Organic Process Research & Development **2010**, *14*, 1480–1484

# *Highlights from the Literature*

## Safety Notables: Information from the Literature

This is the eighth annual literature overview on safety issues which are of interest to process chemists and engineers to appear in *Organic Process Research & Development*. As in the previous years, this review will cover recent articles from the literature which address safety issues, common safety mistakes which seem to be repeated all too often, and major industrial accidents. This paper is not intended to be all inclusive of the safety literature nor should the information presented be used to make decisions regarding safety without reading the full text of the appropriate article. The intent is to give a flavor of the issues facing other chemists and engineers and how they are solving these problems.

### Toxic Tips

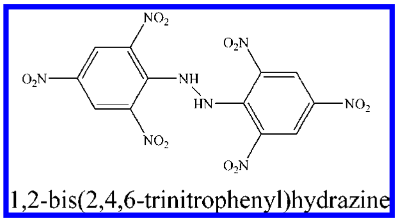
William E. Luttrell continues to write reviews on organic molecules and the precautions which should be taken while working with these compounds. These molecular safety reviews appear in the *Journal of Chemical Health & Safety* and are a good addition to the material safety data sheets (MSDS) which should be read prior to working with any new organic compound. Acetaldehyde (*J. Chem. Health Saf.* **2009**, September/October, 43.), potassium hydroxide (*J. Chem. Health Saf.* **2010**, January/February, 54.), nitrobenzene (*J. Chem. Health Saf.* **2010**, March/April, 24.), cyclohexane (*J. Chem. Health Saf.* **2010**, May/June, 33.) and methyl *tert*-butyl ether (*J. Chem. Health Saf.* **2010**, July/August, 28.) are a few of the more recent chemicals covered.

### Effect of Accidents in the Chemical Industry

The effect of a solitary chemical accident can send shockwaves around the globe and sully the reputation of the chemical sector in a matter of seconds. Andy Brice recently reviewed the impact of explosions at chemical facilities throughout the world (*ICIS Chem. Bus.* **2010**, March 29.). The legacy of these accidents is that lessons will be learned, and new processess and procedures will be put into place to hopefully prevent a recurrence. The article provides a timeline of major chemical explosions starting with the explosion in Feyzin, France, in 1966 and ending with the Texas City, Texas, United States, refinery accident in 2005. Background details of these incidents and additional information on the impact of these incidents can be found on the web-based version of this article (www.icis.com/Articles/2010/03/29/9345517/the-explosive-effect-of-accidents-in-the-chemical-industry.html).

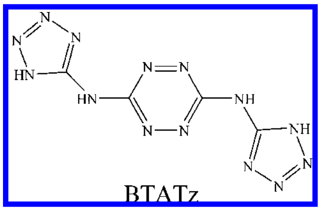
### Synthesis of Energetic Materials

Many readers of this review are looking for ways to lessen, or avoid all together, working with molecules that have hazardous thermal properties. However, scientists working to prepare novel propellants and explosives, such as D. M. Badgujar and colleagues, deal with the dangers and rigors of working with thermally unstable compounds on a daily basis. In one such example, the synthesis, characterization, and evaluation of 1,2-bis(2,4,6-trinitrophenyl)hydrazine (*J. Hazard. Mater.* **2009**, *172*, 276.) is outlined in good detail.



1,2-bis(2,4,6-trinitrophenyl)hydrazine

In a similar article, Sikder and co-workers report the synthesis and characterization of 3,6-bis(1*H*-1,2,3,4-tetrazol-5-ylamino)-1,2,4,5-tetrazine (BTATz) (*J. Hazard. Mater.* **2009**, *170*, 306.). Along with the synthetic and purification details, thermal studies are presented that were carried out to determine the decomposition patterns of this class of highly energetic materials.



BTATz

Belzowski and co-workers published a communication on the energetic characteristics of transition metal complexes of nitrates and perchlorates (*J. Hazard. Mater.* **2009**, *171*, 1175.). Along with their preparations, the authors describe the testing of each material in underwater detonation tests and how these new materials compare to classical explosives.

Last, Didier Mathieu has written a paper on a new software package (MATEO) for the molecular design of energetic materials (*J. Hazard. Mater.* **2010**, *176*, 313.). By using MATEO, chemists involved in the synthesis of new energetic compounds can get immediate estimates for the relevant properties of a new molecule whose synthesis appears feasible. This software gives information to help a chemist select the most promising candidate from a number of compounds that can be synthesized.

