Incident Report

Chemistry 332

Date of Incident October 14, 2011

Report Prepared by

Jared Martin – Laboratory Safety Manager

Texas Tech University Environmental Health & Safety

Introduction

On October 14, 2011 there was an explosion involving catastrophic failure of a sealed waste acid glass storage bottle where excess gas pressure had built up in the bottle. The explosion did not involve a detonation or ignition of combustible gases. The explosion occurred in a Chemistry Building laboratory. The laboratory in question is room number 332. The Lubbock Fire Department (LFD) was notified by the Texas Tech University Police Department (UPD) and the LFD HAZMAT team responded. The chemicals in the laboratory that had spilled were contained and removed.

Chain of Events When Texas Tech University Environmental Health & Safety (EH&S) was notified

EH&S was notified about an explosion in 332 Chemistry at approximately 5:30pm on October 14, 2011, when Emergency Maintenance contacted the EH&S on call staff member (Mike Toombs). Mr. Toombs notified his manager (Paul Cotter) who then contacted the EH&S Laboratory Safety Manager (Jared Martin) at approximately 5:43pm. Mr. Cotter then contacted the EH&S Environmental Protection Manager (Matt Roe), EH&S Waste Specialist (Richard Whitehead) and EH&S Director (Randy Nix). Mr. Toombs then proceeded to the Chemistry Building where he met up with Texas Tech University Fire Marshal (Walter James) at approximately 5:40 pm. Dr. Mayank Shashtri and Mr. Kishor Girase, who work in the laboratory, were outside of the Chemistry Building at approximately 5:40pm. These two individuals are employees of Eburon Organics International, a Lubbock-based organic chemical synthesis company. They are not TTU employees or students. Dr. Eighmy and Dr. Korzeniewski arrived at the Chemistry Building at approximately 5:50pm. Mr. Nix arrived at the Chemistry Building at approximately 5:55pm. Mr. Martin, Mr. Whitehead and the EH&S Laboratory Safety Specialist (Brandon Mount) arrived at the Chemistry Building at approximately 6:20pm. Prior to the arrival of Mr. Martin, the LFD HAZMAT team had entered the building to assess and contain any spilled chemicals or hazards immediately present in room 332. Mr. Martin spoke with Dr. Shashtri and Mr. Girase to try and determine what chemical may be present inside of the fume hood where the incident took place. Dr. Shashtri and Mr. Girase stated that there was Methyl glutaric acid being crystalized in the rotoevaporator, a small plastic bottle of Methanol, Nitric acid waste at approximately 25-30% concentration in an amber glass bottle, Ethyl acetate/Acetone waste in a 20 liter metal can, and Acetic/Hydrochloric acid waste in an amber glass bottle inside of the fume hood. Mr. Martin assisted the LFD HAZMAT command on gathering information on the hazards with these agents. At approximately 7:00pm, the LFD HAZMAT team exited the building and gave clearance to EH&S personnel to enter to assess the extent of the incident. At approximately 7:30pm, EH&S gave clearance to the UPD that the parts of the building not affected by this incident could be reoccupied. Dr. Eighmy, following consultation with the Chemistry Department Chair, ordered the locks to Rooms 332 be changed to facilitate the investigation. At approximately 8:30 pm, the Texas Tech University Lock Shop

and Physical Plant sent their on call personnel to assist with getting the fume hood operational and changing the locks on both the doors to room 332. The fume hood at this time was inoperable and the locks were changed and EH&S took possession of room 332. The following day, Dr. Eighmy and Dr. Korzeniewski inspected the labs with Randy Nix. Dr. Eighmy, following consultation with Dr. Korzeniewski, ordered the locks to rooms 339 and 008 to be changed. October 15, 2011, at approximately 2:30pm, EH&S had the lock changed on room 339 of the Chemistry Building and the back door to the laboratory locked as well and EH&S took possession of this room. Also, room 008 of the Chemistry Building EH&S had the lock changed and EH&S took possession of this room on October 15, 2011, at approximately 2:30pm. Room 339 was locked due to the fact that the individuals who were working in 332 also conducted operations in this room. Room 008 was locked due to the Principal Investigator (PI) who was in charge of 332 and 339 having chemicals stored in this room from rooms 332 and 339. Additionally, room 008 is not authorized to store chemicals and the PI had been repeatedly instructed to not store chemicals there.

