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Compressed Gas Safety at the University

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ABSTRACT: Academic institutions are faced with the complex problem of accurately determining the hazards associated with a proposed or ongoing project. For many reasons, compressed gas safety can be a significant challenge at universities, especially those that involve research using many uncommon materials. This article is a call to action for implementing better safety practices when using compressed gases and helps to fill the gap on resources related to compressed gases. General operating procedures and resources for safe practices when using compressed gases are outlined in this article, as well as lessons learned from incidents involving compressed gases.

KEYWORDS: *General Public, Safety/Hazards, Gases, Chemical Technicians, Laboratory Equipment/Apparatus, Laboratory Management*

INTRODUCTION

At the University of Hawaii, a research lab used a process that combined hydrogen, carbon dioxide, and oxygen gases to create a feedstock gas for growing cells. This mixture had been used in the lab since 2008 and was originally fed directly into the growth chamber using flowmeters for each of the three gases. In 2016, the researchers began to mix these gases together directly by combining the gases from high-pressure cylinders in a lower-pressure container. Three months after they implemented this new process for preparing the feedstock gas, a spark ignited the mixture.[1] The postdoctoral researcher working in the lab lost her arm in the resulting explosion.

This incident is an example of not taking proper safety precautions into account when designing new protocols in the lab. It is well-known that the safety regulations require that a flammable gas be separated from an oxidizer gas by a distance of 20 ft or by a fire partition.[2−4] Basic compressed gas safety guidelines that include this recommendation were printed in the University of Hawaii Report, which have been reproduced in the Supporting Information. At the University of Hawaii laboratory, the hydrogen and oxygen cylinders were separated by the recommended distance. So why was it deemed safe to mix them together in a cylinder at pressure?

In their report, the investigation team concluded that there were numerous deficiencies related to safety protocols that led to the incident.[5] As noted in the report (ref 5, p 3), the

> *Investigative Team concludes that serious deficiencies in the institution's approach to laboratory safety contributed to a lapse in proper risk assessment and lack of a culture of safety that ultimately led to the accident. The Investigative Team noted systemic problems pointing to an overall lack of effective safety oversight at the UH campus, including insufficient training in hazard recognition and risk mitigation, poor gas cylinder safety, a deficient laboratory inspection program, a dated and ineffective chemical hygiene plan, and inadequate standard operating procedures (SOPs). Of particular significance for this accident was an absence of formal risk assessment protocols in place for processes involving highly hazardous chemicals such as explosive gases.*

This tragic incident is not unique. In 2015, a postdoctoral chemist died after a hydrogen cylinder exploded in a lab at Tsinghua University.[6] In a second, similar incident in 2019, a senior Israeli researcher was badly burned and died in a hydrogen explosion.[7] Despite a multitude of accidents in academic research laboratories over the past decade, there is little evidence of change in safety practices.[8] Investigations into the causes of these incidents and the conditions surrounding them point to a number of pressure points related to compressed gas safety at universities.

The Unique Challenge of Compressed Gas Safety

The great variety of compressed gases and their storage presents the first challenge in handling compressed gases. Today, there are over 200 different gases supplied in cylinders that have multiple hazards and can be packaged

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in cylinders ranging in size from small lecture bottles (440 cc) to a large 49 L cylinder for use in university laboratories. In industry, even larger cylinders of up to 40 ft tube trailers can be used. Some of these gases may have little to no safety or environmental information since they are being used in research and are produced in limited quantities on bench scale systems.

Like any chemical, there are risks associated with using compressed gases in the laboratory. An EHS manager must be capable of understanding and assessing the risks for many activities involving compressed gases, some of which are novel. While each college controls the use of compressed gases, they may have a safety policy that differs from university policy. Many major universities have over 1,000 laboratories and several different colleges involved in a diverse range of research activities (e.g., biology, pharmaceuticals, chemistry, batteries). The purchase of compressed gases and EHS oversight is often not centralized even at the college level, with department-specific policies. This can lead to missed information, as different universities, departments, and even laboratories receive differing information related to safety. An example of the challenge that a lack of centralization leads to includes the fact that many researchers may not be aware of fire regulations such as the recently adopted 2018 edition of the International Fire Code (IFC), Chapter 38 Higher Educational Laboratories, and the 2019 edition of the National Fire Protection Association (NFPA) 45, Standard on Fire Protection for Laboratories Using Chemicals, which contain requirements that are applicable to academic laboratories.

