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Unknown Gases Generated from a Silicon Wafer Grinding Filtration Process – Colorado

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DEPARTMENT OF HEALTH AND HUMAN SERVICES
Centers for Disease Control and Prevention
NIOSH *National Institute for Occupational Safety and Health*

The employer shall post a copy of this report for a period of 30 calendar days at or near the workplace(s) of affected employees. The employer shall take steps to insure that the posted determinations are not altered, defaced, or covered by other material during such period. [37 FR 23640, November 7, 1972, as amended at 45 FR 2653, January 14, 1980].

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ABBREVIATIONS

ACGIH®	American Conference of Governmental Industrial Hygienists
CFR	Code of Federal Regulations
CO	Carbon monoxide
CO_2	Carbon dioxide
GA	General area
GC-MS	Gas chromatography-mass spectrometry
GC-TCD/FID	Gas chromatography-thermal conductivity detector/flame ionization detector
HHE	Health hazard evaluation
H_2	Hydrogen
IDLH	Immediately dangerous to life or health
LOD	Limit of detection
LOQ	Limit of quantitation
mg/m^3	Milligrams per cubic meter
mm	Millimeter
NAICS	North American Industry Classification System
ND	Not detected
NIOSH	National Institute for Occupational Safety and Health
OEL	Occupational exposure limit
OSHA	Occupational Safety and Health Administration
PBZ	Personal breathing zone
PPE	Personal protective equipment
PEL	Permissible exposure limit
ppm	Parts per million
REL	Recommended exposure limit
STEL	Short-term exposure limit
TLV®	Threshold limit value
TWA	Time-weighted average
WEEL™	Workplace environmental exposure level

HIGHLIGHTS OF THE NIOSH HEALTH HAZARD EVALUATION

The National Institute for Occupational Safety and Health (NIOSH) received a request for a health hazard evaluation at a semiconductor manufacturing plant in Colorado. The employer submitted the request because unknown gases had pressurized and heated a covered 55-gallon metal storage drum ("drum") containing used filters from a silicon wafer grinding filtration process. The company was concerned that the disposal of used filters in covered drums could pose a health and safety hazard for its employees.

What NIOSH Did

- We visited the plant in February 2008 and March 2009.
- We collected air samples in the filtration process area and from the inside of covered drums. We sampled for hydrogen, carbon monoxide, and carbon dioxide.
- We reviewed the company's air sampling records, including sampling for silane.
- We spoke with the maintenance employees about their symptoms.

What NIOSH Found

- Hydrogen was measured at concentrations up to 118,000 parts per million inside the covered drums.
- Carbon monoxide and carbon dioxide were also found inside the covered drums.
- Silane was measured at concentrations of 20 to 50 parts per million inside a covered drum.
- Maintenance employees reported no symptoms related to their work that would suggest high exposures to carbon monoxide.
- Disposing of used filters from the silicon wafer grinding filtration process in covered drums can pose a health and safety hazard for employees.

What Managers Can Do

- Leave the drums uncovered to prevent the buildup of gases.
- Post signs in the work area to remind employees not to cover the drums.

What Employees Can Do

- Leave the drums uncovered.
- Report all symptoms, including headache, nausea, or confusion to the on-site healthcare professional.

SUMMARY

NIOSH received a request from an employer representative for an HHE at a semiconductor manufacturing plant in Colorado. The request concerned unknown gases that were pressurizing and heating a covered 55-gallon metal storage drum ("drum") containing used filters from a silicon wafer grinding filtration process. The requestor was concerned that disposing of these used filters in covered drums may present a safety and health hazard to maintenance employees working in the filtration process area. No health effects among maintenance employees were listed in the request.

We visited the plant in February 2008 and March 2009. We collected PBZ, GA, and task-based air samples for H_2, CO, and CO_2. We analyzed the samples with different sampling and analytical measurement techniques. We spoke with maintenance employees about their symptoms and reviewed the company's previous air sampling reports.

We found that H_2 was being generated inside the covered drums and that the concentrations increased with time. We also measured CO and CO_2 inside the covered drums, but these concentrations varied drastically depending on the analytical method, making interpreting the results difficult. Employee PBZ exposure to CO could exceed the NIOSH ceiling limit and ACGIH excursion limit when employees evaluate the covered drums for off-gassing filters prior to disposal. Our results indicate that a possible hazard from CO exposure might exist. This hazard can be easily mitigated by keeping the drums uncovered to prevent the buildup of gases. Maintenance employees reported no symptoms related to their work that would suggest high exposures to CO.

On the basis of a scientific literature review and air sampling results, we conclude that the covered drums were pressurized primarily because of H_2 formation and that silane was also formed. The disposal of used filters resulting from the silicon wafer grinding filtration process in covered drums can pose a health and safety hazard to employees. Leaving the drums uncovered will help mitigate these hazards. In addition, if employees experience headache, nausea, or confusion, they should promptly report them to the on-site healthcare professional.

NIOSH was asked to identify unknown gases accumulating in a covered drum that contained used filters from the silicon wafer grinding filtration process. We measured H_2, CO, and CO_2 inside the covered drums. The concentration of H_2 increased with time inside the covered drums. Disposing of used filters from the silicon wafer grinding filtration process in covered drums can pose a health and safety hazard to employees. Leaving the drums uncovered will help reduce this hazard.

Keywords: NAICS 334413 (Semiconductor and Related Device Manufacturing), silicon, silane, hydrogen, carbon monoxide, CO, carbon dioxide, CO_2, wafer grinding

INTRODUCTION

NIOSH received a request from an employer representative for an HHE at a semiconductor manufacturing plant in Colorado. The request concerned unknown gases that were pressurizing and heating a covered 55-gallon metal storage drum ("drum") that contained used filters from a silicon wafer grinding filtration process. The requestor was concerned about the safety and health of the maintenance employees working near the filtration process as a result of exposure to this unknown gas. No health effects among maintenance employees were listed in the request.

In October 2006 a maintenance employee removed clogged filters from the silicon wafer grinding filtration process that were warm to the touch. Because this was an odd occurrence, the maintenance employee called the company industrial hygienist to ask how to dispose of the used filters. He was instructed to place the filters in a drum and to move the drum outside until the industrial hygienist could evaluate the situation. The employee followed these instructions but sealed the drum with a rigid lid. When the company industrial hygienist arrived, he removed the lid of the drum while wearing PPE (a fully encapsulated suit with a self-contained breathing apparatus). When the seal to the lid was loosened, the lid violently "popped" off the drum a reported 5 feet into the air. No one was injured but the incident did reveal a possible explosion hazard for the maintenance employees.

