Special Design Considerations for Pilot Plants: Delivering Scalable Solutions

ZETON®

One of a Kind

The world of pilot plants is unique. A proprietary process with multiple interconnected "chemical loops" with inherent recycles could only be meaningfully tested using the pilot plant pictured below.

Pilot Plants Are Unique

I recall a talk that I attended during my post-graduate work during a break from my lab doing kinetic experiments related to a new commercial hydrogenation catalyst. The speaker boasted about how rich we chemical engineers were with meaningful mathematical models for the processes we worked with: "With computing power becoming cheaper every year and with the wealth of steady-state models at our disposal, I see a day when our simulators are so powerful that the pilot plant will become a thing of the past." Even just starting my career, I was skeptical of such a statement, knowing just how difficult it is to obtain meaningful kinetic data under industrially significant conditions and how pivotal that data is to designing a commercial plant. Twenty-five years later, having helped to develop technology for Zeton's clients from the bench through pilot and onto the commercial scale across the full breadth of the chemical process industry, I am just as sure that pilot plants are here to stay and will very much be a part of our future.

With that said, pilot plants are a significant investment (significantly larger than the investment required for a simulation or lab test rig), so the process technology developer or owner and the designer of a new pilot-plant facility are faced with making high-stakes technical choices. Good choices mean schedule savings in the design, manufacture, and commissioning of the pilot plant or in rapid completion of the experimental program. Poor choices or oversights in the pilot work may kill the entire program outright.

Starting Out on the Right Foot

Making good choices means finding a balance between cost savings and well-conceived planning. The first step to finding this balance is to think about what a pilot plant is. A pilot plant is a

processing system that operates at a scale intermediate between the laboratory and the commercial scale. In many instances, laboratory testing is done batchwise whereas pilot plants generally operate in the same mode as commercial operations as continuous processes. However, even though both are sometimes designed for continuous operation, viewing a pilot plant as a commercial plant in miniature can lead to some incorrect design assumptions.

Different Design Requirements

A full-scale commercial plant's goal is to generate tonnes per hour of product of suitable quality. The pilot plant has an entirely different product; it is designed to generate process knowledge and understanding in the form of both experimental data and operational observations and to do so in an economical and timely fashion. This is a key difference, one that makes the pilot scale a unique undertaking, and one that must be kept clearly in focus during all parts of the design process.

It is helpful to compare and contrast the pilot and commercial scales in terms of objectives and design factors arising from these objectives, as detailed in Table 1. A pilot plant will have

Table 1. Comparison of Commercial and Pilot-Scale Operations

Factor	Commercial Scale	Pilot Scale
Key Objectives	Continuous generation of on-spec product(s)	• Process knowledge and understanding • Operational observations • Scale-up data
Scale	Tonnes per hour	Kilograms per hour
Operation	Continuous, maximizing up-time	Continuous campaigns lasting up to several weeks
Design Life	Tens of years	Between one and ten years
Maintenance	During operation, as much as possible	Between campaigns
Operational Mode	Steady state	Chasing steady state
Data Acquisition and Control	As needed to maintain steady state	To obtain steady state and the necessary process data for scale-up
Operating Temperature and Pressure	Commercially optimal conditions	Beyond commercially optimal conditions (to establish optimum)
Design Points	Single	Multiple
Flowsheets	Single, fixed	Frequently multiple, variable
Source of Design Data	Pilot plant	Laboratory data and simulations, experience
Capital Project Timescale	Several years	One year
Need for Operational Flexibility	Modest	Considerable

different design data sources, objectives, scales, lifespan, operational conditions, and products than a commercial operation and should therefore have a separate, distinct design and project execution approach. By starting from the perspective that a pilot plant has a different set of objectives and a different set of operational conditions than a commercial plant will help the design process make the best choices right from the beginning.

Design Inputs for Pilot Plants

When developing an equipment design project, it is best to start with a fully developed process flow diagram with a complete mass and energy balance evaluated at the desired operating point. Unfortunately, one of the purposes of most pilot-plant projects is to provide that very data for the
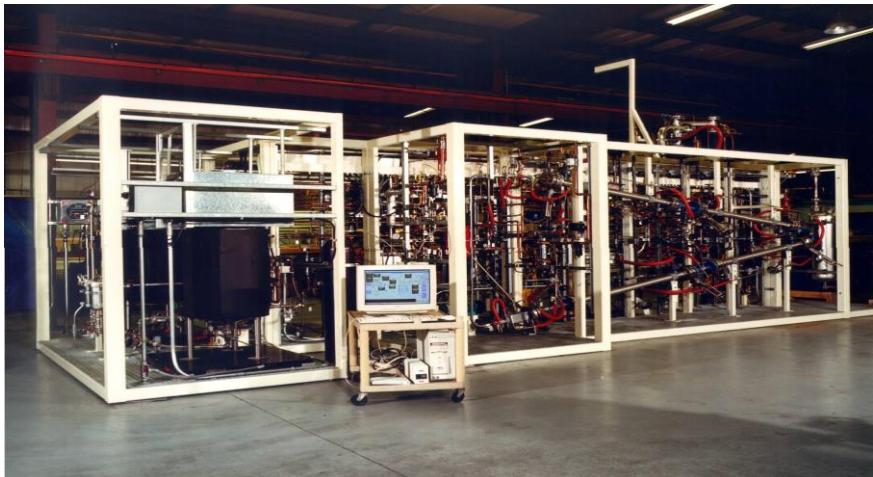

Figure 1. This pilot plant, designed and built by Zeton, produces customer evaluation samples of copolymers.

potential full-scale facility. If you wait for the flowsheet and the mass and energy balances to be nailed down completely before beginning the design exercise for the pilot plant, you will never get started.

Pilot plants are meant to be the safe places where uncertainty and risk are reduced and multiple potential operating cases can be tested. Indeed, operation outside the optimum conditions in pilot plants is necessary to establish where the optimum conditions truly lie for full-scale operations. There is no question

that process flowsheet simulations and designed lab experiments can help avert major pitfalls, but batch lab data and simulations cannot substitute for continuous pilot-plant data.

The difficulty every process development pilot-plant designer faces, then, is how to balance safety with the need for real-life data. This balance is best found in knowing when, where, and how to use the power of human creativity and the power of computer simulation.

Harnessing the Power of Human Creativity and Experience

At Zeton, we begin our design work with the knowledge and ingenuity we find in our engineers. We have a wealth of non-proprietary specialist knowledge about how to accomplish various unit operations efficiently at the pilot scale based on the successful completion of hundreds of pilot-plant projects. We recommend that clients harness this knowledge by using the practice of bench scale engineering in process development [1], which requires bringing together process chemists and technicians and process and design engineers, including Zeton as a preliminary engineering partner, as early as possible in the development program to create a hands-on multidisciplinary team. Together, these team members are able to yield more complete and accurate information for the design of a successful pilot plant than they would by working as separate teams or coming together later in the process.

Harnessing the Power of Simulations

One of the key products of the pilot-plant program should be a validated process

simulation. And this preliminary simulation, augmented with lab data, is the key starting point for the design of the pilot facility.

Lab data and simulations provide the necessary composition information, thermophysical properties, and duties to permit the pilot-plant designer to size vessels, heat exchangers, pumps, and instruments and to select materials of construction. While the reactions themselves are generally a "black box" in the simulator with composition information filled in based on actual laboratory reactor testing, the simulation output is needed to fill in the rest of the flowsheet. It is a richer and more accurate source of information than lab testing data for some process steps,

Getting Started

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particularly heat transfer steps which are confounded at the lab scale due to unfavourable surface-area-to-volume ratios at small scales.

