Stanford | Environmental Health & Safety

# HYDROGENATION EXPLOSION

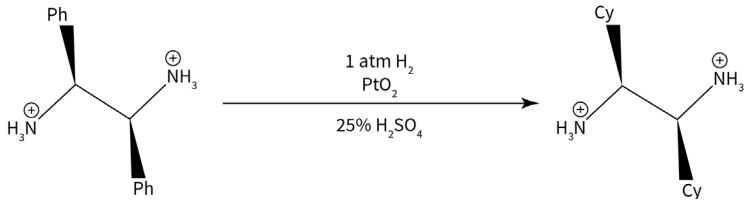
*Lessons Learned*

**This Lessons Learned focuses on a small explosion that occurred while a researcher was working with flammable gas. It demonstrates the importance of thorough safety review during experiment planning, particularly when working with relatively high hazard materials. It also highlights the particular difficulties associated with catalyzed hydrogenation reactions.**

## What Happened?

**Hydrogenations are a notoriously difficult class of reactions to perform safely due to the flammability of hydrogen and the changing hazards of the catalyst. For more information on hydrogenation reactions, see EH&S' information sheet on the topic.**

A researcher ran a hydrogenation reaction in a chemical fume hood while wearing a flame-resistant lab coat, nitrile gloves, safety glasses, and lab-appropriate attire. They added 1.5 g of the hydrochloride salt of (S,S)-1,2-diphenylethylenediamine, 500 mg of PtO2, and 80 mL of 25% H2SO4 to a 250 mL roundbottom flask. Then, they purged the flask of air using nitrogen. The reaction was put under ~1 atm of pure hydrogen gas by attaching a party balloon to a needle through the septum, and they then left the mixture to stir at room temperature overnight. A day later, the researcher replaced the original balloon with a fresh balloon of hydrogen. Two days later, the researcher purged the vessel of hydrogen using nitrogen gas, took an aliquot, and analyzed it using NMR. Upon analysis, less than half the starting material had converted to product, so the researcher replaced the hydrogen balloon and allowed the reaction to proceed. The researcher was concerned that the catalyst had been poisoned (i.e., had become contaminated and was no longer functional), and so they removed the septum and added 420 mg of PtO2 (without purging the hydrogen atmosphere). The researcher was wearing the same PPE described initially at this time. Immediately, a small explosion occurred, generating an audible noise and a fireball with a diameter of 4 - 5 inches above the opening of the flask. After the explosion, the material in the flask continued to burn. The researcher replaced the septum and the fire, starved of oxygen, went out. The researcher, having experienced no personal injury and seeing no damage to the flask, continued with the experiment. They purged the flask atmosphere with nitrogen and then placed a new hydrogen balloon, continuing the experiment. The researcher did not notify their PI, feeling that it was unnecessary given that there was no injury or damage to equipment.




Later, the researcher mentioned the incident to their PI in passing. The PI had the researcher write up the incident and present it at group meeting. Due to a miscommunication, however, no SU-17 incident report

was filed until several months later.

## What Went Right

- The researcher had not performed this reaction before, so they ran it at a relatively modest scale.
- The researcher was able to think quickly and extinguish the fire.
- The researcher felt comfortable discussing the incident with their PI and the rest of the lab. This was due to the PI putting in conscious effort to make safety an important part of their lab and treating incidents as moments for learning. Incident investigation and dissemination of lessons are the responsibility of the PI.
- After the SU-17 was submitted and the incident was investigated, EH&S and the lab collaboratively wrote an information sheet on hydrogenation reactions.

## What Went Wrong

- The researcher did not perform a risk assessment before beginning their experiment, and thus were unaware of the full extent of the hazards involved. Without the crucial knowledge that $PtO_2$ that has been exposed to H2 becomes pyrophoric, they were not aware of the risks of opening the reaction vessel to air.
    - While the lab did have established hazard thresholds for experiments requiring written risk assessments to be submitted to the PI, this incident revealed a gap in some of the threshold "triggers." The lab reviewed and updated their criteria.
- The researcher did not take adequate precautions while working with hydrogen gas. When working with hydrogen, keep a fire extinguisher nearby, avoid exposing it to air, and if it will be confined in a glass vessel or used in any vessel at >1 atm, a blast shield should be used.
    - Catalyzed hydrogenation reactions must always be purged with an inert gas prior to exposure to air (e.g., adding more catalyst, work-up). More information can be found in the EH&S hydrogen safety information sheet.
- The researcher did not follow fire response best practices. Putting the septum on the container and bringing their hands close to the flame was a high-risk action, even though it ultimately extinguished the fire.
- After the explosion, the researcher should have paused their experiment to review its safety and called 9-1-1.
- The researcher and PI did not follow proper incident reporting procedures. Any lab incident should be reported to the PI as soon as possible. The PI is responsible for reporting the incident to EH&S within 24-hours. Any fire and/or explosion requires the submission of an incident report, even if it is successfully extinguished or contained.

## What Lessons Can We Learn?

- Never expose metal catalysts charged with hydrogen to air or another oxidizer. For more detailed guidance on hydrogenation reaction safety, see the EH&S information sheet on this topic.
- While it is always advisable to complete a risk assessment and/or review appropriate SOPs before beginning an experiment, it is especially critical when working with relatively higher hazard materials such as hydrogen gas.
    - Classes of compounds that may merit particularly close review include, but are not limited to:

flammable gases, toxic gases, water-reactive compounds, pyrophoric compounds, oxidizers, explosives, and materials that are highly acutely toxic by inhalation or dermal exposure.

- When working with higher hazard materials, discuss your proposed experiment with your PI before beginning work. You can also reach out to EH&S for further consultation.
- If there is a fire and you are at all uncertain, do not attempt to fight the fire. Evacuate the area, call 9-1-1, and wait outside the building so you can provide information to first responders.
- Submit SU-17 incident reports promptly. These reports help EH&S to support faculty and researchers, to share vital safety information, develop new resources, and help prevent future incidents.