Reading these types of papers gives a solid background on the preparation of thermally energetic compounds. Hopefully, it can lead to insights into the proper amount of data required prior to synthesizing other potentially unstable targets on scale.

### Amide Bond Formations

The formation of amide bonds is a widely used reaction in organic chemistry. There are many combinations of carbonyl

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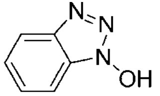
activating agents and additives which can increase the rate of the desired reaction and/or decrease unwanted side reactions in the process. One popular carbonyl activating agent is 1-hydroxybenzotriazole (HOBt), which is normally sold as a monohydrate.



**1-hydroxybenzotriazole**

There are drawbacks of using HOBt in the chemical industry of which users of this material should be aware. The compound is known to explode when heated beyond its melting point (156 °C), and it has a large energy of decomposition (−1715 J/g) when measured by DSC at a heating ramp of 5 °C/min. The drying of the hydrated commercial material should be avoided. HOBt is also classified by the UN committee and the European Chemicals Bureau as a Category 1 explosive when in anhydrous form and as a Category 4.1 desensitized explosive, or ‘self-reactive’ solid, when containing 10−20% water. Thus, HOBt is subject to transportation and storage restrictions because of this explosive potential. An excellent and detailed approach to remove HOBt from a process was recently published by Jon Lorenz, Carl Busacca, et al. of Boehringer-Ingelheim Pharmaceuticals, Inc. (*J. Org. Chem.* **2010**, *75*, 1155.). In the article, the authors go through over 25 different combinations of coupling agents, solvents, and weak acid additives to arrive at an acceptable alternative to HOBt. It is a solid example of the steps of company should take to remove a potentially hazardous reagent from a synthetic pathway.

As an aside, two potential alternatives to HOBt were previously published including 2-hydroxypyridine (*Org. Process Res. Dev.* **2004**, *8*, 1054.) and 2-mercaptobenzothiazole (*Specialty Chem. Mag.* **2005**, September, 50.). Please note that, although comparison of thermal safety analysis was given for the use of 2-hydroxypyridine versus HOBt, no such analysis was provided for the use of 2-mercaptobenzothiazole.

## An Unexpected Explosion

Dmitrii Perepichaka and Shehzad Jeeva submitted a safety letter related to an unexpected explosion of trimethylsilylacetylene (TMSA) (*Chem. Eng. News* **2010**, January 18, 2.). The explosion ruptured a 2-L flask and seriously injured a researcher. The compound was prepared according to the literature procedure of Graham Jones, David Kendrick, and Andrew Holmes (*Org. Synth.* **1993**, Collect. Vol. 8, 63.). The authors of the original report recommend the use of a safety shield, which is a common precaution when working with flammable materials in the atmosphere of oxygen. The procedure involves purging oxygen through a solution of TMSA in acetone in the presence of a copper(I) chloride:tetramethylenediamine complex catalyst at room temperature. In this incident, the explosion occurred as soon as the solution of catalyst in acetone was added to the reaction.

The authors of the safety letter speculate that the cause of the explosion was a discharge of static electricity between the syringe needle and the digital thermometer inside the flask. The incident emphasized once more the potential danger of mixing oxygen gas with flammable solvents or reagents and to be wary of introducing two conductors into a reaction flask.

## Maintaining Safety During Corporate Change

The announcements of mergers and acquisitions (M&A) are standard occurrences in the world of chemical businesses. Chemical companies are often merging, acquiring, divesting, splitting, and even spinning off new companies. The Center for Chemical Process Safety (CCPS) has written a new book titled *Guidelines for Acquisition Evaluation and Post Merger Integration*. This book was written with a strong focus on process safety and process safety management. In an article written by Melhem et al., the safety aspects of the book are reviewed (*ProcessSaf. Prog.* **2010**, *29*, 118.), and the detailed check list of responsibilities for use during and after the acquisition process are discussed. The authors conclude that as companies use the tools described and recognize their value, evaluation of process safety in the M&A process will become a mandatory requirement and not an afterthought. It is hoped that safety will become a core issue and a major factor in the decision making process of any acquisition.

