Blank	-	-	**ND, < 0.6 μg**	-	N/A

ND = non-detectable
< = less than; indicates that the concentration is less than the analytical limit of detection
PEL = Cal/OSHA Permissible Exposure Limit
TWA = Time-weighted-average
μg/m^3 = μg arsenic per cubic meter of air

Table 2. Beryllium Air Sample Results - UC Berkeley IML

Sample #	**Sample Location**	**Time (min)**	**Volume (Liters)**	**Average Conc. (μg/m^3)**	**8-Hour TWA (μg/m^3)**	**Cal/OSHA PEL (μg/m^3)**
SA080702-6	Personal Sample/ Supervisor	448	1231	**ND, < 0.2**	ND, < 0.19	2
SA080702-7	Area Sample/ bench top under MBE opening	447	1224	**ND, < 0.2**	ND, < 0.19	2
SA080702-8	Personal Sample/ Lab Tech.	316	846	**ND, < 0.2**	ND, < 0.13	2
SA080702-16	Blank	-	-	**ND, < 0.2 μg**	-	N/A

ND = non-detectable
< = less than; indicates that the concentration is less than the analytical limit of detection
PEL = Cal/OSHA Permissible Exposure Limit
TWA = Time-weighted-average
μg/m^3 = microgram arsenic per cubic meter of air

Wipe Sample Results

The three surface wipe samples that were collected *before* maintenance on the MBE was performed had arsenic concentrations well below the lowest recommended industry standard of 20 ug/100 cm^2 (see Table 3). However, two of three surface wipe samples collected *after* maintenance on the MBE indicated arsenic concentrations above the industry standard of 20 ug/100 cm^2. After maintenance and cleaning was performed, the wipe result on the bench top under the MBE opening was 42 ug/100 cm^2 and the result from the floor sample under the MBE opening was 101 ug/100 cm^2, which is greater than the higher industry standard of 100 ug/100 cm^2.

Table 3. Arsenic Wipe Sample Results - UC Berkeley IML

Sample Location	**Arsenic Conc. (μg/100 cm^2) Before Maintenance**	**Arsenic Conc. (μg/100 cm^2) After Maintenance**	**Industry Standard (μg/100 cm^2)***
bench top under MBE	**2.69** (1W)	**42.0** (9W)	20-100
floor under MBE	**6.35** (2W)	**101** (10W)	20-100
desktop near keyboard	**ND, < 0.86** (5W)	**1.61** (13W)	20-100
hallway outside lab	N/A	**ND, < 0.86** (14W)	20-100
Blank	N/A	**ND, < 0.22 μg** (15W)	N/A

All samples were collected on 8/7/02, so all sample strings are SA080702-X, e.g. SA080702-1W
ND = non-detectable
N/A = not available
μg/100 cm^2= μg arsenic per 100 cubic centimeters of surface area sampled
< = less than; indicates that the concentration is less than the analytical limit of detection
Before = Before maintenance was performed on the MBE
After = After maintenance was performed on the MBE and work areas were cleaned
* Industry Standard Source: *Hazard Assessment and Control Technology in Semiconductor Manufacturing*, American Conference of Governmental Industrial Hygienists, 1989. p 167.

An additional surface wipe sample collected in the hallway outside of the laboratory after maintenance on the MBE was finished and clean-up had been completed served as a field control. This sample had an arsenic concentration less than the laboratory's detection limit. Although the arsenic level on the desktop after maintenance was performed was low at 1.61 μg/100 cm^2, this result demonstrates that some arsenic contamination occurred away from the immediate work area (see Photo 4).

Photo 4. Desktop away from the MBE work area -
Wipe sample collected in front of keyboard

The wipe samples collected on the bench top under the opening to the MBE were also analyzed for beryllium and found to have levels below the laboratory's detection limit and the industry standard of 1.0 μg/100 cm^2 (see Table 4).