We have found that rather than establishing a single design point based on a simulation, it is often necessary to use simulation data to parameterize the design and establish an operating range for each piece of equipment in the pilot plant. The process design of the pilot plant can then be evaluated across the full parametric range to ensure that the plant has the capacity and turndown needed to handle a wide range of operational cases. Using this data, you may find that you need to select a more flexible device or multiple pieces of equipment or instruments, or you may even need to design different process approaches to cover the range that really needs to be tested. However, sometimes the data may show you that it is simply necessary to re-evaluate the operating range you think you need to test—in terms of

available instrumentation or other process equipment—and set more realistic expectations.

Guidance for Pilot-Plant Simulations

We know that the process engineer tasked with simulating a pilot-plant flowsheet for a new process has a very tough job. For this reason, I did a quick poll of Zeton's design engineers to produce a few cautions for the process simulator. Here is a short list of problems we frequently see and how to avoid them:

1. Basic Pressure Profile

Make sure to implement a basic pressure profile. Fluids don't move from place to place without a driving force, and they don't flow through equipment such as packed beds and heat exchangers without losing some pressure. Applying an elementary pressure profile will help you identify missing pumps or compressors.

We suggest that in the absence of other information for processes operating at substantial pressure, it is best to allow at least 5 psi (1/3 bar) as a starting point pressure drop across each flow control valve, flowmeter, heat exchanger, packed bed, etc. Allowing the same rough pressure drop for frictional loss through each set of lines is also a prudent first guess. However, if you're operating under vacuum or near atmospheric pressure, throw out these rules of thumb and get an equipment design engineer involved to get more realistic estimates. The equipment designer will refine the pressure profile wherever it is tight based on better estimates of the real losses of the selected equipment.

2. Thermal Profile

With few exceptions, you don't need to trouble yourself over a "thermal profile." Tracing can be used to keep streams hot or cold to best simulate the commercial flowsheet (where heat loss or gain isn't nearly as troublesome as it can

be in a pilot plant). However, if there are any lines where heating or cooling or the lack thereof will cause damage or operational problems (e.g., condensation leading to corrosion, solidification, or crystallization leading to plugging), this is necessary information. Often these problems show up in the lab and need to be communicated to the designer. Never assume designers will just know.

3. Use a Heat Exchanger

Don't use stream mixers, valves, or other pieces of passive equipment to add or remove heat. One can "cheat" the simulator to do that, and our designers see this done as an aid to simulation convergence. However, the real process will need a heat exchanger, and the designer will need its duty and the thermophysical properties of each stream to design it. So, it is best to put the exchanger into the flowsheet.

4. Heat Duties

When a piece of process equipment legitimately adds or removes heat as part of its function (e.g., a reactor with heating or cooling or a distillation column with reboiler and condenser), call out the heat duties so the designer knows what needs to be provided.

5. Heat Release Curves

For exchangers used for vapourization or condensation of mixtures or where inerts are also present, heat release curves are needed.

6. Information Missing from Stream Summaries

When in doubt, generate the simulation's basic unit operation report for each heat exchanger and piece of separation equipment, and supply that to the designer as a starting point. This report often contains necessary information missing from the stream summaries.

7. Complete Data

Make sure you provide all the thermophysical data needed for sizing purposes on each stream.

- Minimum for each phase (vapours and liquids): Density, heat capacity, thermal conductivity, and viscosity.

- For liquids: Vapour pressure.

- For gases: Cp/Cv and compressibility.

- For gas/liquid contacting systems: Liquid surface tension.

- For slurries and liquid/liquid mixtures: Mixed stream data can be scarce and untrustworthy, but provide any properties information for the solid and each liquid that are known.

8. Thermodynamic Equation of State

Double check to make sure the right thermodynamic equation of state has been selected.

9. VLE Data

Use real VLE data to validate the simulation's output whenever possible.

A solid simulation with these cautions taken into account will minimize the amount of labour and the cost of preliminary design work for the pilot plant. If your firm doesn't have the simulation skills to do this work, we can help you make the necessary connections.

Choosing the Design Point for Pilot-Plant Equipment

For an established process, choosing the temperature and pressure ratings of equipment is a relatively straightforward exercise. However, choosing the maximum operating pressure and temperature combination for major pieces of equipment in a pilot plant is much more challenging.

Choosing the correct combination, or design point, is one of the key decision processes in any pilot-plant project, meriting significant analysis and thought. Choosing a design point that is too low can render a plant useless, but choosing a point that is inappropriately high can increase the cost and delivery schedule of the plant by a significant fraction without providing any benefit. An inappropriately high point may

Figure 2. This pilot plant had a reactor with an extremely challenging design point: 2500 psig at 1200 °C.

also hamper operation of the full plant by limiting the types of closures, equipment, valves, and instrumentation that can be used, resulting in increased downtime or a reduction in the ability of the plant to deliver the desired measurements, products, and data in a timely fashion.

Using a Breakpoint Analysis

A breakpoint analysis for the proposed materials of construction should be carried out as a preliminary design exercise in an effort to establish an appropriate design point. By identifying the natural limits for pressure and temperature for the required materials of construction, natural breakpoints for the design and pressure and temperature combinations for various pieces of equipment can be determined. It goes without saying that crossing a natural breakpoint can come with a significant cost, schedule, or operability penalty and should not be done lightly.

Figure 3 demonstrates some of the natural breakpoints applicable to process equipment

and piping systems based on materials of construction and utilities.

Note that the thermoplastic materials (PVC, PP, PVDF, and FRP) temperature limits given are based on the practical use of the material as pipe or tubing, which of course greatly depends on pressure. Some of these materials may be extended to higher temperature service as linings on metallic components for corrosion resistance. Other materials, such as PTFE, PFA, ETFE, the elastomers, and PEEK, are listed with the maximum continuous service temperatures at which they can be reliably deployed as sealing materials as gaskets, packings, valve seats or as
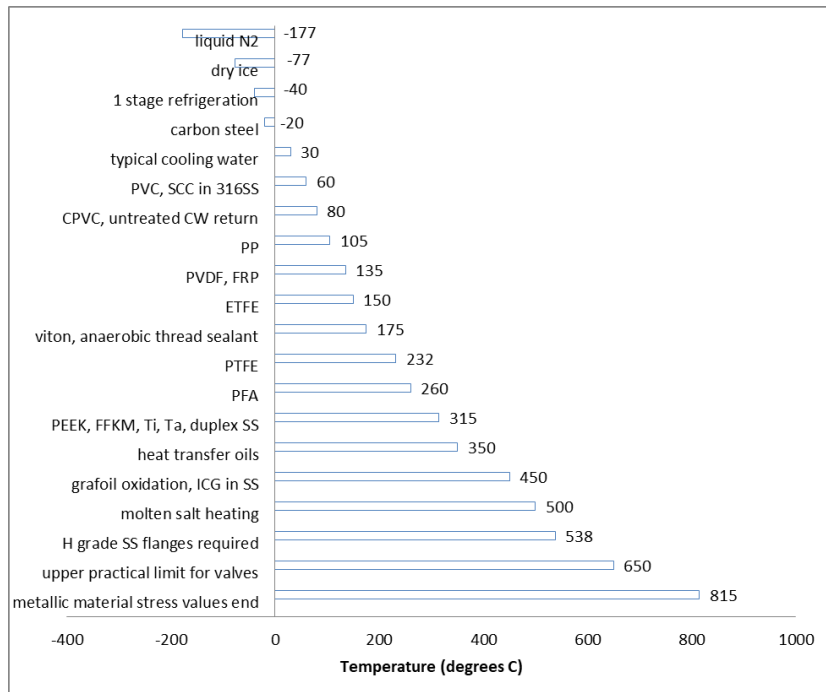

Figure 3. Practical limits of materials.

linings. Also in the figure, a number of "rules of thumb" for the practical upper or lower limit of various utilities (cooling water, hot oil, refrigerant, molten salts) are listed. These are intended for rough guidance only, but they do serve as a useful reference for scoping purposes during flowsheet development.