Findings From Investigation

Inside of the fume hood on northeast side of the east wall of 332 is where the incident took place. The bottles in question were stored on the north side of the fume hood. The sash of the hood was not closed and appeared to be between ¾ to fully open. Clear and amber glass shrapnel was located throughout the laboratory as far as approximately 56 feet from the fume hood where the incident took place. The pattern of glass dispersal is consistent with the accounts of a sealed bottle rupturing from over pressurization inside of the fume hood. The sash of the hood had a couple of strike marks on the lower half where debris had struck the inside of the sash and cracked it. The countertop of the fume hood had two cracks that ran the full depth of the hood going east to west. The seam on the countertop was also disjointed. EH&S was informed that inside of the fume hood there was a 20 liter metal can of solvent waste with (Ethyl Acetate, Acetone, Methanol, Hexane, Toluene and Ethanol), a 4 liter amber glass bottle with (Nitric acid waste that was approximately 25-30% concentrate), a 4 liter amber glass bottle with (Acetic acid waste, Hydrochloric acid waste and possibly Trichlortriacetic acetate), a 500 ml bottle with (Methanol) and a reaction flask on a rotoevaporator with (Methyl glutaric acid). However, during the reconstruction of items collected from the LFD HAZMAT team there was a 20 liter metal can of solvent waste with (Ethyl Acetate, Acetone, Methanol, Hexane, Toluene, Ethanol, Chloroform and Dichloromethane) on the waste label, a 500 ml broken bottle of (Epichlorohydrin 99%), a broken clear glass round bottom flask approximately 200 ml with no markings or identifiable contents, a broken 500 ml plastic beaker with no markings or identifiable contents, a broken 4 liter amber glass bottle (originally containing Chloroform) with a waste label marked as (Hydrochloric acid, an unidentifiable acid and 2 other chemicals that the names were unidentifiable) and a broken 2.5 liter bottle (originally containing Hexane) with a waste label marked as (Nitric acid) and a 500 ml plastic bottle marked as (Methanol). The 2.5 liter bottle that contained the Nitric acid appeared to be the bottle with the more severe damage

and is the most likely suspect as the origin of the incident. All the caps were recovered with the neck of the bottles and the caps appeared to be tightly capped.

Actions Taken By Laboratory Workers Preceding The Incident

The reaction that they were conducting had occurred two times prior to this accident on May 19, 2009 and August 11, 2010. The reaction that was taking place during this incident was started on October 12, 2011. The reaction was conducted in the hoods on the west wall of 332 and the waste was transferred to the hood on the east wall. Once the reaction was completed, the product was placed on the rotoevaporator in the east hood. Inside the east hood there were two rotoevaporators, the one on the south side was being used and the one in the middle of the hood was not being used. During this reaction, they were at the end of the project where they were conducting recrystallization. The recrystallization process was started on October 14, 2011, and at approximately 4:30pm, the rotoevaporator was lifted out of the water bath to recrystallize. Both Dr. Shashtri and Mr. Girase left the laboratory with Dr. Shashtri going to lunch and Mr. Girase going to room 339. The incident occurred at approximately 5:30pm. The process taking place in the hood at the time was not involved in the incident.

Response From Occupants

Dr. Shaw was the first to find the incident. Dr. Shaw's office is directly west of the laboratory and while sitting in his office he heard what appeared to be a detonation. He went to investigate and noticed that the east door of 332 was cracked open and when he looked in he saw debris and liquid on the floor. Dr. Shaw called UPD who, upon arriving on scene, checked for anyone in the laboratory and then activated the fire alarm. When the alarm activated, the building was evacuated and the LFD was dispatched to the Chemistry Building.

Probable Cause of the Incident

The Nitric acid waste storage bottle appeared to be the main suspect for the incident. The probable cause was the mixture of an organic solvent accidently being placed into the Nitric acid waste bottle or the bottle not being totally empty when Nitric acid waste was added. The last time that waste was added to the Nitric acid waste was on October 12, 2011. Since that time there more than likely was a pressure buildup in the bottle until it had a catastrophic failure. The area where the incident happened did not have any signs of fire or burn marks, indicating an over pressurization explosion and not an ignition explosion.