Following this incident the company industrial hygienist attempted to identify the gases in the drum containing used filters. These attempts included sampling the air inside the drum with a colorimetric detector (ChemKey™ TLD toxic gas detector, Honeywell Analytics, Lincolnshire, Illinois) after it was covered with a shower cap and allowed to sit overnight; air sampling for arsine using NIOSH Method 1003 and phosphine using NIOSH Method 6001; and air sampling for CO and hydrogen sulfide using direct reading instruments. The company industrial hygienist concluded that H_2 was present along with some type of hydride gas.

We visited the plant February 6–7, 2008, to learn more about the process, observe work practices, and conduct air sampling to identify the unknown gases inside covered drums. We returned on March 17–19, 2009, to evaluate potential employee exposures to CO.

Facility and Process Description

The company manufactured a variety of microelectronic devices including radiofrequency integrated circuits and transistors. Silicon wafers, an integral part of these devices, are obtained by cutting a single crystal silicon ingot. These wafers can range up to 10 inches in diameter and are ground to a thickness of 0.07 mm [Burgess 1995].

The backs of the silicon wafers are wet-ground in an enclosed machine with deionized water. The water stream containing elemental silicon is sent to a 1,500-gallon storage tank in the basement of the building. The silicon particles are removed from the water with bag filters and polypropylene cartridge filters. Of the four cartridge filter tanks and four bag filter tanks, only half are operational at any one time. The alternate filters are used when the first set of filters become clogged.

A portion of the filtered water was recirculated to the storage tank, and the rest was discharged. Maintenance employees removed both types of filters on the basis of filter back pressure and the amount of water being discharged. The clogged filters were placed in a nearby uncovered drum.

The company industrial hygienist monitored the drum with a ChemKey TLD toxic gas detector, a direct reading instrument with a colorimetric detector to identify whether the filters are off-gassing. The company had ChemKeys that allowed the instrument to detect and provide quantitative air concentrations for arsine and phosphine. These ChemKeys were chosen for two reasons: the detection limit for arsine is lower than for phosphine and silane, thus allowing detection of hydride at lower levels; and arsine and phosphine gas are filtration byproducts generated during the grinding of arsenic- and phosphorous-doped silicon wafers, which are also manufactured at this plant. The drum was covered overnight with a plastic shower cap, and air samples were taken the following morning. If the instrument detected gas above the PEL for arsine of 0.05 ppm (lower than the phosphine PEL of 0.3 ppm), the drum was uncovered, allowed to off-gas for another 2–3 weeks, and rechecked. If the gas concentrations were below 0.05 ppm, the drum was sealed with a rigid lid and shipped to a contractor for disposal in a landfill.

INTRODUCTION (CONTINUED)

Figure 1. Silicon wafer grinding filtration process.

Figure 2. Used cartridge and bag filters in a 55-gallon metal storage drum.

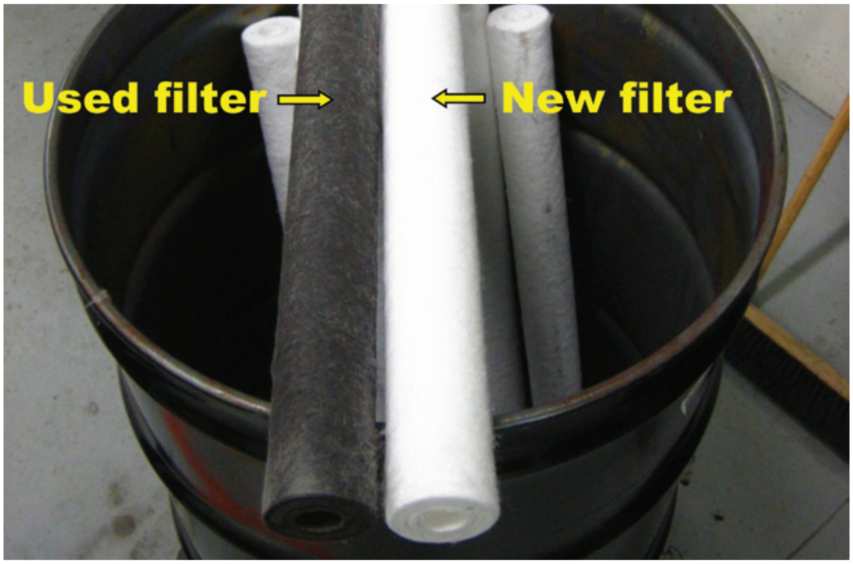

Figure 3. Used and new cartridge filters (Courtesy: Company photo).

February 2008 Site Visit

We met with the employer and employee representatives to discuss the HHE request. We spoke with employees and observed the silicon wafer grinding process, the associated water filtration system, employee work practices, and workplace conditions.

During our visit, a maintenance technician drained the water from the bag and cartridge filter tanks. After waiting approximately a half hour, the technician opened the tops of the bag filter and cartridge tanks. We immediately collected air samples with Vacutainers® (evacuated glass tubes) from inside two of the three cartridge tanks. The filters were removed, placed in a drum, covered with a plastic shower cap, and sealed with tape.

We collected vacutainer air samples from inside the covered drums that contained filters removed on January 18, 2008, and February 7, 2008, in the area around the filtration process, and in office areas on a different floor. We also monitored the temperature inside the covered drum that contained used filters from February 7, 2008. We reviewed air sampling reports dated November 7, 2006, and June 18, 2007, for evaluations conducted by a consultant who was trying to identify the unknown gas.