Maximum Operating Pressure and Temperature versus MAWP/T

Maximum operating pressure and temperature are distinct from maximum allowable working pressure (MAWP) and temperature (MAWT) of vessels, valves, piping/tubing systems, or equipment. The former is determined by process considerations, while the latter is calculated based on actual wall thicknesses, flange ratings, and gasket or seal performance. It is common for pilot equipment to have multiple pairs of MAWP and MAWT for various service conditions.

Depending on the type and specification of relief devices used for overpressure protection and the desired reliability of operation, the maximum feasible continuous operating pressure for a system will likely be between 70% and 85% of the MAWP of the pressure-limiting device(s) protected by the relief valve(s) or rupture disk(s). Setting a maximum operating pressure at less than $1/0.7 = 1.43$ times the maximum desired operating pressure is risky without a detailed knowledge of the overpressure protection strategy for the plant.

MAWT is set as a prudent margin above the maximum sustained mean metal/material temperature anticipated during normal pressurized operation. Brief excursions beyond this temperature are permitted in both ASME VIII and B31.3 under pressure [2], and prolonged exposure while unpressurized may or may not be possible depending on the design and construction of the components. The prudence margin between maximum operating temperature and MAWT depends on the service and the variability expected in that service. Unlike for pressure, passive temperature relief is not a code requirement, leaving the nature and robustness of overtemperature protection to the designer's engineering

judgment—from a code perspective at least. (Note that electric heaters have some electrical code requirements in this regard.)

Pushing the temperature limits of materials is routine in pilot-plant design. At temperatures above 1,000 °F in metallic materials of construction, the properties of materials used in design are typically time dependent, meaning that creep is the primary worry. Creep takes time to occur. In pilot equipment, where campaigns might last a couple weeks to a month, the risk of a creep-related failure may be fairly easily mitigated with proper inspection and maintenance. This is probably not true in the commercial plant, but once you get to that design you will have better data upon which to set appropriate limits.

Flange Ratings as Breakpoints

Flange ratings are often used as a natural breakpoint for pieces of process equipment and for piping, particularly at the scoping or basic design level. The ASME B16.5 flange table for 304 stainless steel (Figure 4) is listed as an example of how these ratings can be used [3].

Note that flanges made of other alloys will have ratings higher or lower than those listed depending on temperature. Often in carbon steel lines or on carbon steel vessels, instruments such as flowmeters and control valves with stainless steel flanges are used, and the MAWP
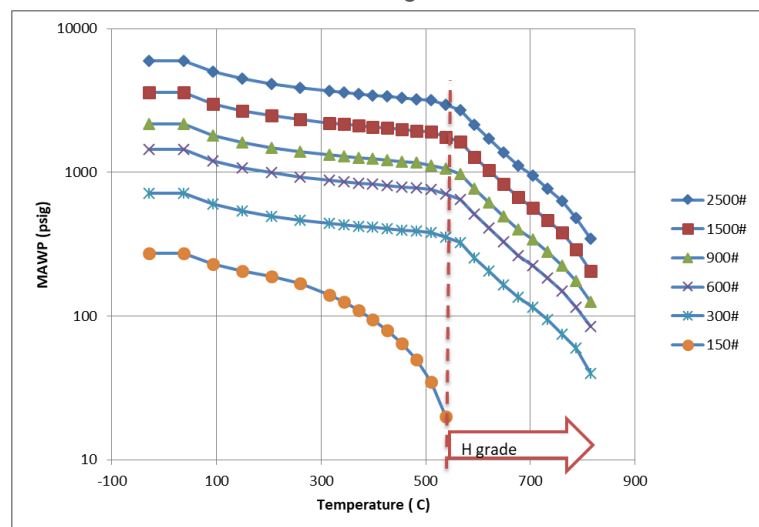

Figure 4. ASME B16.5 (2013) pressure ratings for 304SS flanges.

of these components frequently limit the MAWP of the resulting assembly.

When a vessel or pipe line is designed such that the flanges set the MAWP, the design is termed to be "flange limited." Flange limited design offers certain advantages to both the plant designer and the operator:

• The ASME B16.5 table [3] gives a natural MAWP/T reference for extending the design point to higher temperatures, or higher pressures at more limited temperature, should desired operating conditions change in future.

• If the flanges limit the MAWP, an overpressure event should lead to flange leakage before failure (though there is no guarantee that this will happen in practice).

Flange ratings also serve as a convenient measure of the ease with which other components (valves, instruments, etc.) may be procured and at what cost. While 150# and 300# components are common, each increase in flange class beyond 300# may be thought of as reducing the options for the selection of valves and instruments and some other components by roughly a factor of three with cost sometimes increasing by a similar factor.

Both the ASME VIII-1 pressure vessel code and the ASME B31.3 pressure piping code [2], permit any component to limit the MAWP of the resulting vessel or piping design. A design may be limited by a shell or head or nozzle neck, by flanges or flange bolting, or by the wall thickness of pipe or tubing. It may also, in practical terms, be limited by the MAWP of any

component connected to that piping system: any instrument, valve, pump, or component thereof may set the MAWP for any section of the plant and thus determine the maximum setting of required relief devices to protect against overpressure. In most cases, however, the optimal design for the overall pilot unit will not be fully flange limited, as some other practical limitation will come into play before the flange limit is reached.

Testing

It should not be forgotten that the MAWP not only sets the relief pressure but the MAWP (and MAWT) also set the pressure at which the code-required pressure integrity test (misdescribed in ASME B31.3 as a "leak test" [3]) must be carried out as part of the quality assurance on every piping and tubing system. This test may only be waived by the owner for Category D (nonhazardous) piping systems (air, water, etc.) in favour of an in-service test. The pressure integrity test, which is carried out at a multiple of the MAWP determined by code, may be carried out hydrostatically or pneumatically if hydrostatic testing is not deemed practical (frequently the case in pilot plants). Pressure integrity testing is required after fabrication and is usually required (and strongly recommended) after any substantial alteration of the piping. Some

Figure 5. This demonstration-scale hydromet plant designed by Hatch and built by Zeton had thirteen different wetted materials of construction in piping.

components, including all relief devices, must be removed prior to carrying out this test. By prudent selection of the components based on prior knowledge, it may be possible to leave many components in place for this test without damage.

True leakage testing is generally carried out after fabrication, shipment, and flush-out, with all components installed, and it is carried out at maximum operating pressure or a small multiple thereof. The requirements for pre-operation leakage testing vary greatly with the nature and hazards of the service.

Choosing Materials of Construction

Corrosion Testing at the Lab Scale

If you are in new process territory and corrosion service is a known risk, you cannot trust the charts or the experts. In order to give you reliable advice, a corrosion metallurgist needs to know more about the composition and other conditions of each stream than you likely know at the outset of a pilot-plant design project. From our hard-earned experience, we recommend accelerated corrosion testing done at the lab scale prior to making final materials selections for a pilot plant. If possible, select a material completely immune to the type of corrosion you may face in the pilot, so you can pursue the pilot program in safety and confidence. This is doubly true for mechanisms that may produce localized corrosion such as pitting, stress cracking, or the like. The additional investment required to build pilot equipment from an alloy with known resistance is modest relative to the risk of an uncontrolled corrosion-related failure.

With this said, sometimes the design life of the pilot equipment is short enough to make the corrosion risk of using a more standard material acceptable. Alternatively, periodic preventative replacement of pieces of equipment may be an alternative to a truly resistant alloy. But note that while this may be the best option for off-the-shelf

equipment, it can be a real problem when these items are custom built for purpose.

