FAUSKE & ASSOCIATES, LLC

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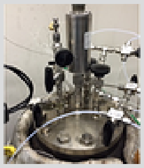

Determining the Limits of Flammability: Which ASTM Method Do I Choose? E918 vs. E681

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Safety Equipment, Process Design, and Operation

When you are a world leader in nuclear and process safety testing and engineering it is not surprising to find yourself in *Perry's Chemical Engineer's Handbook 9th Edition 2019.* Hans. K. Fauske, D.Sc. Emeritus President and Regent Advisor, Fauske & Associates, LLC, Fellow American Institute of Chemical Engineering, Fellow American Nuclear Society, Member National Academy of Engineering provided the following to Section 23 in this newest publication:

- Pressure Relief Systems
- Required Relief Rate
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- Vapor Systems Vent Sizing
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- Pressure Relief Valve (PRV)
- Stable PRV Operation
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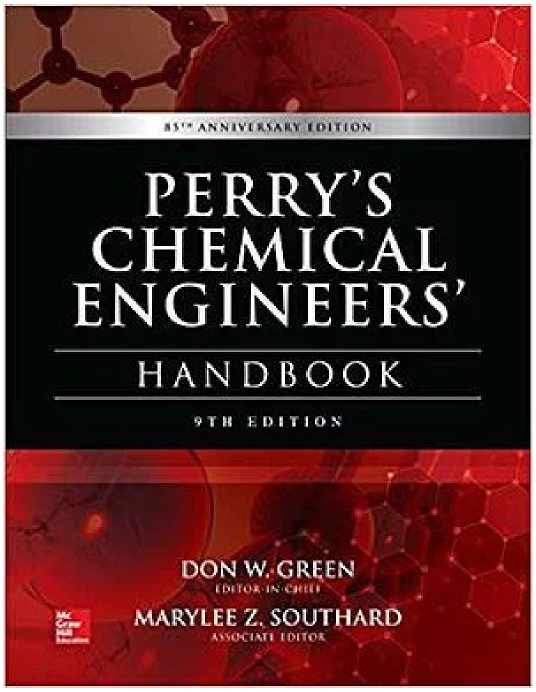


85TH ANNIVERSARY EDITION

PERRY'S CHEMICAL ENGINEERS' HANDBOOK

9TH EDITION

DON W. GREEN
EDITOR-IN-CHIEF
MARYLEE Z. SOUTHARD
ASSOCIATE EDITOR

McGraw Hill Education

["Safety Equipment, Process Design, and Operation" in Section 23]

Letter From The President

Dear Customer,

Did you know we are a subsidiary of Westinghouse Electric Company ? This sometimes surprises customers so we thought we'd point out just how we are able to provide you with holistic and comprehensive services. Many of our nuclear, chemical process, dust explosibility and flammability testing and engineering projects are part of an extensive capability program utilizing the best resources and talent available.

For example, in these areas:

- Facility Siting Analysis
  - Combines Fauske & Associates, LLC (FAI) process safety analysis skills with Westinghouse Electric Company (WEC) world class quantitative risk assessment tools to define cost effective solutions resulting in a more comprehensive analysis which provides a quantitative basis to support investment decisions to reduce event risk, while adding rigor to reduce regulatory review time.
- Natural Gas Risk Assessment
  - Leverages FAI's expertise in fire and explosion analysis with quantitative risk reviews of transmission and distribution systems to identify and mitigate risk areas resulting in clearly defined risk reduction analysis to support large scale investment in programs and capital upgrades. This approach also facilitates regulatory reviews.
- BWR Acoustic Resonance Testing
  - Build's on FAI's thermal hydraulic testing capabilities with specialized WEC acoustic analysis resulting in a streamlined regulator review and achieving uprates in lowest achievable times.

Contact us to see what we can do to test and solve your complex problems in the suite of many services we provide. It is a pleasure to serve you.

Best Regards,

JW Fasnacht

John W. Fasnacht, President

VSP2 – Everything You Need to Know About Setting Up Your Heater Assembly (VIDEO)

Explosion Protection for Volumes Greater Than 8 ft3

Safer Scale-Up of Batch and Semi-Batch Reactions; Part 2 - Quantification of the Desired Reaction

This is the second of four articles Fauske & Associates, LLC will be publishing on the topic of safer scale-up for batch and semi-batch reactions. The first article discussed desktop reviews and preliminary hazard analysis: Safer Scale-Up of Batch and Semi Batch Reactions; Part 1. This second article addresses characterizing the desired reaction and its implications on scale-up.

Donald J. Knoechel, Ph.D., Senior Consulting Engineer

Two of the four reasons for thermal runaways mentioned in the first article are related to the behavior of the desired reaction:

1. Insufficient understanding of the process chemistry and the energy/kinetics for the desired reactions
2. Improper design of the heat transfer capacity required at the plant level

The first reason directly so. The second reason, indirectly, as it is the mismanagement of the desired reaction heat with scale up that leads to the runaway. In fact, in the Process Equipment section of the OSHA Process Safety Management 1910.119 rule, an energy balance is required. While this clause is focused on the plant equipment and its heat transfer capabilities to handle the process, an energy balance cannot be written without knowledge of the heat of reaction and the rate of heat generation from that process chemistry.

Stoessel's Criticality Classes[1] serves as a universal guideline for the safe process scale up of exothermic reactions. In this approach, the Maximum Temperature of the Synthetic Reaction (MTSR) is one of the critical attributes to be determined. The MTSR is simply the process temperature plus the adiabatic temperature rise (ΔT_{ad}) due to the desired reaction heat, the latter of the two occurring in a loss of cooling scenario simulated by adiabatic conditions.

These two examples point to knowledge of the heat of reaction and reaction rate as critical parameters for safe process scale up. Previous FAI newsletter articles have elaborated on theoretical heat of reaction (THOR) calculation and reaction calorimetry (RC) as ways to calculate or measure the heat of reaction, respectively. While the former is sufficient to calculate a MTSR (given a heat capacity for the reaction mass), experimental data of conversion versus time for the process chemistry or assumptions of reaction time would be needed to create a heat rate of reaction for an energy balance. RC provides a solution whereby both the heat of reaction and heat rate are derived from the same experiment.

Theoretical Heat of Reaction (THOR)

The paper by Weisenburger et. al.[2] presents a very comprehensive study on theoretical heat of reaction estimation versus measurement and outlines when calculations can be used and how. Most notably for any heat of reaction estimation to be sound, values for the enthalpy of formations of the species involved or representative model compounds in the appropriate physical state must be available or reasonably estimated. Of course, a balanced chemical equation representing the process reaction of concern must be known as well. When compiled, the pertinent heats of formation are combined according to equation 1

$$\Delta H_{rx} = \Sigma\, \nu\Delta H_f \text{ products} - \Sigma\, \nu\Delta H_f \text{ reactants} \quad (1)$$

where ν is the stoichiometric coefficient for the individual reactant or product in the balanced chemical equation.