In parallel to EH&S investigation a forensics consultant, Rimkus Consulting Group, was hired to assist in the determination of the cause of the explosion. Their report on this accident is appended to this report.

Basically, the consultant determined that:

1. The explosion was most likely caused by an oxidation reaction between nitric acid and ethanol that generated gases.
2. These lab wastes were inadvertently mixed in a glass lab waste bottle that was being stored in the fume hood.
3. The top of the lab waste storage bottle was inadvertently tightly capped, resulting in over pressurization and catastrophic failure of the bottle.
4. The waste bottles were not externally polymer coated to minimize glass fragmentation in the event they were over pressurized or dropped.
5. The waste bottles did not have venting caps that would have mitigated over pressurization.
6. The waste bottles and drum were not color coded or labeled to indicate the type of waste present in each container.
7. The position of the sash of the waste storage hood was approximately 75 percent open, not in its lowest position. The nearly-open sash provided an unobstructed path for glass shards when the waste bottle shattered. It also reduced the velocity of the incoming air, limiting the containment of fumes.

Corrective Measures Recommended To Prevent Similar Events

- Laboratorians need to receive specific training from PIs on how to handle the chemicals and wastes present in the laboratories.
- Written waste handling SOP's need to be generated by the lab to explain how that laboratory specifically handles their waste.
- Waste needs to be segregated by its hazards and its incompatibilities.
- Waste containers need to be disposed of within one month or when they are ¾ full, whichever comes first.
- Waste needs to be properly labeled with orange EH&S labels and legible.
- Smaller containers need to be used when waste generation is slow.
- Bottles used for waste need to be cleaned to remove any residues from the original chemicals in that container.
- If an acid or base chemical bottle is to be used for waste it needs to only have waste of that acid or base added to them.
- Fume hood sashes need to be completely closed when there is no one actively working in the fume hood and the hood must be on.

Results of Resurvey

Room 332

During the resurvey 12 findings were observed, including the chairs were not of a non-fabric material which has been a topic in the department as a whole. There were no designated work areas for work with carcinogenic compounds, however there was no indication that they were being actively worked with at that time. The fume hoods had excess storage of chemicals that were not being work with. Items in the hood were not 6 inches away from the front edge. The fume hood sashes were not completely closed. Not all the chemicals were segregated. Labels were not on all the secondary containers. Not all the flammables were stored in the flammables storage cabinets or flammables refrigerators. A peroxide-forming compound was found with no receipt date, open date, or expiration date. It is important to note that Room 332 had been inspected on September 13, 2011 and at that time no deficiencies were observed. Finally, the two Eburon employees were current with their EH&S required training.

Room 339

During the resurvey 5 findings were observed, including the fume hood sash was not completely closed. Not all the chemicals were segregated. Labels were not on all the secondary containers and not all the flammables were stored in the flammables storage cabinets

The previous issues in these laboratories found in June of 2011 had been addressed on September 13, 2011. The majority of the findings in the resurvey found are day to day chemical handling issues. These types of issues need to be addressed by PIs through proper training of their laboratory staff on how to handle and store chemicals and operate a fume hood.

Room 008

This room was used by Dr. Bartsch to store products that were generated by his graduate students. Prior to the incident the room housed a fairly large collection of sample vials that had been removed by Dr. Bartsch. However there were still 10 small boxes with sample vials with generated products contained in them. The products had a cataloging system but only referred to the structure of the compound and not the name of the compound. Also the containers had no identification as to what hazards the compounds presented, if any. These compounds were removed from room 008 and were stored in a room for chemical storage until the containers can be identified and labeled properly.

Images From Incident

Image 1. Area where incident occurred. Prominent crack in fume hood countertop.