As a final note of caution, be aware that the experience in your laboratory batch reactor may not accurately simulate the conditions experienced in all parts of a continuous pilot plant over the long term. The first compartment in a CSTR train or the first few diameters downstream of a chemical injection point encounter conditions continuously which might be experienced only for seconds to minutes in a batch. Your history of hundreds of batch runs may therefore have given you only a few hours of meaningful experience in those sections of the pilot plant.

Scaling Metallic Materials for a Pilot Plant

When deciding on metallic materials, two important points must be kept top of mind. One, the available selection for a pilot plant is more limited than for a commercial system, and two, a pilot plant does not need to be made of the same material as the current commercial concept to give adequate risk-mitigation on scale-up. A special alloy that you may be thinking about for the commercial plant is probably not available in less than full mill runs of pipe or fittings, especially at the smaller sizes, and may not be available in tubing at all. This means that fittings or even pipe may have to be fabricated from bar stock. While there is always a justification for selecting "unobtainium" as a material of construction for a pilot plant, it is usually a mistake arising from inflexible thinking.

At the pilot scale, any metallic material you select will be significantly more expensive and less readily available than the common grades of stainless steel for valves, instruments and lines, and often for equipment as well. An example is the use of duplex or super-duplex grades of stainless steel or 6% molybdenum austenitic grades. These materials may be the right choice for even pilot equipment once it is beyond a certain size and can offer great economic advantages relative to high nickel alloys or
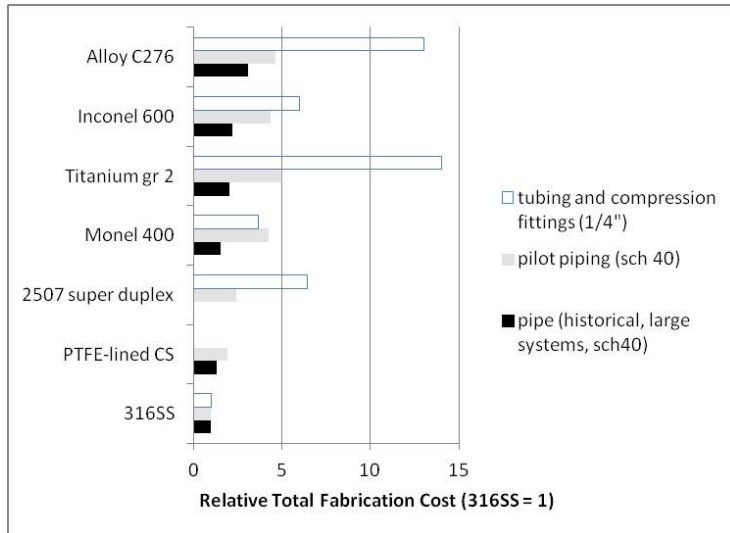
Figure 6. Comparison of total procurement and fabrication cost for various piping materials of construction [4].

best suits the corrosion service requirements for the durability period of the plant and the use of corrosion coupons and/or electrically insulated test spools for materials testing.

Methods of corrosion protection, such as acid brick lining, exotic alloy cladding, electrochemical protection, and lined pipe, are scale-sensitive methods, infeasible below a certain physical size for practical reasons. Thus, the use of solid resistant materials or alternative lining methods and materials must be pursued for pilot projects below a certain size out of physical necessity.

titanium at the commercial scale, but once you're down into tubing, the savings versus nickel alloys or titanium frequently evaporate.

In our experience, the relative cost factors found in commercial plant fabrication (and found in the cost comparison tables presented by vendors of alloys and pipe and fittings) dramatically underestimate the installed cost differences actually observed in pilot-plant fabrication. Figure 6 compares the historical, industry accepted, scoping-level commercial scale ratios of the cost of piping systems [4] versus those encountered in a pilot-plant design study from roughly 9 years ago.

Clearly, the comparative cost between various candidate materials of construction is scale-dependent, and also varies with time as alloy constituent prices fluctuate.

Corrosion Protection and Prevention in Pilot Plants

If knowledge of the service life of materials of construction is key to the success of the pilot program, we recommend constructing the plant from the commercially-standard alloy that

Pilot-plant lines are frequently built in tubing, which offers significant advantages in terms of fabrication cost and operational flexibility relative to welded piping. However, in corrosion service, it is important to realize just how little meaningful corrosion allowance is available in a tubing line before rupture. This is another reason to consider selecting a more resistant alloy than you might ordinarily consider.

Special Considerations for Titanium

Titanium and its alloys are extensively used in pressure hydrometallurgical pilot plants, one of many areas of Zeton's practice in the chemical process industry. Among its various other
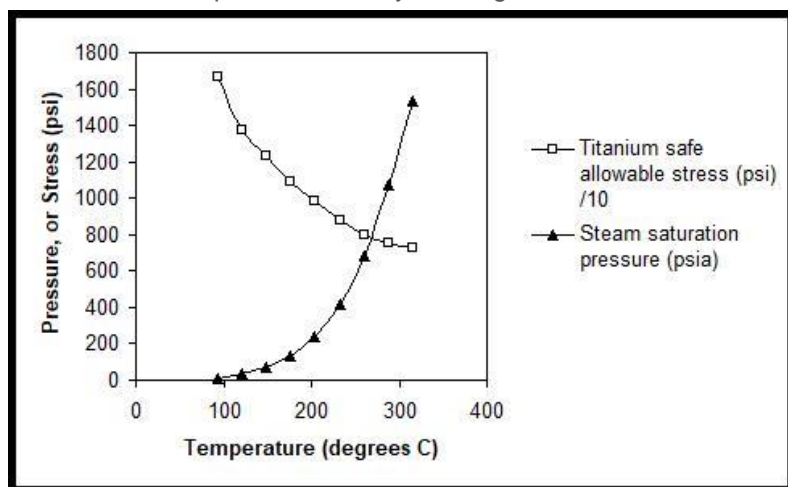

Figure 7. Titanium safe allowable stress [3] versus steam saturation pressure.

properties, titanium in direct pressure-bearing service has a safe allowable stress that is strongly de-rated with increasing temperature to the code limit of 315 °C.

Figure 7 shows the strong temperature dependence of safe allowable stress (S) for commercially pure titanium. The saturation pressure for steam (P) is plotted alongside for comparison purposes. These factors combine such that modest changes in operation temperature can have significant effects on required wall thickness (proportional to the ratio P/S). The effect is most pronounced for pilot plants, where the use of titanium as a lining may be physically infeasible due to the small size of the vessels in question.

Simplify Your Pilot Plant

Unnecessary complexity leads to unnecessary delay and increased costs, money that is better spent on additional training for operators or more campaigns. Indeed, the labour costs of installation and testing, procurement, and engineering specification and selection rise in nearly direct proportion to the number of tagged items on a plant's P&ID drawings, and these labour costs often dwarf the cost of the item itself. Elimination of unnecessary components can result in significant reductions in the cost and delivery lead time for a plant.

The first step toward simplicity is to consider the life cycle and operating mode of your pilot plant. Will it be essential or even permissible to continue operating while you repair or replace key pieces of instrumentation or equipment, or will a shutdown be inevitable? If the latter is true, block and bleed valves on instruments, double-block valves, and bypasses around control valves and the like may merely add unnecessary cost and complexity to the system without adding significant safety or other value.

I know that several readers are already shaking their heads after reading the previous paragraph. "Just try operating a plant without adequate block and bleed valves, and you'll install them on everything!" But you need to HAZOP that assumption for a minute before making up your mind. Take, as an example, the common configuration for a control valve that many customers seem to want as a matter of course: a block valve immediately upstream and downstream of the control valve itself, plus a needle valve (and possibly another block valve) in parallel to bypass the control valve.

