

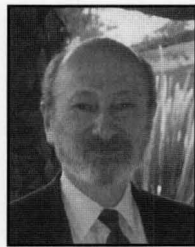
# A LABORATORY EXPERIMENT THAT ENHANCES ENVIRONMENTAL AWARENESS

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The goals of the senior-year chemical engineering laboratory course at Northwestern University are to nurture critical thinking skills so that students can analyze open-ended problems, to develop and improve the student's technical communication skills, and to provide experience with typical equipment and instrumentation. We try to accomplish these goals in one academic quarter by requiring student teams of 3-4 students each to run five different experiments (from a current total of eight, listed in Table 1), to prepare a detailed written technical report for each experiment, and to present an oral report as the final exam. Furthermore, we attempt to stimulate their thinking skills by purposely giving students very brief instructions for most of the more standard experiments so they cannot simply "follow the cookbook" in running the experiment.

A laboratory course essentially similar to ours, frequently designated as a "Unit Operations Laboratory," has been a core course in chemical engineering curricula for many years. Thirty years ago, most chemical engineering instructional laboratory equipment was large and multi-storied, while to-

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day the scale is usually smaller and more compact due to safety and space considerations. The unit operations laboratory has normally been the first opportunity for students, particularly those not in a cooperative education program, to observe and operate larger-scale equipment and to begin to appreciate some of the more realistic situations they might face in industry. Some of the concepts we hope they learn in the course are an appreciation of the difference between steady-state and transient operation and how long it can take to reach steady state in particular equipment, knowing when a computerized data acquisition system is giving realistic and reliable numbers, and how to use and draw reasonable conclusions from data that are limited and far from perfect.

In this connection, Dahlstrom, in a recent article on the history of chemical engineering education,<sup>[1]</sup> described the important role played by Olaf Hougen through his practical approach to education. Hougen's Principles<sup>[2,3]</sup> have particular relevance to the way we prepare chemical engineering students for industrial careers with such laboratory courses. Although Hougen listed twelve principles, the three most applicable to this course are

- *If you can't find relevant problems to give the student, then you shouldn't be teaching the material to the students.*
- *Well-founded and well-tested empiricisms are to be preferred over theories that have only a limited range of applicability.*
- *It is vital for engineers to know how to solve problems with limited or incomplete data.*

We continually try to keep the experiments relevant by phasing out older ones and introducing newer ones that

*Chemical Engineering Education*

expose students to problems of the day. For example, computerized data acquisition is standard in most petroleum refineries and chemical plants, so we have incorporated personal computers with data acquisition hardware/software for all of the more sophisticated experiments. Environmental awareness is another important theme and provides the motivation for the experiment discussed in this article.

## FLUID-BED INCINERATION

We have developed a new fluid-bed incineration experiment for the chemical engineering laboratory that we believe gives students useful exposure to solid and liquid waste treatment. A recent article by J. Mullen<sup>[4]</sup> described the use of fluid-bed incinerators for the destruction of hazardous wastes. Such an incinerator basically consists of a shallow fluid bed in which air, fuel, and combustible waste are fed into the bottom where combustion of fuel and waste takes place in the fluid bed medium, typically sand. The suspended solids/gas mixture has a vigorous boiling action and high heat transfer, which results in rapid and thorough mixing of the air, fuel, waste combustibles, and fluid-bed media. Some of the advantages of the fluidized bed combustor compared to other types of incinerator include efficient combustion, easy control, the ability to handle variable feeds, and much lower emissions of NO<sub>x</sub> and metals; the fluid bed typically operates at temperatures between 1400-1650°F. Combustibles are exposed to full combustion temperature for 5-8 seconds or more.

We decided to build a bench-scale fluid-bed incinerator for incorporation into our laboratory course. The basis for the design was provided by Ecova Inc., a subsidiary of Amoco Oil located in Denver, Colorado. They had developed a bench-scale unit for establishing design parameters on a commercial facility in Kimball, Nebraska, rated at an annual capacity of 45,000 tons of hazardous waste. This commercial unit was started up in the spring of 1994 but has since been shut down due to unfavorable economics and potential liability from its operation.

The laboratory unit was constructed in the Northwestern University shop over a three-month period. The entire unit, complete with instrumentation, was built for slightly less than \$10,000.

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(An additional \$5,000 would be required for analytical equipment, specifically a dual-column gas chromatograph and electronic integrator.) The unit is simple to run and seems to fit well into the framework of the chemical engineering laboratory.

**Experimental Unit Design** • The main elements of the experiment unit are the fluid-bed combustor, the feed systems for air, methane fuel, and waste material, and the analytical equipment. The feed gas monitoring and mixing chamber and the combustion unit are located in a fume hood.