Heats of formation can come from the literature whether they are for actual or model compounds. The more complex the molecule, however, the more unlikely a value for its heat of formation will be found. Rather, this is where model compounds can be effective surrogates for the actual compound. A model compound distills the more complex structure down to just its reacting moiety.

With an estimated heat of reaction in hand, a calculation of the adiabatic temperature rise for a loss of cooling scenario is possible given a process recipe using actual or approximated heat capacities for the reaction mass components.

But not every type of process chemistry lends itself to THOR. In addition, simultaneous heats due to mixing, dissolution, complexation, crystallization, or other physical effects can complicate the calculation to the point of the estimate not being accurate. In these cases, reaction calorimetry is the preferred technique for heat of reaction determination.

Reaction Calorimetry (RC)

Reaction calorimeters come in many flavors: heat flow, heat flux, or power compensation. Regardless of the underlying calorimetric principle, the common deliverable from RC is a heat flow profile. That is the heat rate presented in watts or normalized by reaction mass as watts/kg versus process time during which either some reagent addition was performed to initiate reaction under isothermal conditions or during a temperature ramp or both. The area under the heat flow profile calculates the total heat from which a heat of reaction is determined by normalizing the total heat to reaction mass or mole of limiting reagent. The total heat divided by the thermal mass (mass times heat capacity) affords the adiabatic temperature rise (from which the MTSR is calculated).

The shape of the heat flow profile gives information on the kinetics of the reaction. For semi batch processes where there is a reagent addition, the degree of reagent accumulation is an important concept easily illustrated by the RC heat flow profile. If the reagent reacts nearly instantaneously as it is added, the process is said to be addition limited. The heat rate profile will resemble a square wave with the heat rate level realized dictated by the rate of addition. The heat rate will fall off quickly after the addition is ended. If the reaction is slower than the rate of reagent addition, unreacted reagent will accumulate. In this case the heat flow profile steadily rises during the addition most times but not always peaking at the end of the addition. After the addition has ended, the heat rate decays slowly. In a loss of cooling scenario assuming the addition is stopped when the deviation occurs, the accumulated heat is still available to cause a temperature rise, possibly initiating a thermal runaway.

The reaction calorimeters in the FAI toolbox are the Mettler-Toledo RC1 (heat flow), ChemiSens CPA202 (heat flux), and the Thermal Hazards Technology µRC (heat flux).

Gas Generation From the Intended Chemistry

Many process chemistries intentionally generate non-condensable gases. Reductions utilizing hydrides (sodium borohydride, lithium aluminum hydride, diisobutyl aluminum hydride, sodium triacetoxyborohydride, for example) can generate hydrogen when the reagent encounters acidic protons on the substrate or during a post reaction quench to kill excess reagent. Butyl lithium and some Grignard reagents can evolve low molecular weight hydrocarbons which may or may not be soluble in the reaction mass. Any reaction involving carbonate or bicarbonate can generate carbon dioxide.

Often, determining the amount of gas generated from these types of chemistries is simply an exercise in stoichiometry given a balanced chemical equation. This is an extension of the THOR approach. While the total moles of gas estimate can be fairly accurate, the rate at which it comes off is often in question. A reaction calorimeter fitted with a vent line bubble column, mass flowmeter, wet test meter or scrubber on a balance can be used to follow gas generation from the reactor as well as heat flow.

Simplified Energy Balance

The culmination of the quantification of the desired reaction is the heat rate scale–up calculation whereby a simplified energy balance can be written to demonstrate that the process equipment has adequate cooling capacity (or not) to handle the process chemistry at scale.

The same equations used in the heat flow balance from the Mettler-Toledo RC1 reaction calorimeter used to distill the experimental data down to a heat flow profile can be used to perform a simple heat rate scale up calculation for the plant. The terms in said heat balance are:

Heat **Input** (from additions to reactor) - $Q_{in} = m_{add}Cp_{add}(T_r - T_{add})/t_{add}$

Heat **Generation** (from reactions) - $Q_{gen} = (\Delta H_{rx})$(moles of limiting reagent or kg of reaction mass)$/t_{rx}$

Heat **Removal** (from jacket, condenser, heat exchanger, etc.) - $Q_{out} = -\, UA\,(T_r - T_j)$

Heat **Accumulation** (temperature change of reaction mass) - $Q_{accum} = -m_rCp_r(T_f - T_i)/t_T$

Where m_{add} is the mass of the added stream, Cp_{add} is the heat capacity of the added stream, T_r is the reaction temperature, T_{add} is the temperature of the added stream, t_{add} is the addition time, ΔH_{rx} is the heat of reaction per moles limiting reagent of kg reaction mass (from either THOR or RC), t_{rx} is the reaction time, U is the heat transfer coefficient, A is the heat transfer area, T_j is the jacket temperature, m_r is the reaction mass, Cp_r is the reaction mass heat capacity, and in the case of a nonisothermal process, T_f is the final temperature, T_i is the initial temperature, and t_T is the time of the reaction mass temperature change. We note in the accumulation term that the time derivative of the reaction temperature (dT_r/dt) is approximated by the linearized difference between the initial reaction mass temperature T_i and the final reaction mass temperature T_f over time t_T.

The energy balance is given by: **Input + Generation = Removal + Accumulation**

The various temperature terms can be input as desired values. The heat capacity terms are common outputs of reaction calorimetry experiments though many pure component heat capacities, especially solvents, are known[3]. If not, generalized values of 2000 J/kg-K for an organic component and 1000 J/kg-K for an inorganic component are commonly used. The most elusive term needed in the simplified energy balance is the heat transfer coefficient. For jacketed vessels, values for U depend on the material of construction and jacket utilities though approximate values may be available from the vessel manufacturer (Pfaudler or DeDietrich for instance). The area of heat transfer, A, is easily calculated from the vessel dimensions available from the same source for a given size vessel versus fill volume.

In the absence of an estimate for UA, it can always be derived from a cooling curve of a known heat capacity substance at a specific fill volume and agitation rate in the actual vessel over the temperature range of interest. With no reaction or addition, the simplified energy balance above collapses to just the heat removal and accumulation terms such that $U*A = -mCp(dT_r/dt)/(T_r - T_j)$. For vessels with multiple temperature probes, an average of baffle and bottom valve temperatures can be used for T_r (and dT_r/dt). For the jacket temperature, an average of the inlet and outlet jacket temperature can be used for T_j. For the known Cp substance, either a constant value for Cp (i.e. @ 25°C) or the temperature dependent equation (from DIPPR database[3] for example) may be used.

So with ΔH_{rx}, UA and Cp in hand, the simplified energy balance can be used to predict if there is enough cooling capacity at scale. The easiest version of the energy balance is to assume isothermal conditions are held such that the accumulation term is zero. Then for semi batch processes, given the temperature of the added stream, the addition time, and an assumed reaction time, the jacket service temperature necessary to maintain a desired reaction mass temperature can be calculated. Alternatively, given the temperature of the added stream, an assumed reaction time, and the available jacket service temperature, the minimum addition time necessary to maintain a desired reaction mass temperature can be calculated.

