Incident Report

Nanotechnology 121

Date of Incident October 27, 2011

Report Prepared by

Jared Martin – Laboratory Safety Manager

Texas Tech University Environmental Health & Safety

Introduction

On October 27, 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 Nanotechnology Building laboratory. The laboratory in question is room number 121. 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 121 Nanotechnology Center at approximately 7:15pm on October 27, 2011 when Emergency Maintenance contacted EH&S personnel Paul Cotter. Mr. Cotter contacted the EH&S Laboratory Safety Manager (Jared Martin) at approximately 7:17pm. Mr. Cotter then contacted the EH&S Waste Specialist (Richard Whitehead), EH&S Director (Randy Nix) and EH&S Assistant Director (Cliff Harris). Mr. Whitehead and Mr. Harris then proceeded to Nanotechnology Center. Mr. Martin then contacted EH&S Laboratory Safety Specialists (Seray Elliott and Brandon Mount). Mr. Martin then proceeded to the Nanotechnology Center. Mr. Martin, Mrs. Elliott and Mr. Mount arrived on scene at the Nanotechnology Building at approximately 7:37pm where they met up with the UPD, LFD HAZMAT team, Mr. Harris and Mr. Whitehead who had already arrived and the LFD HAZMAT team was preparing to enter the building to do an assessment and start the hazardous material removal. Dr. Jordan Berg and Dr. Mark Holtz who run the Nanotechnology Center, Dr. Sergey Nikishin who is the professor in charge of room 121, [redacted] who is a student in the Nanotechnology Center and the two students who work in 121 [redacted] and [redacted] were outside the Nanotechnology building by 7:37pm. Mr. Martin then spoke with Dr. Berg who had been speaking with Dr. Nikishin, [redacted] and [redacted] and was told that there were waste bottles under the fume hood (one bottle contained Hydrochloric acid and Hydrogen Peroxide, one bottle that contained Hydrofluoric acid and Methanol and one bottle that contained Nitric acid). The Associate Dean of Engineering (Louisa Hope-Weeks) arrived on scene at approximately 8:00pm. LFD HAZMAT team entered the building at approximately 8:10 to determine the scope of the cleanup. After LFD HAZMAT team's assessment two teams were set up to enter 121 and perform a cleanup. The Vice President of Research (Dr. Taylor Eighmy) arrived on scene at approximately 8:30pm. The cleanup was concluded at approximately 8:50pm and the LFD HAZMAT team exhausted the room with fans prior to handing the room over the EH&S. The materials that were over packed by the LFD HAZMAT team were transported to the EH&S waste building where they could be further investigated. At approximately 12:30am October 28, 2012 the doors to 121 had the locks changed and EH&S took possession of the laboratory until and investigation could be conducted.

Findings From Investigation

In a cabinet under the fume hood in southwest corner of 121 is where the incident took place. The bottles in question were stored on the south side of cabinet under the fume hood. Clear and amber glass shrapnel was located throughout the laboratory 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 cabinet under the fume hood. The cabinetry of the hood had several strike marks on the inside of the door where debris had struck the inside of the cabinet. EH&S was informed that under the fume hood there was a 4 liter amber glass waste container marked as Hydrochloric acid and it also contained Hydrogen Peroxide which was not marked on the container as stated by the laboratorian (this container was not over packed since it was not compromised), a 4 liter clear bottle labeled as Nitric acid, and a 4 liter amber bottle labeled as Hydrofluoric acid and Methyl Alcohol. However, during the reconstruction of items collected from the LFD HAZMAT team there was a 4 liter plastic bottle labeled as Hydrofluoric acid waste that was not compromised, two 500ml broken bottles of Nitric acid, two 500ml punctured plastic bottles of Hydrofluoric acid, a broken 4 liter amber bottle labeled Acetone, a broken 4 liter clear bottle labeled as Nitric acid waste and a broken 4 liter amber bottle labeled as Hydrofluoric acid and Methyl Alcohol waste. The 4 liter amber bottle labeled as Hydrofluoric acid and Methyl Alcohol waste 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 that were recovered with the neck of the bottles 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 in April and August of 2011. The reaction during this incident was started at approximately 10:00am on October 27, 2011 and had concluded at approximately 5:50pm on October 27, 2011. The reaction was conducted in the fume hood in the southwest corner of 121. Once the reaction was completed all waste was stored in the cabinet under the fume hood in the southwest corner of 121. The waste bottles used during this experiment were the same waste bottles used during the experiments that took place in April and August of 2011. After the conclusion of their work [redacted] went home and [redacted] went to work in a laboratory across the hall from 121.