The fluid-bed incinerator is a vertically-mounted 3-inch stainless tube, which is 24 inches long. It is enclosed with two semi-cylindrical electric heaters (3.5-inch ID), with a total heating capacity of 3.4 kilowatts, and 2 inches of insulation. Bed temperature is controlled with a solid-state Omega temperature controller that cycles current through a 220-volt relay and into the electrical heating jacket around the incinerator. The chamber is surmounted by a particle disengaging zone in which the diameter increases to 8 inches.

As shown in Figure 1, there are four ports along the combustor where temperature and pressure drop can be measured. Type K (chromel-alumel) thermocouples have been inserted into the ports, and a pressure tap, connected with a Swagelok® tee, is at the same location. Pressure drops are

measured using three Omega Engineering differential pressure (DP) cells with a full range of 20 inches water. The unit as described has a high height-to-diameter ratio that leads to bed slugging and a small freeboard height. For improvements in the design, we would suggest that the diameter be somewhat enlarged (by about 4 inches) and the freeboard increased to prevent fluidizing media from being blown out of the bed.

A side injection port is provided near the base of the incinerator to introduce wastes such as newsprint, plastic, rubber, and liquid hydrocarbons. It consists of a horizontal stainless-steel pipe with two 1/2-inch ball valves, one to seal the injection chamber from the hot fluid bed and the other to allow access to the injection chamber for loading with solid wastes. A piston is mounted on the end of a 1/4-inch tube

**TABLE 1**  
Northwestern Laboratory Experiments

- ▶ Pressure drop in fixed and fluidized beds
- ▶ Heat transfer in double-pipe and shell-and-tube exchangers
- ▶ Mass transfer in a wetted wall column
- ▶ Sucrose inversion in a plug-flow catalytic reactor
- ▶ Propanol dehydration in a differential catalytic reactor
- ▶ Fractional distillation of methanol-water in a multi-tray glass column
- ▶ Mixing and residence time distribution for a tank-in-series system
- ▶ Unsteady-state heat conduction in solids

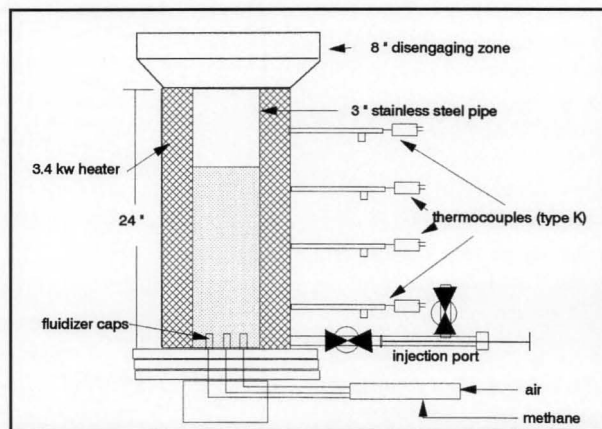


Figure 1. Fluid bed incinerator

that slides through an end cap. For solid-waste injection, the piston is advanced forward through the body of the ball valve and stops with the piston flush with the inside wall of the combustion chamber. If liquid wastes are to be pumped into the incinerator, the piston is left advanced and a small syringe pump feeds liquid into a tee located at the handle end and through the center of the tube.

The bed is filled with Mulcoa 47-20X-50S, an aluminosilicate medium (25x35 mesh) that can withstand higher temperatures than regular silica sand, but sea or river sand can also be used if desired since many commercial incinerators use it.

Air and methane from cylinders are metered through rotameters, premixed in-line in a mixing chamber, and then fed cold into the bottom of the fluid-bed unit through four jets consisting of sintered porous fluidizer caps. The caps are arranged with three in an equilateral triangle pattern and the fourth in the center. Excess air is used to fluidize the bed and lower the combustion temperature. Additional air purges are located on either side of the side injection port to clear the region of fluid bed media when a waste injection is being performed. A layout for the gas distributor is given in Figure 2.

Flue gas from the unit is analyzed with a dual column gas chromatograph equipped with two different columns and thermal conductivity detectors. One column contains molecular sieve 5A and measures oxygen, nitrogen, methane, and carbon monoxide, while the other contains Porapak<sup>®</sup>Q and primarily determines the amount of carbon dioxide and water in the flue gas. A Teflon gas bag is used to collect an average sample of the flue gas and is similar to the bag sample concept used for testing automobile emissions. Typical chromatograms for these two columns are shown in Figure 3. (The most important flue gas components for this experiment are carbon monoxide and carbon dioxide.)

