Academia and Industrial Pilot Plant Operations and Safety

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Academia and Industrial Pilot Plant Operations and Safety

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Foreword

The ACS Symposium Series was first published in 1974 to provide a mechanism for publishing symposia quickly in book form. The purpose of the series is to publish timely, comprehensive books developed from the ACS sponsored symposia based on current scientific research. Occasionally, books are developed from symposia sponsored by other organizations when the topic is of keen interest to the chemistry audience.

Before agreeing to publish a book, the proposed table of contents is reviewed for appropriate and comprehensive coverage and for interest to the audience. Some papers may be excluded to better focus the book; others may be added to provide comprehensiveness. When appropriate, overview or introductory chapters are added. Drafts of chapters are peer-reviewed prior to final acceptance or rejection, and manuscripts are prepared in camera-ready format.

As a rule, only original research papers and original review papers are included in the volumes. Verbatim reproductions of previous published papers are not accepted.

ACS Books Department

Editors' Biographies

Mary K. Moore

Ms. Mary K. Moore is an Innovation Process Manager in Process and Applications Innovation, Strategic Technology at Eastman Chemical Company in Kingsport, Tennessee. Ms. Moore has extensive experience in designing, constructing, and operating lab, bench, and pilot scale units for separation processes, organic synthesis, organic metallic chemistry, and experimental scale-up. She has a broad range of experience in experimental design and data analysis. She has received seven United States Patents. Ms. Moore graduated in 1991 from Northeast State Technical Community College with an Associate of Applied Science degree in Chemical Technology and was the recipient of the 1991 Outstanding Graduate Award. In 2012, she received the Outstanding Alumna Award. She has served in a number of positions in the American Chemical Society (ACS): Division of Chemical Technicians, Committee on Technician Affairs, Industrial and Engineering Chemistry Division, ACS Career Services, Division Activities Committee, Northeast Tennessee Section of the ACS, and Tennessee Government Affairs Committee. She was honored to be named to the 2009 ACS inaugural class of Fellows.

Elmer B. Ledesma

Dr. Elmer B. Ledesma is an assistant professor in the Department of Chemistry and Physics at the University of St. Thomas in Houston, Texas. His expertise and research specialty is in the pyrolysis, gasification, and combustion of fossil fuels and biomass. Dr. Ledesma conducted his dissertation research on the pyrolysis and combustion of coal volatiles at the Commonwealth Scientific and Industrial Research Organisation (CSIRO) Division of Coal and Energy Technology in North Ryde, Australia, and was awarded his Ph.D. by the University of Sydney. He conducted postdoctoral studies in the Department of Mechanical and Aerospace Engineering at Princeton University and worked as a research associate in the Department of Chemical Engineering at Louisiana State University. Dr. Ledesma serves as a program chair in the ACS Division of Industrial & Engineering Chemistry and is a reviewer for *Energy & Fuels* and *Industrial &Engineering Chemistry Research*. He is a member of the ACS (Division of Energy & Fuels and Division of Industrial & Engineering Chemistry), the International Union of Pure and Applied Chemistry, the Combustion Institute, and the American Institute of Chemical Engineers.

Preface

This symposium series volume was developed in order to share papers presented at the 245th ACS National Meeting, held April 7–12, 2013 in New Orleans, Louisiana. We believe this a first publication in this general area. The Industrial and Chemical Engineering, Applied Chemical Technology Subdivision hosted a one-day symposium for industry and academic researchers to present and share their work and best practices on operations and safety in pilot plant environments.

Designing and building "pilot plants" can be a big challenge, both to assure proper operations and to run the plant safely. Regardless if you are designing a plant in academia or industry, the safe operations of the pilot plant should remain a priority. One concern in building a pilot plant is the available real-estate. When designing and building pilot plants, several processes are considered such as chemical separation, chemical reactions, waste generation, process controls, etc. Plant designers and contractors also need to consider ease of operation and maintenance, pinch-points, sampling, etc.

In contrast to workers in large-scale facilities, pilot plant workers in both industry and academia are not only exposed to relatively small amounts of hazardous substances, they are exposed to such materials in a smaller setting and thus to potentially greater concentrations. We feel, therefore, that an opportunity existed to bring together workers and researchers in both industrial and academic pilot plant environments to present and share their best practices as regards operations and safety.