The biggest assumption in the simplified approach assumes approximate addition limited heat evolution behavior (say reagent accumulation < 15%). In this way, the heat of reaction is averaged over the time of addition unless specific knowledge of accumulation of reaction energy is noted. For cases where accumulation is greater than 15%, separate calculations of the handling of peak heat load during the addition and post addition reaction times can be performed. For large volume additions, an average value of A or UA (in the case of known UA vs. volume) is calculated from the actual volume range involved.

There are always many process situations where one or many of the above assumptions are invalid. However, recall that the main purpose of the simplified energy balance is to demonstrate the capability of the intended equipment and utilities to handle the desired process energy. The simplified format easily does this the majority of the time. If the outcome suggests a challenge, the energy balance gives valuable insight into what may be needed to safely execute the process under modified conditions (lower T_j, longer T_{add}, or additional heat transfer area [side loop heat exchanger], for instance). For the cases where a longer T_{add} is needed, it is important to have laboratory confirmation that the longer addition time does not introduce new impurities or other undesired process behavior before carrying out the process at scale.

Characterizing the Undesired Reaction with Adiabatic Calorimetry

Knowing the plant equipment can handle the process under desired conditions is not the end of the story. Protecting the plant equipment in the case of process deviations (loss of cooling, all in additions, fire, etc.) requires characterization of the undesired process behavior and relief vent sizing. Adiabatic temperature rise projections from THOR and RC are limited to just the energy due to the desired reaction. These do not represent the entire runaway scenario but only a minimum possible value. Under actual adiabatic conditions, further reactions may be initiated (with their own heat of reaction) when the actual rise in temperature is experienced which may contribute to a further increase in temperature (and pressure) until all reacting/decomposing components are consumed. Adiabatic calorimetry is required to explore the realm of process upset. The overlap between adiabatic calorimetry and reaction calorimetry lies in the fact that the adiabatic experiment often but not exclusively has the desired reaction as the trigger for runaway. Similarly, if there is off-gassing, quantification of the evolved gas rate is required to ensure the process vent capacity is adequate.

Therefore, the third article in this series will deal with how to characterize the undesired reaction behavior with adiabatic calorimetry and use the subsequent temperature rate and pressure rate data to properly size a relief vent to keep the plant equipment intact even in the event of a runaway.

References

1. Stoessel, F., " Thermal Safety of Chemical Processes: Risk Assessment and Process Design," Wiley-VCH, 2008.
2. Weisenburger et. al., "Determination of Reaction Heat: A Comparison of Measurement and Estimation Techniques," Organic Process Research and Development, **2007**, *11*, 1112-1125.
3. The DIPPR Information and Data Evaluation Manager for the Design Institute for Physical Properties, V 9.0.1, Database Date 2015, Brigham Young University.

Determining the Limits of Flammability: Which ASTM Method Do I Choose? E918 vs. E681

When determining the flammability limits of your product for an SDS or to optimize your process while maintaining high safety standards, it can be somewhat confusing choosing the correct testing method. The two most requested methods are ASTM E918: *Determining Limits of Flammability of Chemicals at Elevated Temperature and Pressure* and ASTM E681: *Concentration Limits of Flammability of Chemicals (Vapors and Gases)*.

TJ Frawley, Project Manager, Flammability Testing and Consulting Services

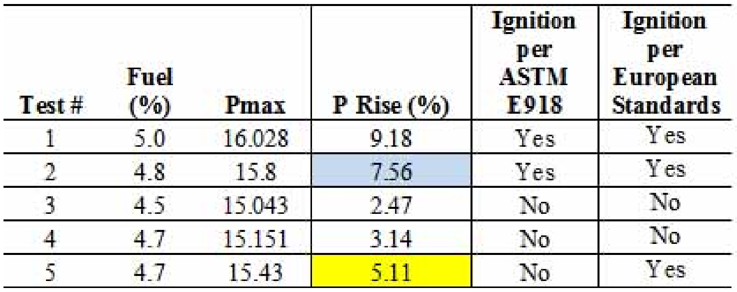
This piece will examine the differences between these two methods and the advantages and obstacles of those differences. As a starting point, the most glaring difference between the two is that E918 is performed in a 5-Liter stainless steel vessel and E681 is performed in a 5-Liter glass flask that incorporates a rubber stopper to seal the vessel. It is important to remember this key difference as it is the basis which most greatly sets the two methods apart and can thus influence data interpretation.

The similarity of the methods are few but deserve mention. Both methods aim to determine the same outcome – boundaries of vapor/gas flammability. Both can be run at elevated temperatures, although E918 allows for a greater range; and E918 can also be run at above, or below, atmospheric pressures.

It is the opinion of the Flammability division here at Fauske and Associates that E918 is the better of the methods because of the increased accuracy, versatility, and safety, that testing in a stainless steel vessel can provide.

ASTM E918 has a greater accuracy because the resolution of an ignition is determined by an established percentage of pressure increase which is measured by calibrated pressure transducers. In the United States a 7.0% rise of absolute pressure constitutes an ignition. At the ambient pressure of 14.7 psia, the 7.0% rise is equal to an increase of 1 psia in pressure. Europeans use a 5.0% pressure rise criterion as the defining threshold of an ignition as is stated by the Swedish Standards Institute in SS-EN 1839.

Below is a chart displaying a dataset of five tests determining the LFL of methane. The yellow highlighted cells illustrate a 5.11% pressure rise (Test #5). This would be considered an ignition in Europe, and the LFL of methane would be reported as 4.7% fuel (assuming testing of 4.6% resulted in non-ignitions). However, in the United States, Test #2 where a pressure rise of 7.56% occurred, would be considered an ignition, and 4.8% fuel would be reported as the LFL (blue highlighted cell).



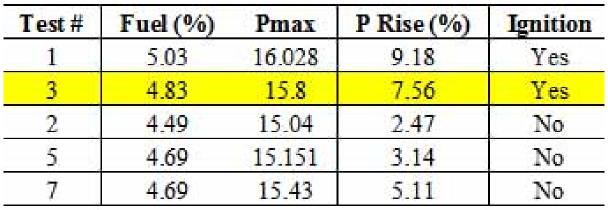
Test #	Fuel (%)	Pmax	P Rise (%)	Ignition per ASTM E918	Ignition per European Standards
1	5.0	16.028	9.18	Yes	Yes
2	4.8	15.8	7.56	Yes	Yes
3	4.5	15.043	2.47	No	No
4	4.7	15.151	3.14	No	No
5	4.7	15.43	5.11	No	Yes

The conception the 7.0% pressure rise and 5.0% pressure rise are not without controversy. The reasoning for those numbers being used as an ignition threshold are still debated today. However, there is consistency for every lab in the United States using a 7.0% pressure rise, and there is consistency across European labs that use a 5.0% ignition indicator. The end user of the LFL data needs to be aware of the exact method used for the determination.

