

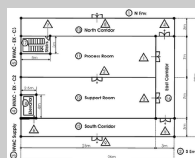


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Process Safety News

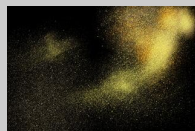
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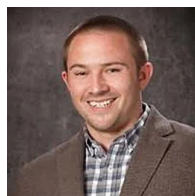


ARTICLE:

Process Safety Scale-Up Aspects of an Epichlorohydrin Hydrolysis Reaction - Part 2

Letter from the President:

When we released our Winter issue of the FAI newsletter in February, the term "social distancing" had little meaning to most of us. Fast forward to today and it's a concept that guides daily interactions in both personal and professional settings. It's pretty amazing how quickly our lives can change. As we work through this pandemic, the need to change



continues to evolve and like many others we at FAI are trying to define what the "new normal" will look like. Yet many aspects remain unchanged. Our collective obligation to safely operate chemical, nuclear, and other industrial facilities is the same, but the way we go about it will likely be different. Change management has always been a critical aspect of a healthy safety program, and it is important to not lose sight of its significance during times of uncertainty. Whether change is triggered by new demand schedules, new product formulations, or the identification of new hazards, a robust management-of-change process is designed to make sure the new way we're interacting with our process environment does not jeopardize safety.

FAI joins everyone in our communities in expressing a sincere thanks to all those who have been battling this pandemic on the front lines, often putting their own lives at risk. We also recognize and appreciate many of our customers who are serving in the background, working in industries that support the global response in everything from vaccine development to PPE manufacture. Finally, I'd like to personally recognize and thank our own team of essential workers who have continued to operate the FAI testing laboratories to produce process safety information that is critical for safe operation of the global supply chain.

- Zachary Hachmeister.

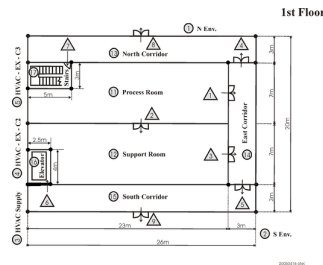
Facility Modeling and Aerosol Transport using FATE Safety Software

By Matt Kennedy, Senior Nuclear Engineer, and Dr. Jim Burelbach, CCO, Fauske & Associates LLC

In these uncertain times, the hustle and bustle of our daily lives has been interrupted, causing many of us to re-evaluate things we take for granted. Vacation planning, family dinner night out, and trips to movies, among other things, have been replaced by social distancing and self-quarantine with a N-95 mask and [an appropriate dose of hand sanitizer](#). Most of us share concerns on how to keep our family and friends healthy during a pandemic. Employers are concerned about the health and safety of employees. Many individuals and companies are finding unique ways to contribute to the effort. For example, individuals with sewing machines all the way up to larger factory production lines are being re-purposed to produce personal protective equipment (PPE) for health care workers and first responders.

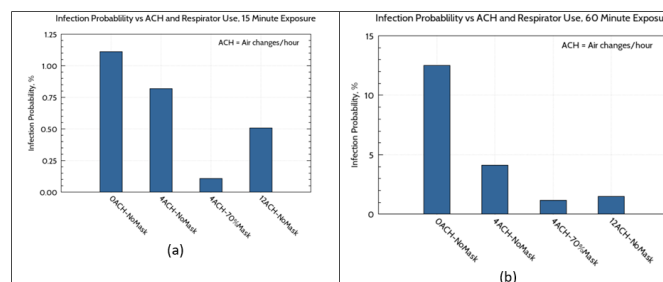
At Fauske and Associates LLC (FAI) it is no different. We have been looking into ways we can apply our expertise in chemical and nuclear process safety to re-focus existing analysis tools and products to help in the fight against the spread of COVID-19. One area where FAI can significantly contribute is in using the FATE computer code to model the spread of a virus within a building. FATE stands for facility Flow, Aerosol, Thermal, & Explosion modeling, and the FATE code (which received a DOE award for technology innovation) was developed under an NQA-1 compliant nuclear quality assurance program.

FATE is a versatile program used to analyze the transient behavior of facilities and buildings during normal and off-normal (accident) conditions. FATE has been widely used in nuclear safety studies to predict hydrogen accumulation and transport, radionuclide and smoke migration, and environmental conditions in nuclear waste storage facilities. Other safety applications have included hydrogen hazards during aluminum smelting, TICl4 process upsets (HCl gas transport), and safety analysis reports for a new medical isotope production facility.



A unique feature of FATE, which is particularly relevant to virus transport, is its ability to characterize and track aerosols, including deposition from settling, impaction, and filtration. The correlation in FATE compares remarkably well to experimental data commonly used to validate aerosol models, for example the Sandia AB5 test for sodium fire aerosols. The ability to model viruses as aerosols indicates that the FATE software can be used to estimate the chance of infection due to inhalation of an airborne pathogen that is spread through inhalation such as COVID-19, whooping cough (pertussis), measles (rubeola), tuberculosis, or influenza.

A simple FATE analysis predicting the probability of infection for a healthy individual in a ventilated room with a single infected person was presciently illustrated in a 2014 FAI blog titled "[A Simplified Airborne Infection Model with FATE Nuclear Safety Software](#)". The below figure from that blog summarizes the healthy individual's infection probability under various scenarios for different exposure times. The example analysis looked at different values for the air changes/hour (ACH): zero, 4, or 12. The larger the ACH the more often the room air is replaced by clean air through the building ventilation system (minimizing the risk of infection), and 0 ACH indicates the ventilation system has completely stopped (failed). One interesting parametric result considered when a healthy individual wore a mask (N95, but poorly fitted so considered only 70% effective). As expected the results show that the mask helps significantly reduce the probability of infection. Of course if the sick person had also worn a mask, consistent with current EHS guidance, the risk of infection would be further reduced.



This illustration demonstrates that virus spread via inhalation can be readily tracked and quantified using an existing validated tool. The analysis can be readily expanded to consider other situations:

1. Multiple rooms connected by a common ventilation system (cruise ships, offices, homes, meat processing plants, etc.)
2. Multiple sources (infected individuals) and receptors (healthy individuals) for analysis of crowded transportation systems (planes, trains, minivans, etc.)

3. Benefits of improved ventilation systems, like portable local HEPA filters, and better PPE practices

This type of analysis can quickly provide technically sound scientific guidance for individuals, companies, and government officials, not only as they develop solutions to mitigate the current pandemic but also to better prepare for other common viruses which pose health risks each year. FATE is well suited to this application since it is a fast running and validated code which captures the essential transport physics (including aerosols) without resorting to computationally expensive CFD calculations. This is particularly important for complex facilities and buildings with many interconnected rooms and for quick optimization of ventilation and filtering upgrades.