On the cover: Picture supplied by Eastman Chemical Company.

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The views presented here do not necessarily represent the views of my employer, and I do not have the endorsement of, or authority to speak on behalf of, my employer.

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Chapter 1

Academia and Industrial Pilot Plant Operations and Safety

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"Academia and Industrial Pilot Plant Operations and Safety" is written for the college or university environment in which students are developing the skills necessary to function within the process industry whether as an operator, researcher or design engineer. Proper management, eclectic exercises, training and hazard awareness are key parts of developing a safe and progressive learning environment. A clear and logical progression of necessary skills are essential to the development of responsible and safe process operations personnel. The student learner should be challenged on a daily basis and a dynamic approach to learning should be exercised by the instructor. This chapter will stimulate ideas while highlighting time tested techniques and methods currently being used.

The operation of a pilot plant in industry provides a necessary scaleup from the benchtop at an intermediate step before beginning full scale production. In most cases, the development of new compounds and their associated reactions will take place in 1-2 liter vessels while under initial research and development. Pilot plant vessels range from 100-500 liters and full scale production vessels are typically 20-40k liters. Pilot plant facilities operated within industry are often dealing with unknown hazards and materials that have not been previously reacted at this scale and often before any long term EH&S data has been compiled. Researchers are highly skilled and every precaution is taken to ensure that employee exposure and and any negative environmental impact are reduced or eliminated. Careful record keeping and strict GLP (good laboratory procedures)

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are followed. High Tech facilities utilize “soft” tools such as PHA (Process Hazard Analysis), PSM (Process Safety Management) and CHP (Chemical Hygeine Plans) to best prepare for any unforeseen – yet predictable occurrence. Focus is on repeatability, predictable and incremental changes, and detailed analysis before any deviation is allowed. While very useful, not every project moves forward with a complete and accurate P&ID (Piping & Instrument Diagram) to aid with their PHA efforts.

Researchers are experienced, most often have worked together in similar environments and have open and functional communication between all team members. Every precaution is considered during pre-task planning in a concerted effort to eliminate any “suprises” that may occur once an experiment begins. Still, accidents and exposures do occur within the pilot plant industry. If one scans the statistists published on the OSHA.gov website some patterns emerge. These include incidents involving poor or inadequate instrumentation, poor or little control of unwanted combustion within confined spaces. This can best be completed by 1 or both of the following:

1) **Nitrogen inerting**-while Nitrogen introduces an additional asphyxiation hazard to the laboratory environment, it has a long history of enhancing safety through the reduction of explosion hazards. Since the use of only non-sparking equipment is very improbable, the removal of oxygen of confined spaces is then a very viable method to reduce/eliminate explosion hazards. Students should be instructed in the hazards associated with nitrogen including:

    - Odorless and colorless gas (poor warning properties).
    - Commonly used throughout the chemical industry.
    - Operations personnel often become complacent in working in areas where nitrogen gas is in use.

2) **Bonding and grounding of equipment**-since the transfer of almost every solid, liquid, gas or slurry creates a static charge, the bonding and grounding of process equipment is essential to prevent unwanted discharge of electrical potential – often the root cause of explosions within pilot facilities. The proper maintenance of this system is key to ensure electrical continuity as equipment and lines are disassembled for inspections and cleanout. This requires well trained operators and a re-commissioning process for putting equipment back online. Often this means the use of a electrician’s multimeter before equipment can be considered “bonded”.

While pilot plant operations within industrial settings involve hazards associated with reactions and mixtures that are relatively “untested”, the hazards associated with pilot plants within academic settings involve potential hazards associated with the lack of knowledge of the students. Unlike the trained professionals that inhabit the real world counterpart, student learners lack the same experience and knowledge. Therefore careful planning, preparedness and open communication must be emphasized in order to minimize hazard.

Ideally, a 50/50 mixture of classroom lecture and hands on laboratory exercises should be considered in order to ensure an optimum learning environment. Through experience we have learned that a 4 hour period is on the average the longest that a group of students should be subjected to. After 4 hours, focus and attention tends to decline and the learning curve begins to decline. Additionally, within the laboratory the incidence of exposure to hazards would also increase.