In a related article, Paul Davidson et al. discuss the key safety roles in organizational change (*Process Saf. Prog.* **2010**, *29*, 11.). With layoffs occurring all too regularly and the implementation of lean manufacturing principles leading to reduced staff and flatter management structures, it is important that safety is not compromised by the removal of key people. This article highlights the Bow Tie methodology to analyze major accident scenarios for high-hazard sites and presents an example from a real-life case study to demonstrate the value of this analysis.

## Learning about Process Safety

An article on the T2 Laboratories runaway reaction in 2007 by Dennis Hendershot (*J. Chem. Health Saf.* **2010**, January/February, 59.) highlights the importance of fully understanding the exothermic nature of the intended chemistry and any potential decomposition pathways. The final CSB report of the incident and its parallels to previous CSB incident investigations is examined. The paper underlines the criticality of producing chemical engineering graduates with a firm grasp of reactive chemical hazards.

## Dust Explosions

An excellent resource article providing an overview of factors that can lead to dust explosions and preventative measures against such events was published by Amyotte and Eckhoff (*J. Chem. Health Saf.* **2010**, January/February, 15.).

## Safety Tip When Using Azide with PTC

Marc Halpern of PTC Organics publishes a monthly newsletter on the use of phase-transfer catalysis (PTC) in synthetic organic chemistry (http://www.phasetransfer.com/). In the July 2010 edition of the newsletter, an article on the use of azides with phase-transfer catalysis was provided (http://ptcorganics.com/ContactUs.htm). The author describes that, along with the usual precautions required for the safe handling of azides, there are additional safety aspects of PTC-azide reactions that one needs to consider. While all organic azides must be treated with great care, methyl azide and ethyl azide are especially hazardous and explosive. When performing reactions with a quaternary ammonium salt (quat) as the phase-transfer catalyst with an azide, the azide can act as a nucleophile that attacks an alkyl group of the quat to produce an alkyl azide and a triakylamine. If the quat contains a methyl or ethyl group (e.g., methyl trioctyl ammonium, ethyl tribenzyl ammonium, or methyl tributyl ammonium), then there is a high likelihood that methyl azide or ethyl azide may be formed in some finite concentration. The author suggests that one of the tests that should be performed during the HAZOP analysis (before running reactions) is a DSC or other very small-scale test to determine the point at which the reaction mixture, containing azide and a phase-transfer catalyst, liberates a lot of energy. You are likely to find that the tolerance of the system for using tetrabutyl ammonium may be higher than for methyl tributyl ammonium. When one searches the literature, one will find publications and patents that use quats containing methyl groups and ethyl groups in an azide reaction. The author asks that one not be complacent and not rely on the luck that the authors may have had during their experimental programs. One must be prudent and perform testing to ensure the safety of using any quat salt with azide, especially if one is working with high concentration, has a significant excess of azide, or intends to heat the reaction. For an example of a phase-transfer-catalyzed azide reaction, please see the work of Malik et al. (Solvent-free process for the synthesis of energeticoxetane monomers. U.S. Patent 5,523,424, 1996). Marc Halpern asks that you please note that the inventors performed very extensive safety testing before performing these reactions and recommends that anyone performing similar chemistry should too.

### 2-Methylpyridine-*N*-oxidation Runaway Studies

Papadaki and co-workers studied the runaway behavior of 2-methylpyridine-*N*-oxide (2-picoline-*N*-oxide) in the Automatic Pressure Tracking Adiabatic Calorimeter (APTAC) (*J. Loss Prev. Process Ind.* **2010**, *22 (6)*, 839.). They studied different combinations of starting materials, product, hydrogen peroxide, and phosphotungstinic acid catalyst in an effort to determine the effects of the different components of the mixture on the decomposition behavior. The reaction is very sensitive to conditions. It is well-known that hydrogen peroxide decomposition is accelerated by a number of different materials. The authors showed that the decomposition of the product, 2-methylpyridine-*N*-oxide, is also accelerated by both the catalyst and hydrogen peroxide, highlighting the need to be aware of synergistic effects for processes with hydrogen peroxide or complex catalysts.