[Contact us to learn more](#)

Can Lessons Learned from Nuclear Power Plant Safety Testing Help Solve a National Water Crisis?

By Dr. Jim Burelbach, CCO, Fauske & Associates LLC, and Howard Heil, President, Heil2O Water Solutions

Water is a precious natural resource and a valuable commodity that is fundamental to life all over the world. But according to the American Society of Civil Engineers, our own water piping distribution networks in the US are graded “D-minus” meaning they are a near “Fail”.

Many of the more than one million miles of water main pipes in the US were installed in the early to mid-20th century with an expected lifespan of 75 to 100 years—and they are breaking at a current average rate of 240,000 annually.* If we do nothing, this number will likely rise as the aging pipes continue to deteriorate due to conditions such as:

- Corrosion from acidic soil and cathodic grounding from utilities
- Uneven weight of soil often caused by freeze-and-thaw cycles bearing down and creating uneven loadings on the aging pipes
- Water temperatures of 38° F or below that make cast iron pipes even more brittle

Even when pipes show no sign of deterioration, they will break when they are unable to withstand the rapid loading transients (water hammer events) caused by rapid starting and stopping of water flow to satisfy the needs of water users.

The American Water Works Association (AWWA) estimates it will cost \$1 trillion to maintain and expand service of US water mains to meet the projected water demands over the next 25 years. With the arrival of COVID-19 and a growing gap in funding, municipalities may be tempted to focus on priorities other than keeping up with the operation and maintenance of those essential pipes that transport clean water to homes, industries, hospitals, etc.

Main line breaks are especially costly and time-consuming for municipalities and dangerous for their work force. Flooding, road closings, water outages and boil-orders can also put residents and businesses at risk. For the sake of both public health and economic growth, utilities need to explore, when possible, less disruptive and less costly alternatives to total replacement of water mains.

Understanding water hammer could lead to a solution

Nuclear power plants (and many other industrial processes) use water in a variety of ways, basically to create steam heat that generates electricity and again to condense the steam once it has delivered energy to the turbine.** Thermal-hydraulic testing conducted by Fauske & Associates, LLC (FAI) in support of nuclear safety analyses has illustrated the potentially destructive effects of a hydraulics-based phenomenon called water hammer on supply pipes. We’ve also seen how suppressing the surge that leads to water hammer helps protect those same pipes from reaching their breaking point.



Could surge suppression then be a solution that, applied to the water industry, greatly prolongs the life of its infrastructure, saving perhaps millions of dollars and avoiding unnecessary water loss?

First, let’s look at what causes water hammer. A water distribution system typically consists of miles of pipes that are obstructed by the bends and T’s that are needed to supply water to the customers. Water systems also have valves and hydrants, with many of the valves being operated by high-volume users such as manufacturers, car washes or fire suppression systems.

When water is flowing at high velocity and a valve is activated quickly, or the water encounters a bend or a T, it is forced to slow down and is then followed by a surge forward with even greater momentum. These uneven pressure changes within the water utility system create a cavitation which leads to water hammer. The water, under immense pressure, seeks the weakest point in the system to find release. That’s when a main break is likely to occur.

Compare this with what happens in another type of liquid transport system. The petroleum industry distribution piping uses 10 times greater pressure (between 800 and 1200 psi) than the water industry and yet experiences very few main breaks. That’s because instead of using the T’s and 90° angles in pipes to serve multiple homes and industries, petroleum industry systems can be designed with more gradual transitions and they have far greater control of their valves.

It’s a common belief that cold temperatures cause mains to break. While we’ve proven that cold temperature (38° F or below) can make cast iron pipes brittle and cause breakage, cold weather isn’t the only factor. Houston averages only 18 days with temperatures of 32° F or less and yet it experiences more main breaks per mile than any other US city.

These examples—the petroleum industry with few main breaks and a warm climate city with many breaks—provide evidence that it’s primarily water hammer/cavitation that is causing much of the damage to our water infrastructure.

Taming water hammer with surge suppression

Engineers from FAI began exploring the role of surge suppression in preventing water hammer in the nuclear industry in the 1990’s. But before that, a public works superintendent for the rapidly growing

Village of Burr Ridge, Illinois, had been noticing early in his career how, with increasing demand for water, more main breaks occurred. After witnessing the repeated risk of water contamination and endangerment of workers' lives, Howard Heil, a farmer's son who grew up solving problems and making his own repairs, observed a pattern. He was especially intrigued by where main breaks *did not* occur—near elevated tanks and pumping stations. He also thought about what he knew about residential plumbing. A banging noise means the water system is missing an air-filled volume that absorbs the sudden change in pressure caused by turning water on or off quickly, as often happens in a kitchen, bathroom or laundry room.

In 1994, Heil began to introduce the same principle of surge suppression that he had noticed with water tanks and in residential plumbing to the Burr Ridge water system. While at a main break site, he observed how the pipe broke. If it was circumference break, which looked like a pencil breaking, or a blow-out break, which is when the pipe split open, he and his crew planted a stainless steel air encapsulation tank as part of the repair work. (He learned that corrosion breaks, evidenced by a rusty hole, can't be as easily prevented with surge suppression but could be postponed by it.) The result of his "Johnny-Appleseed" style work was that Burr Ridge experienced dramatically fewer breaks and less water loss, prolonging the life of their water distribution system and delaying unpopular special tax assessments on residents and businesses.



Science based backing of Heil's theories

In the early 1990's, Heil came to FAI's office which is located in Burr Ridge next to a water plant he supervised. While the purpose of his visit was to service a water meter, he began a conversation with employees about how he had installed a surge suppression device at the water plant next door. He shared his findings about how the device would mitigate the water hammer that, in this case, was increased by activation and deactivation of the water supply due to a recently added well.

Engineers from FAI became intrigued with Heil's work and he immediately recognized that FAI's technical knowledge and water hammer demonstration lab were complementary to his own practical experience. FAI's team worked with Heil and a manufacturer to develop an early model of the product, an informational brochure and even a tabletop display to demonstrate the science behind surge suppression. FAI also joined Heil at some of his early continuing education seminars to help "teach the teacher" in educating the water industry about the power of surge suppression to protect their mains.