The Challenge of an Academic Setting

Despite safety ranking as a top priority for surveyed researchers,[8−10] studies suggest that researchers are unlikely to conduct safety assessments[11] or look for additional safety information for developing their experimental procedures.[12] In fact, survey responses seem to suggest that chemists have come to accept that accidents and near-misses in the laboratory are simply to be expected.[13,14] This is an attitude that is exacerbated in academia, as academic researchers self-reported that they are less likely to assess risk in comparison to their industry or government counterparts.[15]

This culture is largely unchallenged in academic research laboratories. No comprehensive data set of laboratory incidents exists, in part because sharing this information with the public is voluntary. Oversight and responsibility for what happens in an academic research lab vary, as the large regulating bodies such as OSHA do not apply to all employees, and university policies on safety protocols differ across the nation.[8] There are few studies on safety programs that have been implemented at universities, and the safety programs that are published do not always report on their efficacy.[8] In many cases, safety training for researchers consists of anecdotes and informal sharing of safety knowledge.[8,16]

Many have pointed to principal investigators as a major barrier to changing safety practices.[17−22] Variables such as a lack of knowledge related to safety protocols, lack of funding to investigate safe practices, lack of time to engage in safe practices, and apathy toward changing behavior all contribute to this hurdle.[11,14,23,24] The perception of PIs as all-knowing further exacerbates the problem, as principal investigators and researchers are assumed to be knowledgeable in all areas of their laboratory, including safety. Even if they sought assistance, where would they turn? As Dr. Craig Merlic, Executive Director, University of California Center for Laboratory Safety, and others have indicated,[25] we need to build an effective support system for openly talking about laboratory safety practices in order to dispel the negative associations tied to conversations about safety practices. Far too many accidents have occurred because investigators do not participate in these conversations for fear of appearing ignorant.

Chemical research is a dynamic process, presenting additional challenges for safety. As Ralph Stuart, the Environmental Safety Manager for Keene State College, noted in a DCHAS listserv email[25]

> *I have seen many situations where the research process has drifted from the original proposal and a laboratory is doing very different chemical work shortly after a project begins. This drift often occurs under the radar of the institutional oversight process (including the PI of the lab) and with inadequate funding, as funders decide to only partially fund the work being proposed.*

Thus, even when oversight is in place, it is inadvertently circumvented as research protocols are adapted to new findings or changes in funding or instruments. Adapting safety protocols accordingly becomes an issue of timing as well as complexity.

To complicate this situation further, every year there are new students entering the university system. These students either have never handled cylinders and must be trained or are students who may have been at another university with a completely different safety policy. While standard laboratory safety training is generally available (and required), it is typically up to the principal investigator to ensure that students are properly trained on safe practices for the chemicals being used in that lab. At most universities, there is little oversight to ensure PIs are properly training their students on safety practices specific to their research.

Lack of Developed Resources and Training

Many articles have criticized safety practices in academic laboratories and the lack of training and resources for improving lab safety.[26−29] This extends to using compressed gases in the lab. Only recently has lab safety training begun to include information related to gas cylinder safety. A recently designed curriculum for laboratory safety training for graduate students spans 14 class periods, and gas cylinders and compressed gases are touched on in two classes.[30] While it is not necessarily comprehensive of all types of compressed gases academics may encounter, it covers basic handling for gases.

Even when a robust program for laboratory safety like the one developed by Hill and colleagues exists,[30] publications, programs, and training dedicated for compressed gases are rare. A recent search of peer-reviewed chemistry journals revealed that articles that specifically cover compressed gases and cylinder safety were published over 30 years ago and focus on basic handling of gas cylinders only.[31,32] When novel experimental protocols are published that involve the use of hazardous materials, they rarely include information on how to safely handle these substances in the lab.[8,33] This is due in part to the novelty of uses for compressed gases in research, and subsequent training to address these varying uses is slow to be developed. Additionally, some gases are used in conditions that greatly deviate from ambient. Accounting for these different conditions makes safety preparations more complex. At this time, no common training tool for compressed gas safety exists for universities.

Call to Action

From a process safety perspective, we can all agree that a minimum set of safety rules when using compressed gases should be followed, such as labeling for hazards, compatible materials, pressure rating, and cylinder prefill inspection. However, this basic documentation does not exist for many procedures involving compressed gases. Including standard safety procedures and involving the relevant stakeholders in the development of research form the foundation for safe handling of compressed gases.

This article seeks to fill some of the gaps in knowledge about compressed gas safety by providing information related to more specialized compressed gases and cylinders. This builds on the work of previous articles that outline key safety practices[9,10] by expanding on these practices and providing examples of additional safety issues commonly encountered by the first author. First, standard operating procedures to follow when compressed gases are being used are outlined. This is followed with examples of safety violations that the first author has encountered in the field and the lessons that can be learned.