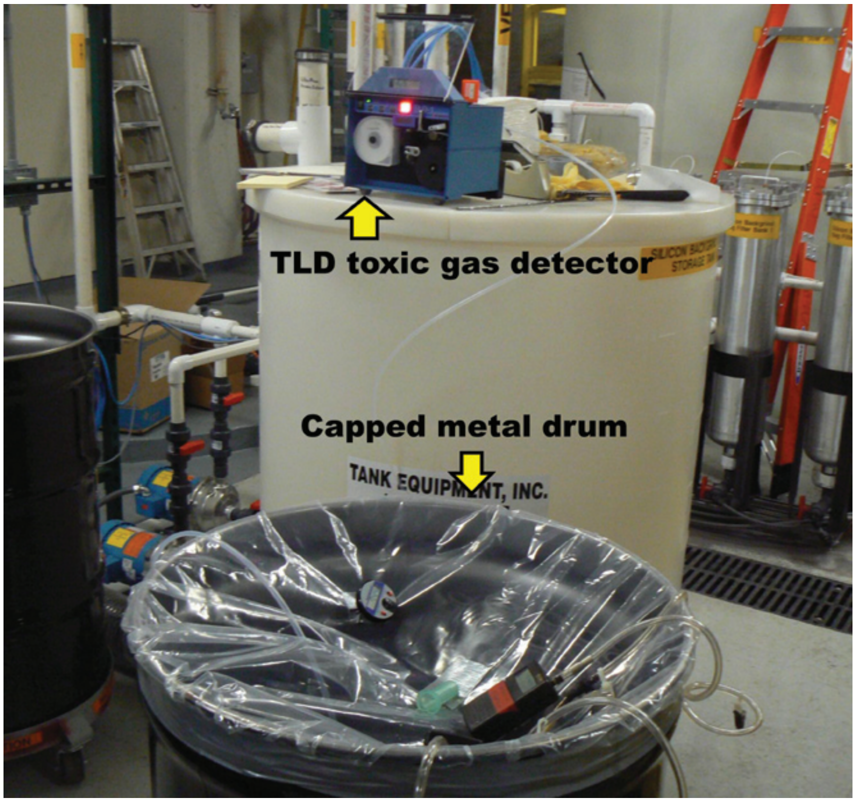

Figure 4. Collecting an air sample from inside a covered 55-gallon metal storage drum that contained filters removed on February 7, 2008.

Figure 5. Collecting a vacutainer air sample from inside a covered 55-gallon metal storage drum.

Analysis of the vacutainer air samples from this visit indicated high concentrations of H_2, CO, and CO_2 inside the covered drums. These findings prompted us to return to the plant in March 2009 to further evaluate CO and CO_2 exposures.

March 2009 Site Visit

We measured airborne CO concentrations in the PBZ of the maintenance employees and the plant industrial hygienist as well as in various areas of the plant with three different direct reading monitors. On March 18, 2009, we measured temperature with a digital thermometer (Fisher Scientific Traceable® Jumbo Display Thermometer, Thermo Fisher Scientific Inc., Pittsburgh, Pennsylvania) and CO concentrations inside two covered drums containing filters removed on March 12, 2009, and March 18, 2009, with one or more sampling techniques: vacutainers, colorimetric gas detection tubes, and a Dräger Pac III direct reading CO monitor (Dräger Safety Inc, Pittsburgh, Pennsylvania). Vacutainers were also analyzed for H_2, CO, and CO_2 by the NIOSH contract laboratory, and a duplicate set of samples was analyzed for CO and CO_2 by the OSHA Salt Lake Technical Center. We evaluated the full-shift CO exposure of the maintenance technician (which included conducting a filter change out) using three different direct reading instruments. During this visit, the company industrial hygienist collected air samples for silane with colorimetric gas detection tubes.

ASSESSMENT (CONTINUED)

At the end of the day on March 18, 2009, both drums (which contained filters from March 12 and 18, 2009) were covered with plastic shower caps and left overnight to simulate conditions under which they would be checked by the company industrial hygienist for off-gassing prior to disposal. On March 19, 2009, we collected vacutainer air samples from the inside of both drums, and we also collected task-based PBZ air samples when the industrial hygienist was checking the drums.

Details on the methods used in this evaluation for H_2, CO, and CO_2, are explained in Appendix A. The OELs and potential health effects from H_2, CO, CO_2, and silane are discussed in Appendix B.

RESULTS

Detailed PBZ, GA, and task-based air sampling results for H_2, CO, and CO_2 are presented in Tables C1–C6 of Appendix C.

February 2008 Site Visit

Table C1 presents the vacutainer air sample data collected from the inside of cartridge tanks 1 and 2; the inside of a covered drum containing filters from January 18, 2008; and in various areas of the plant. The concentrations from inside the cartridge tanks ranged from < LOQ to 64,500 ppm for H_2, 3,500 to 22,400 ppm for CO, and 3,400 to 17,000 ppm for CO_2. The highest concentrations were found when a maintenance technician opened cartridge tank 1; the lowest were from cartridge tank 2. The concentrations from inside the covered drum containing filters from January 18, 2008, were ND (< 5,000 ppm) and 26,200 ppm for H_2; 2,200 and 7,500 ppm for CO; and < LOQ and 5,900 ppm for CO_2. Although the drum cover was not removed, the lower concentration for all three gases was from the second air sample collected from inside the drum, approximately 1 minute after the first air sample. The vacutainer air samples collected in the basement near the process area and on the second floor of the building were all below the LOD.

Table C2 presents the vacutainer air sample data collected from the inside of another covered drum containing filters removed on February 7, 2008. Thirteen vacutainer air samples were collected over approximately 4 hours. The concentrations ranged from ND to 84,700 ppm for H_2; ND to 19,900 ppm for CO; and ND to

16,700 ppm for CO_2. As displayed in Figure 6, H_2 concentrations inside the covered drum fluctuated but increased over time and reached 84,700 ppm at the end of the sampling period. The concentrations of CO and CO_2 did not increase over time, but did seem to mimic each other (i.e., an increase in CO reflected an increase in CO_2). Both CO and CO_2 approached 20,000 ppm at the beginning of the sampling period but were not detected at the end of the sampling period. Temperature readings taken along with each vacutainer air sample indicated an overall temperature increase of 3.0°F.

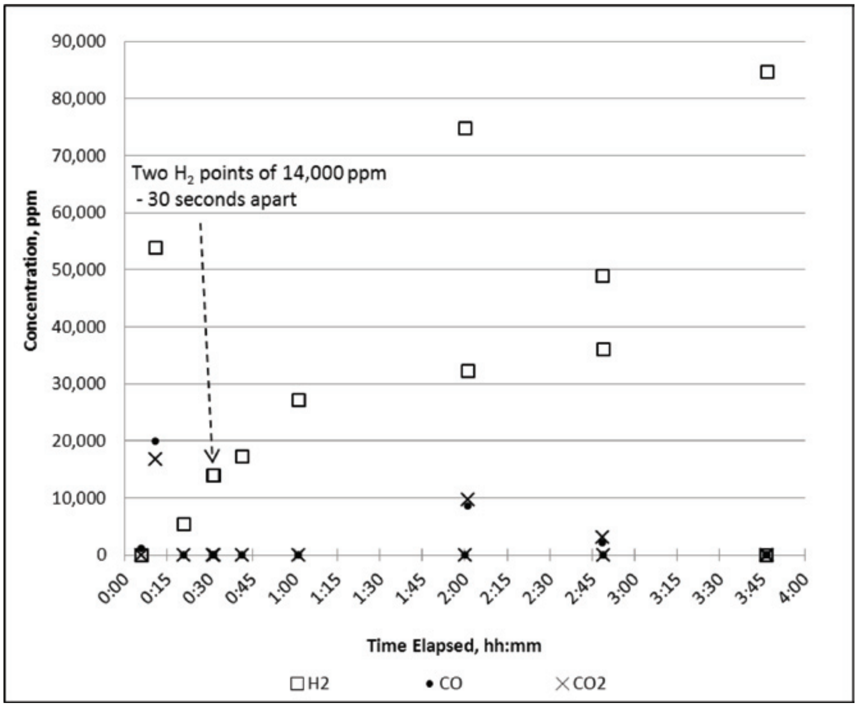

Figure 6. Time plot of H_2, CO, and CO_2 concentrations inside a covered 55-gallon metal storage drum containing filters removed on February 7, 2008.