In 1995, one of Fauske & Associate's founders, Dr. Robert E. Henry, a mechanical engineer and widely recognized expert in two-phase flow, concluded that surge suppression could greatly extend the life of water distribution systems and reduce maintenance costs. You can read his white paper report [here](#).

Heil had some success convincing other municipalities in and outside the Chicago area to try the surge suppression that worked so well for the village of Burr Ridge. He would follow up with them later to add data to his findings and develop ways to continuously improve his product. Early adopters include DuPage County Water, Oak Park and Hickory Hills, all in Illinois; plus, Ann Arbor, Michigan, and Green Bay, Wisconsin, and a handful of others throughout the country who have been using surge suppression for 20 years or more. Heil's follow up conversations confirm that these systems are experiencing fewer main breaks than before they installed the surge suppression devices.

Opening up to "out-of-the-box" thinking

Heil did not have the resources to keep detailed records and quantify the results. But he insists that if municipalities did so, they'd learn (like he did) where to place surge suppressors to calm water hammer and avoid the damage it causes to any vulnerable water distribution systems.

Technology to measure exactly when and where peaks and high pressures occur in liquid transport has long existed. Today this information can easily be viewed and tracked via computers. Why couldn't this data be used to more quickly and efficiently determine problem areas and proactively prevent a potential disaster? This may even be an opportunity to apply available machine learning technology from FAI parent Westinghouse LLC.

It is unclear why surge suppression is not being used more broadly to protect our nation's essential but near-failing water distribution systems. Heil continues to advocate for his patented water solution and educate himself further, including attending one of FAI's water hammer training courses, where he was the only attendee from a water utility organization. For him surge suppression is a common sense, no-brainer solution to a growing, worrisome problem. He believes that, unfortunately, the water industry is slow to change and too many decision-makers are stuck in the status quo.

But the need for life-sustaining water isn't going away. Essential infrastructure will continue to deteriorate as we struggle to regain economic stability and find the funding needed to operate and maintain water supply systems. One way to move forward is to learn from testing and scaling innovations such as surge suppression that would cost much less than entirely replacing mains and could save millions of dollars. In Heil's own words, "Prove to me it doesn't work."



Fauske & Associates, LLC (FAI) is pleased to support Heil2O Water Solutions in broadening the application of proven surge suppression technology.

For more information on surge suppression or water hammer issues in municipal water systems please contact Fauske & Associates LLC, info@fauske.com, or visit heil2owatersolutions.com.

Sources:

*Source: <https://www.infrastructurereportcard.org/cat-item/drinking-water>

**Source: <https://nuclear.duke-energy.com/2014/01/22/importance-of-water-at-nuclear-plants>

Don't Get Burned by Battery Fires, Test to New UL 9540A Standard

By TJ Frawley, Project Manager, Flammability Testing and Consulting Services, Fauske & Associates LLC

It is a perfect November morning in Glassboro, New Jersey. The alarm on your phone wakes you up fifteen minutes before class starts. More than enough time to roll out of bed, brush your teeth, throw your Rowan University sweat shirt on along with a pair of jeans, and make it to class with time to spare. As you walk out the door, you sub-consciously grab your headphones to set the mood for the not-so-leisurely stroll to class. But the mood won't be set because the battery on your phone is too low. Not a problem. Your slightly more responsible classmate has a portable charger. "Thanks," you say as the now charging phone and charger slide into your pocket.

An hour later as you chat about Thanksgiving plans with your peers, your pants catch fire.



Ever since the wide spread use of the steam engine in the 1800's, to the gasoline powered automobiles invented a century later, and all the way to a 2016 incident with a portable phone charger, energy storage devices have carried an associated fire and explosion risk. Given the number of personal devices that people carry on themselves, whether it is a phone, laptop, Bluetooth headphones, a smart watch, or a vaporizer, the quantity of batteries produced has increased exponentially. And so has the risk. The risk of injury. The risk of a lawsuit. The risk of a fatality.

"The U.S. Fire Administration declared batteries the "root cause" of at least 195 separate fires and explosions from 2009 to 2017. The Federal Aviation Administration has reported a few hundred incidents of smoke, fire, extreme heat, or explosions involving lithium-ion or unknown batteries in flight cargo or passenger baggage. And there were 49 recalls of high-energy-density batteries from 2012 to 2017, according to the Consumer Product Safety Commission, concerning more than 4 million devices, including mobile phones, scooters, power tools, and laptops." (The Atlantic, April 30, 2019)

To better understand the fire characteristics of a battery energy storage system, Underwriters Laboratory (UL) released the 4th Edition to standard UL 9540A. This edition of the standard requires the generation of specific data to determine the fire and explosion properties of an energy storage device. This is where Fauske and Associates LLC (FAI) can help.

The UL 9540A Ed. 4 standard requires intensive testing on multiple aspects of battery usage, from the battery cell level to the module level, and establishes guidelines on installation. In total the UL 9540A Ed. 4 standard is a cornucopia of requirement codes that references an American Society of Heating, Refrigerating and Air-Conditioning Engineers (ASHRAE) standard, four ASTM standards, two Canadian standards, an EN standard, an ISO standard, and a plethora of NFPA standards. Keeping track of which testing you may need can be daunting, but the Flammability department at FAI is here to help you.



Among some of the newer requirements found in Edition 4 are a series of flammability tests that must be performed on the battery cell when that cell is heated to a point of venting and beyond to thermal runaway.

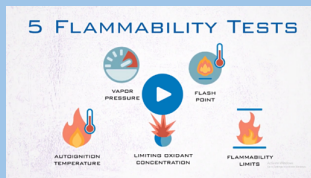
Section 7.4 describes these four tests. These include two lower flammability limit (LFL) tests. One of the LFL tests is done at ambient temperature, and the other LFL test is conducted at the temperature the cell begins to vent gas. These tests are to be run in accordance with ASTM E918, a method that falls within Fauske's ISO 17025 Scope of Accreditation. The third required test is an explosion severity test. The explosion severity test determines the maximum overpressure and the maximum rate of pressure rise of an explosive event. The method used for this test is EN 15967, which is scheduled to fall within Fauske's ISO 17025 scope this year. Finally, the fourth test is the burning velocity test. There are two methods to choose from when generating this last data, ISO 817 and NFPA 68 Annex E. Currently FAI tests to the NFPA method, which will also be added to Fauske's ISO 17025 scope, but plans are underway to include ISO 817 to the Fauske arsenal of testing as well.

The results from these four flammability tests will satisfy the requirements necessary for Sections K, L, and M of a Cell Level test report.

