The Versatile VSP2 – Still Cooking After 26 Years

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Fauske & Associates, LLC (FAI) is well established as a complete one-stop shop for process safety testing and consulting. The Fall 2011 DIERS* Users Group meeting hosted by FAI, was a great opportunity to showcase our expanded facilities. Anyone who visited us in the early '90s would attest that our lab has come a long way since back in the day when the VSP and RSST ruled the roost. As we continue to add to our services, we also continue to improve our own calorimetry products. As DIERS veterans know, FAI is not just an instrument user but also a developer and manufacturer of adiabatic calorimeters since we designed and built the original DIERS Bench Scale Apparatus (forerunner of the VSP/VSP2) in the early 1980's. The VSP2 is now used worldwide by top chemical and pharmaceutical companies, and our own laboratory includes three VSP2 systems (Figure 1).

* Design Institute for Emergency Relief Systems, a subgroup of the American Institute of Chemical Engineers

Figure 1. Basic VSP2 system

The VSP2 uses recommended and established DIERS technology to identify and quantify process safety hazards so they can be prevented or accommodated by process design. It was introduced to DIERS in 1985 as the first pressure balancing and low thermal inertia ("low phi-factor") adiabatic safety calorimeter. This breakthrough meant that a lightweight test cell could be used to adiabatically contain a pressure-generating chemical runaway.

The 116 ml test cell in a standard VSP2 (Figure 2) is large enough to accommodate a representative 80 ml sample and a flexible test procedure (for example, metered or step-wise additions), but small enough to minimize waste and maximize safety. Similar to a process scale vessel or reaction calorimeter, the VSP2 measures temperature and pressure and has controlled dosing, excellent stirring and active temperature control to readily simulate normal process conditions. However the innovative design allows the VSP2 to also simulate upset (abnormal) conditions which might lead to a runaway chemical reaction (e.g., loss of cooling, mischarge of reagents, etc.). The unique features which allow direct (scalable) experimental measurement during such a runaway chemical reaction are the lightweight test cell, pressure balancing and adiabatic tracking.

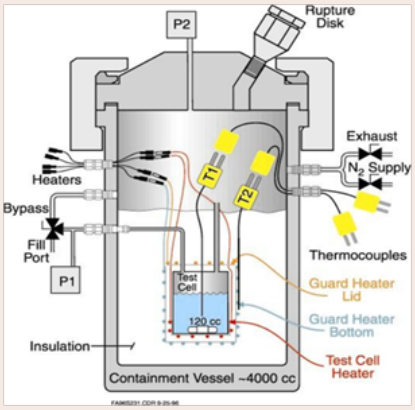

Figure 2. Schematic of VSP2 vessel

As more people use the VSP2 to study processes at higher temperatures and pressures (such as hydrogenations) we have seen increasing use of the Multizone Guard Heater option. This option provides localized and higher power heater control, resulting in significantly better adiabatic operation with minimal drift and better onset detection. For some customers use of the Multizone Guard Heater has become their standard lab practice.

Test cell design and materials of construction are virtually unlimited. We have supplied glass cells and cells with cooling coils. Two recently used designs are shown in Figure 3. Although still available, mechanically agitated test cells are rarely used anymore since comparable agitation can be achieved with the Fauske-designed "Super Magnetic Stirrer" (shown in Figure 1) which uses rare earth magnets to maintain coupling even for hard to stir samples. Newly developed large-opening EZ-seal test cells, convenient for loading solids, are available with O-ring or graphite gasket seals.

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Figure 3. Example Test Cells for H_2O_2 Contamination Study (left) and Emulsion Polymerization (right)

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How the VSP2 Can Help You Operate a Safer Chemical Process

Chemical process vessels require overpressure protection. Insurance statistics show that a too-common cause of vessel overpressure is an uncontrolled (runaway) exothermic chemical reaction. Example runaway reaction data from the VSP2 is shown in Figure 4 (iron contamination of H2O2).

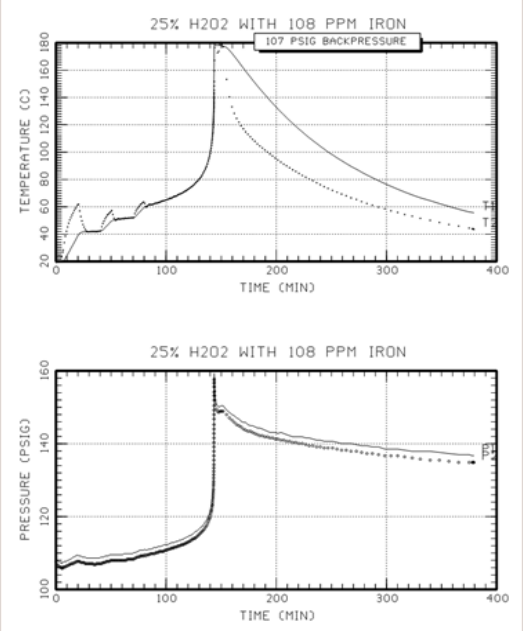

Figure 4. Example VSP2 data

Documented causes for runaway reactions include:

- Inadequate cooling or excess heating
- Incorrect charging
- Unknown exotherm or decomposition
- Contamination
- Incorrect agitation
- Incorrect batch temperature or pressure control

Some things to keep in mind:

- An adequate cooling system may no longer be adequate if the batch size is increased, if a partially reacted batch is transferred to another vessel without cooling or if a reactant or catalyst is overcharged or charged at the wrong time/ temperature.
- The reaction rate (and thus the energy/heat rate) typically increases by about a factor of 2 for every 10°C rise in temperature.
- Deviations above the normal process temperature not only increase the rate of the normal reaction but can lead to secondary (unintended) reactions and decomposition.
- The VSP2 is often used to run a normal recipe in adiabatic mode (loss of cooling scenario); the system may experience an exothermic runaway from normal process conditions or it may be necessary to heat the sample by steps to find the higher temperature where a runaway can occur.