■ STANDARD OPERATING PROCEDURES

Work with highly hazardous chemicals (including compressed gases) requires clear and established standard operating procedures (SOPs) that are uniformly implemented by all individuals throughout the entire organization who are working with the same hazard. These procedures should take into account the severity of the hazard and aim to minimize the risk of an incident. They should include the following:

- SOPs with a step-by-step breakdown of the experiment including a hazard analysis dependent on the hazard concentration
- A description of the amount, concentration, and circumstances in which the chemical is known to create a hazardous event (e.g., toxicity, explosion, fire)
- The equipment to be used with a justification for safety selection
- Appropriate safety barriers and other worker protection (PPE)
- Emergency procedures in case of an unforeseen event

An example of the types of questions researchers should be able to answer before using compressed gases in their laboratory is outlined in Box 1.

Researchers should be trained and demonstrate proficiency in performing the SOP before working with the hazardous chemicals. In the event that the procedures involving the hazardous materials need to be modified, a written amendment should be provided. This is called a "Management of Change Amendment" and describes the planned change to the SOP.

Other important SOPs include involving relevant safety personnel at the university. Principal investigators should involve safety personnel in the proposal stage of their research operations to make sure they are prepared to safely conduct the work they are proposing. As Ralph Stuart noted[25]

Box 1. Examples of Questions to Answer before Using a Compressed Gas, Such as Anhydrous Hydrogen Chloride

- What size cylinder is needed?
- How do you purge the system?
- What are the materials of construction for the system? Pressure rating?
- How will the compressed gas be used?
- Will the compressed gas react with other chemicals in the system?
- Will there be gas detection to monitor for leaks?
- Is there emergency response equipment available that is appropriate for responding to the hazards of this compressed gas?

I often find that my role as the safety professional is to help develop a more realistic estimate of the cost associated with specific lab work than the best case scenario that research budgets are often built around. These new estimates are often necessary because of code requirements and regulatory expectations that are not part of the scientific planning done by the researcher... Stakeholders such as emergency planning and response services, the institutional waste disposal program, and ventilation system managers all have legitimate interests in assessing whether the work is being done in ways that conform to their plans and capabilities.

Continued engagement with safety personnel will help ensure that SOPs are up-to-date and are being implemented effectively. A regular unannounced walk-through should be done by a safety committee member to emphasize the importance of safety and to gain a realistic impression of ongoing operations. Stop work protocols require the researcher or PI to cease all work involving a highly hazardous chemical or process in the event of a near-miss, unexpected problem, or otherwise observed highly unsafe situation.

Working with Unique Compounds

Some researchers synthesize unique compounds that have little to no safety and health information. A large, R1 (research 1) university has the following policy for ordering new chemicals for the cleanroom:

Before placing an order for any gas, determine whether it falls within the hazardous gas definition. If it does, verify that it appears on the current List of Approved Gases and Quantities. Any gas not listed is prohibited for use and may not be ordered without a prior Process Hazard Review application and subsequent approval for use by the Cleanroom Operations Advisory Committee. In case of an urgent need, temporary approval can be granted subsequent to a review and approval of the emergency proposal by the Cleanroom Coordinator and one Cleanroom Operations Advisory Committee member who is not directly associated with the laboratory making the emergency request. The temporary approval will undergo a mandatory full review within 30 days and may be subject to revocation. If approved, it will be added to the List of Approved Gases and Quantities. The total quantities of gas in use may not exceed the approved amounts without another Process Hazard Review.

This protocol can be used by other institutions and is a good first step to ensuring safe practices for working with new compressed gases in the laboratory.

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Available Safety Training and Resources for Working with Compressed Gases

Safety training for the more common gases such as nitrogen, helium, or hydrogen is available from many gas suppliers. The Airgas ACE program is an excellent training tool as well as a resource for the gases and packages that they sell.[34] Once registered, the user can download information cards on cylinder valves, cylinders, and specific gas properties.

The Compressed Gas Association (CGA) has several web-based training presentations on compressed gas safety.[35] These presentations cover the following major areas of compressed gas safety:

- Cylinder and container examples;
- Cylinder and container markings;
- Equipment examples;
- Understanding labels, symbols or pictograms, and product classification;
- Moving and storing cylinders and containers;
- Connecting and using cylinders and containers;
- Emergency response and site security; and
- Regulations and additional references.

While many researchers rely on local vendors to provide them with the more common gases, for many of the exotic gases they have little to no knowledge of the gas or package which they source from a larger gas supplier. With the consolidation of the gas industry, many of the industry experts have retired, and their knowledge has not been passed along. References that contain safety information for more unique compressed gases are included in the Supporting Information, as well as a list of additional excellent reference sources that are no longer in print but may be found online. The authors recommend that readers utilize the suggestions related to compressed gases in this article and the Supporting Information in conjunction with the cited references that outline the basic protocols for safely handling compressed gases.

■ LESSONS LEARNED IN THE UNIVERSITY CONTEXT

The following sections contain examples of safety problems that have been encountered during 35 years of contracted expert visits to university laboratories. The examples do not cover well-known safety issues, such as not using a cylinder cap during storage and handling of compressed gases or a lack of proper separation of flammable gases and oxidizers. Instead, these examples focus on more specialized incidents and lessons that can be learned from them.