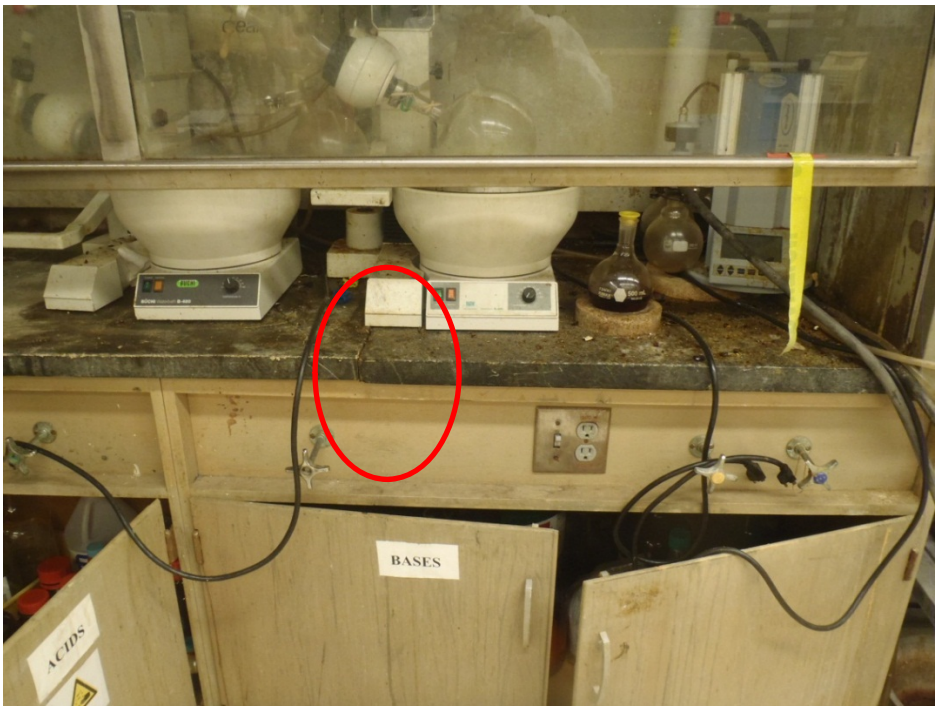


Image 2. East fume hood where rotoevaporators were located. Countertop union is disjointed.

Image 3. Floor of laboratory approximately 56 feet from incident. The red marks are where amber glass shards were located.

Image 4. Looking east towards the fume hood where incident occurred approximately 56 feet away.

Image 5. Work area looking south. Tape marks indicate where glass shards were located.

Image 6. Work station on north wall next to fume hood. Tape marks indicate where glass shards were located.

Image 7. Work area looking south. Tape marks indicate where glass shards were located.

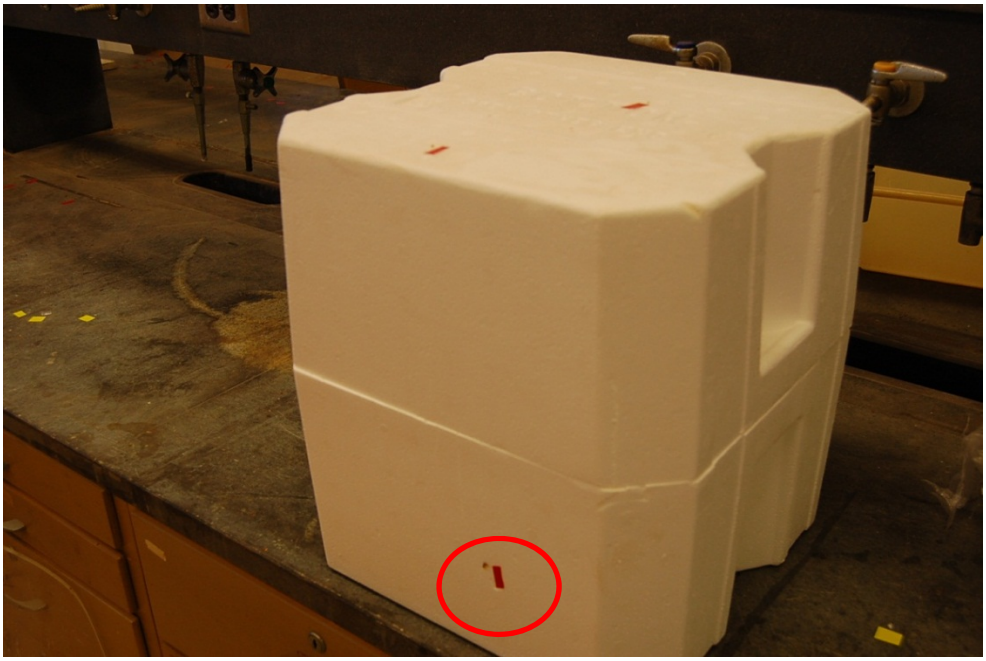


Image 8. Styrofoam box located directly in front of fume hood. Tape marks where there was imbedded glass shards.