While E918 has an objective, clear, and distinct definition of an ignition, E681 relies on subjective visual observations to make the distinction between an ignition and a non-ignition. The standard states in section 3.1.2, "propagation of flame – as used in this test method, [is] the upward and outward movement of the flame front from the ignition source to the vessel walls or at least to within 13 mm of the wall, which is determined by visual observation." This creates bias when determining "what is and what isn't an ignition." What one person may interpret as an ignition, another person may disagree. Herein lies the uncertainty in the end results.

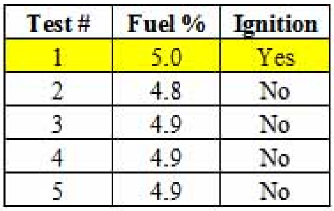
Below are two charts comparing the LFL results of methane obtained being using each standard. The top chart has data from ASTM E918, and the bottom chart was obtained from ASTM E681. Please note the difference of fuel percentage that correspond with the lowest ignition. Using ASTM E918 the reported LFL is 4.83%. The result using ASTM E681 in the glass flask is about 0.2% higher.

Lower Flammable Limit of Methane in a 5-L Stainless Steel Vessel



Test #	Fuel (%)	Pmax	P Rise (%)	Ignition
1	5.03	16.028	9.18	Yes
3	4.83	15.8	7.56	Yes
2	4.49	15.04	2.47	No
5	4.69	15.151	3.14	No
7	4.69	15.43	5.11	No

Lower Flammable Limit of Methane in a 5-L Glass Vessel



Test #	Fuel %	Ignition
1	5.0	Yes
2	4.8	No
3	4.9	No
4	4.9	No
5	4.9	No

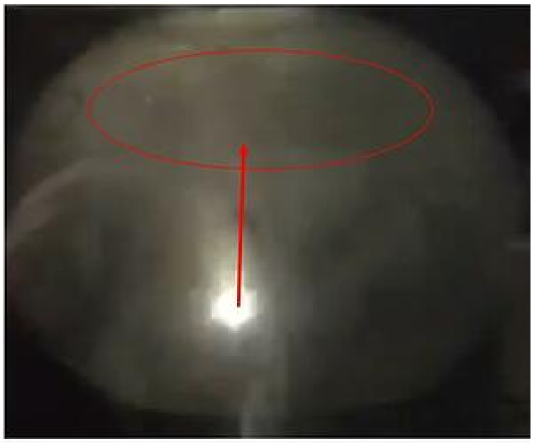
Correlation does not equal causation, therefore, the difference in results may not stem from the differences in the standards. However, based on the data in the top chart using a 7.0% rise as the threshold for an ignition, we can clearly distinguish an ignition from a non-ignition. The lower chart is more ambiguous.

Even when using video to record testing of E681, the outcome is not always as dependable in practice as the standard describes.

In this picture our ignition source is fired. It appears that a flame is in the beginning stages of propagating.

Here a flame has been identified. However, it must propagate upward and outward to be considered an ignition.

This picture illustrates the flame moving upward in the vessel. It may be difficult to view the flame clearly due to residue from previous tests.



Finally, through the haze the flame can roughly be seen reaching the top of the flask.

How would you characterize the result of this test? Ignition or no ignition?

The pictures above depict a typical test ran according to ASTM E681. While this test is more visually appealing than watching a line move on a graph, it can be difficult to view and interpret. These pictures were specifically chosen to illustrate the difficulty of determining an ignition visually. This is not an uncommon scenario.

Any material that may produce a residue or a solid product in decomposition, such as chemicals with a saline group or one that generates soot or tar, will decrease the technician's visibility and impair their ability to distinguish between an ignition and a non-ignition.

E918 also has more versatility and can replicate more processes by being able to reach higher pressures and temperatures. Here at Fauske and Associates using E918 we have been able to perform testing above 300 psia. The other method cannot test at any pressures above ambient levels. According to E681, the 5-L glass flask is vacuum sealed with a rubber stopper.

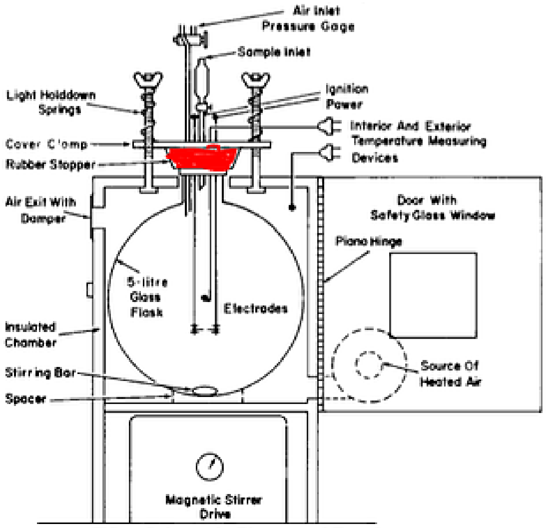
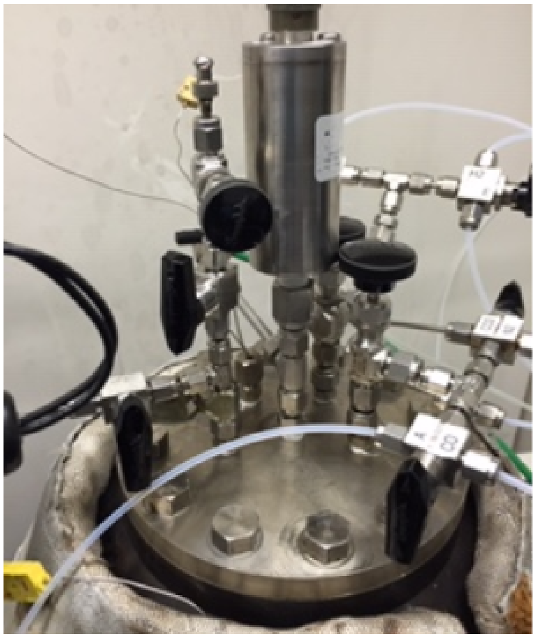


FIG. 1 Schematic Diagram of Test Apparatus



On the top: a diagram of E681 setup with a rubber stopper sealing the glass flask
On the bottom: a stainless steel 5-L vessel sealed with a stainless steel lid

If pressurized above ambient pressure, the rubber stopper will pop, and the test mixture will be compromised. Not only will this test have to be repeated, if the sample is hazardous, a safety concern now exists.

Finally from a safety perspective, testing in a completely sealed stainless steel vessel is overwhelmingly preferable to testing in a glass flask that relies on a vacuum sealed rubber stopper.

Stainless steel will not crack or shatter; glass might. Although engineers and technicians are trained and experienced at avoiding dangerous scenarios, the possibly remains. We are igniting chemicals that may result in high pressure explosions. It is not outside the realm of possibly for the glass flask to crack or break.

As previously mentioned, a steel vessel can be pressurized. This is also very important for purging the vessel after a test. Any hazardous decomposition products can be purged by pressurizing the vessel with nitrogen and then evacuated into a scrubbing system or into a fume hood. This cannot be done in a glass flask. As soon as the pressure inside the flask becomes greater than the atmosphere outside the flask, the rubber stopper will break its seal with the glass flask and hazardous gases or vapors will be ejected from the flask in an uncontrolled manner into the surrounding environment. Therefore, the purging process must rely on vacuum purging alone, which most likely will increase the time to fully purge. This adds to the total turnaround time of the test and an increase in cost.