### Compressed Gases: Managing Cylinders Safety

Compressed gas cylinders are widely used in research, pilot plant, laboratory, and small-scale manufacturing and processing operations as a way to provide a convenient, economical, and safe source of high-pressure gases for a variety of applications. Richard Palluzi has written a set of recommendations to ensure the safe handling, storage, and use of gas cylinders (*Chem. Eng.* **2009**, October, 52.). The article begins with a solid introduction of cylinder basics which outlines the key components of an appropriately appointed compressed-gas cylinder, a review of the critical information that can be found on a manufacturers label, and a listing of common cylinder dimensions and capacities. A significant review on the proper storage of these cylinders is also given with specific attention applied to regulatory limits of the different kinds of compressed gases. The author also discusses proper labeling of cylinders, inventory control of compressed gases, and how to handle empty cylinders. Palluzi concludes by stating that, despite the widespread use of these cylinders in research, they always hold the potential for accidents and injuries if not handled properly.

### Hydrochloric Acid Tank Explosion

Chandrashekhar Chandwadkar et al. have published an overview of an explosion that occurred at their facility (*Process Saf. Prog.* **2009**, September, 293.). The article describes the explosion of an HCl tank having a 250 kL capacity, 60% filled with 33% HCl. The tank took off from the ground, went approximately 9 m high in the air and landed 50 m away from its foundation while spraying its contents all over the nearby area. The authors walk the reader through early warning signs that there may be an issue, a narration of the event itself and what occurred, conclusions on the event, and recommendations on how to prevent future explosions in similar tanks. All too often incidents and accidents of this type are kept quiet and never used to instruct others on how to prevent them. These authors and their organization, National Petrochemical Company of Iran, should be commended for sharing this information with the public.

### University Laboratory Safety

Last year, details of the fatal fire at UCLA involving *tert*-butyllithium were extensively described by Jyllian Kemsley (*Chem. Eng. News* **2009**, *87*, 29−31.). This article not only shows the lab notebook entries, but also has a plethora of information and web links regarding the incident and associated safe handling practices. A follow-up report which detailed lax safety practices at UCLA and the subsequent fines that they were assessed appeared this year (*Chem. Eng. News* **2010**, March 22, 9.). The accident at UCLA has led to an increase in awareness of potential issues that may exist at university chemical laboratories throughout the United States.

As an example of this increased awareness, the Chemical Safety and Hazard Investigation Board (CSB) announced the first investigation into a university lab accident (*Chem. Eng. News* **2010**, January 25, 7.). The accident occurred at Texas Tech University, involved an explosion of a mixture of hydrazine nickel perchlorate, and led to one student being seriously injured (lost three digits on one hand, severe burns and lacerations to the hands and face). It is stated in the article that the CSB is very selective about what it investigates and that publicizing this particular incident may be a wake-up call to other institutions that there are hazardous materials and equipment in their laboratories. An internal investigation determined that Texas Tech University violated many of its own policies and procedures which played a role in this accident (*Chem. Eng. News* **2010**, August 23, 34.). An expansion of the CSB into other laboratory accidents in academia could be on the horizon (*Chem. Eng. News* **2010**, February 1, 25.).

In another example, the American Chemical Society’s Division of Chemical Health & Safety (CHAS) commissioned, conducted and released the results of a survey aimed at evaluating the laboratory safety culture within hundreds of academic chemical departments (*Chem. Eng. News* **2010**, June 21, 33.). This survey was created from scratch as no previous survey of academic laboratory safety culture could be found. The complete survey results are available online (www.dchas.org click on "Culture Survey") and the department is seeking comments on the results to help determine what the next steps in this process may be.

In a last example, many schools are expanding safety training for undergraduate students in chemical departments (*Chem. Eng. News* **2010**, June 28, 14. and *Process Saf. Prog.* **2010**, June, 97.). In response to incidents in their laboratories or to comply with new educational guidelines issued by the American Chemical Society's Committee of Professional Training (CPT) for ACS-approved bachelor's degree programs, schools are exploring a variety of ways to augment laboratory safety instructions for undergraduate students. Examples from several schools and universities on how they are addressing these issues are provided.

## Handling Pyrophoric Reagents

A useful short article by Neal Langerman (*J. Chem. Health Saf.* **2009**, September/October, 51.) discusses the safe handling of *tert*-butyllithium in a laboratory setting. It includes an interesting review of the appropriate PPE to be utilized and some ideas on equipment setup.

## Inherently Safer Technology

Trevor Kletz in his book on plant design (*Plant Design Safety*; Hemisphere PublishingCorporation: New York, 1991) stated, "The essence of inherently safer approaches to plant design is the avoidance of hazards rather than their control by added-on protective equipment." Kletz also proposed the five fundamental principles of inherent safety; minimization, substitution, attenuation, simplification, and limitation of effects. This year, as usual, many articles were published which discussed the principles of inherently safer technology (IST) or inherently safer design (ISD) of chemical processes and process plants.