Response From Occupants

[redacted] was the first to find the incident. The laboratory [redacted] was working in was across the hall to the north of 121 and while working in the laboratory he heard an explosion from 121 at approximately 6:55pm. He went to investigate and was able to see the incident through the window of 121. [redacted] called Mr. Mill who was on his way to see Dr. Berg

and [redacted] notified Dr. Berg of what just occurred. Dr. Berg then had the UPD called who, upon arriving on scene, activated the fire alarm. When the alarm activated, the building was evacuated and the LFD was dispatched to the Nanotechnology Center.

Probable Cause of the Incident

The Hydrofluoric acid and Methyl Alcohol appeared to be the main suspect for the incident. The probable cause was the accidental mixing of two waste streams (Nitric acid waste being placed in the Hydrofluoric acid and Methyl Alcohol waste). With the mixture of the Nitric acid with Methyl Alcohol there more than likely was a pressure buildup due to Nitric acids ability to oxidize alcohols. The pressure more than likely increased 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's investigation a forensics consultant, Rimkus Consulting Group, was hired to assist in the determination of the cause of the explosion. Their report on the 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. Lab waste containing these materials were inadvertently mixed in a glass lab waste bottle being stored in the cabinet beneath the fume hood.
3. A likely contributing factor to the incident was the lack of understanding of the printed text on the laboratory waste bottles. The graduate student who likely mixed the wastes is non-native English speaking/writing and demonstrated a lack of understanding of chemical names printed in English.
4. The top of the lab waste storage bottle was inadvertently tightly capped, resulting in overpressurization and catastrophic failure of the bottle
5. The waste bottles were not externally polymer coated to minimize glass fragmentation in the event they were overpressured or dropped.
6. The waste bottles did not have venting caps that would have mitigated overpressurization
7. The waste bottles were not color code labeled to indicate the type of waste present in each container.

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.

Images From Incident

Image 1. Area where incident occurred before LFD HAZMAT team cleanup.

Image 2. Area where incident occurred after LFD HAZMAT team cleanup.

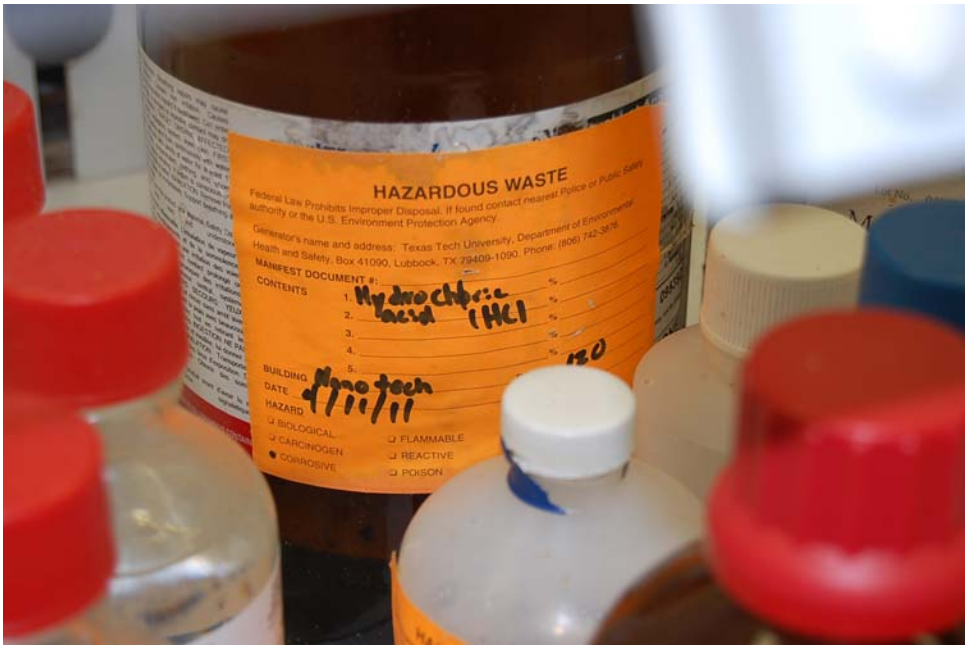


Image 3. 4 liter waste bottle with Hydrochloric acid and Hydrogen Peroxide that was not compromised.