There are benefits to E681. It is visually more appealing because a person can actually see reactions in the vessel. Also the equipment for E681 is less expensive and may be less expensive to perform testing.

It is always important to know the pros and cons when choosing a testing standard. Please contact flammability@fauske.com or 630-323-8750 for more information.

Maximum Experimental Safe Gap Test

Process industries that handle flammable gases and vapors may involve concentrations that can potentially form an explosive atmosphere. In a situation where the right precautions are not taken into consideration, a flammable mixture may be exposed to an ignition source such as a static electric discharge, an electrical arc or a spark, which may ignite and potentially cause an explosion which can then result in personnel injury or damage to property.

Michael Lim, Flammability and Dust Operations

Various regulations and standards have been developed to ensure a high level of safety in these hazardous conditions. The testing standards that have been established are aimed to provide information that can be used in engineering designs. An important piece of data that is valuable in designing explosion protection is the safe gap value or the maximum experimental safe gap.

The Maximum Experimental Safe Gap (MESG) is defined as the maximum gap between two flat surfaces, under specified test conditions, that prevents an ignition of a flammable gas/air mixture propagating from an inner chamber through a 25-mm long path into a secondary (outer) chamber. The data generated from MESG testing is commonly used as a guideline in installing properly sized flame arresters on process equipment. Additional information regarding flame arresters is found in NFPA 69 and ISO 16852.

The gases and vapors are classified into different explosion groups. Per NFPA 70, National Electrical Code, Class I locations are those in which flammable gases, flammable liquid-produced vapors or combustible liquid-produced vapors are, or may be, present in the air in quantities sufficient to produce explosive or ignitable mixtures. Class I locations are divided into divisions and zones depending on current or expected conditions. Material Groups, based on the MESG, are also used to further classify the explosive characteristics of specific gas/vapor air mixtures. The Material Groups are as follows:

Class I, Division	Maximum Experimental Safe Gap

Group A	Acetylene[a]
Group B	MESG ≤ 0.45 mm
Group C	0.45 mm < MESG ≤ .075 mm
Group D	MESG > 0.75 mm

a. The standard did not provide an MESG value

Per the European standard IEC 60079-20-1, the equipment is classified into groups in accordance with the properties of explosive atmospheres for which it is intended. The groups for equipment for explosive gas atmospheres are as follows:

Group I: Equipment for mines susceptible to firedamp

Group II: Equipment for locations with explosive gas atmospheres other than what was stated for Group I

Group II equipment is then subdivided into three sub-groups. For the purpose of classification of gases and vapors, the MESG limits are:

Class I, Zone	**Maximum Experimental Safe Gap**
Group IIA	MESG ≥ 0.9 mm
Group IIB	0.5 mm < MESG < 0.9 mm
Group IIC	MESG ≤ 0.5 mm

FAI has added testing capability to determine the MESG values of flammable gases or vapors. The test is performed in accordance with IEC 60079-20-1 and the setup is shown in Figure 1.

Figure 1: Setup for MESG Testing

The test equipment was verified by performing tests using propane and methane. The MESG values of the reference samples were determined in accordance to the IEC 60079-20-1 standard. Results obtained for MESG are compared with other reported literature values.

MESG Results of Propane and Methane

Material	**Concentration (vol.%)**	**Literature Values (mm)**	**FAI Results (mm)**

Propane	4.2	0.90[a]	0.94
Methane	9	1.16[a]	1.14

a. Data was obtained from IEC 60079-20-1 (2010)

For more information about MESG testing, please contact flammability@fauske.com or 630-323-8750

VSP2 – Everything You Need to Know About Setting Up Your Heater Assembly (VIDEO)

Ensuring that the VSP2 heater assembly is set up properly is crucial to performing a successful test. While the heater and heater glands are reusable parts, testing their integrity prior to beginning a test and replacing any questionable parts will help to ensure success. Further, care should be taken to insulate the test cell and heater assembly inside of the containment vessel as well as possible. The heat loss of the assembly during the test will be a determining factor in the overall quality of the test data. This article and video discusses the proper electrical testing and insulating procedures for the heater assembly and provides some setup tips and tricks.

Elizabeth Raines, Chemical Engineer

Heat Assembly Background

The VSP2 heater assembly is comprised of two separate heaters; the "Main" or "Auxiliary" heater (the "driver" heater used to raise the test cell temperature), and the "Guard" heater (used to maintain the adiabatic environment). The Auxiliary heater has approximately three turns of heater wire over the lower quarter and base of the test cell. The nominal resistance of this heater element is 18 ohms. After inserting the test cell in the Auxiliary (Main) heater, a layer of insulation is wrapped around the cell. The Auxiliary heater has wires with male prongs that are shrink wrapped in yellow tape. This insulated cell assembly in turn fits inside the Guard heater assembly. The Guard heater consists of two separate, connected circuits: the larger comprising the bottom and walls, and the top comprising the lid. These are connected using the smaller male and female prongs (these are wrapped with red shrink tape while the Guard heater's other connectors are larger male prongs and the standard assembly uses green shrink tape along these fittings). The nominal resistance of the Guard heater assembly (both elements connected) is 58 ohms. A dual zone "Multizone" Guard heater divides the Guard heater into two zones. The lower zone (controlled by TC2, green tape) comprises the base and lower half of the Guard heater while the top zone (controlled by TC3, and now includes additional blue wrapped male prongs) comprises the upper half. The nominal resistance of the base zone is 29 ohms and of the top zone is 33 ohms. This video and article focuses on the standard heater assembly, but the procedure and principles apply to the Multizone heater assembly as well.

Electrical Checks

Note, in the following pictures, the locations where the multimeter leads should make contact are identified by blue and red dots, respectively.

Heater

There are four primary checks to be performed to ensure your heater is in good working condition:

A. Verify that the resistance across the Auxiliary heater is approximately 18 ohms

- Turn your multimeter on and set to read resistance (might be shown with the Greek letter omega, Ω, or the word "ohms")
- Connect each of the leads from the multimeter onto each male prong on the Auxiliary heater

- The resistance across these two locations should read approximately 18 ohms

B. Verify that the Auxiliary heater is not shorted out and that the resistance is > 10 Mega ohms

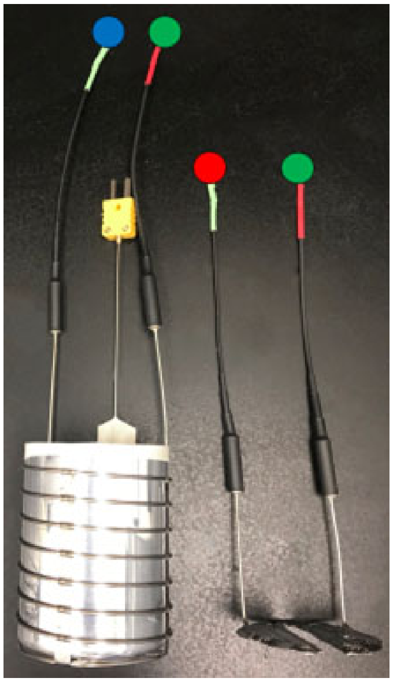
- Turn your multimeter on and set to read resistance
- Connect one of the leads from the multimeter to one of the Auxiliary heater male prongs and the other lead to a part of the Auxiliary heater coil