Filling Cylinders

Gas cylinders are typically owned by the gas supplier. The US Department of Transportation (DOT) regulations require that the cylinder owner approve any refilling of their cylinders. Under 49CFR173.301(b)1, there is no prohibition against charging a cylinder without the consent of the owner of the cylinder, provided the charged cylinder is not offered for transportation in commerce. This has led to the common practice of researchers filling cylinders without proper training or safeguards. Very few universities have procedures in place to control this activity.

Dangerous Gaseous Mixtures Created in the Lab

Researchers improperly filling cylinders can lead to many hazardous conditions. The most frequently observed problem with researchers filling their own cylinders is the creation of dangerous gaseous mixtures. Although they used their own cylinders and not those of a gas supplier, the incident at the University of Hawaii is an example not taking into account whether the gases were safe to mix together in a cylinder.

Researchers may accidentally create a mixture that, while not dangerous on its own, can damage the cylinder itself. It is common knowledge that it is not safe to mix an acidic gas such as hydrogen chloride with an alkaline gas such as ammonia. Very few are aware, however, that a pollution calibration gas mixture containing carbon monoxide and carbon dioxide can be extremely dangerous when pressurized in a carbon steel cylinder. This can cause stress-corrosion cracking of the carbon steel, which creates numerous microscopic cracks throughout the cylinder. The mechanism is proposed to be local dissolution of iron due to the carbonic acid formed between water and carbon dioxide, with general corrosion being inhibited by carbon monoxide. This phenomenon normally leads to transgranular cracks with branching. It is difficult for researchers to know that these cracks are forming. A carbon steel cylinder filled with a mixture that includes carbon monoxide and carbon dioxide could catastrophically fail without warning,[36] as shown in Figure 1. This is an example of why researchers must consider the interactions of the mixtures they are creating with the material of the cylinder they are storing them in.

Figure 1. Catastrophic cylinder failure from stress-corrosion cracking.

As a result, the US Department of Transportation has limits on the pressure that it can be filled to, 49CFR173.302-(c). The CGA also has guidance on how to fill these cylinders safely: CGA P-57, Avoidance of Failure of Carbon Monoxide and of Carbon Monoxide/Carbon Dioxide Mixture Cylinders.

Dangerous Gaseous Mixtures Prepared by Suppliers

Other incidents include mixtures that were prepared by the gas suppliers and not safe for use. The Indian Institute of Science (IISc) in Bengaluru conducts shock wave research using a high-pressure cylinder containing a stoichiometric mixture of hydrogen (67%) and oxygen (33%) at 120 bar. On December 5, 2018, this cylinder exploded, killing the operator and severely injuring 3 others as well as causing severe damage to the facility. This mixture had been used for many years without incident.[37]

Explosive gas mixtures can exist indefinitely without ignition; even impact and transport will not ignite it. In 1970, a silane safety testing program mixed silane, oxygen, and argon in a cylinder. After 30 min, a 25 lb weight was dropped onto the cylinder without igniting the mixture. In two tests, the mixture ignited when the valve was opened to vent it. One cylinder shattered into pieces while the second one bulged.[38] The silane/nitrous oxide mixture cylinder that killed 3 people and severely injured one in the Gollub incident on March 17, 1988, had been shipped around the US for over 4,000 miles.[39] It was not until the cylinder valve was opened one more time that the adiabatic compression heat ignited the mixture and the cylinder exploded.

Explosive gas mixtures continue to be prepared and shipped around the world. Tylar Gas was a California-based company that prepared and sold stoichiometric mixtures of hydrogen and oxygen in high-pressure cylinders for fuel cell research. Despite two explosions, they continued to ship these cylinders around the world until there was a third explosion.[40] The difficulty in igniting these explosive gas mixtures creates a false sense of security, and researchers must take into consideration the hazards of the gas mixtures they are using in their work.