  The exponentially increasing reaction rate and the adiabatic temperature rise are directly measured, as is any subsequent decomposition and gas evolution rate.
- Knowledge of the self-heating and gas evolution rates under such upset conditions can be used to develop a safer process, design appropriate emergency control or venting systems and train plant personnel in how to respond to the upset scenario.
- Emergency vent design should accommodate the worst credible upset. Such was not the case in the T2 Laboratories incident (2007). In that metallation process the operators did not recognize that the adiabatic temperature rise following a loss of cooling would lead to an undesired runaway reaction and decomposition. Failure to recognize the runaway reaction hazard had catastrophic results and put the company out of business.

VSP 2 Applications – Beyond Vent Sizing

Over the years the VSP2 has proved useful for developing a variety of data and knowledge about chemical substances and chemical processes, not only for relief system design. Although in many applications the VSP2 is not the only tool that can do the job, an impressive number of safety parameters can be measured, approximated or validated using VSP2 data. These applications include:

- Determine an appropriate temperature or pressure set point for a safety cutoff system
- Verify adequacy of injected chemical "short stop" or "poison" solutions to stop a runaway
- Determine required cooling rates to control a chemical runaway at a given temperature
- Identify onset of decomposition and decomposition products (samples can be taken for analysis during or after an experiment)
- Measure rates of gas generation during normal or upset conditions
- Optimize process design (charge amounts, charge rates, solvent, operating procedures)

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operators and production supervisor lacked a complete understanding of safe operating parameters and the potential consequences. The potential for a runaway reaction to occur if cooling (or power) was lost had not been investigated, documented or understood.

Process Hazard Analysis

The owner did not review the PHA. None of the original participants of the PHA still worked on site. The PHA had been completed by a previous owner over ten years earlier and then, only revalidated.

The revalidation was little more than a verification that the PHA report could be found if OSHA arrived on site. There was no evidence that recommendations were tracked, evaluated, implemented or rejected.

The objective of the PHA is to determine areas of excessive risk where preventative and mitigative measures may be warranted to better control the hazards. Merely performing a PHA is not enough if it is not properly inclusive and the recommendations not followed up.

Operating Procedures

Procedures did not include steps for emergency situations including a loss of power.

Good procedures provide clear written instructions for safely conducting activities of each covered process. These instructions should address operating limits, safety and health considerations and safety systems and their functions. Procedures need to exist for every mode of operation such as routine start up and shut down and emergency situations. Note that this element also includes maintenance procedures.

Mechanical Integrity

A written MI program did not exist but a preventive maintenance schedule did. The PM schedule included pumps, heat exchangers, the emergency generator and the pressure relief valves. However, accurate records of PM were not maintained.

A robust MI program will ensure the integrity and safe operation of process equipment through inspection, testing, preventive maintenance and quality assurance.

This element is one of the most cited PSM elements by OSHA.

Management of Change

There was an MOC Program. Unfortunately there was no apparent linkage between MOC, PSI, Training and PHA.

The plant operators must establish and implement written procedures to manage changes (except for replacements in kind) to process chemicals, technology, equipment and procedures and to facilities that affect a covered process. Changes that effect or alter PSI must be integrated through the other elements.

PSM Due Diligence in Mergers & Acquisitions

The company that experienced this loss had recently acquired the reactive chemical processing plant approximately 6 months earlier. A due diligence process was completed that considered environmental and plant safety factors. The ASTM Phase 1 and 2 inspections provided no show stoppers. The safety review efforts raised no workman compensation flags and no history with OSHA. In fact, the incident rate bettered the industry average.

The owner accepted responsibility for PSM as part of the due diligence process. He only considered two of the PSM elements and did not have the time or expertise to address them properly. The cost of PSM compliance was not understood because the owner did not perform a gap analysis.

Conclusion

FAI is focused on providing customers safety solutions to identify and prevent such high risk events from causing devastating consequences such as these.

FAI offers PSM development and support as well as due diligence assistance for Mergers and Acquisitions or compliance auditing purposes.

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- Thermal stability (for processing, storage, or transport) and Arrhenius kinetic parameters guide emergency response procedures by understanding timing and products of a runaway reaction
- Verify adequacy of quench system liquids to condense/neutralize potentially vented reactants/products
- Material compatibility
- Measure adiabatic temperature rise (ATR) and estimate heat of reaction, mixing or neutralization

In Conclusion

Even as our service and testing menu expands to meet the changing needs of our customers, we continue to rely upon the demonstrated excellence of the versatile VSP2. Like a traditional Chicago style pizza, now available with a variety of toppings, it remains a specialty of the house.

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