Image 9. Strike mark on fume hood sash where it is cracked.

Image 10. Strike mark on fume hood sash where it is cracked.

cc: Dr. Eighmy, Dr. Young, Dr. Korzeniewski, Dr. Mayer, Dr. Zak, Dr. Schovanec, Mr. Ron Phillips

Lab Safety/Incident Reports/Room332CHEM_110911

Rimkus Consulting Group, Inc.
Eight Greenway Plaza, Suite 500
Houston, TX 77046
(800) 580-3228 Telephone
(713) 623-4357 Facsimile
Certificate of Authorization No. F-1545
Certification Expiration Date September 30, 2012

November 23, 2011

Dr. Taylor Eighmy
Vice President for Research
Texas Tech University
169 Administration Building
Lubbock, Texas 79409

Re: Subject: **Report of Findings, Incident of October 14, 2011**
RCG File No.: 11004383

Dear Dr. Eighmy:

On October 14, 2011, an explosion occurred in room 332 of the Chemistry Building at Texas Tech University. The explosion resulted in damage to the hood in the laboratory. Fortunately, no one was injured in the incident.

Rimkus Consulting Group, Inc. (Rimkus) was retained by Texas Tech University on October 17, 2011, to determine the cause of the explosion. In the course of our work, we reviewed the materials and performed the tasks described in the attached **Basis of Report**.

Conclusions

1. The explosion was most likely caused by an oxidation reaction between nitric acid and ethanol that generated gases.

2. These lab wastes were inadvertently mixed in a glass lab waste bottle that was being stored in the fume hood.

3. The top of the lab waste storage bottle was inadvertently tightly capped, resulting in overpressurization and catastrophic failure of the bottle.

4. The waste bottles were not externally polymer coated to minimize glass fragmentation in the event they were overpressured or dropped.

5. The waste bottles did not have venting caps that would have mitigated overpressurization.

6. The waste bottles and drum were not color code labeled to indicate the type of waste present in each container.

7. The position of the sash of the waste storage hood was approximately 75 percent open, not in its lowest position. The nearly-open sash provided an unobstructed path for glass shards when the waste bottle shattered. It also reduced the velocity of incoming air, limiting the containment of fumes.

Discussion

During the week of October 10, 2011, two employees of Eburon Organics were working in room 332 of the Chemistry Building at Texas Tech University to synthesize 3-hydroxy-3-methyl-glutaric acid from 3-methyl-1,3,5-pentanetriol per U.S. Patent #4,966,993. The senior researcher was Dr. Mayank Shashtri and his assistant was Kishore Girase. Dr. Shashtri is a chemist with several decades of industrial and academic experience.

The synthesis reaction had been carried out mid-week at the west end of the lab room in a fume hood. The crystallization of the product and storage of lab wastes was being conducted in a fume hood at the east end of the lab room. There were four containers being stored in the fume hood, a 4-liter amber bottle for 20 percent to 25 percent aqueous nitric acid waste, a 20-liter metal drum containing organic wastes including acetone, ethyl acetate, chloroform, hexane, toluene, methanol, ethanol, and dichloromethane, a 2.5-liter bottle of ethanol and ethyl acetate waste, and an ethanol wash bottle.

None of the waste containers were completely full. The last time that wastes were added to the bottles was the evening of Wednesday, October 12, 2011.

On the evening of Friday, October 14, 2011, at approximately 5:30 p.m. a loud explosion was heard by graduate students and faculty working in rooms adjacent to room 332. Upon investigation a faculty member, a chemistry professor, found the door to room 332 ajar and a very strong smell of acetic acid. Campus police were summoned, the room was searched for any potential victims, and the building was evacuated. The fire department also responded to the incident. University Environmental Health and Safety staff responded to the incident and performed an initial site assessment. A hazardous materials crew was brought in to clear the lab of any potentially dangerous materials. No one was injured in the incident, because the lab was unoccupied at the time of the explosion.