- The resistance across these two locations should read a very large resistance indicating that the heater is not shorted to the sheath (>10 Mega ohms)

C. Verify that the resistance across the Guard heater is approximately 58 ohms

- Turn your multimeter on and set to read resistance

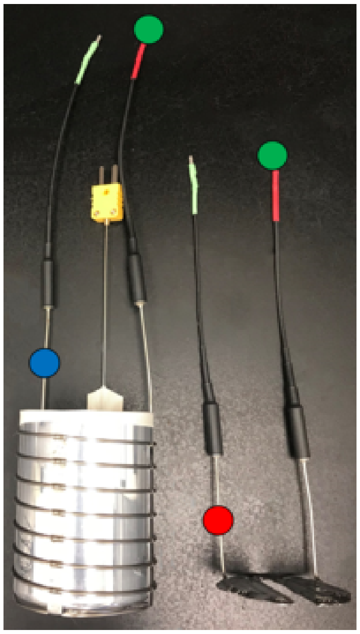
- Connect the lid heater to the base and lower half heater by connecting the two red smaller female and male prong (the green dots in the photo below)
- Connect each of the leads from the multimeter onto each of the green Guard heater prongs



- The resistance across these two locations should read approximately 58 ohms

D. Verify that the Guard heater is not shorted out and that the resistance is > 10 Mega ohms

- Turn your multimeter on and set to read resistance
- Connect the lid heater to the base and lower half heater by connecting the two red smaller female and male prong
- Connect one of the leads from the multimeter to one of the Guard heater male prongs and the other lead to a part of the Guard heater coil



- The resistance across these two locations should read a very large resistance indicating that the heater is not shorted out (>10 Mega ohms)

Heater Gland

There are four primary checks to be performed to ensure your heater gland is in good working condition:

A. Verify the heater gland continuity through the vessel

- Turn your multimeter on and set to read continuity (it might look this this symbol •))), otherwise set to resistance)
- Connect one of the multimeter leads into one of the female heater gland locations (on the interior of the vessel) and connect the other lead to the male prong on the exterior side of the vessel (touch the metal part on each side)

- When a complete path is reached, the multimeter will beep (and show a very small resistance of ~0.3-0.5 ohms). Only one pair of male/female parts should be detected as a complete path
- Swap pairs and test that the other wire is intact
- Repeat this procedure on the other set of wires

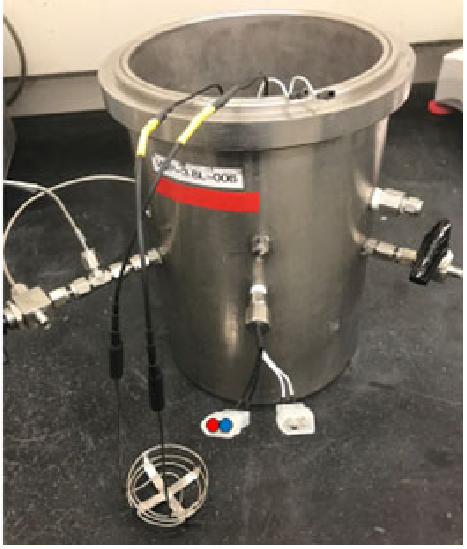
B. Verify the heater gland wires are not shorted to the vessel and that it is > 10 Mega ohms

- Turn your multimeter on and set to read resistance
- Connect one of the multimeter leads onto one of the male heater gland prongs and connect the other lead to the vessel

- The resistance across these two locations should read a very large resistance indicating that the heater gland is not shorted to the vessel
- Repeat this procedure on the other set of wires

C. Plug the Auxiliary and Guard heaters into the heater gland and verify that the resistance across the Auxiliary heater is 18 ohms and the resistance across the Guard Heater is 58 ohms

- Turn your multimeter on and set to read resistance
- Connect each of the leads from the multimeter onto each male prong on the exterior heater gland that is connected to the Auxiliary heater



- The resistance across these two locations should read approximately 18 ohms
  Note, the heater glands consist of two black wires and two white wires. The two Auxiliary heater wires (yellow) should be connected to either two white or two black. The two Guard heater wires (green) should be connected to the other two wires not used for the Auxiliary heater. Then, on the exterior of the vessel, the resistance across the two male prongs should match the resistance of the heater that is connected to it.
- Connect each of the leads from the multimeter onto each male prong on the exterior heater gland that is connected to the Guard heater

- The resistance across these two locations should read approximately 58 ohms

D. Plug the Auxiliary and Guard heaters into the heater gland and verify that the heater assembly is not shorted out to the vessel and that the resistance is > 10 Mega ohms

- Turn your multimeter on and set to read resistance
- Connect one of the leads from the multimeter onto one of male prongs on the exterior heater gland and connect the other lead to the vessel

- The resistance across these two locations should read a very large resistance (>10 Mega ohms)
- Repeat with the other prongs

Test Cell Installation

The recommended method of insulating and installing the VSP2 test cell is as follows:
Tip—lay out all the required materials before you begin wrapping the test cell to make the process as quick as possible.

1. Place the test cell into the Auxiliary heater.

   Note, it is good practice to record the weight of the test cell before (for phi-factor *calculations) and after (for measuring mass loss during the test) installing the required fittings.*

2. Wrap the test cell and test cell heater with 2 3/4" wide paper insulation. Use one full-length strip (24") followed by 1/4 of a strip (6"). Use masking tape to hold the insulation in place.

   Note, when wrapping the test cell with the paper insulation, it is helpful to tuck the starting end of the insulation strip underneath the vertical leads of the test cell heater. Subsequent wraps are on top of the test cell heater leads. This provides a snugger fit of the test cell in the insulation, minimizing the air gap between the test cell and the insulation. Elimination of the air gap prevents possible convective heat transfer and thereby tends to improve insulation performance particularly at high temperatures and pressures. Normally there is no problem seating the new test cell into the test cell heater, but a twisting motion is necessary for all-welded test cells that have a weld bead on the bottom lip.

   Tip—begin by using 3 ~2-3" pieces of masking tape to seal the seams of the paper insulation. Then use 5~6" pieces of tape to wrap the bottom of the test cell. This creates a smoother barrier that makes it easier to slide the test cell into the Guard heater.

   Note that the test cell should be insulated from the Guard heater on all sides – along the wall, top, and bottom with the same thickness insulation layer.

3. Place a 1/2-thickness disk of Fiberfrax insulation (about 1/4" thick) inside the Guard heater can.

   Note that the insulation can irritate the respiratory tract or skin if it comes in contact with it so use caution and the proper PPE.