Table 4. Beryllium Wipe Sample Results - UC Berkeley IML

Sample #	Sample Location	Area Sampled (cm^2)	Beryllium Conc. (μg/100 cm^2)	Industry Standard (μg/100 cm^2)*
SA080702-1W	Before - bench top under MBE	232	**ND, < 0.43**	1
SA080702-9W	After - bench top under MBE	232	**ND, < 0.43**	1
SA080702-15W	Blank	-	**ND, < 0.11 μg**	NA

ND = non-detectable
μg/100 cm^2= μg arsenic per 100 cubic centimeters of surface area sampled
< = less than; indicates that the concentration is less than the analytical limit of detection
Before = Before maintenance was performed on the MBE
After = after maintenance was performed on the MBE and work areas were cleaned
* Industry Standard Source: DOE, Lawrence Livermore Laboratories

Semiconductor Industry

To learn what control methods are typically used in the semiconductor industry, the author visited a MBE facility in the San Francisco Bay Area. Health and Safety specialists with Agilent Technologies offered information about their arsenic program and allowed the observation of the type of control methods they have in place. As shown in Photo 5, local exhaust ventilation (LEV) trunks attached to chamber view ports would draw air away from the worker's breathing zone at the chamber access port, thereby reducing airborne arsenic exposure and potential arsine gas exposure. One facility had air monitoring data showing that LEV reduced airborne arsenic concentrations by about ten-fold. Workers don Tyvex coveralls and either supplied air line respirators or respirators with HEPA and organic vapor cartridges to collect particulates and arsine gas. The probe of a gas monitor capable of detecting arsine gas is placed near the chamber opening.

Photo 5. Local Exhaust Ventilation on MBE view ports
Courtesy of Agilent Technologies

QuickTime™ and a
Photo - JPEG decompressor
are needed to see this picture.

Special hazards exist with large MBEs, such as the one shown in Photo 6. This MBE has a chamber diameter of four feet; the bottom portion is removed for chamber cleaning. Because the workers must clean overhead there is greater potential for high arsenic exposure. These workers are therefore wearing supplied air line respirators.

Photo 6. Two workers cleaning inside of a MBE chamber
Courtesy of Agilent Technologies

Medical monitoring via annual urinary arsenic analysis is conducted. Because non-occupational sources (e.g. seafood; Klaasen, 1996) may contribute to elevated organic arsenic levels, urine is analyzed for both inorganic and organic arsenic. The surface standard used for inorganic arsenic is 100 μg/100 cm^2, based on the logic that workers inhale roughly 10 m^3 during an 8-hour shift. Using the airborne Cal/OSHA PEL of 10 μg/m3, an effective dose of 100 μg arsenic is calculated. This logic is valid for 4,4'-methylenebis, which has a Cal/OSHA surface standard of 100 μg/100 cm^2 and airborne PEL of 10 μg/m^3. Tacky mats are placed at doorways to reduce contamination.

Academic Use

University of California at Santa Barbara (UCSB) has a facility with six MBE machines. A unique control method they use is a glove bag (see Photo 7). Initially designed to keep internal components of the MBE chamber protected from ambient air, the glove bag also acts as an arsenic control method because it creates a barrier between the contaminated materials and the workers. Positive pressure is maintained inside the glove bag by supplying nitrogen through an access sleeve. As shown in the photo, workers wear respirators with organic vapor and HEPA cartridges. For wet cleaning and contamination control, the UCSB facility also uses "Swiffers", a commercial household cleaning tool that sprays liquid ahead of the cleaning surface, thereby minimizing aerosolization of dust particulates.

Photo 7. Glove bag used during MBE maintenance task
Courtesy of UCSB

QuickTime™ and a
Photo - JPEG decompressor
are needed to see this picture.

Recommendations for UC Berkeley IML

Air sampling results indicate that arsenic and beryllium exposures during maintenance of the MBE were below Cal/OSHA Permissible Exposure Limits. However, the arsenic surface results on the bench top and floor below the MBE indicate that arsenic deposition onto the surfaces occurred during maintenance work and that clean-up procedures were not adequate in reducing the contamination levels to below recommended industry standards. The employees reported that the activities performed were relatively clean compared to some previous maintenance tasks on the MBE. Thus, the arsenic surface levels measured probably do not represent the worst-case contamination levels. Although arsenic is not easily absorbed through the skin, surface contamination is of concern because the hazardous dust may be inhaled, ingested, or tracked out of the work area. In order to reduce arsenic exposure during maintenance of the MBE, the following actions are recommended:

1. Clear floor area near MBE opening of wires that may collect contaminated dust or interfere with cleaning.

2. Place tacky mats at entrances to prevent particulates from being tracked out of the immediate MBE area.

3. Wet wipe the floor and bench top under the MBE opening after thorough vacuuming with a HEPA vacuum. All towels used during wet wiping should be double-bagged and disposed of as extremely hazardous waste. A "Swiffer" can be used for floors, but the cleaning pad will need to be disposed of as hazardous waste.