## OPERATING PROCEDURES

### Start-up of Unit:

- Air flow is initiated at a moderate rate, somewhat less than the fluidization velocity.
- The temperature controller is then turned on and set to 800°F. In about thirty minutes that temperature is reached, and the temperature set point is increased to 1300°F, which is slightly higher than the auto ignition temperature of methane (1200°F).
- Once the temperature reaches 1300°F, the air flow is increased to start fluidization of the bed and methane is slowly introduced into the mixing chamber outside the bed. Ignition of the methane starts immediately. For the 3-inch diameter bed, we use 1 SCFM of air and 0.07 SCFM of methane (air/fuel ratio of about 16).

A small mirror is positioned above the top of the unit so that the bed can be observed during operation. "Light-off" of the incinerator is usually accompanied by a soft pop, after

which the bed begins to glow red. The set point to the temperature controller can now be shut down since methane combustion will sustain the temperature in the bed with no external heating. The adiabatic flame temperature of methane with a stoichiometric amount of air (9/1 air-to-fuel ratio) is 3500°F, so it is important to feed the fluidization air in excess or the incinerator will get too hot and severely damage the steel combustion chamber as well as fuse the bed medium into large aggregates. We suggest that a temperature controller with a high-limit switch be used to shut off fuel gas flow with a normally closed solenoid valve.

Once the bed has reached steady-state operation, wastes may be injected by means of the injection port at the bottom of the fluid bed. As noted earlier, the injection port has two stainless-steel ball valves (1/2-inch). The valves are mounted so that one serves as a vertical loading port, while the other is used to keep fluid bed media out of the injection chamber.

- To load the port with waste, the vertically mounted ball valve is opened and about 10 ml of waste material is dropped in.
- The valve is then closed and an air purge is started on both sides of the horizontal ball valve to keep the valve free of granular solids.
- The horizontal ball valve is then opened and a push rod is manually advanced toward the bed to pass through the ball valve and inject the solid waste into the fluid bed.

Injection of solid wastes is clearly an *unsteady-state* process; consequently, the observations made with solid wastes

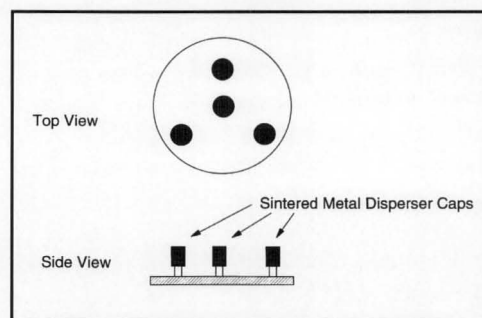


Figure 2. Gas distributor layout.

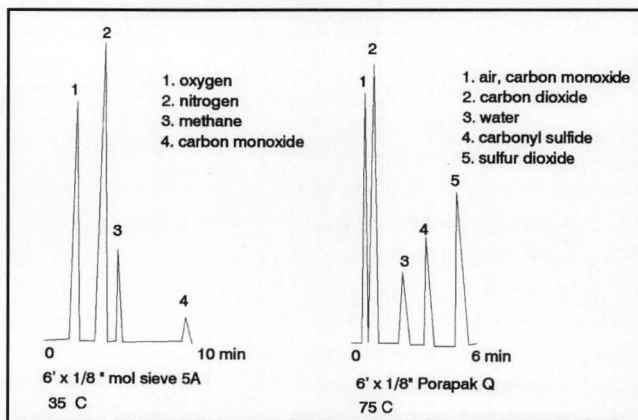


Figure 3. Gas chromatograph analysis of flue gas.

will be mostly qualitative. But steady-state conditions can be achieved with liquid wastes. The push rod consists of a hollow tube connected to a disc with a small hole drilled in it; this can be used with liquid wastes that are pumped continuously into the incinerator. It should be noted that the combustion reactions of the fuel as well as the waste materials produce large heat releases; dealing with these is considerably more stimulating and challenging for the student than the more typical use of air and water as process media in the laboratory setting.

## THEORY AND DATA ANALYSIS

This experiment provides opportunities for application of several different types of theory and data analysis.

**Minimum Fluidization Velocity and Pressure Drop** • The student needs to estimate the minimum fluidization velocity before starting operation of the incinerator. We find that you cannot exceed this value too much or granular material is entrained and blown out of the bed. Once the fluidization air rate is established, this controls how much methane fuel can be added for heating the unit.

### Combustion and Adiabatic Flame Temperature

$$U_{mf} = \frac{d_p^2(\rho_s - \rho_g)g}{1650\mu} \quad \text{Re}_p \leq 20$$

$$U_{mf}^2 = \frac{d_p(\rho_s - \rho_g)g}{24.5\rho_g} \quad \text{Re}_p \geq 1000 \quad (1)$$

where  $U_{mf}$  = minimum fluidization velocity  
 $d_p$  = particle diameter  
 $\rho_g$  = gas density  
 $\rho_s$  = particle density  
 $\mu$  = fluid viscosity  
 $g$  = gravitational constant  
 $\text{Re}_p$  = particle Reynolds number,  $(\rho_g U_{mf} d_p) / \mu$

**Stoichiometry** • We typically use methane as the fuel gas, but propane will also function well. But the heating value of propane is significantly higher than methane, so this must be taken into consideration.