4. Slide the wrapped test cell down into the Guard heater can.

   Tip—mark the thermocouples 1 for the sample thermocouple and 2 for the Guard heater (If using Multizone Multizone heater, mark 3 for the upper zone heater). This makes it clear on the proper connections when the test cell is installed in the containment vessel.

5. Place a full thickness Fiberfrax disk on top of the test cell. *Note, for the thermocouple or if a test cell with a vent is used, the disk on top of the test cell should be cut in half to make room for the different parts.*

   Tip—it is helpful to have a separate pair of scissors for cutting tape and for cutting the Fiberfrax disks. Cutting the Fiberfrax disks dulls the scissors making it difficult to cut tape over time.

6. Install the lid heater on top of the test cell assembly, crimping over the top edge of the Guard can if desired to hold the lid heater in a fixed position. A strip or two of white glass-cloth insulation tape can be used to help hold the lid heater securely on top of the assembly.

   Tip—it is helpful to align the test cell, Auxiliary heater, and Guard heater assembly so that *the heater wires remain close to each other to make it easier to make the required connections.*

7. Place a large Fiberfrax disk on the containment vessel floor. *Note, this current video shows the heater gland checks and the test cell insulation. A future video will walk through installing the test cell into the containment vessel and making the required connections.*

8. Install several large doughnuts (have a whole cut out from the middle for the test cell) of insulation in the vessel to just below where the fill lines connect.

9. Slide the Guard heater/test cell assembly down into this nest.

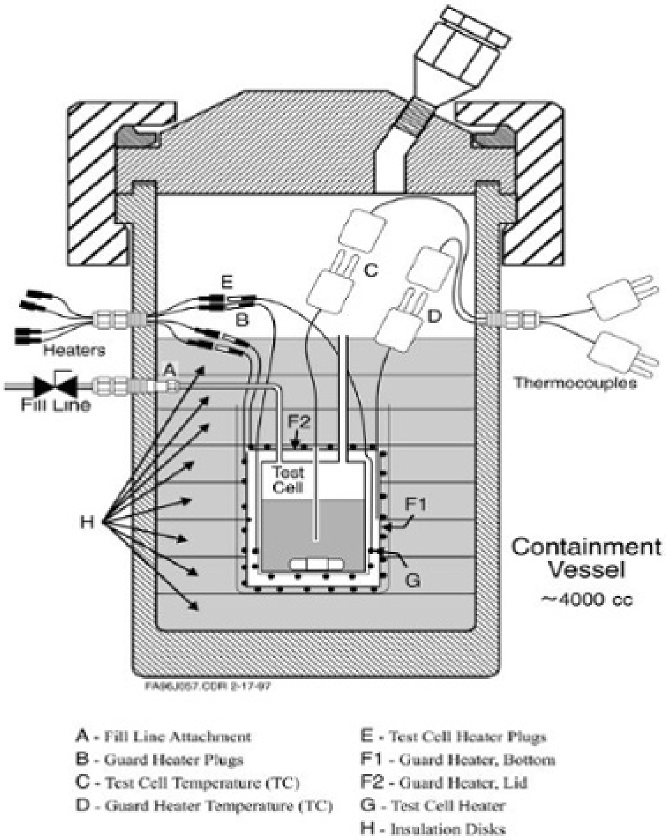
10. Connect the fill line(s) and Guard heater ground strap(s).
    Note, for the standard heater assembly, this can be accomplished by using a three way ground strap and connecting one of the lines to the fill line, and connecting to each of the red heater sheath.

11. Tighten the nuts to the fill line and the pressure line.

    Note, if used, support, the 1/16" fill line when bending it to avoid a sharp bend in the tube. Also, ensure that the tube are aligned with the 1/16" or 1/8" bulkhead fitting by first rotating the nut counterclockwise until a click is heard. Then tighten the nut finger tight. Using a wrench tighten the nut an additional 3/4 of a turn.

12. Continue installing doughnut segments (usually cut in two or three pieces so they can be fitted around obstacles).

13. Use small Fiberfrax discs on top of the test cell assembly, cutting them in half or notching as needed to fit around thermocouples and fittings. Insulate up to the upper containment vessel wall penetration. The overall assembly is illustrated below.



Adiabatic calorimeter testing provides data for relief system design, safe scale-up of chemical processes, and changes to process recipes. Safe process design requires knowledge of chemical reaction rates, character and energy release - all of which can be obtained from a low phi-factor adiabatic calorimeter such as the VSP2^{TM} (Vent Sizing Package 2) or ARSST^{TM} (Advanced Reactive System Screening Tool). If you are interested in purchasing either the VSP2^{TM} or ARSST^{TM} check out our store by clicking below.

Visit Our Online Store

Meet Engineering Intern Alexander H. Kaffka

I'll be entering my senior year this fall at The University of Iowa, where I study Chemical Engineering with my focus area in Energy & the Environment. I've always had a love for chemistry, and engineering has always been an interesting field to me. When I began chemical engineering courses at The University of Iowa, it was clear that I made the right choice. I enjoy collaborating with other engineers and creating solutions in a team-driven environment. Outside of school, I like to play guitar and to have game nights with friends. My family is big on community theatre, from painting and building sets to performing and directing.

-What will you be working on at FAI this summer?

So far at FAI, I have had the privilege of working in both nuclear and chemical aspects of the company. I am receiving a lot of useful modeling experience with programs such as MathCAD and FATE. I have also begun the process of archiving printed documents and creating searchable pdf documents from them. Beyond my desk, I have received a lot of great instruction on how to operate and understand the results of ARSST beyond what I have been taught in Iowa. My summer-long project is in the thermo-hydraulics lab, where I will be cleaning and maintaining a vortex suppressor unit. After the cleaning process is completed, next month I will be adding two filtration units to the overall unit. Receiving this diverse load of work has really been an interesting and exciting venture so far, and I look forward to continuing all of my current projects and starting new ones here at FAI.

-What classes in school do you think will useful for your projects here/what are your favorite subjects or labs?

The most useful classes that I took at The University of Iowa are Chemical Process Safety and Heat & Mass Transfer. Chemical Process Safety taught me a great deal about industrial safety topics, such as flammability limits and dust explosions. The course also covered the applications of ARSST instruments and the analysis of the data that they produce. Heat & Mass Transfer provided me with useful background for some of the MathCAD modeling that I am doing right now, including radiation and diffusion. My Organic Chemistry Laboratory and Thermodynamics & Transport Laboratory classes have helped me feel more comfortable within the lab setting. They have given me a lot of understanding on the handling and cleanup of chemicals during testing. My favorite course that I have taken so far is Green Chemical and Energy Technologies, where I learned a great deal about green energy alternatives and how to conduct environmental impact analysis from fuels and chemicals.

-Tell us about your experience using the ARSST at school?

One of the laboratory experiences that we experienced in our Chemical Process Safety course revolved around runaway reactions, which greatly utilized the ARSST instrument. Our goal was to run reactions (such as toluene and styrene in DTBP) at high and low pressures. The information found through experimentation was then used to calculate vent sizes for vessels. We were told to determine whether the reactions contained non-condensable gases, vapors, or a hybrid. The focus of the experiment was to relate the results of this experiment to applications in industry, and what measures a company should take to ensure that the reaction does not run away. I am grateful for my previous experience in the subject, and I am enjoying the more hands-on experience with ARSST that I am getting here at FAI.