Improper Labeling of Gas Cylinders

Another safety hazard that arises from researchers filling their own cylinders is improper labeling. Marking cylinders using a sticky note or other impermanent label on the cylinder body or tag is inadequate to convey information about the contents of the cylinder to others working in the area, safety professionals, or first responders. These can easily fall off, and the only information remaining will be the shoulder label indicating what the cylinder was originally filled with. It likely will be returned to the gas supplier with this gas mixture in it possibly creating a dangerous condition if they connect it to a manifold with an oxygen cylinder, believing that it still contained an inert gas. In other cases, the cylinders may be sent back overfilled or not have the proper valves or pressure relief devices attached. This creates potentially dangerous situations for responders, gas suppliers, and others encountering the cylinder. The cylinder in Figure 2 was originally filled with an inert gas (nitogen) by the gas supplier, but it now contains a 95% mixture of hydrogen and carbon dioxide a flammable gas mixture. If the tag labeling the cylinder is removed, responders and gas suppliers will not realize that the inert gas has been replaced with a flammable gas mixture. The cylinder has a CGA 580 outlet, which is prescribed for nitrogen; hydrogen has a CGA 350 outlet connection. The pressure relief device (PRD) required for nitrogen is a CG-1 rupture disk, whereas hydrogen must have a CG-4 PRD a fusible metal/rupture disk combination. Because the cylinder was originally designated to be filled with nitrogen, its connections are all designed for nitrogen and give no indication of the new mixture. Should a supplier connect this cylinder to a system that contains an oxidizer, it could become an explosive gas mixture.

Best Practices for Filling Cylinders

Before filling a cylinder, researchers should be able to answer the following questions regarding the gases they are using:

- What is safe to mix together?
- How much can be put into the cylinder?

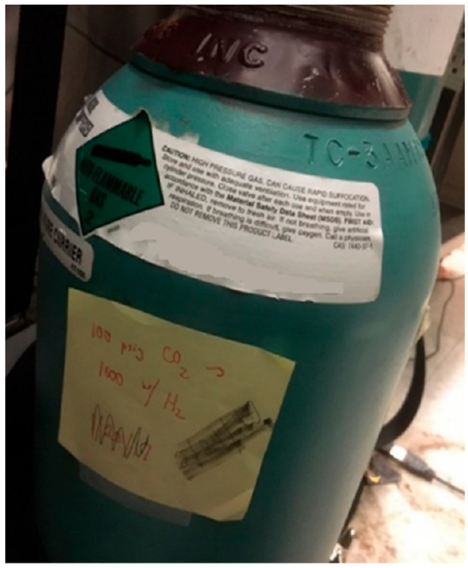

Figure 2. Illegally filled nitrogen cylinder.

- What type of valve/cylinder is compatible with the gas?
- How often must the cylinder be tested?

Universities must prohibit the filling of a gas supplier's cylinder unless the gas supplier has specifically requested it. Before filling any cylinder, including one authorized by a gas supplier, a Hazard Operability study should be conducted to define the following:

- Types of cylinders and valves that can be used
- Whether the cylinder is within the hydrotest date
- Filling safeguards
- Labeling and markings
- Disposal

Chemophobia

Many researchers use toxic and highly toxic gases without an understanding of the symptoms of exposure or how to medically treat for an exposure. Of all the toxic gases, arsine (AsH_3) psychologically is of greatest concern due to its toxicity. As a result, arsine is handled very carefully. Arsine has an olfactory threshold of 0.5 ppm and has an odor that is not unpleasant or irritating.

While many other gases in common use (e.g., hydrogen selenide, diborane) have acute exposure levels well below that of arsine, most people believe arsine is more dangerous. People associate it as a chemical warfare gas that was used in World War I, or confuse it for the chemical arsenic, which can act as a poison. These negative associations lead to additional fear of using arsine in the laboratory. The symptoms of acute exposure are also not immediately apparent; as a result, one can be acutely exposed and not be aware. This likely increases fear of handling arsine.

As a result, a "non" incident in 1993 resulted in bad news coverage and public perception at a major university in the US. The university was starting up a new gas supply system for the metal organic chemical vapor deposition (MOCVD) laboratory. While installing an arsine cylinder, the gas detection system went into alarm at 1/2 the permissible exposure limit (PEL) level of 25 ppb. The emergency response (ER) team responded but could not find a leak. Some graduate students thought they smelled arsine. A few hours after the alarm, they contacted the hospital about being

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exposed to arsine and drove themselves to the emergency room. The hospital went into a panic, thinking they had victims contaminated with a highly toxic gas that could cause secondary exposure. They quarantined the victims, proceeded to evacuate the wing of the hospital, and called in additional workers to deal with the crisis. Hours later, no one had checked on the "victims" due to the chaos, so they simply walked out of the emergency room. The first author was contacted to be part of the investigative team, which never found any evidence of arsine exposure or a leak.

In another incident at a German university on Jan 19, 2012, an accident occurred during a demonstration for a freshman chemistry class. Some students reported smelling a garlic-like odor. People became concerned that it was arsine. Almost 100 people (mostly students) were transported by 30 buses and ambulances to area hospitals for observation, many staying overnight. Over 100 firefighters and 70 other government agencies responded. No evidence of arsine was found. The first author was the lead accident investigator for a large (65 lbs) arsine release that occurred on July 11, 2001 in Catoosa, OK. Over 100 people reported to the local hospitals for possible arsine poisoning. None were found to have elevated arsenic levels.