Let's first look at the needle valve bypass. It can serve a very useful purpose while you're shutting down or doing clean-out operations by giving you a valve with a much larger Cv than that of the control valve to do the last bit of depressurization or to flush waste out of a line.
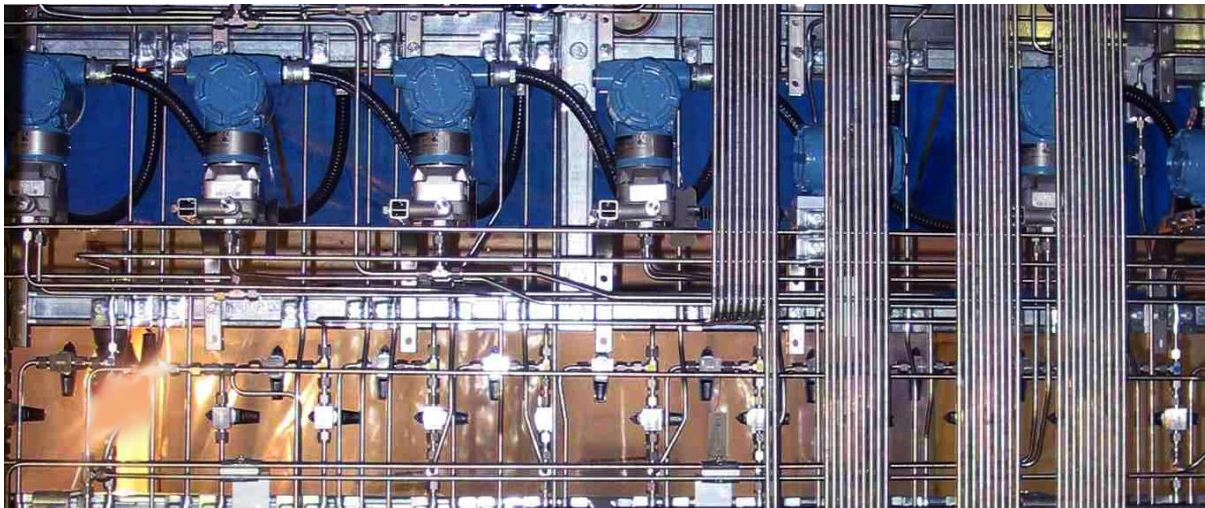

Figure 8. Sometimes complexity is necessary to function, as with this DP monitoring system for a fluid bed reactor.

But let's think about the situation when the control valve's trim has become clogged with debris or the valve is otherwise non-functional. Are you really going to continue to operate with an operator manually adjusting that needle valve? One hand on the valve handle, the other

Keep Moving Forward

Focus on what you need to pilot and do not pilot the rest. If it adds complexity without improving the plant's ability to give you the data or product you need, eliminate it.

on the radio to the control room? Would that be safe? The answer is almost always no, except in cases where you probably should have replaced the control valve itself with a manual globe or needle valve. Does the needle valve improve operability enough to warrant the safety risk of using or misusing it? That depends on how robust your procedures are and how well trained your operators are, but the decision also has implications for the pilot-plant designer's relief calculations. That needle valve is almost certainly going to cost you a lot more than you expect, on many levels.

Now let's look at the upstream and downstream block valves. There's almost always another block valve upstream and downstream, and on a pilot plant that valve is probably only a few feet away, even though it might be on the previous P&ID drawing and hence may be easily overlooked. And since we've likely already concluded that you're not going to continue operating with a defective control valve, do you really need valves to isolate both sides of the control valve as if you were going to pull that valve for maintenance during operation? And if the answer to that question is yes, is the isolation provided by a single valve safe enough?

In our opinion, setting arbitrary rules such as "we want blocks, bleeds, and bypasses around every control valve" is a sure way to make your pilot

plant more complex and expensive than it needs to be without adding proportionate value. We will do it if you insist and are willing to pay for it, but in our view, it is far better to give some thought to operational, maintenance, and isolation strategies or philosophies for the pilot plant and then let the designer assess the valve needs for each line based on the service. Those decisions should then be checked carefully during the HAZOP review.

Focus on what you need to pilot and do not pilot the rest. If it adds complexity without improving the plant's ability to give you the data or product you need, eliminate it.

Pilot the Process, Not the Equipment

There have been many occasions when an evaluation of a client's flowsheet has led me to suggest the complete elimination of whole steps or trains from a proposed pilot flowsheet to be replaced with a simpler but commercially uneconomic alternative with lower capital costs or, in some cases, even with the very cheapest option: analysis and simulation. Where this can be done without significant impairment of the process development goals of the pilot project, it is a very attractive option.

It is understandable that customers may have a strong desire for similarity between the pilot plant and their ultimate commercial plant, but sometimes this extends to similarity that is actually detrimental or impractical. And sometimes the similarity desired is an aesthetic one rather than one which has true technical significance. It is important to remember that you're piloting the process, not the equipment. (There are cases where testing the function or efficiency of a particular type of equipment is itself a key goal of the pilot program, but these are the exception rather than the rule.) A clever and experienced pilot-plant designer is not looking to create a miniature version of your commercial plant, but is out to find a cheap and

reliable means to accomplish each unit operation in your flowsheet with the turndown required for successful piloting.

Heat Integration

A primary example of how a good designer sees differences between pilot plants and commercial plants is in heat integration. Heat integration (i.e., the use of hot process streams to transfer heat to cold process streams via cross exchangers) is something commonly practiced in commercial designs where it offers significant energy savings. However, energy consumption in a pilot plant is typically a third-order priority at best, well behind

Figure 9. This pilot plant had 700 pieces of equipment with extensive use of creative scale-specific solutions.

the cost of labour to operate the unit, the opportunity cost of pilot program schedule delay, and the venture capital cost of the pilot plant itself. Typically, a cross exchanger also requires a trim utility exchanger on at least one and sometimes both streams to make up the difference between the performance of the cross exchanger (particularly once it becomes fouled) and the needs of process control. Then there's the problem of start-up, particularly the time to steady state. On start-up, process effluent will not be hot enough to transfer meaningful heat to the feed, necessitating either that the start-up heater be designed for the full duty (cross exchange plus trim duty) or that the start-up be done at a greatly reduced flow. This means two and sometimes three exchangers rather than one. Though there are always exceptions, the correct approach for a pilot plant is to simplify by specifying separate heaters and coolers for the feed and products with utilities supplying and absorbing the required heat.

Rotating Equipment

Rotating equipment also offers an opportunity for simplification. A centrifugal pump used as a pressure source can also be used to effect mixing and even gas/liquid contact by combining it with a venturi eductor. The result is to use some of the energy typically wasted in a pilot plant by operating a centrifugal pump well to the left of its best efficiency point for a truly useful purpose while also saving the capital and installation cost of a mixer or compressor and its motors, seals, and controls.

An Example of Simplicity

Here is an example of what you can do on a hydrometallurgical pilot plant, downstream of the pressure leaching equipment where there are large numbers of repeated equipment items such as multiple CSTRs in series:

• Where height permits, use gravity overflow instead of pumps.

• Standardize on a few tank sizes with variable overflow levels.

• Eliminate level controls in favour of air-tolerant pumps.

• Use a single variable speed motor to drive multiple agitator shafts, a single peristaltic pump driver to drive multiple pumpheads, or a single VFD to drive multiple motors.

These suggestions may seem simple, but they have been used to drive thousands of dollars of unnecessary costs out of pilot units for such systems. This can sometimes mean the difference between proceeding with a project and abandoning all the lab work due to insufficient budget to carry it forward at the pilot scale.

Scale Up or Scale Down

A good pilot-plant design must balance the need for physical and equipment similarity to the commercial process with the other goals of the pilot-plant program. In essence, this means careful and creative design is necessary to ensure that the equipment will continue to work reliably for the duration of a campaign while maintaining sufficient process similarity to give reliable pilot-scale experimental data and observations.