For more information on explosion severity, LFL testing, and burning velocity determination to comply with UL 9540A Ed. 4, please contact the Fauske and Associates flammability department at flammability@fauske.com.

Flammability Testing at FAI

One of the most common causes of loss in the process industries for both equipment and life is fire. At Fauske & Associates LLC we can help you assess your risk exposure by characterizing the flammability potential of your combustible vapors or gases. Curious to learn more? Watch our new video on Flammability Testing:



Schedule to Perform a Dust Hazards Analysis (DHA) During the Worldwide Shutdown

By Ashok Dastidar, PhD, Fellow Engineer, VP Dust & Flammability Testing & Consulting, Fauske & Associates LLC



It has been observed that many of the root causes of industrial dust explosions extends back to the awareness of the hazard in the specific process setting. This lack of hazard awareness and inability to identify risks is fundamentally a knowledge-based safety failure. This lack of safety awareness can be easily rectified by a systematic stepwise safety review of the process. This was the logic behind the requirement in NFPA 652 to conduct a Dust Hazards Analysis (DHA) of any process handling combustible dusts so as to identify fire, flashfire and explosion hazards at a facility. The DHA requirement stems from an earlier recommendation in NFPA 654 to conduct a Process Hazards Analysis (PHA) of powder/dust handling operations. The NFPA felt that the recommendation to conduct a PHA would be confusing to the user of the standard since it may be confused with the requirement to conduct a PHA under the OSHA Process Safety Management (OSHA PSM) program. The intention was not to force companies into a PSM program when such a complex and onerous plan was not warranted. As a result the NFPA, when authoring the new 652 document, changed the term PHA to DHA and made it a requirement, not a recommendation. The requirement in the Standard, which came into effect September 7, 2015, is that every new process has a DHA performed BEFORE it is started up for the first time and existing operations have a DHA completed before September 7, 2018. Additionally, the commodity specific standards; i.e. 654 for general chemicals/plastics, 61 for agricultural dusts, 664 for wood dusts, 484 for metal dusts and 655 for sulfur process, adopted a similar requirement and timeline.

As the 2018 deadline approached it became apparent that many companies, who in good faith, started the DHA process would not be able to meet the timetable and complete the DHAs in time. As a result the NFPA extended the deadline to 2020 to give those companies additional time with the understanding that they would continue to diligently work towards completing the work given the extended time.

Fast-forward to March 2020; many facilities were forced to limit access to their facilities or close down their facilities altogether. With such a global "shutdown" in place, does that mean that the September 2020 extension of the previous deadline will be extended again? No, in recent discussions with several NFPA staff member, their intentions were made clear. The September 2020 extended deadline would not be extended again. This, however, does not mean that Code Enforcers like OSHA, Building and Fire Department inspectors, will be visiting your facility in the autumn. Code Enforcers may choose to issue warnings and not citations or fines until later in the year – however, this is no guarantee and may vary jurisdiction to jurisdiction. As a result, even the current stay-at-home order shutting down facilities it would be a good idea to continue working to meet the September 2020 deadline.

Therefore, given the fact that the facility may be restricted to company personnel only, or may not have current operations actively producing, how can an engineer inspect your facility for compliance to the NFPA standards? The DHA is not only about the site visit but also includes reviews of procedures and equipment design! The DHA team can meet virtually and review documents such as Standard Operating Procedures and practices (housekeeping, hot work, electrical area classification etc.), training records and existing safety audit documents to see if they address combustible dust risks. Getting these documents collected and reviewed will speed up any onsite review that will need to be performed at a later date. One of the many time-sucking pitfalls that can delay a DHA during the on-site visit is the frequent pauses that occur when the audit needs to stop so that relevant or crucial documents or reports can be found and disseminated to the team.

Of these reports that can cause delays, the greatest delay can occur if there is insufficient information available about the combustible nature of the dust(s)/powder(s) the company is processing or generating. The flowchart in Figure 1 is discussed in detail in "Chapter 4 Dust explosions: Test methods" of Methods in Chemical Process Safety vol. 3 Dust Explosions. It is also discussed in a presentation titled "Introduction To Combustible Dust Test Methods" at the [Digital Dust Safety Conference February 24-27, 2020](#) and addressed in the

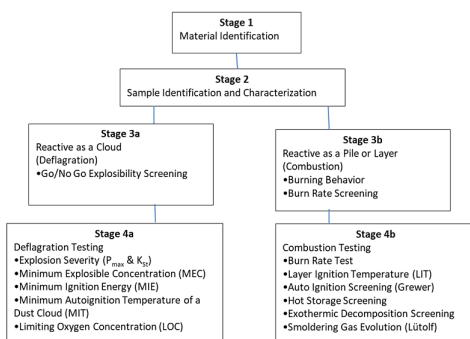


Figure 1: Combustible and Deflagrable Dust Testing Strategy.

webinar "Schema for Combustible Dust Testing to Provide Data for a Dust Hazards Analysis (DHA)" presented on March 31, 2020. The flowchart discusses the types of tests that can be used to characterize a potentially hazardous dust for explosibility and combustibility. Each of the tests described in the chart has merit for establishing dust safety hazards by establishing explosion/combustion violence and the ease at which the dust will undergo this hazardous reaction. The information may be necessary for all the dusts/powders handled at the plant and all the data from the various tests may be required. The process of collecting the sample, contracting the tests and obtaining the data will take several weeks thus delaying the delivery of the final DHA report. However, the DHA on-site team can review the process flow and estimate the number of samples that need to be tested and what subset of the tests identified in Figure 1 need to be conducted before the field visit and which ones can be deferred to a later time.

Ultimately, it is important to start the DHA process now, even though the actual field visit will need to be conducted later, the documentation can be reviewed and the test data obtained (with time to fill gaps in either).

For further information, or if you would like assistance with your DHA and Combustible Dust Hazards Training Program, please contact DHA@fauske.com.

Process Safety Scale-Up Aspects of an Epichlorohydrin Hydrolysis Reaction - Part 2 - Heat Rate Scale-Up Calculations from Reaction Calorimetry Data

By Donald J. Knoechel, PhD, Senior Consulting Engineer, Growth Leader for Reaction Calorimetry & Testing Services, Fauske & Associates LLC

In the last newsletter we showed the tremendous amount of energy contained in a post epichlorohydrin (EPI) hydrolysis reaction mass. For our generic recipe (27.1% wt epichlorohydrin, 72.4 wt% water, 0.5 wt % acid catalyst [69% aqueous nitric acid]), the total adiabatic potential was +195.8°C as shown by reaction calorimetry and differential scanning calorimetry (DSC) which included intended reaction heat and higher temperature activated polymerization/decomposition heat. Ultimately, this total energy potential and the overlap, if any, between the desired reaction energy and the undesired polymerization/decomposition energy is to be confirmed by adiabatic calorimetry (Fauske VSP2) which will be the subject of the third part in this newsletter series.