Best Practices for Providing Medical Treatment Information

As a minimum, users of highly toxic gases should have available medical treatment information that is available to all users and responders. An excellent source is the US Health Dept Agency for Toxic Substances and Disease Registry (ATSDR) Medical Management Guidelines (MMG).[41] These are comprehensive medical treatment protocols developed by medical doctors primarily for use by emergency medical technicians (EMTs), paramedics, and medical doctors to diagnose and treat acute chemical exposures. These are complementary to Safety Data Sheets (SDS) since they focus on issues that a responder or medical professional would be concerned with such as secondary exposure or decontamination. MMGs for the highly toxic gases arsine, diborane, hydrogen cyanide, nitrogen oxides, phosgene, and phosphine, along with other toxic gases, are available as PDF files.

Lecture Bottles

Standard cylinders have been designed with safety in mind. In the US, CGA outlet connections were designed to prevent incompatible gases from being connected together on a system (e.g., hydrogen with oxygen). Over 54 connections have been established for industrial/specialty gases, which cannot interconnect with each other to form a gas-tight seal. Unique connections are formed by using various combinations of the following: male/female threads, varying diameters, different nipple shapes, and right/left-handed threads. For example, hydrogen cylinders typically use a CGA 350 connection, which is a male exterior left-handed thread, while oxygen is a CGA 540 connection, which has a similar diameter exterior thread but is right-handed. A gas-tight seal can be created via different methods, such as bullet nose deformation or by using a flat gasket, which can also be used to make unique connections. Medical gases have Pin Index outlets, which is a yoke-type connection utilizing a flat gasket and a series of pins or holes to prevent interchange of incompatible cylinders. More information on the appropriate valves and connections for pure and mixed gases can be found in the CGA Reference: V-1 "Standard for Compressed Gas Cylinder Valve Outlet and Inlet Connections".

Lecture bottles are one type of cylinder for compressed gases. Lecture bottle are high-pressure cylinders typically made to DOT 3 × 10-1800 specifications. They are approximately 440 cc in volume and hold less than 500 g of gas. These are convenient for a university as they contain a very small quantity of the gas. Lecture bottles are often used for classroom demonstrations.

Lecture Bottle Connection Compatibility

Lecture bottles do not follow the same specifications as standard gas cylinders. Lecture bottles are exempt from having pressure relief devices by DOT. In addition, there are only two valve outlet connections, CGA 170 and 180. These types of outlet connections are shown in Figure 3. Another

Figure 3. Lecture bottle valve with CGA 110 F and 180 M outlet connections.

type of cylinder is the 7X. 7X cylinders are lecture bottle bodies, but they have standard cylinder valves with the required outlet connection and pressure relief device to prevent incompatible gases from being connected. A typical cylinder is shown next to a 7X cylinder in Figure 4.

The design of lecture bottles presents potential problems that have been accounted for with the design of standard cylinders. In Figure 5, the lecture bottles contain nitric oxide and nitrogen dioxide which are highly toxic and strong oxidizers, carbon monoxide which is toxic and flammable, and hydrogen which is flammable. Since they all have a CGA 110 F and a 180 M connection, a researcher could connect these incompatible gases together in a system. High-pressure hydrogen could backflow into the nitrogen dioxide cylinder since it is a low-vapor-pressure liquefied gas, creating an explosive gas mixture.

Forgotten Lecture Bottles

While lecture bottles are technically refillable, many gas suppliers will not take them back due to the cost to clean and refill them. Because the lecture bottles are not returnable, most researchers do not have the funds to pay for disposal of the cylinders. As a result, lecture bottles are stored away in a laboratory drawer and forgotten. This creates a very unsafe

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Figure 4. Lecture bottle (left) and 7X cylinder (right).

Figure 5. Lecture bottles containing highly toxic, oxidizer gases and flammable gas.

condition where the lecture bottle can leak and corrode over time or the labels become unreadable. Some gases such as hydrogen cyanide have a stabilizer that degrades over time.

Anhydrous hydrogen fluoride (HF) stored for long periods of time (20 years) in lecture bottles has been involved in a few explosions[42−44] that have caused significant laboratory damage. Figure 6 contains a photo of a cylinder from one such incident. Hydrogen fluoride is a liquid (0.8 psig vapor pressure at 21 °C) that slowly reacts with the carbon steel cylinder and moisture to form hydrogen and iron fluoride. This will thin the cylinder walls and pressurize the cylinder until it ruptures after many years in storage. A waste disposal company in the US started to monitor the pressure of all HF cylinders sent for disposal. Of the 60 lecture bottles received, 14 had pressures >1,000 psig while 3 cylinders had pressures >3,000 psig. It should be noted that this may not be an

Figure 6. HF cylinder rupture at a major US university July 2005.

accurate reflection of the dissociation as it was not known which cylinders were still full and unused. Since HF is a low-pressure liquefied gas, most systems used to handle the gas will have a pressure rating of 100 psig or less. These cylinders could have violently ruptured the systems. HF cylinders that are more than 2 years old must be handled with care. The best practice is to return the cylinder after 2 years unless it is in use and the pressure is relieved on a regular basis.