March 2009 Site Visit

Table C3 presents the vacutainer air sample data collected from the inside of bag filter and cartridge tanks, from the inside of a covered drum containing filters from March 12, 2009, and from the basement. The concentrations inside the bag filter and cartridge tanks ranged from < LOQ to 15,800 ppm for CO and ND to 8,930 ppm for CO_2; H_2 was not detected. The CO and CO_2 concentrations varied between bag and cartridge tanks and did not show a trend within each tank (e.g., bag filters did not always have the highest CO or CO_2 concentration nor did an elevated CO concentration in a cartridge tank have a corresponding elevated CO_2 concentration). The concentrations from inside the covered drum containing filters from March 12, 2009, ranged from < LOQ to 34,000 ppm for H_2, 5,280 to 19,400 ppm for CO, and 3,750 to 15,000 ppm for CO_2. Although the drum cover was not removed, the highest H_2 concentration was from the last vacutainer air sample collected from this drum; in contrast, the CO and CO_2 concentrations decreased over time. The first vacutainer air sample collected in the basement near the process area found all three gases, but the second air sample (collected immediately after the first) identified only CO.

Table C4 presents data from 15 vacutainer air samples collected over a 5-hour period from inside a covered drum containing filters from March 18, 2009. The concentrations ranged from ND to 118,000 ppm for H_2; ND to 21,600 ppm for CO; and ND to 18,800 ppm for CO_2. The results displayed in Figure 7 show similarities to those in our previous visit. The H_2 concentrations increased over time (118,000 ppm at the end of sampling), and CO and CO_2 concentrations mimicked each other; both had peak concentrations around 20,000 ppm. Temperature readings taken along with each vacutainer air sample indicated an overall temperature increase of 14.5°F.

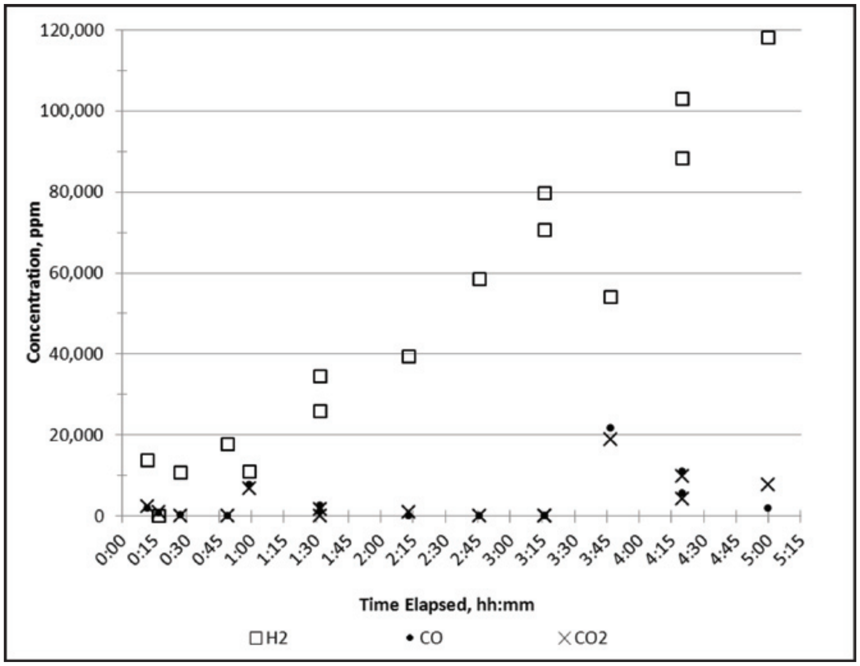

Figure 7. Time plot of H_2, CO, and CO_2 concentrations inside a covered 55-gallon metal storage drum containing filters removed on March 18, 2009.

Figure 8 presents CO concentrations inside a covered drum containing filters from March 18, 2009. In addition to CO data from vacutainer air samples analyzed using GC-TCD/FID (data presented in Table C4 and Figure 7), Figure 8 presents data from vacutainer air samples analyzed by GC-MS, colorimetric detection tubes, and a Pac III direct reading instrument. The OSHA Salt Lake Technical Center analyzed the duplicate vacutainer air samples via GC-MS and reported all CO concentrations as less than 100 ppm and CO_2 concentrations as approximately 400 ppm. Of the six colorimetric detection tube air samples collected during this period, the concentration of CO inside the covered drum ranged from 8 to 40 ppm. The Pac III measurements showed an increase in CO concentrations over time that ultimately reached the upper limit of the instrument (10,000 ppm).

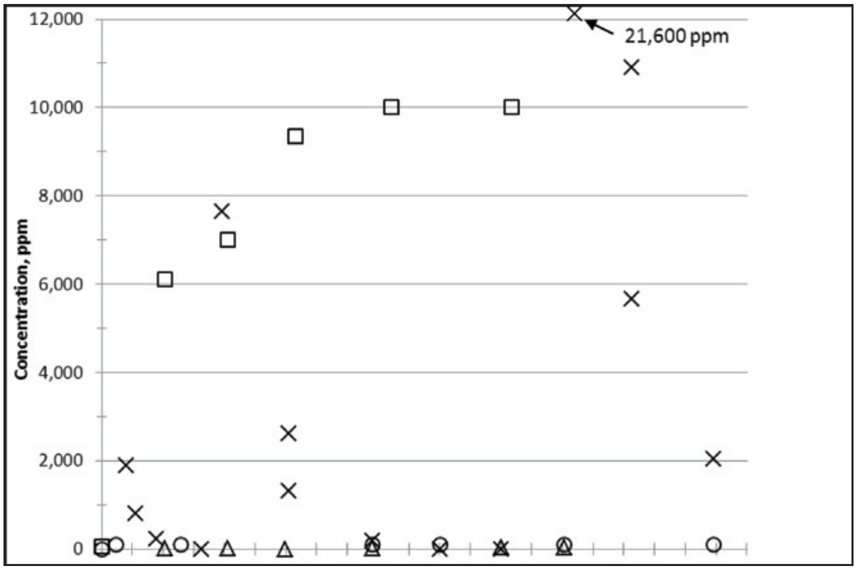

Figure 8. Time plot of CO concentrations measured using different analytical methods inside a covered 55-gallon metal storage drum containing filters removed on March 18, 2009.