4. If clearing the area of wires is not feasible, it will be difficult to adequately clean this floor space. The floor area under the MBE may need to be covered before maintenance work is started. The covering should be disposed of as extremely hazardous waste after the maintenance tasks on the MBE are completed. Large sheets of lint-free paper or tacky paper for covering surfaces are recommended instead of aluminum foil.

5. To capture arsenic-contaminated dust when the MBE is opened, a glove bag may be appropriate. The glove bag could be sealed over the access port of the MBE before it is opened. The opening cover and internal components that need to be serviced could then be contained within the glove bag. After the task is finished the glove bag can be HEPA vacuumed or disposed of as extremely hazardous waste. Glove bags are economical and

often used in asbestos abatement work. Glove bags can be designed with multiple sets of gloves (as shown in Photo 7) which may be necessary if multiple workers are required to open the MBE or work on internal components.

6. A standard control method in MBE facilities is local exhaust ventilation connected to the duct system (as shown in Photo 5), but because maintenance tasks are not performed very frequently, this may be uneconomical for an academic research lab. An alternative would be to connect a portable dust extractor to a chamber port other than the port that is being accessed in order to create negative pressure in the chamber. This would minimize the amount of arsenic-contaminated dust that escapes into the room. Numerous portable dust extractor models are available with HEPA filters and adequate flow rates (Lab Safety Supply, 2003).

7. Although air concentrations were well below the Cal/OSHA PEL, the continued use of full-face respirators with HEPA cartridges during maintenance work is recommended. The respirators should be supplemented with organic vapor cartridges designed to capture potential arsine gas.

8. The HEPA vacuum should be supplemented with an activated charcoal canister to remove arsine from the HEPA filter exhaust.

9. The HEPA vacuum and special tools used for maintenance on the MBE should be stored separately in the lab and labeled with stickers "Arsenic Contaminated - for MBE use only". Additionally, all waste and contaminated materials should be labeled "DANGER - CONTAINS INORGANIC ARSENIC - CANCER HAZARD - HARMFUL IF INHALED OR SWALLOWED."

10. Improve written Standard Operating Procedures (SOP) for maintenance activities on the MBE to establish minimum guidelines for adequate preparation and clean-up each time the MBE is serviced. This SOP should also include personal protection requirements, hazard communication, a description of engineering/ventilation controls, spill and accident procedures, waste disposal guidelines, and a plan for periodic wipe sampling to verify that surfaces are clean.

In addition, if maintenance on the MBE is conducted often, medical monitoring of employees for arsenic exposure at the UC Berkeley Tang Medical Center may be appropriate. The UCSB facility conducting MBE research has a medical monitoring program in place, but employee maintenance tasks are more frequent than at UCB.

Conclusion

MBE has primarily been used as a R&D tool, but because of demand for high quality light-emitting diodes (LEDs) and other MBE high purity and precision products, MBE is increasingly being used in production. According to Global Information, Inc., the total market for MBE equipment, services, and materials is estimated to increase to $1.05 billion in 2006 at an average growth rate of 33.3% (GII, 2002). Arsenic exposure control is important in both academic/R&D settings and production. Academic settings may use older or donated equipment without the resources for engineering controls such as LEV. Thus, lower cost control options such as glove bags or portable dust extractors may be appropriate. In summary, this paper has evaluated arsenic exposure during a maintenance task on a MBE system on the University of California Berkeley (UCB) campus, summarized some arsenic controls methods used in industry and academic settings, and provided ten recommendations for arsenic control in an academic or R&D MBE facility.

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