Image 4. Plastic 4 liter waste bottle of Hydrofluoric acid waste that was not compromised.

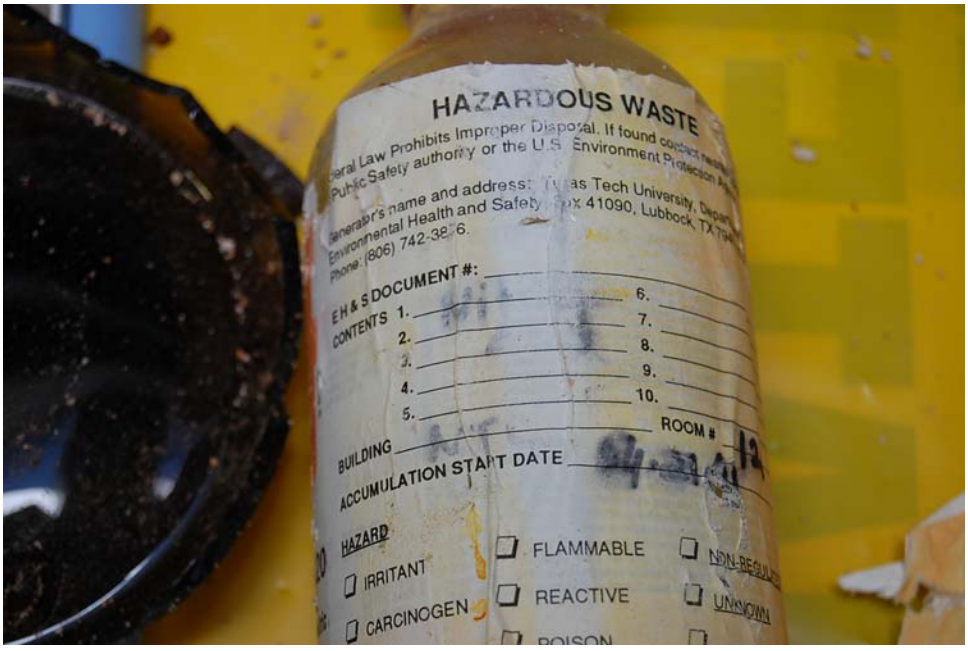


Image 5. One of two 500ml Nitric acid bottles that were broken.

Image 6. 4 liter bottle of Acetone that was broken.

Image 7. 4 liter bottle of Nitric acid waste that was broken.

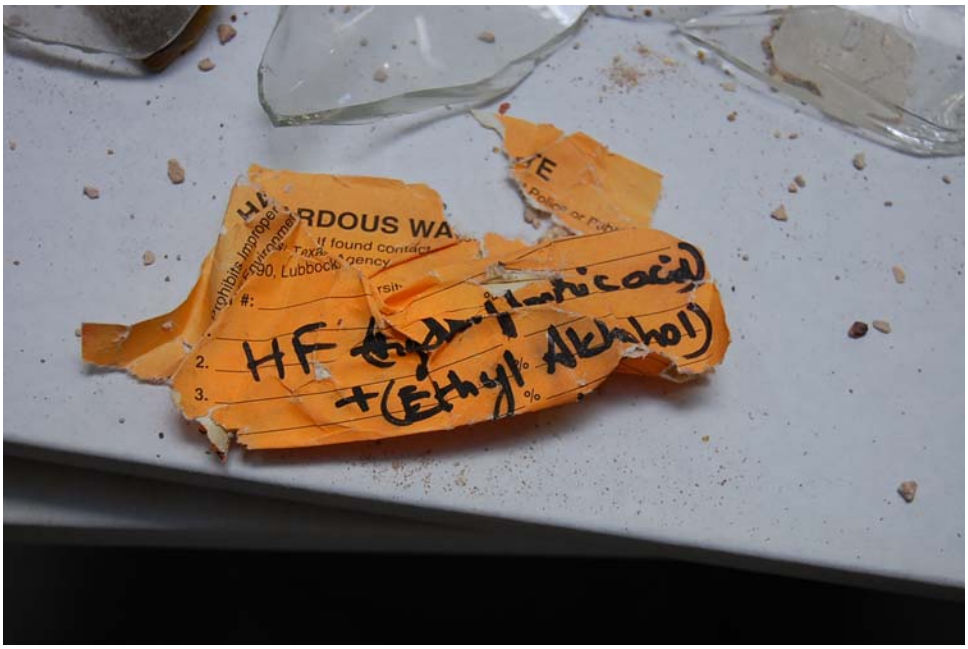


Image 8. Label from the 4 liter amber bottle with Hydrofluoric acid and Ethyl Alcohol waste that is the possible cause of the incident.