In addition to the CO air samples we collected inside a covered drum containing filters from March 18, 2009, the company industrial hygienist collected air samples for silane inside the same drum with a colorimetric detection tube (Sensidyne, Mülheim, Germany). The colorimetric detection tubes showed silane concentrations ranging from 20 to 50 ppm. The inside of the drum was also sampled using a ChemKey TLD toxic gas detector that showed that hydride gases were present.

Table C5 presents vacutainer air sample data from two covered drums containing filters from March 12 and March 18, 2009. Both drums were left covered overnight and air samples were collected on March 19, 2009. The concentrations inside the drum containing filters from March 12, 2009, ranged from 13,900 to 25,400 ppm for H_2; ND to 21,400 ppm for CO; and ND to 18,600 ppm for CO_2. The concentrations inside the drum containing filters removed on March 18, 2009, ranged from 138,000 to 153,000 ppm for H_2; 643 to 2,900 ppm for CO; and ND to < LOQ for CO_2.

On March 18–19, 2009, using three different direct reading instruments, we evaluated the full-shift CO exposure of a maintenance technician who conducted a filter change out. We also evaluated the CO and H_2 exposures of the company industrial hygienist while he was checking drums prior to disposal (Table C6). The CO measurements of the maintenance technician did not exceed applicable OELs. The peak CO measurement was 29 ppm, which is well below the NIOSH ceiling limit of 200 ppm. This air concentration was recorded when the technician had left the filter change out process area to complete other routine tasks. All three instruments' CO concentration measurements were consistent with each other. Two of three direct reading instruments showed peak CO measurements for the company industrial hygienist that exceeded the NIOSH ceiling limit of 200 ppm for CO, and all three exceeded the ACGIH excursion limit of 125 ppm. We also collected two PBZ air samples with vacutainers during this task; one showed the presence of CO at a concentration below the LOQ (228 ppm), and the other did not detect CO above the LOD of 100 ppm.

Document Review

The company suspected that a hydride gas caused the October 2006 incident, so they asked the consultant to sample for silane and phosphine inside the covered drums and workplace air. Air sampling for phosphine (four GA air samples) was conducted using OSHA Method 1003. Silane (four GA air samples) was sampled using an in-house method developed by the consultant. This method consisted of a 37-mm mixed cellulose ester prefilter in line with an impinger containing a 0.01N potassium hydroxide solution. The air samples were analyzed to detect the presence of silicon using inductively coupled argon plasma-mass spectrometry. Silane was detected in the air samples, but phosphine air concentrations were not detected above an LOD of 0.030 ppm. Silicon, however, was detected in the field blank sample, and the consultant attributed it to silicon possibly being etched from the glass impinger. The consultant recommended further investigation to determine if the unknown gas was truly silane.

The consultant also performed drum and GA air sampling in May 2007 with Teflon® impingers (instead of glass) and the same sampling and analytical methods. Silane concentrations

RESULTS (CONTINUED)

inside a covered drum were 0.99 mg/m^3 and 0.12 mg/m^3, and the silane concentrations in two comparison air samples collected in a room south of the silicon wafer grinding process were 0.029 mg/m^3 and 0.024 mg/m^3. Because the air concentrations were below applicable OELs no recommendations were made.

DISCUSSION

Crystalline silicon doped with different elements is a base semiconducting material for many electronic products. Upon grinding, crystalline silicon ingots produce fine silicon powder, which has a high surface area. Theoretically, silicon reacts with water to produce silicon dioxide (silica) and hydrogen, and this reaction (wet oxidation) can be expected to occur during the wet grinding process, as shown below [Doremus and Breed 1976; Stephen and Riley 1989; Chemguide 2010; Gatech 2010].

$$\mathrm{Si} + 2\mathrm{H_2O} \longrightarrow \mathrm{SiO_2} + 2\mathrm{H_2}\ (\mathrm{g})$$

$$\mathrm{Si} + 2\mathrm{H_2} \longrightarrow \mathrm{SiH_4}\ (\mathrm{g})$$

It is possible that some silicon can react with the generated hydrogen gas to form silane as shown above. Experiments involving the reaction of water vapor with freshly cleaved, etched, or polished silicon surfaces have shown silane in ppm concentrations [Lampert et al. 1986]. Salonen et al. studied the oxidation of porous silicon in water, methanol, and ethanol. Silane was not detected when porous silicon reacted with water. However, the reaction of porous silicon with methanol and ethanol showed traces of silane, and pressurization of glass ampoules used in the experiments was also observed [Salonen et al. 1997]. Silane is a pyrophoric gas that poses a severe explosion hazard [ACGIH 1989].

Our air sampling of the drums showed that H_2 gas was being generated, and the concentrations increased with time inside a covered drum. The increasing H_2 concentrations inside a tightly sealed drum could cause it to become pressurized. Additionally, the drum temperature increased over time, suggesting exothermic chemical reactions inside the drum. The presence of silane is theoretically possible and was shown by air sampling (impinger and colorimetric tube) conducted by the company industrial hygienist. The presence of a pyrophoric gas such as silane and a combustible gas such as H_2 in the same covered drum could be a safety hazard.

This hazard could be easily minimized by ensuring that the drums are left uncovered at all times thus preventing a buildup of such gases.

In our first site visit, we were surprised to measure high CO and CO_2 concentrations inside the covered drums because there was no apparent source to generate these gases. However, the concentrations reported in the tables are not indicative of employee PBZ exposures. The different measurement and analysis techniques we used during the second site visit did not clarify whether CO and CO_2 were truly present as our results continued to show a wide range of concentrations. The high CO concentrations measured with the Pac III direct reading instrument tended to agree more with the vacutainer air samples analyzed with GC-TCD/FID. These results, however, conflicted with the low CO concentrations detected on the colorimetric gas detection tubes and the vacutainer air samples analyzed with GC-MS by the OSHA Salt Lake Technical Center.

An additional challenge in interpreting our direct reading CO results is that many of the chemical-specific sensors can positively or negatively bias the readings in the presence of interfering gases or when measuring a gas mixture. For the Pac III, the manufacturer indicated that a CO sensor in a H_2-only environment can have up to a 40% positive bias, i.e., in an atmosphere containing only H_2 at 1,000 ppm, the instrument will indicate a CO concentration of up to 400 ppm [Walker 2011]. Similarly, the Biosystems ToxiUltra has a 40% positive bias, and BW Technologies reports a 20% positive bias for its CO sensors [Biosystems 2011; Heckathorn 2011]. Therefore, the presence of H_2 could have affected our direct reading CO results. In addition, the manufacturers were not aware of the sensors' biases when measuring gas mixtures, making it difficult to interpret the instrument readings.

