On June 17, an explosion in a chemistry lab at the University of Minnesota injured graduate student Walter Partlo. He was making trimethylsilyl azide, starting with 200 g of sodium azide. The incident originated in lack of hazard awareness, school representatives say, and the department response focuses on identifying hazardous processes and communication.

The synthesis was based on previously published methods, with some alterations—in particular, the solvent Partlo used was polyethylene glycol (PEG), says chemistry department chair William B. Tolman. Partlo is a fifth-year graduate student in professor T. Andrew Taton’s group, which had run the reaction at least ten times previously, Tolman says. When the explosion happened, the reaction was on its second day. Partlo was on his way from the lab office to the hall when he noticed that the thermometer was askew. He stopped and reached into the hood, but he didn’t have time to touch anything before the experiment exploded, says Anna Sitek, a research safety specialist in UMN’s department of environmental health and safety. Partlo wasn’t wearing any personal protective equipment. The explosion left him with second-degree burns and glass injuries to his arm and side; he also injured an eardrum. The explosion also destroyed the experimental apparatus and hood.

Tolman, Sitek, and other investigators have not been able to definitively identify what went wrong with the reaction, Tolman says. One explanation is that the explosion was from hydrazoic acid, which could have formed from wet PEG providing water to react with sodium azide or the PEG itself reacting with sodium azide. Another explanation is that the sodium azide overheated.

More important than the reaction, Tolman emphasizes, is the deeper root cause of the incident: insufficient recognition of the reaction’s hazards. Warnings included with literature protocols were “pretty lame,” he says. He also thinks that the lab group became complacent after doing the reaction several times without incident. “While they were aware of the hazards, concern about them became less up front,” he says.

Also, as people modified the protocol, they didn’t appear to understand how changes might affect the risk of the synthesis. “There was a real reason to use PEG,” Tolman says. The reaction involved a heterogeneous mixture and people had trouble with clumping, and literature indicated that using PEG would help. “But they hadn’t thought through that maybe the PEG was wet or might react itself,” Tolman says.

“It was not a case of blowing off safety,” Sitek adds. “They thought they were making the right changes, but they didn’t know the questions to ask to recognize when they were moving in the wrong direction.”

“Overall, there was clearly a lack of proper hazard assessment,” Tolman continues. “They didn’t stop and say, ‘This is a really dangerous procedure, should we be doing this at all, or should we be taking extra precautions?’” Tolman notes that there are no engineering controls available in any lab in his department that would allow the reaction to be performed safely at 200 g scale. He has set a limit of 5 g.

For his part, Partlo says that “I think that the biggest lesson that I have taken away from the experience is that though a synthetic procedure is well-documented in the literature, the inherent safety concerns may not be.” He continues: “A corollary of this is that researchers need to be sure that they are properly heeding the warnings that they do have, and properly recognizing the risk of everything that could go wrong in a particular synthesis, even if those risks seem unlikely. Unforeseen problems to consider include a transformer or thermostat failing, the water supply to a condenser being interrupted during a reflux, or stirring being stopped. When planning a reaction, these things should be considered and the equipment and scale of the reaction should be adjusted accordingly to ensure proper management of potential risks.”

Going forward, Tolman hopes that a review of standard operating procedures (SOPs) and new communication efforts will help ensure that something similar doesn’t happen in the future.
Tolman has ordered lab groups to assess their standard operating procedures and update them if necessary by Aug. 15. The goal is to get everyone to stop and think about whether they’re doing anything that is potentially hazardous, whether they have an SOP for that activity, and whether the SOP is correct, he says. Meanwhile, the department’s safety committee and its Joint Safety Team with chemical engineering and material science are working out how to review the procedures as well.

Additionally, lab groups will now be required to use safe operation cards on hoods to communicate who’s running a reaction, what it is, and its hazards. Safe operation cards are something that the department learned about through interactions with Dow Chemical to promote lab safety, but logistical challenges had held up department-wide implementation. “This incident spurred me to say, ‘Enough, let’s stop dithering,’” Tolman says. Now, each group has until Sept. 2 to develop a card and set a lab-specific policy for using it. Tolman has already implemented a card in his lab.

Tolman is also now requiring groups to hold at least monthly meetings at which safety must be discussed. “Larger group seem to be communicating effectively. They have regular group meetings and safety moments and discussions,” Tolman says. “Smaller groups were perhaps not doing this as well.” A large group is one with more than the department average of eight people.

Taton and his group members are also working on safety letters that will be sent to C&EN and the journals involved, Tolman says.

Overall, “I think our safety culture has improved a lot” in recent years, says Tolman, who held a department-wide meeting on July 11 to discuss the incident and response. “But like any culture, there’s variable adhesion to it, so you have people who are strong players and leaders who are ahead of the game, and people just going along for the ride.” With 300 people in the department, it’s a challenge to ensure that everyone is fully tuned in, he says, but that’s where the community as a whole has to step up. “We have to look out for each other and help each other,” he says.

Tolman, Sitek, and colleagues also have recommendations for the chemistry community at large:

1. Update risk assessment procedures
   (a) to identify factors affecting the probability and severity of an energetic event occurring
   (b) to consider the capabilities of available safety controls.

To paraphrase the limitations of the Laboratory Hazard Risk Assessment Matrix according to the ACS guidance document Identifying & Evaluating Chemical Hazards in the Research Lab, a higher degree of training is required to consistently and accurately rate the severity of consequences and probability of occurrence for a given risk and may also require a secondary assessment and or tool.

2. Warn researchers not to assume journals include complete risk control information. Encourage researchers to check multiple sources for information about hazards and include safety sources other than the SDS and published procedures. Examples: ToxNet’s Hazardous Substances Data Bank and Bretherick’s.

3. Encourage researchers to perform complete risk assessments on all potentially hazardous experiments.

4. Develop additional tools and training to help researchers assess the severity of consequences, probability of occurrence and capacity of controls.