**Adiabatic Temperature Rise** • The target temperature for the unit is 1400-1800°F, so the student must calculate the adiabatic temperature rise for some typical fuel gas feed rates. The approximate equation for temperature rise with an energy balance yielding the more exact expression is

$$\Delta T = \frac{-\Delta H_r}{C_p} \quad (2)$$

where  $\Delta T$  = temperature rise  
 $\Delta H_r$  = heat of reaction  
 $C_p$  = specific heat of feed stream

## GENERAL PROCEDURE FOR TESTING AND ANALYSIS

The fluid-bed incinerator experiment is reasonably challenging for even the best students. Consequently, we recommend that it be placed midway or later in the laboratory course so that the students will be more experienced with operating procedures and not require such close supervision. We usually schedule the laboratory experiments so that the students have operated the fixed/fluid bed experiment first and have some appreciation of what fluidization is by observing it in a clear tube with small glass packing. They are then better prepared to run the fluid-bed incinerator unit and to address the aspects of fluidization, combustion, and incineration of industrial wastes.

The experiment is performed over two laboratory periods of eight hours each. During the first period, the students start up the unit, measure temperature and pressure drop across the bed and analyze the flue gas by filling the Teflon bags, and then inject samples into both columns of the gas chromatograph with a gas syringe. During the second period, wastes are fed into the fluid bed through the injection port. Small pieces of polystyrene, plastic eating utensils, and rubber stopper are first dropped into the top of the bed to observe incomplete combustion at short residence times (typically yielding black particulates or soot). Then the same materials are injected into the bottom port of the fluid bed incinerator for essentially complete combustion under conditions of longer residence time and better heat transfer. Also, a bag sample of the exhaust is taken while the waste injection is performed to compare to the operation with no waste incineration. The following questions are proposed for the students to think about and answer in their reports:

- Does an energy balance on the unit predict an outlet temperature close to what was observed?
- How do the measured pressure drops and temperature distribution relate to theory?
- Does the minimum fluidization velocity calculation agree with what you actually observed?
- Discuss the flue gas composition and the distribution of carbon monoxide/carbon dioxide.
- Do you think significant nitrogen oxides (NOx) formed in the fluid bed incinerator?
- What about dioxin formation? Besides hydrogen and carbon, what is the key element needed to make dioxin in the flue gas and what temperatures favor its formation?

## CONCLUSIONS

The new fluid-bed incinerator experiment has lived up to our expectations and has given a new spark to the array of teaching experiments used for the unit operations laboratory. It seems to satisfy a number of the criteria in Hougen's Principles. More specifically, it is a relevant industrial prob-

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personal problems among students). The engineering faculty advisor is unlikely to possess the requisite special training and skills to deal with such issues effectively, and a well-intentioned attempt to help might even worsen the problem. It is best to refer such problems to trained counselors available at any university. To make sure that the student receives help, the advisor may insist that an appointment be made right then and there. Afterward, the advisor should follow up with the counseling office to make sure that the student kept the appointment, as well as check with the student periodically so the student knows someone is concerned. In order to deal with such crises, the advisor should be aware of all the relevant campus resources, along with contact names and phone numbers—before any crisis occurs.

## CONCLUSION

The central premise of this article is that the advising process is an integral part of the educational process. Unfortunately, it is too often misinterpreted as a purely clerical task and receives only limited attention by the faculty, students, and administration. A valuable systemic change would be the separation of the clerical and developmental sides of advising; the former can be handled by staff, allowing the faculty's full attention to be devoted to the intellectual growth of the students.

Faculty advisors should strive to improve the strategies they follow in encouraging student contact, acting in a teaching and supportive role, allowing the students ultimate decision-making and responsibility, and helping students to focus on the greater educational and professional decisions and objectives and the means for accomplishing them.

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## LABORATORY EXPERIMENT

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lem, particularly for the 1990s, it is based on well-tested combustion phenomenon and not limited to a special limited situation, and the data from the unit are not complete, forcing the soon-to-be practicing engineers to solve problems and perform an analysis based on their best judgment.

The experiment is best performed over an entire day, so trying to carry it out in a half-day session is not recommended. We strongly advise that the entire experiment be located in a fume hood so that the flue gas is swept out of the unit and no dangerous or noxious odors are emitted into the laboratory. Finally, the potential to overheat the fluid-bed unit from feeding too much fuel gas means that the students need to be monitored periodically to be sure they are operating the unit in a controlled and safe manner. The use of a high-temperature limit switch will eliminate this potential problem.

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