Nevertheless, if one wanted to scale up this process, the management of the desired heat of reaction is crucial to process safety. While these reactions are easy to control in a laboratory reaction calorimeter via jacket cooling, just how easy is it to control with scale? Eventually, we will perform some simple calculations to show how these thermal challenges offered by the epichlorohydrin hydrolysis process would be handled at scale.

First, the full series of EPI hydrolysis reaction calorimetry (RC) experiments will be presented to get a feel for the kinetics of the semi-batch process as seen by the heat flow profile. Figure 1 shows a series of four reaction calorimetry runs covering three different addition rates of EPI (1, 3, 5 g/min) and two different temperatures (60°C, 80°C). Specifically, runs at 80°C with EPI feed rates of 1 g/min (80-1 HF), 3 g/min (80-3 HF) and 5 g/min (80-5 HF) and a run at 60°C with EPI feed rate of 3 g/min (60-3 HF) are shown. Also shown are the respective addition profiles. Table 1 summarizes the RC data derived from the four runs.

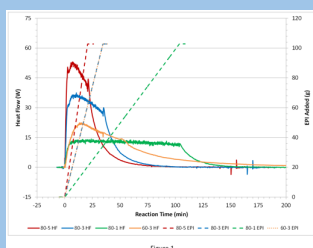


Figure 1

Table 1				
	80°C, 5 g/min	80°C, 3 g/min	80°C, 1 g/min	60°C, 3 g/min
Reaction Mass (g)	380.18	379.86	379.86	379.90
Total Heat (J)	-85,803	-86,970	-86,338	-89,652
EPI added (g)	102.74	102.74	102.76	102.76
EPI added (g-moles)	1.108	1.108	1.108	1.108
Heat of Reaction (kJ/mole EPI)	-77.4	-78.5	-77.9	-80.9
Addition Time (h:min:sec)	00:20:55	00:34:45	01:43:45	00:34:43
% Accumulation	33.2	24.0	11.0	58.8
Pre-Reaction Cp (J/g°C)	4.360	4.365	4.361	4.210
Post-Reaction Cp (J/g°C)	3.823	3.758	3.782	3.648
ΔT_{ad} (°C)	59.0	60.9	60.1	64.7
Dynamic ΔT_{ad} (°C)	19.6	14.6	6.6	38.0

Out of the four sets of process conditions, not surprisingly, the lower temperature 60°C with 3 g/min feed rate results in the highest amount of accumulation (58.8%). That is, the largest amount of energy yet to come due to unreacted EPI after the end of the addition. Even at 80°C with a 1 g/min feed rate there is a slight accumulation of EPI, but only 11%. At 80°C, it is easily seen that the longer the addition the less accumulation, and the more the heat flow profile approaches an addition-limited profile (square-wave) where ultimately, the EPI would be reacting as soon as it is added.

Typically, in batch equipment one would rely on jacket cooling to remove the reaction heat and control temperature. If we scale our epichlorohydrin hydrolysis batch process to 2000 kg of EPI and run it in a 12,000-liter (diameter 2.4 m) reactor, the stirred volume for our generic recipe would be 7,250 liters corresponding to a heat transfer area of 15.1 m² (A). If the tank was glass lined steel (GLS), a typical heat transfer coefficient (U) would be ~300 W/m²K. Assuming a maximum temperature difference between the reactor and jacket (DT) of 50°C, our maximum cooling capacity would be UADT = 300 x 15.1 x 50 = 226,500 W. For the 2000 kg EPI batch size, the normalized cooling capacity is 113.3 W/kg EPI.

If the reactor material of construction was Hastelloy C (HC), a slightly larger U could be applied (500 W/m²K). Similarly, then for the case of HC, the cooling capacity would be UADT = 500 x 15.1 x 50 = 377,500 W. For the 2000 kg EPI batch size, the normalized cooling capacity is 188.8 W/kg EPI. Please note that all the values assumed for the heat transfer coefficient are at the upper end of the performance range for each material of construction. Certainly, jacket fouling and choice of heat transfer fluid can affect these assumed U values, as well.

Figure 2 shows the now normalized (W/kg EPI) heat flow profiles for our series of four RC runs together with the normalized cooling capacities for the 2000 kg batch size at the 12,000 scale for glass lined steel (GLS) and Hastelloy C (HC) reactors.

One can easily see that jacket cooling in either the glass lined steel reactor or Hastelloy reactor is inadequate for both the 80°C 5 g/min and 3 g/min cases as a significant portion of the heat flux profile exceeds the cooling capacity lines. While one may not run the process this way due to the large amount of accumulation (58.8%), one can see that the HC reactor is close to handling the 60°C, 3 g/min case, if the addition was longer, say > 1 hour. Similarly, the glass lined steel reactor is close to handling the 80°C, 1 g/min case if the addition was say, > 2 hours. Clearly, an extended, controlled addition of EPI is necessary to perform this process at scale.

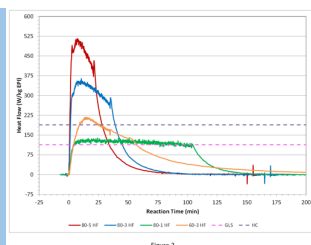


Figure 2

In fact, an 80°C run with a 0.822 g/min addition (126 minutes) was done and the results are shown in Figure 3 (an updated version of Figure 2).

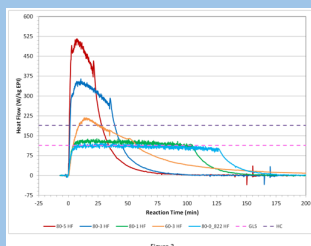


Figure 3

Indeed, with the greater than 2-hour EPI addition at 80°C, the resultant heat rate is at or below the cooling capacity line for the glass-lined steel reactor.

Relevant RC data (a la Table 1) derived from the 80°C, 0.822 g/min run were total mass 379.8 g, total heat -91,578 J, EPI added 102.68 g (1.108 moles), heat of reaction -82.7 kJ/mol EPI, addition time 2:06:09, % accumulation 9.53%, pre-reaction Cp, 4.378 J/g°C, post-reaction Cp 3.819 J/g°C, projected adiabatic temperature rise +63.1°C, and dynamic DT_{ad} +6.0°C.