An investigation by this author did not reveal any visual evidence that this incident involved any explosive compounds or combustion-initiated explosions. A possible chemical hazards reaction matrix of the known chemicals in the hood indicates that the most likely cause of the explosion was a gas-evolving oxidation reaction of ethanol with nitric acid. The evolving gas led to an increase in the pressure of a tightly capped bottle.

From the literature, this reaction, under similar time scales and circumstances, has been documented numerous times in the past. Nitric acid maintains its ability to oxidize alcohols, even when diluted to the concentrations present in this incident and at ambient temperature. The oxidation is exothermic (releases heat), which would have increased the temperature of the contents of the bottle and contributed to overpressurization.

The sash of the hood was approximately 75 percent open (see **Photographs**). This allowed numerous shards of glass (measuring approximately 1 inch) to be projected about 50 feet to the opposite end of the lab. The force of the explosion was sufficient to fracture the 2-inch thick, stone base of the hood. The three glass bottles in the hood did not survive the explosion. The drum of waste organic compounds was dented but maintained its integrity.

Other alcohols from the synthesis or known to be present in the hood may have also been oxidized in the incident including methanol, 3-hydroxy-3-methyl-glutaric acid, and 3-methyl-1,3,5-pentanetriol. Further, the author cannot rule out the possibility that some of the more than 900 chemicals being stored in room 332 were involved in the explosion, if they were used in the research being performed in the lab in the weeks prior to the incident.

This report was prepared for the exclusive use of Texas Tech University, and is not intended for any other purpose. Our report is based on the information available to us at this time, as described in the **Basis of Report.** Should additional information become available, we reserve the right to determine the impact, if any, the new information may have on our opinions and conclusions and to revise our opinions and conclusions if necessary and warranted.

Photographs taken during our work are retained in our files and are available to you upon request.

Thank you for allowing us to provide these services. If you have any questions or need additional assistance, please call.

THE ORIGINAL OF THIS REPORT, SIGNED AND SEALED BY THE PROFESSIONAL WHOSE NAME APPEARS ON THIS PAGE, IS RETAINED IN THE FILES OF RIMKUS CONSULTING GROUP, INC.

Sincerely,
RIMKUS CONSULTING GROUP, INC.

Robert Scates/mmk

Robert M. Scates, Ph.D.
Principal Consultant

Harmon Kirkpatrick

Harmon L. Kirkpatrick, P.E.
Vice President

/mmk

Attachments: Basis of Report, Photographs, CVs

November 23, 2011
RCG File No. 11004383

Basis of Report

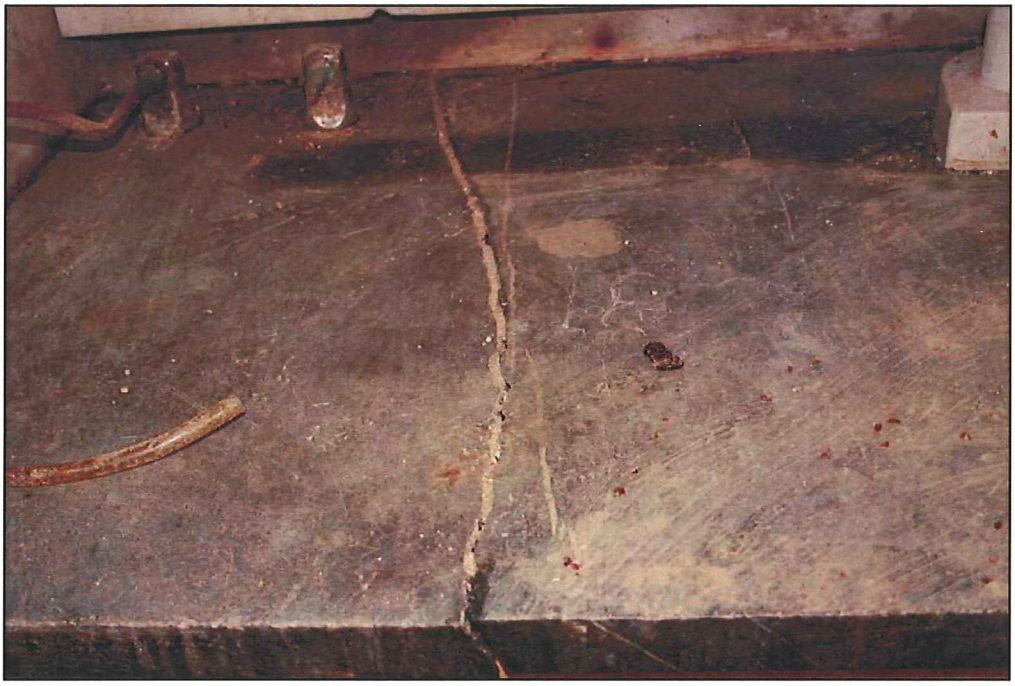
1. Inspection, interviews and photographs by the author, October 20, 2011.