The first time I really thought about scale, was when my high-school physics teacher, Richard Haiser, gave a lesson slightly outside the curriculum. He started his lesson by raising his deliciously Hungarian-spiced baritone, and asking us to **"Consider the Lilliputians!"** That class turned out to be strangely prophetic of my future career, as scale is the stuff and trade of the pilot-plant business.

Reaching back to the eighteenth-century novelist Jonathan Swift's classic satire of *Gulliver's Travels*, he compared factors associated with the scale of the tiny Lilliputians and giant Brobdingnags to show us how the physics of scale affect the living things around us.

First he had us consider the average Lilliputian, which Swift describes as a person 1/12th the scale of a regular human.

The Lilliputian's volume (and thus mass) scales with the cube of the scale dimension, whereas its surface area scales with the square of the scale dimension. The Lilliputian's ratio of mass (i.e., the number of cells available for heat

Figure 10. Photograph of a mural of Gulliver with the Lilliputians on a toy shop in Bremen, Germany. Painter unknown. Photo by Javier Carro.

generation) to surface area (i.e., the area through which he would lose heat to the surroundings) was 1/12th that of a regular human. This meant that food intake would need to be twelve times as high in relative terms. This is the same reason that mice and small birds have to eat a large fraction of their body mass in food each day to stay alive.

He then had us consider the Brobdingnags, who are twelve times as large in all dimensions as a regular human.

Here the problem is that the volume (and thus mass) was 12^3 as high, but the strength of the bones varied in their cross-sectional area, which would only increase by 12^2. A Brobdingnag, then, would be in serious trouble unless his physical form changed as he was scaled up.
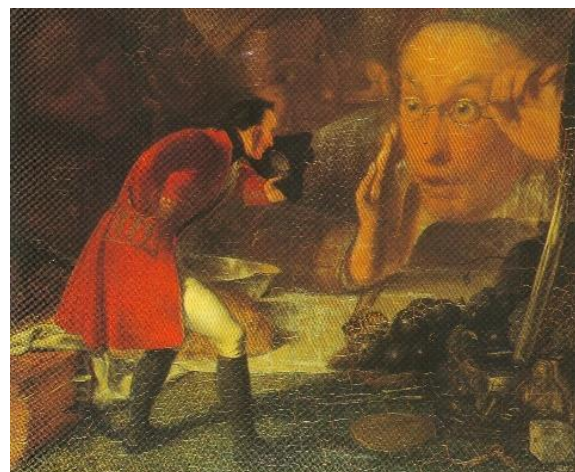

Figure 11. Illustration of *Gulliver's Travels* by Richard Redgrave, Permanent Collection, Victoria and Albert Museum, London.

This is why there are limits on the size and shape of land animals, and goes some distance toward explaining why we find the largest animals in the sea.

The pilot-plant designer's key problem is exactly what Mr. Haiser was trying to get across to us with his lesson: some factors change with scale and others don't. In fact, even within a process there may be intermixing of factors that scale with those that will not. For example, residence time and solid feed particle size are generally kept constant with scale, but the volume and linear dimensions of reactor vessels, the area of settlers and heat exchangers, and the sizes of piping/tubing and throttling valve orifices vary with scale and to different powers of the scale factor. When the variables and the constants collide, severe processing problems, such as plugging, inadequate gas contact time, or excessive heat loss, can result.

Sometimes a particular scale cannot be achieved for a pilot plant at all. There will be cases where at a certain minimum scale, geometric scale-down of the commercial unit operation or equipment becomes fundamentally infeasible and alternative approaches must be used for the pilot unit. Often these approaches involve switching some unit operations (e.g., solids feeding and slurry letdown) from continuous to semi-batch, but sometimes it's more complicated than that. At Zeton, we encounter scaling obstacles on nearly every project to some degree, and after 750 projects, we have created a whole library of specialist knowledge related to how such problems may be successfully solved.

As chemical engineers, the most powerful tool in our scale-up library is dimensional analysis and its result—the dimensionless number. We have

a host of dimensionless numbers that we work with every day (e.g., Reynolds, Prandtl, Nusselt), and there are many more if we need them (e.g., Peclet, Bodenstein, Froude, various Damkohler numbers). The rule here for scale-up is pretty simple: pick a number and keep it constant with increasing scale. But which one? Not so easy!

As an illustrative problem, take the plug flow

Figure 12. An example of a pilot-scale PFR for a confidential client and process.

reactor (PFR). To get something approximating plug flow, one needs fully developed turbulence with a Reynold's number of at least 10,000. But there is a minimum scale below which a PFR becomes impractical due to pressure drop. Even if you were to manage, however, you'd do well to dig out your copy of Octave Levenspiel's *The Chemical Reactor Omnibook* and do the calculations related to axial dispersion. Plug flow is an idealization, and axial dispersion may give you a residence time distribution very different than what that idealization might suggest.

Conversely, in a large pipe, a Re of 10,000 may be achieved at surprisingly low velocities, rendering a PFR practical at scales where it might be dismissed out of hand. Or you could add a little viscosity and conclude the PFR concept goes straight out the window. But we at Zeton have done many PFRs for polymer applications and other applications for viscous

liquid processing. How are they done? Static mixers. Static mixers don't fully approximate plug flow, but they do a decent job, and most importantly they constantly move material from the heated or cooled shell to the interior.

Mechanical mixing also changes with scale, getting worse as scale increases. This means that the usually spectacular mixing achievable in a pilot unit must be impaired so that it approximates what you will get in your commercial unit. There's more to it than merely varying the mixer RPM during your experimental work, so mixing consultants and specialists who work for some of the mixing manufacturers can help with this.

Heat loss and heat gain are the most obvious problem for pilot-scale equipment. Because the heat loss situation for pilot units is often extreme, electric heat trace, cold tracing, or even vacuum insulation may be required. In some cases, the stream may just be allowed to heat or cool to ambient and then be "tempered" before entering the next unit operation. In other cases, the effectiveness of heat tracing may determine almost entirely how long your campaign will last before you get a blockage and are shut down.

The process of selecting the scale multiplication factor between pilot and demonstration or between either of these steps and the

commercial plant is a complex topic–too complex to be covered here–but it is enough to be reminded of how deep and intertwined this complexity is in the design, and how important it is to move slowly and thoughtfully through each step.

The Design–Build Approach and Modular Construction

The development timescale for the design, fabrication, and commissioning of a new pilot plant is characteristically much shorter than for the commercial facility, and the commercial pressure to meet these tight schedules can be enormous. However, the technical and financial risk of carrying out an inadequate pilot program or skipping the pilot step entirely is also quite clear.

Coming in to a project, then, pilot-plant designers and project managers must face down accelerated project schedules with thorough and efficient execution strategies. One of the main issues comes from the uniqueness of the pilot-plant scale, which has individual pieces of equipment that are smaller than those of the commercial plant by orders of magnitude, but no reduction in complexity. The number of pieces of equipment that must be designed, procured, installed, plumbed, wired, and tested is not reduced, and on top of everything else, custom-engineered equipment is often needed.

At Zeton, we have found that an integrated design–build approach offers significant advantages over the conventional strategy of detailed design followed by fabrication by a contractor. Figure 14

Figure 13. This 60 million litres per year continuous biodiesel production plant was executed by Zeton using our design–build modular approach.

compares a typical high-level conventional engineering, procurement, and construction management (EPCM) project schedule and a fast-track design–build project schedule. Both examples assume preliminary design is complete.