The goal of the classroom lecture is to keep pace with the current hands on exercise. Incrementally, new skills are added at a pace determined by student learning progress and that of an experienced teacher. Common themes that must be emphasized and supported from the very first day include safety, teamwork, communication, troubleshooting skills, documentation, and record keeping. Every attempt should be made to emulate the standards and culture of the industry you are preparing the students for when seeking employment. For technical schools and colleges this usually involves local industry. For universities, a broader based approach may best serve the student learner.

Industrial facilities within your geographic area most likely will be very helpful, if not enthusiastic in helping your staff with setting up program parameters. The involvement of these companies as members of an "advisory board" not only provides real time input, but also develops a liason for spare parts, jobs for your graduates and often a pool of well qualified guest speakers or adjunct instructors. The utilization of SME's (Subject Matter Experts) when developing your program, or when making changes to your program, is the best way to stay current with area hiring needs and may provide a cost effective way for handling expensive repairs as well as reduction in the financial impact of capital projects. If asked, most area companies may willingly loan skilled trades workers to help with your maintenance needs. For example: most well equipped pilot facilities have instrumentation and DCS systems that are far beyond the knowledge of a "general electrician". In these cases, access to a journeyman "instrument technician" is invaluable. Since this trade is so specialized, they are most likely employed only by mid-sized to larger industrial facilities.

Therefore, the major difference between an industrial pilot plant and an academic pilot plant lies in the "unknown" factor involved. In industry this involves the hazards associated with unknown reactions, compounds/chemicals, and the pressures associated with deadlines and corporate culture. With pilot plants within academic settings, the unknown lies with the student learner.

Safety

Safety, in any setting is a mindset. Safety within a workgroup, is a culture. When workers are asked 2 simple questions, this is easily exhibited:

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Q1: What percentage of workers in your plant comply with all safety standards and PPE requirements during the day shift when management is present?

Q2: What percentage of workers in your plant comply with all safety standards and PPE requirements during the off shift when there is no management present?

Employees will agree that there is a moderate to significant drop in safety compliance when workers are not being observed. It is important to note that the hazards in the workplace actually increase during the nighttime and afternoon shifts. Worker fatigue, lack of adequate lighting, fewer employees to share tasks, as well as many other factors are all contributors. <u>Therefore, one can easily extrapolate that as the potential for a hazard increases during the "off shifts", the actual compliance with safety standards and PPE requirements decreases.</u> Data supports that the likelihood of an accident to occur is much more likely during the night shift. Most famously are major catastrophes such as the Exxon Valdez, Chernobyl, Titanic, Piper Alpha Rig, and Three Mile Island disasters.

Therefore, it is extremely important that students learn safety from day one and that it is emphasized at all aspects of their learning process. Students must learn how to recognize hazards, this is done through training and the consistent application of all standards by their instructors. Students should also learn to intervene when fellow students are at risk due to improper PPE and/or compliance with laboratory rules and safety standards.

The culture of safety within a work environment is an accumulation of behaviors and the attitude of those workers. This may be affected in a positive or negative manner by the following factors:

1. Workplace Culture
2. Complacency
3. Training
4. Hazard awareness
5. Distractions/motivation
6. Focus
7. Communication

The implementation of BBS (Behavior Based Safety) has helped to revolutionize safety for a large portion of the chemical processing industry both within the United States and abroad. BBS also fits very well in the academic laboratory setting and helps the student to build the necessary safety awareness and attitude coveted by future employers. Simply put, BBS safety requires that the students and instructors:

1. Keep a positive environment
2. Celebrate the good points (large or small)
3. Set goals – success mapping
4. Utilize teamwork and positive intervention
5. Set the right example

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6. Develop a safety "culture"
7. Develop "habit strength" behaviors towards safety performance

"Everyone likes to hear they are doing a good job!" Reinforcing behaviors POSITIVELY is the driving force to the success of BBS in the reduction of accidents in the workplace. While BBS provides the vehicle in which a successful safety culture thrives, several tools are also necessary and available to keep safety performance in the forefront:

Measure

Keep a spreadsheet of the team's safety performance history and always make it visible to the students. Use a formula of (# of students X laboratory hours X laboratory days X Classes being held) to get "Total Consecutive Student Laboratory Hours Without an Accident" Students will respond positively and take pride in these statistics.

Celebrate Success

Set safety goals and celebrate when they are reached. Students will associate safety performance with rewards.