2. Bretherick's Handbook of Reactive Chemical Hazards, 6th Edition, P.G. Urben, ed., 1999.

3. Prudent Practices in the Laboratory, Handling and Management of Chemical Hazards, National Research Council, 2011.

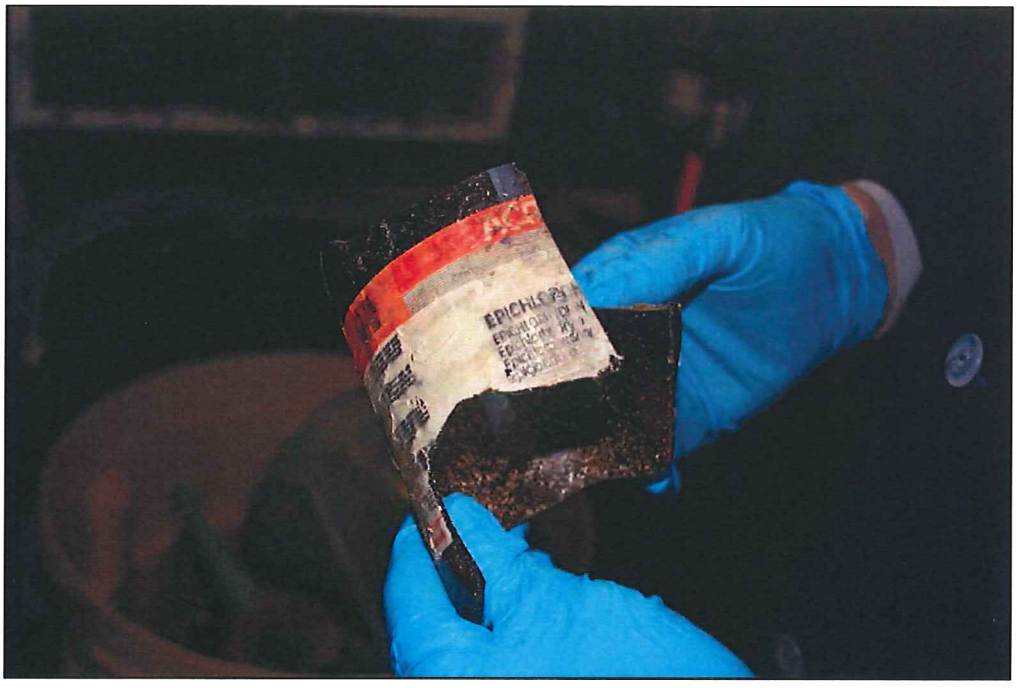
Photograph 1
Corner of Hood where Explosion Originated

Photograph 2
Cracked Stone Floor of Hood from Force of Explosion



Photograph 3
Partial Remains of Glass Waste Bottle

Photograph 4
Partial Remains of Glass Waste Bottle



Photograph 5
Remains of Waste Organic Solvent Drum

Photograph 6
Label from Organic Solvent Drum

Photograph 7
Line of Sight, From Explosion Origin to Far End of Lab

November 23, 2011
RCG File No. 11004383

CVs

RIMKUS
CONSULTING GROUP, INC.

ROBERT M. SCATES, Ph.D.
PRINCIPAL CONSULTANT

Dr. Scates is a 2003 graduate of the University of Houston, with a doctorate in Chemical Engineering. He has over 20 years experience in industrial and consulting assignments in the chemical, petrochemical, refining and nuclear industries. His background includes plant engineering and design, economics, construction, and operations, accident analysis and prevention, research and development, environmental evaluations, and environmental and industrial safety regulations.