It is important to note that the standards outlined earlier apply to US cylinders. Cylinders from other countries may have outlet connections as defined in their national standard. For specialty gases, the more common connections from Europe are German DIN (Duetch Industri Normen) or from Asia Japanese JIS (Japan Industrial Standard).

Best Practices for Using and Storing Lecture Bottles

Lecture bottles should be purchased only from gas suppliers that will take them back. When received, they should be adequately labeled with their hazards and the date of purchase. NFPA 45 allows a laboratory to have up to 25 lecture bottles in use. Most lecture bottles should be located in a laboratory hood with a clamp or stand to hold them upright. Lecture bottles not in use shall be stored in a well-ventilated area, with a fire sprinkler; this area should be dedicated for compressed gas storage as per the requirements of NFPA 55. They are separated by hazard class: flammable, oxidizer, corrosive acid, corrosive alkaline, highly toxic, and pyrophoric. The lecture bottles should be visually inspected weekly for leaks and returned after two years to the supplier.

Spun Pressure Gauges

Working pressure gauges are an important tool for safe handling of compressed gases. For safety, researchers using compressed gases must know the pressure at any point in their system to ensure that the design pressure of the system is not exceeded. Most research systems are not designed for full cylinder pressures of 2,000 psig or higher. Quite frequently, the first author has found pressure gauges that have been mechanically damaged and the needle has been spun (Figure 7). This happens when the maximum pressure of the gauge has been exceeded. This damages the needle or bourdon tube so that they are no longer working. When this happens, the following questions must be answered:

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Figure 7. Spun pressure gauges.

- Why did this happen?
- Will it happen again?
- Has it been fixed?
- Has it damaged anything else in the system?
- How does the user know what pressure the system is at?

Sometimes the system might not have the appropriate pressure relief, causing the gauge to spin. After the cause of the spun pressure gauge has been determined and system has been fixed, a new pressure gauge must be installed. The new pressure gauge should be double the expected operating pressure, and if necessary, a pressure relief system should be installed.

Regulators

Regulators are used to lower the cylinder pressure to a safe use pressure. Typically, a high-pressure cylinder at 2,000 psig is lowered in pressure using 2 single-stage regulators. Many users would have a single-stage regulator at the gas cylinder to step it down to 100 psig. A second regulator in the instrument or system steps it down to the desired use pressure.

Incorrect Regulator Attached to Cylinder

During an inspection at a US university, the first author was horrified to find a single-stage regulator attached to a high-pressure cylinder (see Figure 8). The PI found this regulator and adapted it for use.

Issues with this setup include the following.

1. A station regulator is designed to be installed in a piping system after the cylinder pressure is reduced to pressures of <200 psig. This regulator was clearly marked with a maximum rating of 200 psig. In general, pressure equipment will burst at 4 times the service pressure, in this case 800 psig. Typical high-pressure cylinders like the one in Figure 10 are filled to 2,000 psig.
2. A CGA 580 connection was attached to the station regulator which would allow it to be connected to any high-pressure cylinder; however, as noted earlier, the regulator is meant to be installed in a low-pressure piping system.
3. A piping adapter was fabricated to connect the CGA 580 to the cylinder CGA 296. CGA prohibits the use of adapters for connecting cylinders.

Figure 8. Single-stage regulator adapted for high pressure.

4. An NPT pipe thread was used rather than the appropriate CGA 296 connection. A CGA 296 connection is a straight threaded connection that seals at the nipple. An NPT thread is tapered, sealing is at the threads which are mechanically deformed as it is threaded onto the connection.
5. An oxygen mixture with a concentration >23.5% is an oxidizer. This cylinder, which had a 25% mixture, was appropriately labeled as an oxidizer. Gas equipment for oxidizer gases must be designed and cleaned for the oxidizer service as described in the following section.

Contamination of Valves and Regulators

Adiabatic compression occurs every time the cylinder valve of a high-pressure gas cylinder is opened and gas flows into the low-pressure downstream piping, rapidly pressurizing that system. Adiabatic compression heat occurs because the pressurization of the gas occurs so rapidly that there is no time for the heat of compression to dissipate into the surrounding piping and valves.[45] This process is illustrated in Figure 9.

Oxidizer systems should also be designed without the use of quick opening valves such as ball valves. This rapid compression of the gas generates elevated temperatures that can ignite a flammable contaminant in the system due to the lower autoignition temperature in the presence of the oxidizer or initiate the decomposition reaction of a gas like nitrogen trifluoride (NF_3). NF_3 cylinder pressures have been limited to 1450 psig to decrease the potential for the decomposition reaction to be initiated due to the heat.