A couple of points should be emphasized when scale-up requires an extended addition for cooling capacity reasons. First, the inherent safety offered by an extended addition minimizing accumulation is only realized if in the event of the loss-of-cooling, the addition is stopped. Also, with extended additions, it is very important to perform a validation run using that projected addition time in the laboratory to make sure material of desired quality and purity is still made with the longer addition.

In summary, we hope this article has given the reader an appreciation for the challenges faced by scaling up the exothermic EPI hydrolysis process to manage the desired reaction energy while avoiding the secondary polymerization/decomposition. This is also an example of how RC data can be used to optimize a process. The data make it possible to determine the fastest practical addition rate of reactant given the available cooling capacity or one can determine the required cooling for a desired process temperature or addition rate.

The next article will cover the actual runaway scenario (all-in EPI addition at 80°C) as seen by adiabatic calorimetry (VSP2), and its implication on relief system design. Furthermore, we will revisit the time-to-maximum-rate curve (derived from DSC screening of post-reaction mixtures) presented in the first article in this series to see where the ARC, VSP2, and the Thermal Activity Monitor (TAM) instruments detect the onset of secondary reactivity. Finally, we will modify the criticality class assessment of the process, if needed, given the additional data from the other instruments.

Reaction Calorimetry was performed in a Mettler-Toledo RC1.

If you have process scale up concerns or reaction calorimetry needs, please contact Don Knoechel at (knoechel@fauske.com or 630-887-5251) to discuss your process.

VSP2 Pressure Transducer Calibration & Maintenance Guide

By Aaron Ruiz, Thermal Hazards Technician, Fauske & Associates LLC

Introduction

The key parameters measured during Vent Sizing Package 2 (VSP2) experiments are temperature and pressure. Temperature measurements are taken using K-type thermocouples connected to the VSP2 data collection system through a thermocouple gland. Pressure measurements are made using pressure transducers. These important parameters are used to make critical decisions related to safety. The key to collecting reliable and reproducible data is properly calibrating and maintaining the instrumentation used to collect it. The pressure transducers used with the VSP2 are the DP15/ DP215 Variable Reluctance Pressure Sensors from Validyne Engineering Corporations. These transducers were primarily selected because they are designed to withstand various corrosive liquids and gases, have replaceable diaphragms for various pressure ranges, and have high accuracy and precision.

Calibration

The calibration system within the VSP2 control box requires the user to adjust a zero and gain reading based on a separate calibrated source. In order to calibrate the VSP2 pressure transducer, perform the following steps:

1. Secure the pressure transducers that will be used for the experiment (with a diaphragm that will cover the pressure range of interest) to the calibration tree. The calibration tree should be connected to a high-pressure inert gas (i.e. nitrogen) source, a vent line, and a calibrated source. The FAI calibration kit comes with a pressure gauge that can be separately calibrated. This gauge acts as the "calibrated source" that the transducers are calibrated against. See Figure 1 for the FAI calibration tree with pressure gauge and transducers installed. Note; **do not** place the pressure transducers on the Super Magnetic Stirrer when it is enabled.



Figure 1: The FAI calibration tree with pressure gauge as the "calibrated source" for calibrating the pressure transducers. The left-hand-side shows the "zero"

point and the right-hand-side shows the "gain" measurement for a 2,000 psig transducer (i.e., 847 psig)

2. Within the VSP2 software, select the button that says "Calibrate Temperature and Pressure" to open up the calibration window (see Figure 2).

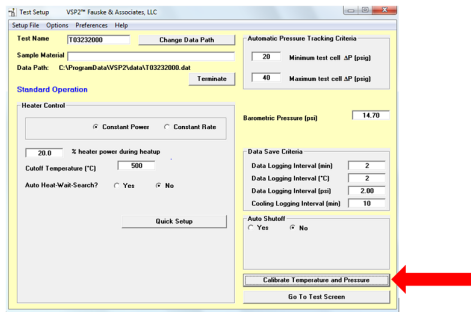


Figure 2: The "Calibrate Temperature and Pressure" button is shown by the red arrow when the software is opened. When clicked, it will redirect the user to the calibration window

3. The full output of the transducers is 10V. In order for the VSP2 software to correlate the measured voltage from the deflection of the diaphragm to a pressure, the user must input the full range of the diaphragm divided by the total voltage (10V). For example, if a 2,000 psi transducer is used, the VSP2 user should input 200 psi/V inside the "Pressure Sensitivities" window for each transducer that is used. Figure 3 includes a table indicating the pressure range based on the number imprinted on the diaphragm used. Figure 4 shows where the pressure range divided by the total voltage needs to be specified.

Pressure Diaphragm Selection Chart						
Range Dash No.	PSI	IN HG	IN H ₂ O	KPA	TORR	CM H ₂ O
20	125	25	3.5	86	6.5	8.80
22	20	41	5.5	140	10.3	14.0
24	32	65	8.9	22	16.5	22.5
26	50	102	14.0	3.5	26.5	35.0
28	80	18	22.2	5.5	41.4	56.0
30	125	25	35.0	8.6	65.0	88.0
32	20	41	55.0	14.0	103	140
34	32	65	90	22.0	165	225
36	50	102	140	35.0	258	350
38	80	18	222	55.0	414	560
40	125	25	350	86.0	650	880
42	20	41.0	550	140	1030	1400
44	32	65.0	890	220	1650	2250
46	50	102	1400	350	2580	3500
48	80	180	2220	550	4140	5600
50	125	250	3500	860	6500	8800
52	200	410	5500	1400	10300	14000
54	320	650	8900	2200	16500	22500
56	500	1020	14000	3500	25800	35000
58	800	1800	22200	5500	41400	56000
60	1250	2500	35000	8600	65000	88000
62	2000	4100	55000	14000	103000	140000
64	3200	6500	8900	22000	165000	225000

Figure 3: A table from the Validyne Pressure Transducer Manual showing which dash number corresponds to what pressure range/

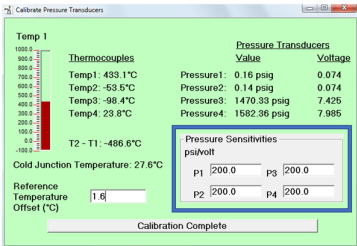


Figure 4: a screenshot from the VSP2 control software after the "Calibrate Temperature and Pressure" button is selected on the main page. The "Pressure Sensitivities" box is where the psi/volt needs to be input.