-What do you think is the most useful safety lesson you've learned in school/how does Iowa incorporate process safety into their curriculum?

Chemical Process Safety is a mandatory course for all Chemical Engineers at the University of Iowa. It stretches across many aspects of safety within the chemical industry, and focuses mostly on accident prevention. We have studied a lot of different types of accidents that occurred across the world, what the source of their error was and how it could have been prevented. I think that the greatest lesson that we all learned from that experience was to analyze the mistakes from the past in great detail to prevent them from happening again.

Join our Relief Systems Design Course!

Relief Systems Design Course

Location: Fauske & Associates, LLC, 16W070 83rd Street, Burr Ridge, IL

Date: October 16 - 18, 2019

CEUs: 2.0 (20 PDH)

Time: October 16 and 17 (8:00 am - 4:00 pm)
October 18 (8:00 am - 12:00 pm)*

Price: $2,000

** A laboratory session demonstrating experimental techniques will be held the afternoon of October 18, but is optional.*

Course Description

Unlike other emergency vent sizing courses, this curriculum highlights simplified calculation methods capable of giving safe - but not overly conservative - relief system designs, with an emphasis on reactive chemistries and the role of two-phase flow.

Benchmarking of these methods will be illustrated with incidents and available plant data. Utilization of methods and equations will be demonstrated through practical design examples, covering vapor, gassy and hybrid systems.

Attendees will participate in group workshops and complete an independent quiz at the end of the course in order to ensure comprehension of the material.

**A laboratory session demonstrating experimental techniques will be held the afternoon of October 18, but is optional.*

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PROVIDER

Course Topics - Day 1

- Introduction to Vent Sizing and Case Study
- Vent Sizing Fundamentals
- Codes and Standards Explanation
- History of DIERS
- Two-Phase Flow Considerations
- Experimental Considerations
- Vent Sizing Based on All Gas or Vapor Venting

Course Topics - Day 2

- Vapor System Vent Sizing
- Gassy System Vent Sizing
- Hybrid System Vent Sizing
- Simplified Two-Phase Flow Methods for Vapor, Hybrid, and Gassy Systems
- Non-Reactive Fire Sizing

Course Topics - Day 3

- Stable Relief Valve Operation
- Discharge Coefficient Evaluation
- Containment and Disposal Considerations
- Lab Demonstrations (optional)

Learning Outcomes

- Understand up-to-date DIERS vent sizing methodologies and models, as well as the role of single and two-phase flow in venting behavior
- Perform vent sizing calculations using the correct models and methodologies
- Apply adiabatic calorimetry data
- Be able to use hands-on techniques and "rules of thumb" to ensure that realistic vessel and vent size conditions are specified

For hotel information or to register, please contact: FAIUniversity@fauske.com
Please direct course related questions to the FAI Thermal Hazards team: thermalhazardsgroup@fauske.com

www.fauske.com + + + **(630) 323-8750**

NFPA 652– An Introduction to Dust Hazard Analysis - Special Course at Aerodyne!

NFPA 652

– An Introduction to Dust Hazard Analysis

Special Presentation at Aerodyne Environmental

October 29 - 30, 2019

Aerodyne Environmental Offices
17387 Munn Road
Chagrin Falls, OH 44023

Course Description

Day 1	***Time: 8 am - 4:30 pm***	***CEU's: 0.7***

This course will ensure all participants are aware of important issues associated with NFPA 652 and describe how this standard interacts with other relevant NFPA codes and guidelines. A special emphasis will be placed on explaining the requirements for a Dust Hazard Analysis (DHA) and an overview of the methodologies that can be employed to perform a DHA. The course will also include a logical approach to characterizing a powder's hazardous dust properties, as well as a description of various techniques used to control and/or avoid dust explosions in a safe and compliant manner.

Scheduled Agenda

- Introduction
- Overview of NFPA 652
- Fundamentals of Dust Explosions
- Introduction to DHA methodology
- Mock DHA on a Small Blending Operation

Outcomes

- Protection Options
- Daily Learning Assessment
- Questions and Answers
- Course Evaluation Instruction

Day 2	***Time: 8 am - 4:30 pm***	***CEU's: 0.7***

Advanced DHA Workshop

The Advanced DHA Workshop will focus on how to organize, lead, and implement the DHA study. This will include how to utilize appropriate test methods to determine potential dust hazards; as well as how to apply appropriate mitigation techniques to prevent or control combustible dust hazards. During the workshop, participants will have the opportunity to apply DHA methodologies to realistic combustible dust scenarios.

ACCREDITED IACET PROVIDER

Special Aerodyne Environmental Pricing

Two Day Course: $895

Fauske & Associates, LLC is accredited by the International Association for Continuing Education and Training (IACET) and is authorized to issue the IACET CEU

To register, please contact: FAIUniversity@fauske.com, or (630) 323-8750
Please direct instructor or course related questions to Ashok G. Dastidar - dastidar@fauske.com

www.fauske.com **www.dustcollectorhq.com**

UPCOMING EVENTS IN 2019

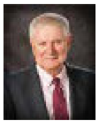
Visit with representatives from Fauske & Associates, LLC at these tradeshows and conferences in 2019:

- **NSC 2019 Expo**
  - September 9-11, San Diego, CA
- **Fall 2019 DIERS Meeting at FAI**
  September 16-18, Burr Ridge (Chicago), IL

DiERS

Come and see Fauske & Associates, LLC (FAI) at the

Fall 2019 DIERS Meeting
September 16-18
Chicago Marriott Southwest at Burr Ridge Burr Ridge, Illinois



Don't miss this opportunity to hear featured keynote speaker, Dr. Hans K. Fauske, present on predicting equilibrium and non-equilibrium flow.

- **Relief Systems Design Course**
  October 16-18, Burr Ridge (Chicago), IL
- **NFPA 652– An Introduction to Dust Hazard Analysis - Special Course at Aerodyne!**
  - October 29-30, 2019
- **2019 AIChE Annual Meeting**
  - November 10-15, 2019, Orlando, FL

We look forward to seeing you!

Dr. Fauske provided overall technical direction for the AIChE's acclaimed DIERS program formed in 1976 as a consortium of 29 companies to develop methods for the design of emergency relief systems to handle runaway reactions. Currently, Dr. Fauske is performing a key role in resolving potential process safety issues and the development of inherently safe nuclear and chemical process reactor concepts.

On Tuesday, September 17, FAI will host DIERS meeting attendees at our Burr Ridge, IL campus for lunch and a tour of our state of the art laboratory facilities.

To learn more about the Fall 2019 DIERS Meeting and how to register visit: https://www.aiche.org/design-institute-emergency-relief-systems-diers

We look forward to seeing you in September!

Fauske & Associates, LLC 16W070 83rd Street Burr Ridge Illinois 60527 USA