CHEMICAL ENGINEERING

April 2011

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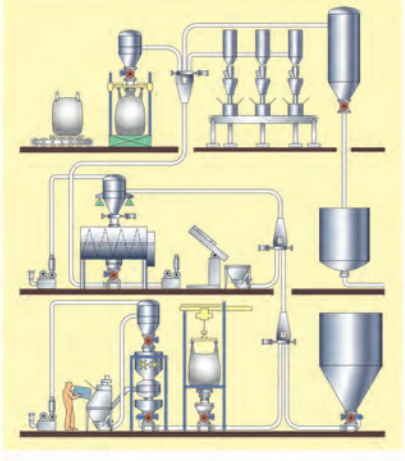

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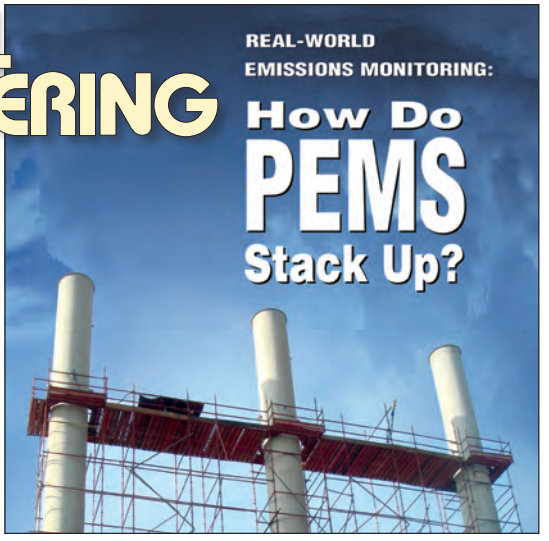

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Ten troubleshooting tips

Good troubleshooting skills are invaluable in chemical engineering practice. While their value is clearly gleaned from the impact they have on plant performance, it is also a reflection of the intangible process of learning them. Last month, at the AIChE Spring meeting in Chicago, Ruth Sands, former *CE* author and mass transfer consultant at DuPont Engineering Research & Technology, presented attendees of the Distillation Topical Conference with troubleshooting tips from those who do it best*. They just so happen to amount to ten, and have relevance across the profession.

1. Safety first: Assess the SHE (safety, health and environment) issues and implement a temporary solution to give time for troubleshooting. Rushing can lead to safety hazards.

2. Good troubleshooters understand the basics well. Use calculations, models, experiments and so on to check your theories. Know what is important and what is not.

3. Know what to expect before you start. What temperature, pressure profile should you expect? What quality of separation should you expect? Have a feel for what is reasonable based on a variety of experiences. And, last, but not least, "Don't ever be afraid to do a simple material and energy balance. It will tell you a million things."

4. Do not overlook the obvious. However, correcting obvious problems does not necessarily solve the whole issue. Be patient.

5. The so-called mental model both helps and hurts the troubleshooting process. "Good troubleshooters have a willingness to accept the data rather than their own theories." But, "Don't let the data get in the way of a good theory."

6. Think of ways to challenge the mental model. Is the process at steady state? Visualize what is happening. Imagine yourself as a pocket of liquid or vapor looking for the easiest path. Think of everyday analogies. Processes are the same that occur in the kitchen, bathroom or yard. And as Izak Nievwoudt, R&D director, Koch-Glitsch, commented, "A lot of troubleshooters look for the smoking gun, but it can be a cluster problem."

7. Testing strategy advice varies considerably, and depends on your situation. Testing should begin with the easiest to prove or disprove and not be based on how likely the theory is. Do all practical tests before making a permanent change.

8. Believe your instruments, unless you have a good reason not to. Don't start by questioning your instruments. What scenario could cause these data to be true? Instruments report information as they see it.

9. Use people as sources of information. Operators can be powerful resources. They know how to make their own equipment work. What do you see? What do you do in response? What do you do to fix it? Listen to all sources. See value from different viewpoints (level, experience, function personalities). Seek out unsolicited opinions and contrary views. Listen more than talk. Use a learning attitude. Implying that someone screwed up is a sure way to get no cooperation.

10. Learn through examples. Incident investigations give ideas for modes of failure. Good investigations cover people, equipment and the system. Participate in startups, especially in new plants. It compresses the learning. Startups from shutdowns are also opportunities. A person that learns from others' failures is wise. ■

Rebekkah Marshall

* Sands pulled suggestions from three books (see references in the online version) and interviewed the following troubleshooting experts: Jose Bravo, chief scientist, Shell Global Solutions; Henry Kister, director of Fractionation Technology, Fluor Corp.; and several of her DuPont colleagues: Ed Longwell, principal engineer, Jeff Loomis, senior engineer, Nick Sands, technology fellow, Teresa Thomas, senior engineer, Jean Trotter, consultant and Bob Trotter, engineering fellow (retired).