Figure 10 shows the type of damage that can occur when a contaminated regulator is connected to a system with an oxidizer gas.

Best Practices for Using Regulators

The best practice for using regulators is to dedicate them for specific gas services. Valves and regulators used for oxidizer service must be cleaned for oxygen service before they are assembled by the supplier. Once they are in that service, they should be marked and dedicated for that service. To avoid contamination when they are no longer installed on a system, they must be stored properly. Researchers should avoid changing the outlet connection. Researchers should avoid

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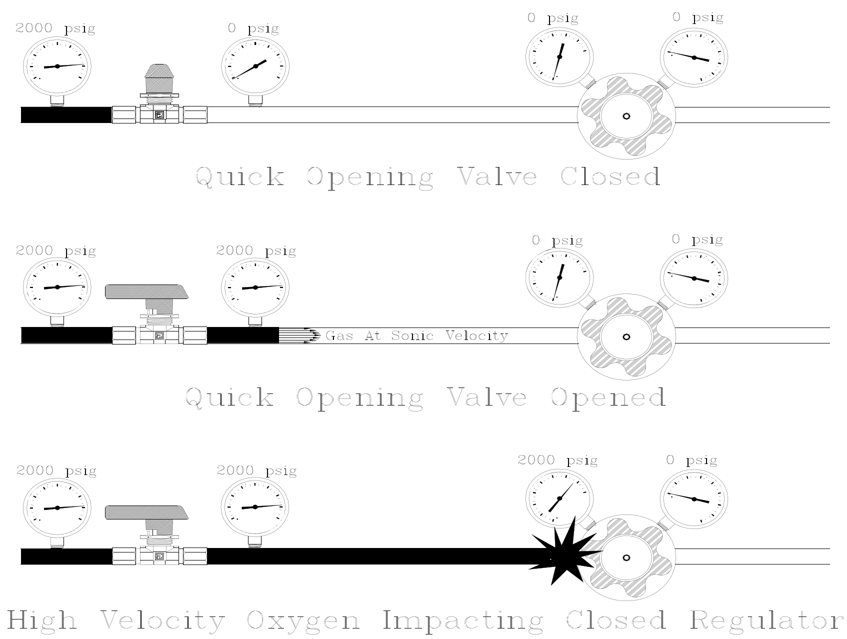

Figure 9. Adiabatic compression.

Figure 10. Regulator used for another gas service was adapted for oxygen use; it exploded when the cylinder valve was opened.

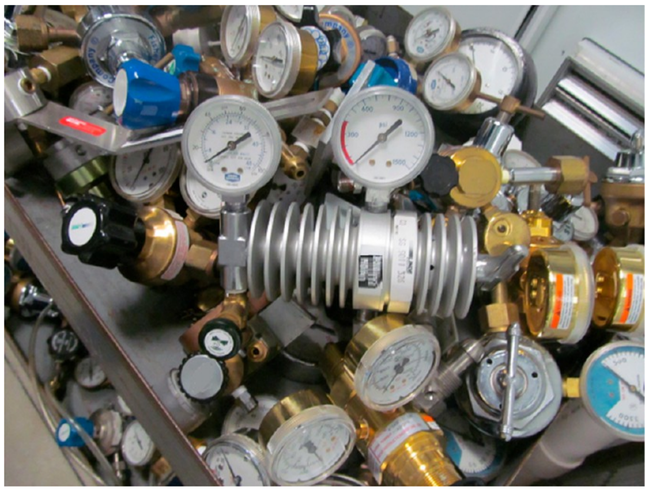

Figure 11. Regulators in a cart.

accumulating used regulators without properly cleaning or labeling the regulators, as seen in Figure 11. Researchers or students will pick through the cart for their use with little to no knowledge of its history or the materials of construction.

■ CONCLUSION

In conclusion, it is recommended that universities develop and implement a robust safety program that involves the relevant stakeholders. Many of the incidents outlined in this article happen infrequently, so people are unaware of them. Valuable information about chemical safety is often published in the form of an alert that do not always make it to the end users. Looking forward, communication is important for establishing a clear understanding of incidents and how to prevent them in the future. A forum such as the ACS DCHAS Listserv[25] or the website The Safety Net[46] has value for communicating safety issues as well as bringing together people who are knowledgeable in specific areas. For this type of knowledge dissemination to be effective, however, researchers must be willing to contribute their expertise. This can aid in keeping safety training up-to-date for using chemicals such as compressed gases, ensuring that this knowledge reaches all end users.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.0c00138.

Full email text from the January 1, 2019, listserv as well as additional recommended resources for compressed gas training (PDF, DOCX)

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Notes

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