Maintain a POSITIVE Environment

This helps to reduce the negative effects of the factors related to cultural and industry related influences.

Accountability

Ensure students are accountable for their own performance. This includes sign in/sign out sheets, attendance, safety compliance, record keeping and ability to follow established SOP (Standard Operating Procedure). It is suggested that each of these are measured, and become a part of each student's scoring rubric.

Involve Students in Decision Making

- Develop a discussion panel that encourages student input (safety, technology, budget).
  - Involve students in RCI's (Root Cause Investigations)
  - Encourage student participation in advisory panel discussions.
  - Require the writing of SOP (Standard Operating Procedure) as part of graded curriculum.

Stay Current with Industry/Regulations

Instructors should visit/tour area plants and discuss the needs of local industry. Training in EH&S (Employee Health and Safety) is readily available and state OHSA extensions offer free or relatively inexpensive training options.

Incorporate Industry SME's (Subject Matter Experts)

The involvement of area experts is invaluable to your program, your students and therefore the industry as a whole. Most area employers are very willing to assist you with your needs – including providing expert help (instruction, maintenance, inspections, advisory) whenever asked.

Preparing Students for Employment in Industry

The best way to satisfy employment needs, as well as to best prepare the student for real world employment, is to utilize all of the tools being used by area industry.

Good Laboratory Practices (GLP)

Proper documentation, log entrys and corrections as per industry standards. Once introduced, the student's laboratory writeups must be closely monitored so as to prepare them for proper documentation compliance once employed. This includes the use of black ink, mistake correction (single line, initialed and dated), not skipping data entry boxes, not erasing and the elimination of correction fluids (*1*).

Standard Operating Procedures (SOP)

Students must learn to interpret, adhere to and troubleshoot with SOP's. Students should learn to write, test, develop and publish SOP's. These include P&IDs (Process and Instrument Diagrams), startup and shutdown procedures (*2*).

BOLS (Breaks-Odors-Leaks-Spills)/LOPC (Loss of Primary Containment)

Forms obtained from local industry and used in the pilot facility will help students prepare for employer's expectations for environmental release reporting and participation with maintenance trends. Students have to identify, log in, clean up and report any spills resulting from Loss of Primary containment. A monthly review of these forms as a class helps to identify redundant issues, target PM (Preventative Maintenance) efforts and open communication between work groups.

Preventative Maintenance (PM)

Students should learn to perform all preventative maintenance to keep pilot plant in peak operating condition. Once PM interval spreadsheet is developed (with student input), students should be given responsibility. Tasks include lubrication, non destructive testing, line labeling, valve maintenance/packing, vibration testing, inventories and other tasks unique to your laboratory (*3*).

Troubleshooting Methodology

Troubleshooting is an essential competency to any employee in the chemical processing field. This is a valuable tool that will enable a student to approach an abnormal condition in a logical, organized and calm manner. The student must learn to recognize, investigate, prioritize and find a solution to process problems. Below is a suggested seven step process:

1. <u>Identify the problem</u>

    - The first step to solving any problem is to recognize that a problem exists, and then define it.

2. <u>Identify any potential causes</u>

    - Once a problem is found, this step is used to identify all potential sources/causes. It is important in this step to not omit any potential causes/sources of the problem step to ensure that all potential sources are considered.

3. <u>Narrow the possibilities to the "likely" cause</u>

    - Implementation of past experience with this equipment, worker's knowledge, conditions specific to this equipment will lead operator to the most likely cause of problem.

4. <u>Draw preliminary conclusions</u>

    - In this step a scenario should be developed that could reasonably explain how the likely cause in step 3 could result in the identified problem from step 1. This will lead personnel to consider all ancillary causations including those that may affect quality or safety.

5. <u>Prove conclusions</u>

    - In this step, the operator uses multiple process indicators (e.g., DCS (Distributed Control System) indications, field indications, sensory information, etc.) to verify that his or her conclusions

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are correct before taking action. This is the most important step in the methodology because it prevents taking an incorrect action. This step, more than any other, requires an in-depth understanding of the process operation and variable interactions

6. Implement corrective action(s)

    - Here, personnel will take action to correct the original problem and return the process to safe – or steady state condition.