On November 6, 2009, the United States House of Representatives passed the Chemical and Water Security Act of 2009 (H.R. 2868), and the bill will be presented to the Senate for consideration in 2010. This legislation would significantly expand the Department of Homeland Security's (DHS) authority to regulate security practices at thousands of facilities nationwide that produce, use, or store chemicals. A provision of the legislation requires an evaluation of IST for facilities which have the potential for a toxic release which has the potential to impact the surrounding communities. A review of the legislation and a discussion of several of the myths associated with IST was written by Dennis Hendershot (*J. Chem. Health Saf.* **2010**, March/April, 29.), and an editorial on this legislation was written by Rudy Baum, the Editor-in-Chief of *Chemical and Engineering News* (*Chem. Eng. News* **2009**, November 23, 3.). All this debate has led to the question of what does IST actually mean? Along with this has been an increased public interest in IST (*Chem. Eng. News* **2010**, August 23.). The United States DHS "asked AIChE's Center for Chemical Process Safety (CCPS) to develop a formal scientific and technical definition of inherently safer technology". A working group was formed of government, academia, and industry professionals. Hendershot presents their results and discusses some of the aspects of IST that are commonly misunderstood. Defining a technology as an IST is necessarily a comparison versus an alternative technology and must include a description of the factors being considered (chemical reactivity, chronic toxicity, acute toxicity, thermal stability, flammability, etc.), their location, and the affected population. There are typically trade-offs between the different dimensions. Attempts have been made to quantify the overall assessment of ISTs, but there is no consensus of methodology at this time. References are given for further reading.

The second edition of the book, *Inherently Safer Chemical Processes: A Life Cycle Approach*; John Wiley & Sons: Hoboken, NJ, ISBN: 978-0-471-77892-9 (written by the AIChE Center for Process Safety, Inc.) was published in 2009. With several jurisdictions having mandated consideration of IST in certain facilities and the aforementioned federal legislation looming, this new and expanded edition is a most welcome addition to the process safety and loss prevention literature. In a book review, Stanley Grossel states that this edition builds on the same philosophy of the first edition, but clarifies and enhances the concept by adding recent research, practitioner observations, examples, industry methods, and discussion on regulatory and security issues (*Process Saf. Prog.* **2009**, September, 297.).

## Process Safety and the Human Factor

A number of papers were published in 2010 addressing the human element of process safety and the importance of not overlooking this key factor. Human error still appears to be a root cause in the majority of processing incidents. One article by a team from Celanese (*Process Saf. Prog.* **2009**, *28*, 325.) describe how a series of near misses in 1996 led to the adoption of a "conduct of operations system" designed to address one of the root causes, human error. The main elements of the code, namely training, operations and procedures are described in detail.

Another paper from workers at Engro Chemical (*Process Saf. Prog.* **2010**, *29*, 174.) tackles a near miss in 2007 involving a syngas compressor train and highlights in depth the subsequent people development program they initiated to avoid any future catastrophe.

A further paper (*Process Saf. Prog.* **2009**, *28*, 347.) takes the consideration of the human element to the next level. This article describes an approach to minimizing the accidents caused by human error, by rigorously evaluating human factors to help drive the design phase of a process plant.

The fourth paper on the theme of the human factor in safety matters (*Process Saf. Prog.* **2009**, *28*, 210.) focuses on safety management. The article notes that many organizations have been proven to have poor retention of lessons learned and argues that the key findings of failure investigations should be distributed more widely to prevent repeat occurrences. It also discusses various challenges to sustaining safe operation of process equipment.

Finally a short article by Dennis Hendershot (*J. Chem. Health Saf.* **2009**, November/December, 37.) titled "Do we really ever learn?" underscores the fact that, 25 years after Bhopal and the Mexico City LPG explosion, we continue to see accident investigation reports which point to the same contributing factors involved in those tragedies. The paper acknowledges the advances in process safety technology, but stresses that the challenge lies in using these tools effectively and not forgetting lessons learned previously.