Dr. Scates' areas of expertise include reaction engineering, particularly in catalyzed systems; reactor, distillation column, heat transfer, and separations equipment design; process technology assessment and selection; process economic analysis and equipment costing; project scheduling, construction, and start-up; process operations support including equipment assessment/improvement and debottlenecking, and process safety assessments for new and existing facilities including audits and accident root cause analysis. His experience includes research and process/product development in laboratory, pilot plant, and industrial-scale operations.

Dr. Scates has performed research, modeled, and reported on the fate-and-transport of chemicals in the environment including measuring and estimating emissions from chemical plants, predicting their fate-and-transport in the environment via air, surface water and groundwater pathways, and estimating their impacts on the surrounding community.

EDUCATION AND PROFESSIONAL ASSOCIATIONS

B.S. Chemical Engineering, Texas A&M University, College Station, Texas
Ph.D. Chemical Engineering, University of Houston, Houston, Texas

EMPLOYMENT HISTORY

2010 – Present	Rimkus Consulting Group, Inc.
1996 – 2010	Scates Engineering Consultants
1990 – 1996	Fina Oil and Chemical Company
1984 – 1990	E. I. du Pont de Nemours and Company, Inc.

RIMKUS
CONSULTING GROUP, INC.

HARMON LEE KIRKPATRICK, P.E.
VICE PRESIDENT – INDUSTRIAL/INTERNATIONAL DIVISION

Mr. Kirkpatrick is a 1967 graduate of the University of Cincinnati with experience in the refining, petrochemical, gas processing and chemical process industries. His background includes process and project engineering design; construction management; plant operations and technical service; economic analysis; scheduling; pipeline and process plant safety; and process development. He has worked over 40 years in consulting and industrial assignments.

Mr. Kirkpatrick's areas of expertise include project management for projects approaching $200 million; economic evaluation; process design; pipeline station design; scheduling and cost control; process technology evaluation and selection; compressors, gas turbines and other rotating equipment; mechanical design and equipment selection, and plant safety and capital cost estimation. Mr. Kirkpatrick has executed numerous design/installation assignments in chemical, petrochemical and refinery plants. He has also conducted detailed operations analyses and process safety audits for new and existing plant processing units. His experience includes cost estimation of manufacturing and environmental remediation facilities; cost control; scheduling; cause and origin of fires, explosions, plant fatalities, equipment failures, and environmental spills; contract negotiations and settlement; technology licensing; distillation design; and cogeneration evaluations. He can lead a team or work independently. He can clearly and effectively present complex technology and events to the nontechnical audience.

In addition to his technical expertise, Mr. Kirkpatrick has prepared expert reports, assisted in mediations, testified in domestic arbitrations, open court and international arbitrations. He has completed both domestic and international field assignments in Canada, Australia, Honduras, Venezuela, Mexico, Korea, Thailand, Singapore, Indonesia, Trinidad, Suriname, Brazil, Argentina, Chile, Colombia, Ecuador, Puerto Rico, Egypt, Germany, Holland, Italy, China, Santo Domingo, Turkey and The Philippines.

EDUCATION AND PROFESSIONAL ASSOCIATIONS

B.S. - Chemical Engineering - University of Cincinnati
M.S. - Chemical Engineering - University of Cincinnati
M.B.A. - Business Administration - Western New England College
Professional Engineer (PE) licensed in Texas, Ohio, Mississippi, Arkansas, Louisiana, Oklahoma, New Mexico, California, and Kentucky. Documents on file with NCEES.
Completed HAZWOPER 8-hour Refresher Course (29CFR 1910.120), 2009
Department of Transportation Hazardous Materials Training HM-126F (49CFR 172.704), 2009
OSHA 40-Hour HAZWOPER Training
American Institute of Chemical Engineers – Member
Institution of Chemical Engineers (U.K.) – Member
Project Management Institute – Member
Alpha Chi Sigma – Member

EMPLOYMENT HISTORY

1991 - Present	Rimkus Consulting Group, Inc.
1990 - 1991	PCI Engineers, Inc.
1982 - 1990	Enterprise Products Company
1967 - 1982	Monsanto Chemical Company