Image 9. 2-500ml bottles of Hydrofluoric acid that were punctured.

Image 10. Over pack of items removed from 121 by the LFD HAZMAT team.

cc: Dr. Eighmy, Dr. Young, Dr. Berg, Dr. Holtz, Dr. Sacco

Lab Safety/Incident Reports/Room121NANO_102711

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 27, 2011**
RCG File No.: 11004383

Dear Dr. Eighmy:

On October 27, 2011, an explosion occurred in the nano-scale research laboratories at Texas Tech University. The explosion resulted in minor damage to the fume 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 29, 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. Lab wastes containing these materials were inadvertently mixed in a glass lab waste bottle being stored in the cabinet beneath the fume hood.

3. A likely contributing factor to the incident was the lack of understanding of the printed text on the laboratory waste bottles. The graduate student who likely mixed the wastes is non-native English speaking/writing and demonstrated a lack of understanding of chemical names printed in English.

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

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

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

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

Discussion

Nano-scale research was being conducted by the Mechanical Engineering Department at Texas Tech University. As part of their research, two graduate students were preparing silicon substrate for Gas Source Molecular Beam Epitaxy (GSMBE). This preparation involved boiling and etching silicon chips in (1) nitric acid, (2) hydrofluoric acid, (3) hydrochloric acid and peroxide, and finally, (4) hydrofluoric acid with ethanol.

Each of the four wastes was being disposed of in four separate waste bottles. Each of the waste bottles was believed to be labeled with a waste label, but not a color coded waste label. Each of the waste bottles was stored beneath the hood in an integral cabinet.

None of the waste bottles were completely full. Preparation of the silicon wafers was performed jointly by the two graduate students the day of the incident. Additions were made to the waste bottles throughout the course of the day. The explosion happened several hours later, at approximately 6:50 p.m. the evening of October 27, 2011. The explosion was heard by one of the graduate students, who was across the hallway from the laboratory room. No one was injured in the incident.

The blast was sufficient to blow open the doors to the cabinet, and propel 0.5-inch glass shards the length and breadth of the laboratory, approximately 35 feet (see **Photographs**). Large glass windows on one interior wall of the laboratory were not damaged.

An investigation by this author did not reveal any visual evidence that this incident involved an explosive compound or a combustion-initiated explosion. A possible chemical hazards reaction matrix of the known chemicals in the cabinet 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 waste 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 oxide 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.

It became evident during the course of this investigation that one of the graduate students had some degree of difficulty with the English language. He is a non-native English speaker/writer. More than half of his laboratory notebook was written in his native tongue. On the label of a fresh reagent bottle clearly marked 'Hydrochloric Acid', he had written 'HCl' in heavy black pen, ostensibly because he understood the shortcut chemical formula but had difficulty understanding the full printed text.

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

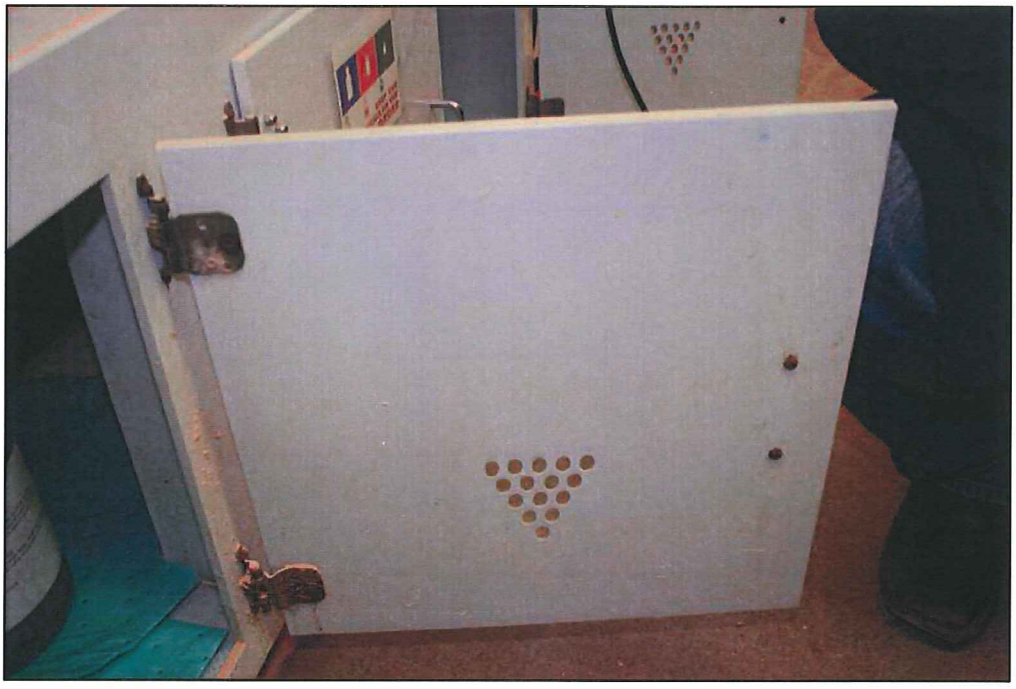
Attachments: Basis of Report, Photographs, CVs