7. Document

    - A Key step to operations excellence! Once the problem has been corrected, the last step is to document the problem, its solution and, most importantly, the troubleshooting process. Proper documentation prevents future occurences, streamlines your process and helps eliminate unnecessary downtime.

Pre-Task Analysis

A pre-task analysis is a formal way to ensure that a team is focused and best prepared to perform a task on a specific piece of equipment. Typicall in a written form, it is a tool to help a workgroup focus on the task at hand, identify hazards and communicate with one another. If properly implemented, the PTA will help a workgroup transition into a focused and coherent group. For example: discussing emergency shutdown procedures in a calm and deliberate setting will prove invaluable in times of emergency shutdown, often under duress and with limited time. Studies show that a PTA significantly improves a teams preparedness in times of emergency and in preparing for an upcoming task/job (*4*).

Laboratory Sign in/Sign out

In industry, all workers are required to diligently sign in and out of a building so as to be accounted for in the occurrence of a plant evacuation or assembly exercise. There should be no exception to this when it comes to the academic setting and will not only ensure accountability, but also help student to prepare for a position post graduation.

Tailgate Sessions

Each lab class should begin with a 5-15 minute tailgate session which helps the student transition mentally to being the best prepared student possible. In these brief instructor led sessions, the introductions of new skills, reinforcement of previously learned tasks and a reiteration of safety focus are recommended.

Safety Shower/Eye Bath

Students should learn how to test and take care of the safety shower and eye bath stations. This includes a weekly water clarity and temperature check. Area should be clean and clear of debris so there are no obstacles to impede a person with impaired vision attempting to get to the safety station. The eye cups should be in place at all times on the eye bath station so no foreign objects would additionally injure the person seeking help. As expected in industry, future operators should learn how to maintain this essential safety equipment (*5*).

Lock Out – Tag Out

The objective of the Lockout – Tagout process is the control of hazardous energy. All operating personnel must become familiar with the Lock Out/Tag Out standard. Whether as a Authorized or Affected worker, a functional understanding of this standard is key to a safe career as a process operator/laboratory operator. Students should learn to plan and perform Lock Out/Tag Out and learn how to return equipment that has been secured back into service (*6*).

Root Cause Investigations (RCI)

An invaluable tool implemented post-incident to help team to determine pathology of a safety or quality issue. It is recommended that instructor facilitate the procedure and solicit input from students representing the work team. Provides a lucid method of gathering ideas and forming a solution. Promotes buy-in from all team members and helps form a positive platform in which to encourage new ideas.

Personal Protective Equipment (PPE)

PPE standards must be developed to address all hazards that may be encounted within the laboratory environment. Additionally, students must be taught how to don, doff and properly clean and store PPE equipment. Included in the training should be a classroom portion to discuss theory and a practical application on the laboratory floor. It is suggested that students learn to complete everyday tasks (sampling, using hand tools, filling out runsheets, etc) while wearing PPE so as to understand the barriers/restrictions that can arise while in the Pilot Plant environment (*7*).

National Fire Protection Act (NFPA)

Participation in the labeling of process equipment as per the National Fire Protection Act will aid the student in recognizing and adhering to the recommended standards. Theory should be emphasized in the understanding of the color and numbering system as well as the icons for special hazards (*8*).

Sampling Safety

The safe sampling of process fluids, gasses and solids is a key part of a process operators position. Students in the pilot plant environment must learn how to sample safely, sanitarily, and utilize good GLP methods. A sampling procedure should be written to aid the student (figure 1).

An example of sampling procedure:

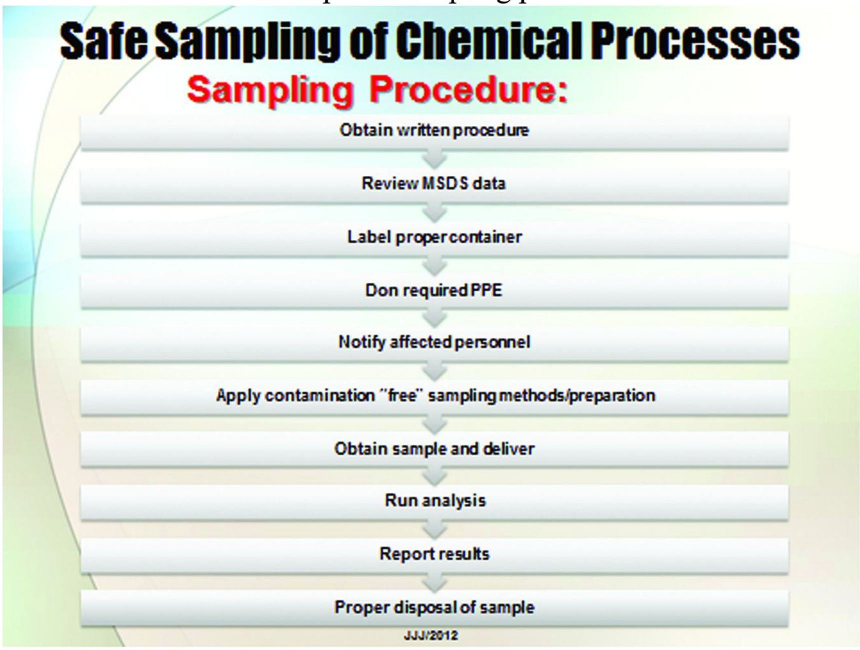

Figure 1. Field Sampling Method

The chart below can be utilized to help the student understand the matricies involved with the sampling of production streams within a chemical plant (figure 2).

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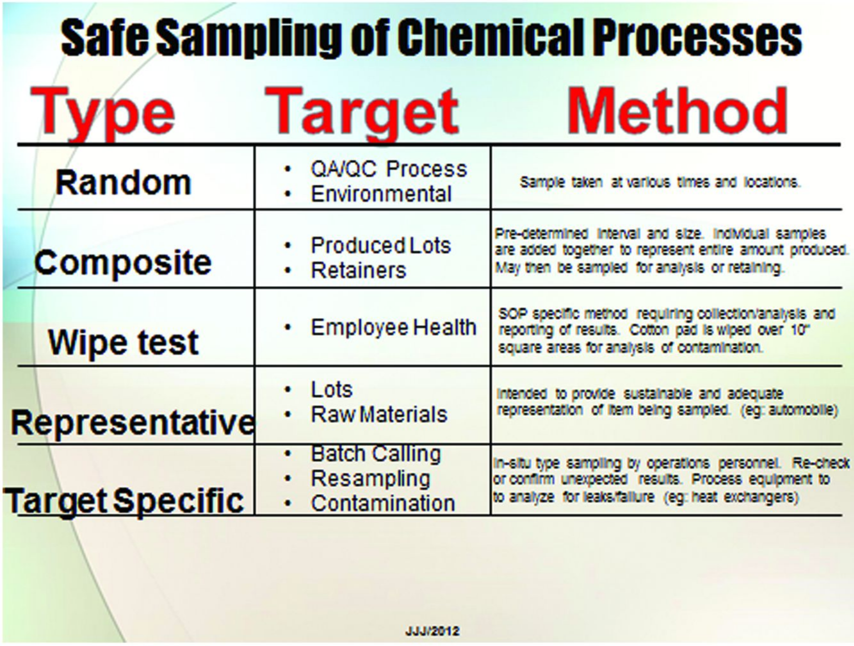
Types of in situ sampling to be completed in laboratory:



Safe Sampling of Chemical Processes

Type	Target	Method
Random	• QA/QC Process • Environmental	Sample taken at various times and locations.
Composite	• Produced Lots • Retainers	Pre-determined interval and size. Individual samples are added together to represent entire amount produced. May then be sampled for analysis or retaining.
Wipe test	• Employee Health	SOP specific method requiring collection/analysis and reporting of results. Cotton pad is wiped over 10" square areas for analysis of contamination.
Representative	• Lots • Raw Materials	Intended to provide sustainable and adequate representation of item being sampled. (eg: automobile)
Target Specific	• Batch Calling • Resampling • Contamination	In-situ type sampling by operations personnel. Re-check or confirm unexpected results. Process equipment to to analyze for leaks/failure (eg: heat exchangers)

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Figure 2. Process Sampling Types

In addition, students can run analysis on a variety of process samples and depending on the equipment available to them. Some examples of "typical" analysis performed by process operators:

- Mass Spectrometry
- Gas Chromatography
- Liquid Chromatography
- Particle Size Distribution
- Percent Actives
- pH
- Titration
- Percent Solids
- Percent Moisture

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