Letters

High-purity MnO_2 production

Regarding the February Chementator article, A new process for high-purity MnO2, (p. 15), I wanted to point out that the process proposed by Mesa Minerals is, for practical purposes, identical to one developed by my company, Kappes, Cassiday & Associates (KCA), in the 1990s to recover MnO2 from manganese bearing ores. The work was completed with the goal of applying the process to a mineral property known as Berenguela in southern Peru, which was owned by KCA at the time. The flowsheet proved successful in the lab for that material. After the process was developed, the property was put to market where the details of the flowsheet were scrutinized by colleagues in the industry and the process was found to be valid. The flowsheet was available on a public Website for some years and it now resides in a page of our Website (www.kcareno.com). The property was eventually sold to Silver Standard of Vancouver, B.C. who owns it today.

Our flowsheet utilizes SO_2 as a reductant to put low-grade MnO_2 into solution and recovers a more pure form of MnO_2 via electrowinning, just as in Mesa's proposed process. There are additional stages designed to recover other metals present in the particular feed material. So, the idea of adding SO_2 gas to an acidified slurry to leach low grade MnO_2 for recovery via electrowinning is not novel. The removal of alkali metals and iron via the Jarosite and Goethite methods in the context of this process is not novel either, as it was studied and proven out in the lab by KCA also.

Thank you for your attention to this matter. I am a fairly new reader of *Chemical Engineering* and I find it very informative.

Jake Ward, engineer
Kappes, Cassiday & Associates (KCA), Reno, Nev.

The Mesa process does not claim to be the first to achieve leaching of manganese dioxide by SO_2. The key achievement of the process is in managing byproduct formation. — Ed.

Postscripts, corrections

March, Bookshelf, p. 8: It should be clarified that the book reviewer, Daniel H. Miller, Esq., is a licensed attorney in the State of Maryland, not Virginia. Although Mr. Miller resides in Virginia, he does not represent himself as a licensed attorney there.

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Calendar

NORTH AMERICA

2011 Materials Research Society Spring Meeting. Materials Research Soc. (Warrendale, Pa.). Phone: 724-779-2754; Web: mrs.org
San Francisco, Calif. **April 25–29**

The 5th Annual Energy Efficiency Finance Forum. American Council for an Energy-Efficient Economy (Washington, D.C.). Phone: 704-341-2376; Web: aceee.org/conferences/2011/eeff
Philadelphia, Pa. **May 3–4**

Powder & Bulk Solids International. Canon Communications LLC (Los Angeles). Phone: 310-445-4200; Web: canontradeshows.com
Rosemont, Ill. **May 8–10**

The 2011 ACEEE Hot Water Forum. American Council for an Energy-Efficient Economy (Washington, D.C.). Phone: 202-507-4000; Web: aceee.org/conferences/2011/hwf/registration
Berkeley, Calif. **May 10–12**

Coating and Drying Technology Seminar. Edgar B. Gutoff, Consulting Chemical Engineer. Phone: 617-734-7081; Email: ebgutof@coe.neu.edu
Waltham, Mass. **May 10–12**

Electric Power Conference. TradeFair Group (Houston). Phone: 832-242-1969; Web: electricpowerexpo.com
Chicago **May 10–12**

International Thermal Treatment Technology (IT3) Conference. Air & Waste Management (Pittsburgh, Pa.). Phone: 412-904-6010; Web: awma.org
Jacksonville, Fla. **May 10–13**

Valves & Actuators 101. Valve Manufacturers Assn. (Fredericksburg, Va.). Phone: 540-785-8901; Web: vma.org.
Calgary, Alta. **June 6–7**

NFPA 2011 Conference. National Fire Protection Assn. (Quincy, Mass.). Phone: 888-397-6209; Web: nfpa.org
Boston **June 12–14**

Valve World Expo Americas 2011. Messe Düsseldorf North America (Chicago, Ill.). Phone: 312-781-5180; Web: mdna.com
Houston **June 21–22**

Powder & Bulk Solids Canada. Canon Communications LLC (Los Angeles). Phone: 310-445-4200; Web: canontradeshows.com
Toronto **June 21–23**