The vast difference in the gas concentrations measured by different analytical and measurement techniques makes it difficult to have confidence in the results, especially the high CO concentrations. In addition, employees working near the process and conducting tasks such as removing cartridge tank filters reported no health symptoms such as headaches and nausea that might indicate high CO exposures. However, there is not sufficient evidence to suggest the CO concentrations found should be disregarded.

Because H_2 was present inside the covered drums, the true PBZ CO concentrations would be hard to determine using the direct reading instruments. However, our air sampling results indicate that

DISCUSSION (CONTINUED)

a possible hazard from CO exposure might exist; this can be easily mitigated by keeping the drums uncovered to prevent the buildup of gases in the drums.

The company has installed a new horizontal belt vacuum filtration system that uses a paper filtration process. The new process generates wet silicon mud, which is stored in an open 55-gallon plastic storage drum. This process change has obviated the use of polypropylene filters, and open drum storage prevents the buildup of gases. Although we have not evaluated the new process, the company continues to use the colorimetric detector to monitor the drum for hydride gases.

CONCLUSIONS

On the basis of a scientific literature review and air sampling results, we conclude that the covered drums were pressurized primarily because of H_2 formation and that silane was also formed. We could not confirm that the apparent high CO concentrations inside covered drums were accurate because of conflicting results obtained with different measurement and analytical methods. Regardless, disposing of used filters from the silicon wafer grinding filtration process in covered drums may pose a health and safety hazard to employees. This hazard can be minimized by leaving the drums uncovered, and/or by a process modification, as is now done by this company.

RECOMMENDATIONS

On the basis of our findings, we recommend the actions listed below to create a more healthful workplace. We encourage the company to use a labor-management health and safety committee or working group to discuss the recommendations in this report and develop an action plan. Those involved in the work can best set priorities and assess the feasibility of our recommendations for the specific situation at your workplace. Our recommendations are based on the hierarchy of controls approach (refer to Appendix B: Occupational Exposure Limits and Health Effects). This approach groups actions by their likely effectiveness in reducing or removing hazards. In most cases, the preferred approach is to eliminate hazardous materials or processes and install engineering controls to reduce exposure or shield employees. Until such controls are in place, or if they are not effective or feasible, administrative measures and/or PPE may be needed.

Administrative Controls

Administrative controls are management-dictated work practices and policies to reduce or prevent exposures to workplace hazards. The effectiveness of administrative changes in work practices for controlling workplace hazards is dependent on management commitment and employee acceptance. Regular monitoring and reinforcement is necessary to ensure that control policies and procedures are not circumvented in the name of convenience or production.

1. Leave the drums uncovered to prevent the buildup of gases.
2. Post signs near the work area to remind employees not to cover the drums.
3. Instruct employees who experience symptoms like headache, nausea, or confusion to promptly report them to the on-site healthcare professional.

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Carbon Monoxide, Carbon Dioxide, and Hydrogen in Air

NIOSH investigators measured CO air concentrations with ToxiUltra Atmospheric Monitors (Biosystems, Inc., Middletown, Connecticut), a BW Technologies GasAlert Extreme single gas detector (Honeywell Analytics, Calgary, Alberta), and a Dräger Pac III single gas monitor (Dräger Safety Inc., Pittsburgh, Pennsylvania). All these instruments were fitted with manufacturer recommended CO sensors. All the direct reading instruments were zeroed and calibrated before each use according to the manufacturer's recommendations. These monitors are direct-reading instruments with data logging capabilities. The instruments were operated in the passive diffusion mode, with a 1-minute sampling interval. The ToxiUltra has a nominal range of 0 to 500 ppm with the highest instantaneous reading of 1,000 ppm, BW Technologies has a nominal range of 0 to 1,000 ppm, and Dräger Pac III has a nominal range of 0 to 10,000 ppm.

Vacutainer® (BD, Franklin Lakes, New Jersey) air samples were analyzed by two laboratories. Bureau Veritas North America, the NIOSH contract laboratory, analyzed a set of vacutainer air samples using GC-TCD/FID. A set of duplicate air samples was sent to the OSHA Salt Lake Technical Center where they were analyzed by GC-MS. The analytical LOD and LOQ for the three gases analyzed by GC-TCD/FID are presented in Table A1.

Table A1. Analytical method LOD and LOQ for vacutainer air sample analysis

Site visit date	Analytical method GC-TCD/FID	H_2 (ppm)	CO (ppm)	CO_2 (ppm)
Feb 2008	LOD	5,000	100	1,000
	LOQ	17,000	330	3,300
Mar 2009	LOD	10,000	100	1,000
	LOQ	33,000	330	3,300

CO concentrations were also measured using Dräger Tubes®, colorimetric detection tubes, model 8/a (range of 8 to 150 ppm) (Dräger Safety Inc, Pittsburgh, Pennsylvania). Tube 8/a is suitable for measuring CO concentrations when H2 gas is also expected to be present. Accuro® pumps were used for drawing air through the colorimetric tubes.

APPENDIX B: OCCUPATIONAL EXPOSURE LIMITS & HEALTH EFFECTS

In evaluating the hazards posed by workplace exposures, NIOSH investigators use both mandatory (legally enforceable) and recommended OELs for chemical, physical, and biological agents as a guide for making recommendations. OELs have been developed by federal agencies and safety and health organizations to prevent the occurrence of adverse health effects from workplace exposures. Generally, OELs suggest levels of exposure that most employees may be exposed to for up to 10 hours per day, 40 hours per week, for a working lifetime, without experiencing adverse health effects. However, not all employees will be protected from adverse health effects even if their exposures are maintained below these levels. A small percentage may experience adverse health effects because of individual susceptibility, a preexisting medical condition, and/or a hypersensitivity (allergy). In addition, some hazardous substances may act in combination with other workplace exposures, the general environment, or with medications or personal habits of the employee to produce adverse health effects even if the occupational exposures are controlled at the level set by the exposure limit. Also, some substances can be absorbed by direct contact with the skin and mucous membranes in addition to being inhaled, which contributes to the individual's overall exposure.

Most OELs are expressed as a TWA exposure. A TWA refers to the average exposure during a normal 8- to 10-hour workday. Some chemical substances and physical agents have recommended STEL or ceiling values where adverse health effects are caused by exposures over a short period. Unless otherwise noted, the STEL is a 15-minute TWA exposure that should not be exceeded at any time during a workday, and the ceiling limit is an exposure that should not be exceeded at any time.