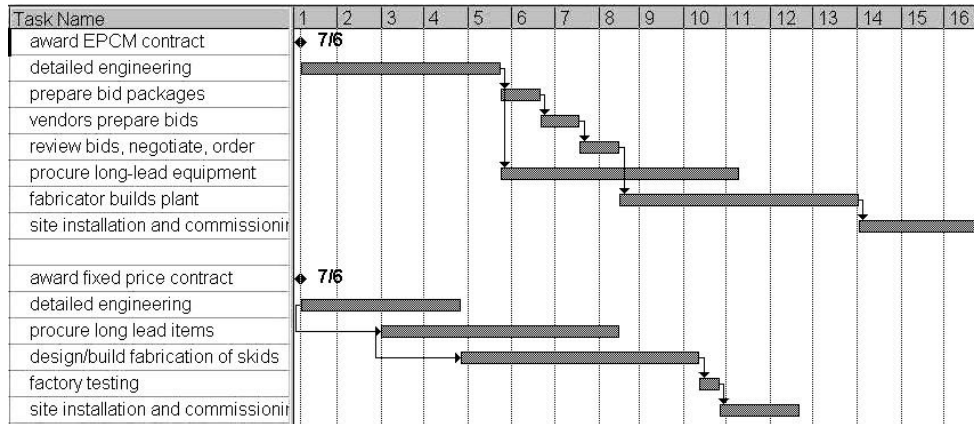Using the design–build model means that the engineers who carry out the design directly

- Schedule savings are realized through simultaneous execution. For instance, the plant fabrication can occur at the same time as the construction or modification of the facility to house the plant without interruption or delay.

- Factory testing reduces commissioning time considerably, as problems can be diagnosed and rectified more quickly in the factory than on the plant site.

Skid-based modular factory construction is familiar to the mining industry, which frequently uses this method for equipment that must be shipped to remote mine sites. Modular construction is particularly well suited to pilot plants due to the scale and size of the individual pieces of equipment. Frequently, entire pilot facilities can be fit onto a single module. Modules can also be sized to permit physical reconfiguration of a flexible pilot-plant space, permitting the testing of multiple flowsheets without the need to remove and re-install individual pieces of equipment.

Figure 14. Comparison of EPCM/fabrication (top) and design–build (bottom) project duration in months.

supervise the fabrication, and this means large numbers of detailed drawings and specifications are rendered unnecessary. The approach is also flexible to change, permitting the design of long-lead items to be frozen earlier in the schedule with less fear of schedule impact. Perhaps most important of all, direct supervision provides feedback to the design engineers, who refine their skills and improve their design decisions on every project they execute.

Skid-Mounted Modular Construction

Our approach to the design of pilot plants is predicated on the use of individual horizontally or vertically oriented modular steel frameworks, or skids, that are sized to both fit the facility and to maximize the efficient use of common, rapid modes of shipment to destination.

Skid-based modular factory construction reduces cost and schedule in several key ways:

- It permits fabrication in an efficient factory environment rather than on a construction jobsite, and productivity and accuracy of fabrication are thus increased.

For larger semi-works and demonstration-scale plants, the selection of module sizes is an optimization exercise between keeping the number of skids as small as possible to minimize reassembly labour on site and the cost and complexity of shipment. This modularization exercise must take place before the sizes of all equipment are frozen and possibly even before a throughput capacity is determined.

Seemingly minor differences in the size of individual pieces of equipment can make successful modularization possible without affecting the function of the plant in a detrimental way. As an example, a recent Zeton basic

design study for a multi-skid demonstration project allowed us to reduce the cost of shipment from over 7.5% for the client's original module concept to under 2.5% of module capital cost. We did so by optimizing the layout around module sizes so that they could be transported using readily available road transport equipment under normal road permits. To do this, we altered the dimensions of certain pieces of equipment to fit the necessary module dimensions without affecting their process function. More important than these cost savings, a schedule savings of over two months (from 14 to 12 months from start to delivery on site) was realized solely as a result of an optimized layout.

Whenever clients permit it, Zeton designs and builds modules which are fully integrated, with electrical and control hardware on every skid containing more than a handful of I/O. Modern networked controls combined with switchgear cabinets on every skid make it possible to carry out a full I/O checkout prior to shipment, reducing reconnection labour on site considerably. The client ideally has to add only one or two supply voltages to each skid and connect two Ethernet cables between each skid and the central controller (on another skid) and

between the central controller and the control room. Another benefit of electrical modularization is a considerable reduction in the length and cost of cables needed to connect field devices to their controllers and switchgear.

Strategies to Enhance Design–Build Quality

Caution should be used in the application of corporate engineering standards for components, materials, and methods of construction to pilot-plant projects. The application of inappropriate standards or the misapplication of standards can result in unnecessary cost and schedule delays without improving the quality of the pilot plant in a meaningful way. In fact, the inappropriate use of commercial plant specifications may limit the options available to the plant designer to the point where key pilot-plant project goals may themselves be compromised [5].

Pilot and demonstration-scale plants typically have a design life considerably shorter than that of a commercial facility. There is also typically a risk component to the project, meaning that some or even many items may need to be removed and replaced after initial start-up to accommodate the needs of the pilot testing

Figure 15. This project for a major oil/chemicals company used objective-oriented project specs plus codes and standards to control quality.

program. A shorter design life and the enhanced need for flexibility toward future change should significantly affect the decisions about what methods of quality control and inspection are used on the project. They should also affect the methods and materials selected for the pilot plant itself.

I've given a few examples of decisions which could be entirely out of place for a commercial plant but which make a lot of sense for pilot plants.

Avoid Painting Carbon Steel Pipe

If exterior corrosion is an issue, consider using non-metallics, galvanized steel, or 304 stainless steel with 150# fittings. Done properly with a blast and then multiple coats of properly selected industrial coatings, painting pipe is both expensive and extremely disruptive to the production of small plants. The labour and schedule savings from eliminating it will almost certainly pay for the additional cost of stainless steel pipe and fittings for line sizes below 3" NPS. Where sch10S pipe and fittings may be used, stainless steel may be a cost-effective substitution for painted carbon steel pipe even up to 6" NPS. Of course this is not a viable solution where exterior chloride exposure to hot lines is an issue, such as in some facilities located outdoors immediately near a seacoast. Under those installation conditions, the risk of chloride stress corrosion cracking of hot lines exists and may dominate the decision.

Use Tubing for Process Lines

When you do appropriate line sizing on a pilot plant, you will often find that the required line size is smaller than ½" NPS pipe. While small pipe might seem like an acceptable alternative,

when lines on a pilot plant are made larger than necessary, the dead volume in piping can become significant, affecting both the quality of results and the speed with which meaningful results may be obtained.

At Zeton, we use stainless steel tubing and compression fittings up to ¾" OD, which has an ID approximately equal to ½" pipe. Tubing and compression fittings have numerous advantages over pipe for pilot projects:

• Bends replace most elbows, reducing the number of joints and leakage points, or welds, and associated NDE if welding is required.

• Every joint is a union, permitting easy disassembly and reconfiguration.

• Leakage integrity is higher than threaded joints, particularly when temperatures are above 175 °C.

• Lines can be efficiently field run, rending isometrics unnecessary.

• Speed and labour productivity of fabrication is dramatically higher than that of pipe.

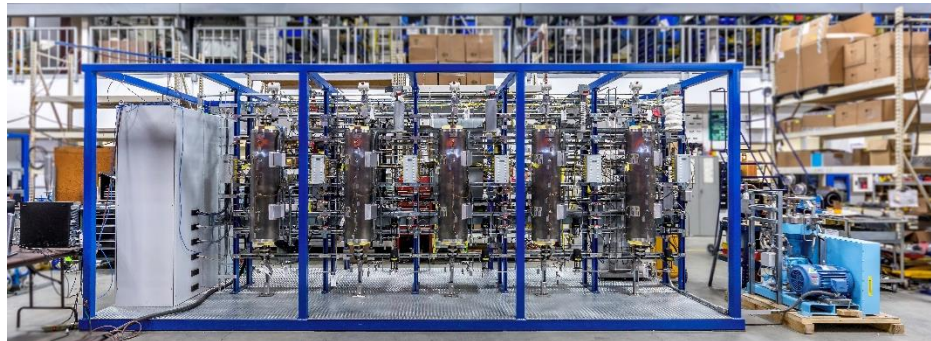Note that many of the benefits of tubing can

Figure 16. Pilot plants such as the one pictured use tubing for most if not all process lines.

evaporate above ¾" OD, and for line sizes even smaller than ¾" OD for exotic materials. We frequently switch to piping valves at even the ¾" OD size, using male connectors to adapt these valves for use in tubing lines. In many cases, piping valves at ½" NPS size are superior in terms of both performance and price to those available with integral compression fitting ends. Compression fittings—particularly tees and

crosses—and compression fitting valves in exotic materials increase dramatically in cost as the size of the fittings increase.