Basis of Report

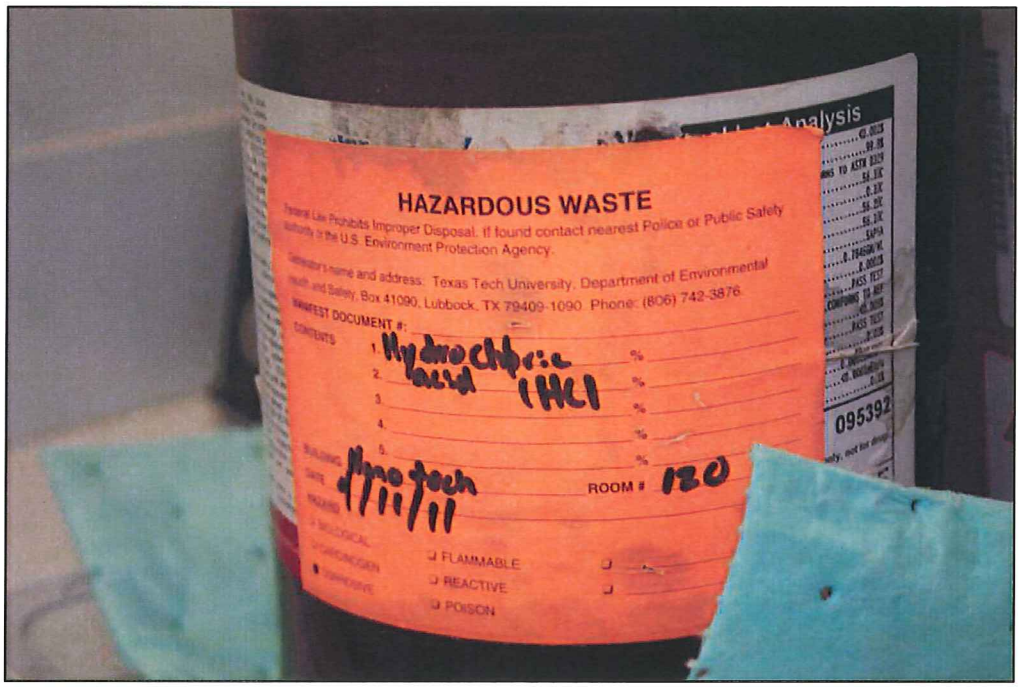
1. Inspection, interviews and photographs by the author, November 2, 2011.
2. Bretherick's Handbook of Reactive Chemical Hazards, 6th Edition, P.G. Urben, ed., 1999.
3. Prudent Practices in the Laboratory, Handling and Management of Chemical Hazards, National Research Council, 2011.

Photograph 1
Left Cabinet Door Showing Cuts from Glass Shards during Explosion

Photograph 2
Right Cabinet Door Showing Cuts from Glass Shards during Explosion



Photograph 3
Surviving Hydrochloric Acid Waste Bottle



Photograph 4
From End of Lab Looking toward Origin of Explosion, Red Dots on Floor indicate Locations of Glass Shards

Photograph 5
Red Dots on Floor Indicate Location of Glass Shards at End of Lab Further Away from Explosion In Front of Hood and Chemical Storage Cabinet

Photograph 6
Glass Shard Locations to Side of Hood and Chemical Storage Cabinet

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