In the United States, OELs have been established by federal agencies, professional organizations, state and local governments, and other entities. Some OELs are legally enforceable limits, while others are recommendations. The U.S. Department of Labor OSHA PELs (29 CFR 1910 [general industry]; 29 CFR 1926 [construction industry]; and 29 CFR 1917 [maritime industry]) are legal limits enforceable in workplaces covered under the Occupational Safety and Health Act of 1970. NIOSH RELs are recommendations based on a critical review of the scientific and technical information available on a given hazard and the adequacy of methods to identify and control the hazard. NIOSH RELs can be found in the NIOSH Pocket Guide to Chemical Hazards [NIOSH 2010]. NIOSH also recommends different types of risk management practices (e.g., engineering controls, safe work practices, employee education/training, personal protective equipment, and exposure and medical monitoring) to minimize the risk of exposure and adverse health effects from these hazards. Other OELs that are commonly used and cited in the United States include the TLVs recommended by ACGIH, a professional organization, and the WEELs recommended by the American Industrial Hygiene Association, another professional organization. The TLVs and WEELs are developed by committee members of these associations from a review of the published, peer-reviewed literature. They are not consensus standards. ACGIH TLVs are considered voluntary exposure guidelines for use by industrial hygienists and others trained in this discipline "to assist in the control of health hazards" [ACGIH 2011]. WEELs have been established for some chemicals "when no other legal or authoritative limits exist" [AIHA 2011].

Outside the United States, OELs have been established by various agencies and organizations and include both legal and recommended limits. The Institut für Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung (IFA, Institute for Occupational Safety and Health of the German Social Accident

Insurance) maintains a database of international OELs from European Union member states, Canada (Québec), Japan, Switzerland, and the United States. The database, available at http://www.dguv.de/ifa/en/gestis/limit_values/index.jsp, contains international limits for over 1,500 hazardous substances and is updated periodically.

Employers should understand that not all hazardous chemicals have specific OSHA PELs, and for some agents the legally enforceable and recommended limits may not reflect current health-based information. However, an employer is still required by OSHA to protect its employees from hazards even in the absence of a specific OSHA PEL. OSHA requires an employer to furnish employees a place of employment free from recognized hazards that cause or are likely to cause death or serious physical harm [Occupational Safety and Health Act of 1970 (Public Law 91–596, sec. 5(a)(1))]. Thus, NIOSH investigators encourage employers to make use of other OELs when making risk assessments and risk management decisions to best protect the health of their employees. NIOSH investigators also encourage the use of the traditional hierarchy of controls approach to eliminate or minimize identified workplace hazards. This includes, in order of preference, the use of (1) substitution or elimination of the hazardous agent, (2) engineering controls (e.g., local exhaust ventilation, process enclosure, dilution ventilation), (3) administrative controls (e.g., limiting time of exposure, employee training, work practice changes, medical surveillance), and (4) personal protective equipment (e.g., respiratory protection, gloves, eye protection, hearing protection). Control banding, a qualitative risk assessment and risk management tool, is a complementary approach to protecting employee health that focuses resources on exposure controls by describing how a risk needs to be managed. Information on control banding is available at http://www.cdc.gov/niosh/topics/ctrlbanding/. This approach can be applied in situations where OELs have not been established or can be used to supplement the OELs, when available.

Below we provide the OELs and surface contamination limits for the compounds we measured, as well as a discussion of the potential health effects from exposure to these compounds.

Carbon Monoxide

CO is a colorless, odorless, tasteless gas produced by incomplete burning of carbon-containing materials such as gasoline or propane fuel. The initial symptoms of CO poisoning may include headache, dizziness, drowsiness, or nausea. Symptoms may advance to vomiting, loss of consciousness, and collapse if prolonged or high exposures are encountered. If the exposure level is high, loss of consciousness may occur without other symptoms. Coma or death may occur if high exposures continue [NIOSH 1972, 1977, 1979, 2010; Proctor et al. 1988; ACGIH 2011]. The display of symptoms varies widely from individual to individual, and may occur sooner in susceptible individuals such as young or aged people, people with preexisting lung or heart disease, or those living at high altitudes.

Exposure to CO limits the ability of the blood to carry oxygen to the tissues by binding with the hemoglobin to form carboxyhemoglobin. Once exposed, the body compensates for the reduced bloodborne oxygen by increasing cardiac output, thereby increasing blood flow to specific oxygendemanding organs such as the brain and heart. This ability may be limited by preexisting heart or lung diseases that inhibit increased cardiac output.

Occupational criteria for CO exposure are applicable to employees who may be at risk of CO poisoning. The NIOSH REL for CO is 35 ppm for full-shift TWA exposure, with a ceiling limit of 200 ppm that should never be exceeded [NIOSH 1992]. The NIOSH REL of 35 ppm is designed to protect workers from health effects associated with carboxyhemoglobin levels in excess of 5% [NIOSH 1972]. NIOSH has established the IDLH value for CO of 1200 ppm [NIOSH 2000]. An IDLH value is defined as a concentration at which an immediate or delayed threat to life exists or that would interfere with an individual's ability to escape unaided from a space.

The ACGIH recommends an 8-hour TWA TLV of 25 ppm on the basis of limiting shifts in carboxyhemoglobin levels to less than 3.5%, thus minimizing adverse neurobehavioral changes such as headache, dizziness, etc., and to maintain cardiovascular exercise capacity [ACGIH 2011]. ACGIH also recommends that exposures never exceed five times the TLV (thus, never to exceed 125 ppm) [ACGIH 2011]. The OSHA PEL for CO is 50 ppm for an 8-hour TWA exposure [29 CFR 1910.1000].

Carbon Dioxide

Carbon dioxide is a clear and odorless gas and is a simple asphyxiant [LaDou 2004]. It is a product of respiratory metabolism and is not considered a building air pollutant. The NIOSH REL and ACGIH TLV for CO_2 is 5,000 ppm as a full-shift TWA exposure and a STEL of 30,000 ppm [NIOSH 2010; ACGIH 2011]. The OSHA PEL for CO_2 is 5,000 ppm for an 8-hour TWA exposure [29 CFR 1901.1000].

Hydrogen

Hydrogen gas is the lightest gas and is colorless, odorless, and tasteless. Hydrogen gas is highly combustible and will burn at concentrations ranging from 4% to 75% with the lower explosive limit being 4%. Because of its explosive nature it poses a safety hazard, and at high concentrations it can also act as a simple asphyxiant [LaDou 2004]. Requirements governing safe storage and delivery of hydrogen are covered under the OSHA standard 29 CFR 1910.103.

Silane

Silane or SiH_4 is a colorless gas with a strong odor at room temperature that acts as a respiratory irritant. It is a pyrophoric gas, i.e., it spontaneously combusts in air. Concentrations greater than 3% silane in nitrogen or greater than 0.5% in hydrogen may ignite spontaneously in contact with ambient air [ACGIH 1989]. When released from a cylinder silane may generate enough static electrical charge to initiate a flame. Silane is a very hazardous gas with high potential to cause damage to life and property.