4. With the pressure transducers at atmospheric pressure, adjust the "zero" on the VSP2 potentiometer screw for each transducer (note, P1 measures the sample pressure and P2 typically measures the primary containment vessel pressure for pressure balancing), shown in Figure 5, until the display on the VSP2 screen (Figure 6) reads 0 psig. The manufacturer specification for accuracy of the pressure transducers is $\pm 0.5\%$ of the full scale for pressure ranges less than 2,000 psig. For diaphragms 2,000 psi and above, the stated accuracy is $\pm 1\%$ of the full scale. Note, adjusting the screws clockwise will increase the pressure reading on the VSP2 control box, and adjusting the screws counterclockwise will decrease the pressure reading. Be careful not to apply pressure when adjusting the potentiometer screws as they can be pushed past the control box surface.

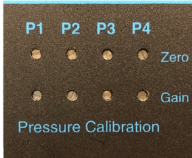


Figure 5: VSP2 potentiometer screws where the user can adjust the "zero" and "gain" for any pressure transducers that are being used to match the pressure reading from the gauge in Figure 1

Figure 6: The display from the VSP2 software.

Pressure Transducers		Pressure Transducers	
Value	Voltage	Value	Voltage
Pressure1: 0.16 psig	0.074	Pressure1: 847.31 psig	4.310
Pressure2: 0.14 psig	0.074	Pressure2: 847.72 psig	4.312

5. adjust the "gain", pressurize the transducers to near the peak pressure of interest (e.g. potentially 800 psig for a 2,000 psi transducer or 600 psig of an 800 psi transducer; these are recommended ranges, but it depends on the experiment and what pressure one expects). Be careful not to overpressurize the diaphragms above their measurement range, because this can permanently damage them. Adjust the "gain" potentiometer screws until the pressure readings (Figure 6) matches the reading on the pressure gauge (Figure 1).

6. Depressurize the transducers back to 0 psig, check the reading, and adjust the "zero" potentiometer until the VSP2 screen reads zero. Repeat with the "gain". This may take several iterations before the transducers have been fully calibrated.

7. When the calibration is complete, it is suggested to incrementally increase the pressure from 0 psig to the peak tested pressure and check that the pressure of all transducers are within the accuracy range for each increment.

MAINTENANCE

General Cleaning

The most important thing for properly maintaining the VSP2 pressure transducers is to promptly clean them after every experiment. The simplest way to clean them is to remove the bleed screw and flush a suitable solvent through the channel. To remove the bleed screw, use a small hex tool, and carefully twist to loosen it. *Figure 7* shows where the bleed screw is located.

- Be careful not to lose the orange washer inside the bleed screw port.
- Flush the channel using a non-residue solvent from the negative side of the transducer, and out through the positive side to flush out any remnants of the sample.
- Utilizing low pressure compressed air or other inert gas, dry out the channel.
- Replace the bleed screw and tighten.

Thorough Cleaning

Periodically it is important to completely disassemble and clean the pressure transducer. This can be for a few reasons:

A. It is expected that the transducer was exposed to a chemical that warrants a thorough cleaning (such as a sticky or viscous difficult to clean material or something that is toxic or severely corrosive).

B. The pressure range of the diaphragm needs to be changed.

C. The pressure transducer is not performing as it is expected, for example:

- The pressure increments from full scale to zero are non-linear and deviate causing excessive hysteresis.
- The zero reading shifts after being calibrated.
- The outputs are not within the stated accuracy.
- The outputs are not balanced despite after making multiple iterations from full scale to zero.

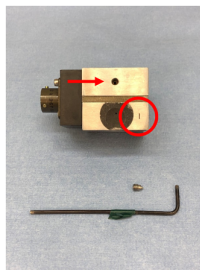


Figure 7: The bleed port is located on the negative port side and shown by the arrow and can be removed by using a small hex tool as depicted. The circled area indicates that this is the negative port of the transducer by the (-) symbol.

Disassembly

1. Carefully secure the pressure transducer within a vise (see *Figure 7*, note we have brass on the vise faces to prevent damage to the transducer body).

2. Using a hex or star key wrench (depending on the screws used), loosen the bolts, by slowly loosening and alternating the bolts incrementally until they are removed.

3. Gently pull open one side of the transducer. Be careful when opening the transducer because thin gauge wires are secured near the top of the transducer and can be snapped off. Now, the transducer is in a position that it can be cleaned with a suitable solvent, or parts can be replaced as needed.

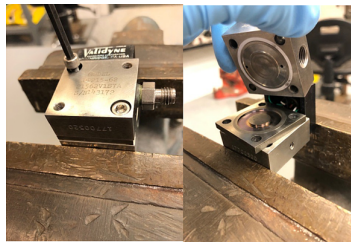


Figure 8: A vise clamp holding a transducer, so the user can unscrew the bolts to remove the diaphragms or the O-rings.

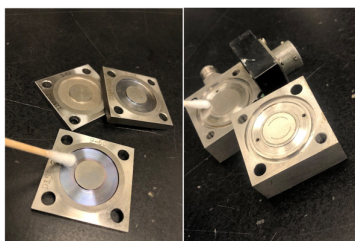
There are few key features of the transducer to note:

1. Diaphragms

The adaptability of the transducer is due to its economical feature allowing the user to replace the sensing diaphragms without purchasing multiple transducer bodies. This allows for numerous pressure ranges to be looked at accurately. For example, closed cell VSP2 experiments can be performed to high pressures by implementing a high pressure diaphragm. To run experiments in lower pressure ranges (like in an open cell orientation) with improved accuracy, lower range diaphragms can be implemented. A benefit of this transducer and diaphragm type is they can withstand extreme shock, vibrations, and generally reasonable laboratory abuse from the stainless steel construction. Further, the stainless steel construction is ideal for use with the VSP2 due to the protection against corrosion and oxidation. Some notes on the transducer diaphragms (shown in *Figure 9*):

- Check for signs of warping or damage to the diaphragms, and periodically replace the them. They can become damaged over time from overpressure, or long exposure to numerous chemicals without prompt cleanup.
- Make sure to utilize the correct diaphragm that correlates with the desired testing range (see *Figure 3*). The accuracy of the transducers is based on the full scale, and thus the lower the pressure range, the more accurate the pressure readings will be.
- Tip: label what diaphragm is being used on the outside of transducer, so it is clear what the range is without disassembling it.

Figure 9: Each diaphragm has a dash number and this case, the diaphragm being cleaned has a dash number of 60, indicating that this diaphragm has a maximum pressure limit of 1250 psig. The diaphragms are compressed by the coiled pads inside of the transducer bodies where the applied pressure compresses internal coils.