### Lessons Learned from a Thermal Runaway Incident Involving an Organic Peroxide Intermediate during a Power Outage

Morrison et al., reported at the AIChE Spring Meeting and Sixth Global Congress on Process Safety held on March 21−25, 2010, on their incident investigation into a runaway oxidation of cumene to cumene hydroperoxide. Parallels are drawn to the runaway incident at T2 Laboratories in Jacksonville, Florida, United States (December 2007). In the runaway investigated by Morrison there were a number of contributory factors that caused a minor incident (lightning strike) to lead to a large pool fire in the plant. A lightning strike led to an extended power outage, but since the plant did not have back-up power to the external circulation and cooling system the operators had no ability to cool the oxidation reactor. The lightning also damaged instruments and control systems which caused the operating personnel to be unaware of the dangerous self-heating that was occurring in one of the reactors. The damaged high-temperature and high-level alarms failed to notify the operators of the impending emergency release to allow them sufficient time to properly evacuate the plant. Although the reactor vented without compromising the vessel, the flammable materials ignited and led to a large pool fire, which impinged on nearby reactors. Although the oxidation of cumene to phenol has a significant runaway potential and there is a history of such incidents, sufficient safeguards were not put into place to prevent or mitigate the runaway.

### Burn Injury Caused by Mixing Incompatible Chemicals with Sodium Permanganate

Ogle and Morrison presented, at the AIChE Spring Meeting and Sixth Global Congress on Process Safety held on March 21−25, 2010, the findings of an investigation into an incident caused by the inadvertent mixing of two incompatible chemicals in a 5 gallon open bucket. Aqueous sodium permanganate (40%) was unintentionally mixed with either solid sodium thiosulfate pentahydrate or solid sodium metabisulfate. A violent reaction would be expected from this mixture, but the peculiar aspect of the incident was the time delay between mixing and eruption. During their investigation Ogle and Morrison reproduced the time delay and even observed eruptions from a bucket after approximately two weeks of contact time. They propose that the mass transfer of the sodium thiosulfate becomes limited by a coating of manganese dioxide (MnO). It was only after the bucket was lifted to transport it, that the MnO layer was disturbed and the violent reaction occurred. Thermodynamic calculations and experience clearly identify the potential for a violent reaction. However, this incident highlights the need to consider the reactivity hazard that results from mass transfer limitations not only in waste handling but also while designing a chemical process.

### Aminodifluorosulfinium Salts: Selective Fluorination Reagents with Enhanced Thermal Stability and Ease of Handling

Fluorine is finding increasing use in pharmaceuticals and agrochemicals. Unfortunately, fluorinating reagents can be difficult to handle and are often thermally unstable. Michel Couturier and co-workers present two promising fluorinating reagents diethylaminodifluorosulfinium tetrafluoroborate (XtalFluor-E) and morpholinodifluorosulfinium tetrafluoroborate (XtalFluor-M) (*J. Org. Chem.* **2010**, *105*, 3401.). The reagents are free-flowing solids that do not generate HF and show increased selectivity in reducing elimination side products. They are significantly more stable than the two most widely used deoxofluorinating reagents, DAST and Deoxo-Fluor. The Accelerating Rate Calorimetry onset temperatures for XtalFluor-E and XtalFluor-M are 119 and 141 °C, respectively, in comparison to the 60 °C onset observed for commercially available DAST and Deoxo-Fluor. XtalFluor-E and XtalFluor-M can also be prepared directly from crude DAST, eliminating the need to distill the energetic, thermally unstable material. The two new reagents are excellent fluorinating agents for alcohols and carbonyls when used with promoters such as DBU, $Et_3N\cdot 2HF$, or $Et_3N\cdot 3HF$.

### Mining the Web for Safety Information

The following is a list of Web sites which the authors find useful for finding information on process safety and hazard analysis. If any readers have additions to this list we would be most interested in seeing them and perhaps including them in next year's review.

1. ACS Green Chemistry Institute: http://www.acs.org/greenchemistry.
2. ACS Division of Chemical Health and Safety: http://www.dchas.org.
3. National Institute of Occupational Safety and Health (NIOSH) Eye safety: http://www.cdc.gov/niosh/topics/eye/default.
4. American National Standards Institute (ANSI): http://www.ansi.org.
5. NIST WebBook: http://webbook.nist.gov/chemistry/.
6. Chemical Compatibility Chart: http://rehs.rutgers.edu/pdf_files/Chemical_compatibility.html.

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