The NIOSH REL for silane is 5 ppm as a full-shift TWA exposure [NIOSH 2010]. The ACGIH also recommends an 8-hour TWA TLV of 5 ppm and an excursion limit of 25 ppm [ACGIH 2011].

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APPENDIX C: TABLES

Table C1. Vacutainer air sampling results (February 7, 2008)

Location (Date filters were removed)	Time of sample collection (military)	Air concentration (ppm)		
		H_2	CO	CO_2
Cartridge tank 1	09:33	64,500	22,400	17,000
	09:37	32,300	8,600	5,600
Cartridge tank 2	09:49	(12,000)*	3,500	3,400
Drum (1/18/08)	10:14	26,200	7,500	5,900
	10:15	ND	2,200	(2,900)*
Basement	—	ND	ND	ND
	—	ND	ND	ND
Office (Building 1, 2nd floor)	14:28	ND	ND	ND
	14:28	ND	ND	ND

ND = Not detected; the concentration is below the LOD.

*Concentrations between LOD and LOQ are listed in the table in parentheses to acknowledge that there is more uncertainty surrounding concentrations below the LOQ.

Table C2. Drum vacutainer air sampling results (February 7, 2008)

Location (Date filters were removed)	Time elapsed after covering the drum (hh:mm:ss)	Drum interior temperature (ºF)	Air concentration (ppm)		
			H_2	CO	CO_2
Drum (2/7/08)	0:06:00	74.9	ND	1,100	ND
	0:11:00	74.1	53,800	19,900	16,700
	0:21:00	74.0	(5,400)*	ND	ND
	0:31:00	74.1	(14,000)*	ND	ND
	0:31:30	74.1	(14,000)*	ND	ND
	0:41:20	74.5	17,300	ND	ND
	1:01:30	74.6	27,100	ND	ND
	2:00:10	76.6	74,700	ND	ND
	2:01:18	76.6	32,300	8,500	9,600
	2:48:45	77.3	48,900	2,000	(3,000)*
	2:49:10	77.4	36,100	ND	ND
	3:46:30	77.8	ND	ND	ND
	3:46:50	77.9	84,700	ND	ND

ND = Not detected; the concentration is below the LOD.

*Concentrations between LOD and LOQ are listed in the table in parentheses to acknowledge that there is more uncertainty surrounding concentrations below the LOQ.

APPENDIX C: TABLES (CONTINUED)

Table C3. Vacutainer air sampling results (March 18, 2009)

Location (Date filters were removed)	Time of sample collection (military)	Air concentration (ppm)		
		H_2	CO	CO_2
Bag filter tank 1C	07:17	ND	15,600	8,930
Cartridge tank 1A	07:38	ND	3,140	(1,240)*
Cartridge tank 1B	07:53	ND	15,800	ND
Bag filter tank 1A	08:09	ND	3,270	(2,070)*
Bag filter tank 1B	08:16	ND	(303)*	ND
Drum (3/12/09)	09:32	(32,100)*	19,400	15,000
	09:33	(27,900)*	11,000	6,790
	09:33	34,000	5,280	3,750
Basement	15:03	46,400	1,410	(1,700)*
	15:03	ND	(128)*	ND

ND = Not detected; the concentration is below the LOD.

*Concentrations between LOD and LOQ are listed in the table in parentheses to acknowledge that there is more uncertainty surrounding concentrations below the LOQ.

Table C4. Drum vacutainer air sampling results (March 18, 2009)

Location (Date filters were removed)	Time elapsed after covering the drum (hh:mm)	Drum interior temperature (ºF)	Air concentration (ppm)		
			H_2	CO	CO_2
Drum (3/18/09)	0:12	66.7	(13,800)*	1,880	(2,310)*
	0:17	66.7	ND	792	(1,040)*
	0:27	66.9	(10,700)*	(229)*	ND
	0:49	68.0	(17,700)*	ND	ND
	0:59	68.5	(10,900)*	7,650	6,860
	1:32	70.6	(25,900)*	2,610	ND
	1:32	70.7	34,400	1,320	(1,740)*
	2:13	73.5	39,400	(192)*	(1,070)*
	2:46	75.6	58,500	ND	ND
	3:16	77.3	70,700	ND	ND
	3:16	77.3	79,600	ND	ND
	3:47	78.8	54,100	21,600	18,800
	4:20	79.9	88,200	10,900	9,710
	4:20	79.9	103,000	5,670	4,310
	5:00	81.2	118,000	2,030	7,890

ND = Not detected; the concentration is below the LOD.

*Concentrations between LOD and LOQ are listed in the table in parentheses to acknowledge that there is more uncertainty surrounding concentrations below the LOQ.

Table C5. Vacutainer air sampling results (March 19, 2009)

Location (Date filters were removed)	Time of sample collection (military)	Air concentration (ppm)		
		H_2	CO	CO_2
Drum (3/12/09)	07:44	(15,500)*	ND	ND
	07:44	(13,900)*	21,400	18,600
	07:44	(24,400)*	6,780	4,900
	07:44	(25,400)*	4,620	3,430
Drum (3/18/09)	07:46	138,000	2,900	(3,120)*
	07:46	150,000	1,050	ND
	07:46	153,000	643	ND

ND = Not detected; the concentration is below the LOD.

*Concentrations between LOD and LOQ are listed in the table in parentheses to acknowledge that there is more uncertainty surrounding concentrations below the LOQ.

Table C6. PBZ air sampling results (March 18–19, 2009)

Date	Job title	Instrument	Sampling time (minutes)	Air concentration (ppm) Mean (Min–Max)		
				H_2	CO	CO_2
3/18	Maintenance technician	Toxi Ultra	380	—	1.94 (0–27.3)	—
		BW	380	—	4.50 (0–29.0)	—
		Pac-III	380	—	0.078 (0–25.0)	—
3/19	Industrial hygienist	Toxi Ultra	18	—	8.70 (2.1–230)	—
		BW	18	—	34.2 (3–140)	—
		Pac-III	18	—	19.0 (0–375)	—
3/19	Industrial hygienist opening drum containing filters removed on 3/12/09	Vacutainer	Grab	57,800	(228)*	ND
3/19	Industrial hygienist opening drum containing filters removed on 3/18/09	Vacutainer	Grab	(29,800)*	ND	ND
NIOSH Ceiling/STEL					200	30,000
ACGIH STEL					125†	30,000

ND = Not detected; the concentration is below the LOD.

*Concentrations between LOD and LOQ are listed in the table in parentheses to acknowledge that there is more uncertainty surrounding concentrations below the LOQ.

† ACGIH excursion limit = 5 x ACGIH TLV of 25 ppm

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