2. O-rings

Besides the diaphragm, there is also an o-ring on either side of the body of the transducers between the diaphragm and transducer body (*Figure 10*):

- Replace the o-rings when they swell-up, appear damaged, or have been inside the transducer of a long period of time, they do not have to be replaced every time the transducer is opened.
- Ensure the o-rings are compressed securely in their grooves.
- Avoid pinching the o-rings (this can damage them).
- It is suggested to use Teflon o-rings instead of Viton-A, which are the default for when purchasing a pressure transducer. Teflon can resist corrosives better than Viton-A.

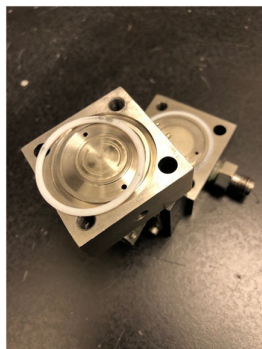


Figure 10: Close-up of Teflon o-rings inside the pressure transducer. This is an example of o-rings that are ready to be changed.

Reassembly

After the required cleaning or replacing of parts, the transducer can be reassembled for use, by retightening the bolts:

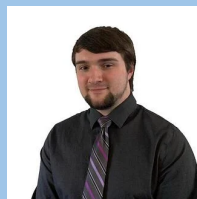
- Apply anti-seize as a sealer on the bolts to prevent any potential leaks from the transducer, protects them during the metal-to-metal contact, and helps prevent the bolts degrading from corrosion.
- Replace any bolts that are damaged.
- Align the bolts carefully with the diaphragm holes.
- Tighten the bolts snug and uniformly applying equal torque throughout. The threads from the bolts should never catch the holes of the diaphragms.

For any further assistance or questions, feel free to contact us at paris@fauske.com.

Contact Us

Meet Thermal Hazards Technician William Spreadbury

*Answers by William Spreadbury, Thermal Hazards Technician, Fauske & Associates LLC
Interviewed by Marc A. Cramer, Digital Marketing Intern, Fauske & Associates LLC*



What is a thermal hazards technician?

Thermal Hazards technicians are the main people that perform experiments for the Thermal Hazards team. On top of performing the experiments we keep the lab stocked with supplies, manage our chemical inventory, and generally maintain the lab to keep it running smoothly.

How would you describe your role at FAI?

My main role is to perform thermal stability experiments, and we have a few instruments to test with different ASTM standards. Performing these experiments involves calibrating the instruments' readings to ensure the values are accurate, ensuring all the safety features on the instrument are functioning properly, preparing the sample, setting up and running the tests, and then cleaning up after the test is complete. I keep the lab stocked with supplies required to run the experiments, such as waste containers, tools, nuts and other fittings, wipes and other cleaning supplies, and PPE to make sure everyone in the lab is safe. I am also the calibration custodian, which means it is my responsibility to make sure the equipment stays within calibration and to trace back any problems or wrong numbers when the equipment is found to be out of calibration. I am proud that our lab is ISO 17025 certified.

What was your background before coming to FAI?

I was an undergraduate at The University of Illinois in Urbana-Champaign. I got my Bachelor's in Chemical Engineering with a concentration in Biomolecular Engineering in May 2019 and started at Fauske & Associates LLC in August 2019. Before working here I did not have any professional experience, so I have learned a lot in the past few months.

What is it that brought you to FAI?

In all honesty, I only learned about Fauske shortly before I started working here. But after researching what Fauske does, it was clear their work was important. It also seemed fun and interesting. I don't get to know all the details of customers' processes, but I do get glimpses into the chemistry of the reactions, and I find that very interesting. And because every reaction is different, every test is different in some way.

What drew you to the Chemical/Nuclear process industry?

Chemical process safety is an important line of work. There are countless incidents where peoples' lives could have been saved if the hazards were properly managed. We can't change what has happened in the past, but we can learn from it and stop it from happening again. Working in process safety is also almost selfless; you don't get recognition or fame for the good work you do. When the emergency relief system you helped design is successful, the result is practically nothing happening (no serious consequences). "Nothing happened" is absolutely better than the unfortunate alternative, but it won't make headlines. And that's fine, I can be satisfied knowing I helped prevent people from getting hurt.

What do you enjoy most about thermal hazards?

I like knowing that the work I do helps keep people and communities safe and healthy. I also enjoy that each test is never identical to another. The lab instrument doesn't physically change a whole lot, but each test is a different process using different chemicals which can yield vastly different results. And even when the recipes are similar, it's interesting to see how slight alterations can drastically change the results.

What have been some of the highlights of your career?

There haven't been any big events that I would consider a highlight, but I've learned a lot in my time here at Fauske, as well as getting valuable technical experience, which I am grateful for. My colleagues have been great to work with; they're understanding and appreciative and have plenty of advice and knowledge to share. They make it easy to enjoy working here.

What are some of the skills and characteristics that are most important for those who work in thermal hazards?

Attention to detail, patience, and communication are all important. Our work directly affects peoples' lives, so making sure everything is correct for that specific test is important. Sometimes the set-up can be long, or the sample frustrating to work with, so being patient will help make sure the results are accurate. Good communication skills affects multiple facets of the work we do; we need to be able to ask the right questions so our tests can be designed to correctly simulate the customers' processes, we need to be able to communicate the hazards associated with the materials we are working with to the other people in the lab to make sure they know how to be safe, and we need to communicate exactly how the test was performed to the customer so they know the conditions that generated these results and are aware that changing any of those conditions in their process can have consequences not shown in our results.

Do you have any advice for future or aspiring people who might consider a career as a thermal hazards technician?

Mistakes will happen as you learn new skills such as setting up a thermal hazards test. It is important to have a questioning attitude and be willing to put in the time to make sure things are done right. It is not ideal repeating tests, but it is important that the experiments are done correctly because process safety decisions may be influenced by our test data.

UPCOMING EVENTS:

Meeting the DHA Deadline in the Time of COVID

Presented by *Ashok Dastidar, PhD, Fellow Engineer, VP Dust & Flammability Testing & Consulting, Fauske & Associates LLC*

June 24, 2020, 1:00 pm CST

Webinar Link: <https://hastingsair.com/seminar-registration/>

Webinar TBA

Presented by *Ashok Dastidar, PhD, Fellow Engineer, VP Dust & Flammability Testing & Consulting, Fauske & Associates LLC*

August 26, 2020

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