We've also found more than sufficient evidence that the product of most of the major two-ferrule compression fitting manufacturers is not only more or less equivalent in terms of meaningful metrics of quality, but that these components are also interchangeable and intermixable without meaningful degradation in the quality of the assembled joint. This opinion is based on our review of significant third-party testing. The one exception is the Gyrolok fitting made by Hoke, which has a different ferrule geometry rendering it incompatible with the industry standard 2-ferrule design offered by such companies as Swagelok, Parker, and Ham-Let.

We buy a lot of stainless steel tubing and have done so for decades. In our experience, we have found that welded seam stainless steel tubing can be obtained with suitable quality for use with compression fittings at a small fraction of the cost of seamless tubing. We do stock both, but when given a choice between the two, I will select welded seam tubing almost all of the time. When selecting seamless tubing, there are very few mills we will buy from. We have encountered problems due to poor cleaning and annealing practices at the mill, which become obvious only after cutting full random lengths in half and inspecting the bore. We recommend this inspection be done on a representative basis with every batch of seamless tubing.

Don't Fear Threaded Pipe

For line sizes 2" NPS and below, and service temperatures 175 °C (350 °F) or below, we make extensive use of NPT threaded pipe and fittings. If the right pipe thread sealant system is selected, threaded pipe offers significant benefits in terms of labour productivity and ease of re-work for future modification (the latter point being absolutely key for pilot operations). We use threaded pipe and threaded joints in this range for both nonhazardous and moderate hazard

duty services, including flammable and moderately toxic services, and have done so with good success for twenty years. Above 175 °C, we tend to limit our use of threads because the thread sealant system options diminish. Note that the most effective thread sealant system uses both a bulk gap-filling and high pressure lubricant material (i.e., Teflon tape with a specific gravity of at least 1.3) and an anaerobic pipethread sealant, such as the many excellent sealants offered by Henkel under the Loctite brand. We have found that using either sealant

Figure 17. Zeton's pre-engineered vertical and horizontal modules stack together quickly and efficiently.

on its own results in inferior performance when compared to the combination. In particular, the anaerobic pipethread sealant provides thermal cycling resistance, which is extremely important to the long-term leak tightness of the resulting NPT joints.

Above 2" NPS, for nonhazardous services such as air, water, low pressure nitrogen, drain, and vent, we use galvanized pipe and roll-grooved

fittings, such as those offered by Victaulic and Shur-Joint. This provides external corrosion resistance without the need for welding, blasting, and multi-coat paint systems.

Heat Tracing and Insulation

On most pilot plants, line sizes are small and heat loss can be critical to successful operation. The main challenge with heat tracing and insulation isn't installing it in a neat and professional manner in the factory. Rather, the issue is with ensuring that whatever system is used can be easily and completely removed, modified, and replaced correctly by the operations and maintenance crew. We have, over the years, come across a number of insulating methods and materials that make

The Zeton Advantage

Since 1986, Zeton has been a premier supplier of modular pilot plants, introducing new ideas and methodologies to pilot-scale applications.

removal and replacement easier and more likely to be complete. However, on a project where it is important to keep small lines hot, the best piece of insulating advice we can give is to install the pilot plant in a building so that the key problem—the ingress of rain and snow—can be controlled without requiring extensive and laborious re-work every time a hot line needs to be removed to clear a blockage or altered to provide a new feature.

Electrical Area Classification

At Zeton, we have frequently seen a pattern of misapplication of electrical hazardous area classification with respect to lab-scale and pilot-scale projects over the years. On many pilot-scale projects, the money spent on matters related to electrical hazardous area classification generates extremely limited benefits in terms of meaningful safety improvements for operators. Alternative methods may offer superior, effective

protection for a much lower cost. In some cases, a misunderstanding of the requirements and scope of protection offered by electrical area classification has resulted in an increase in real operational hazards. Zeton draws heavily on its well-earned skills and experience to conduct a thorough review and evaluation of NFPA 496, NFPA497, NFPA 70, API500, etc. and their application to pilot plants any time a pilot plant processing flammable materials is being considered.

Corporate Specifications

Zeton's mission is to design and builds custom projects and is flexible to using the methods, materials, suppliers, and processes that our clients value most.

That said, in our experience, even the largest oil and chemicals companies do not have appropriate and fully developed specifications optimized for pilot-plant projects. What many large companies have is corporate standards intended primarily for equipment and piping with a very long design life. These specifications can sometimes contain extremely valuable advice based on hard lessons from the company's past. However, they can also be excessively complex and self-referential, sitting in a web which ultimately requires access to the entire set of corporate specifications and design guidance documents to be truly useful. A proper review of the entirety of a set of corporate standards may take many man-months of labour that must be paid for, and in our experience, does not satisfy a good return on investment, as corporate specifications of this sort are almost never appropriate to the size, scale, and design life of a pilot project, and frequently include requirements that drive up the cost and schedule while not bearing fruit during the design life. Corporate specifications, then, should be used in an extremely judicious manner.

Using the Pilot Plant Designer's Expertise to Best Effect

If you've selected a specialist company such as Zeton to design and build your pilot plant, you've made a good choice. You've selected the benefit of working with a company with 750 projects of a similar scale in their experience list and a host of repeat customers. That scale-specific experience is invaluable to a successful pilot-plant project. But to take maximum advantage of that benefit, you have to be careful about how much you tie the hands of the people who design and build such systems for a living. Be careful what you put into hard specifications on a project. Instead of focusing on telling designers how you want a project done in detail, it is better to spend that effort to transfer the learnings from the lab work to the designers, to set appropriate objectives for the resulting pilot plant, and to review the results of basic engineering and the scope of work for the detailed design and fabrication of the project. By focusing on objectives rather than itemizing and specifying the details, the pilot-plant designer can make best use of their experience to help you make scale-appropriate selections of materials and methods to best match your objectives.

In Conclusion

A pilot-plant project has a different design data source and different objectives, scale, lifespan, operational conditions, and product than a commercial plant project for the same technology and therefore should follow a separate, distinct design and project execution approach.

Design for operational flexibility and rangeability is key to pilot-plant success. Multiple operating points and parametric design established through the use of lab experiments and simulations are the rule rather than the exception.

Natural break points for temperature and pressure arising from materials selection should be taken into consideration for design, and these breakpoints should not be crossed without due consideration. Particular care must be given toward the selection of operating temperatures for titanium equipment, as minor changes in temperature can have significant impact on required titanium wall thickness.

Notably, some design factors change with scale while others do not. Accordingly, some unit operations of the commercial plant, as well as some methods of fabrication and materials of construction, may be limited to minimum physical size or throughput scale and require alternative approaches to be successful at the pilot scale. In particular, the small size, significant complexity, and tight schedule of pressure hydrometallurgical pilot-plant projects make them ideally suited to a modular, skid-based design–build approach. Zeton's customers have benefited tremendously from this approach, finding it to be a proven way to decrease costs and increase efficiency over the course of a project.

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Contact Zeton to find out how our proven design–build approach to modular pilot plants can transform your next project.

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