AWMA Annual Conference & Expo. Air & Waste Management Assn. (Pittsburgh.). Phone: 412-904-6010; Web: awma.org
Orlando, Fla. **June 21–24**

Semicon West. Semiconductor Equipment and Materials International (San Jose, Calif.). Phone: 408-943-6900; Web: semi.org
San Francisco, Calif. **July 12–14**

BioPlastek 2011. The Chemical Development and Marketing Assn. (Mount Laurel, N.J.). Phone: 856-439-9052; Web: bioplastek.com
New York, N.Y. **June 27–29**

EUROPE

Blowing Agents and Foaming Processes. Smithers Rapra Technology Ltd. (Shrewsbury, U.K.); Phone: +44-1939-250383; Web: polymerconferences.com
Düsseldorf, Germany **May 10–11**

5th Annual Global Refining Summit. WTG Energy, a div. of World Trade Group (London); Phone: +44-207-202-7690; Web: refiningsummit.com
Rotterdam, Netherlands **May 18–20**

Techtextil. Messe Frankfurt GmbH (Frankfurt am Main). Phone: +49-69-75-75-0; Web: techtextil.com
Toronto, Ont. **May 24–26**

19th European Biomass Conference & Exhibition 2011. ETA-Renewable Energies (Florence, Italy) and WIP-Renewable Energies (Munich, Germany); Phone: +39-055-500-22-80; Web: conference-biomass.com
Berlin, Germany **June 6–10**

2nd Indo-German Catalysis Conference. Dechema e.V. (Frankfurt am Main, Germany); Phone: +49-69-7564-254; Web: processnet.org/igcc2011
Rostock, Germany **June 19–22**

Conference on Energy Process Engineering — Efficient Carbon Capture for Coal Power Plants. Dechema e.V. (Frankfurt, Germany). Phone: +49-69-7564-0; Web: icepe2011.de
Frankfurt am Main, Germany **June 20–22**

3rd European Process Intensification Conference. Dechema e.V. (Frankfurt am Main, Germany). Phone: +49-69-7564-0; Web: icheme.org/EPIC2011/
Manchester, U.K. **June 20–23**

ASIA & ELSEWHERE

CPhI Japan. UBM Japan Co. (Tokyo). Phone: +81-3-5296-1020; Web: cphijapan.com
Tokyo **April 18–20**

Global Conference on Energy Sustainability. AIChE (New York) and Dechema e.V. (Frankfurt am Main, Germany); Phone: 203-702-7660; Web: aiche.org/conferences/specialty/ESPI.aspx
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Suzanne Shelley

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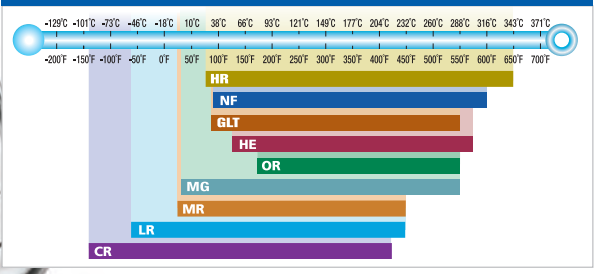

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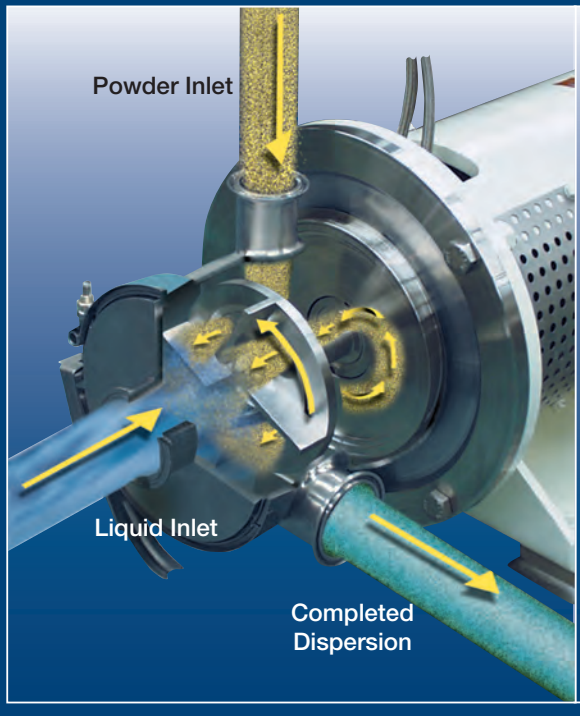

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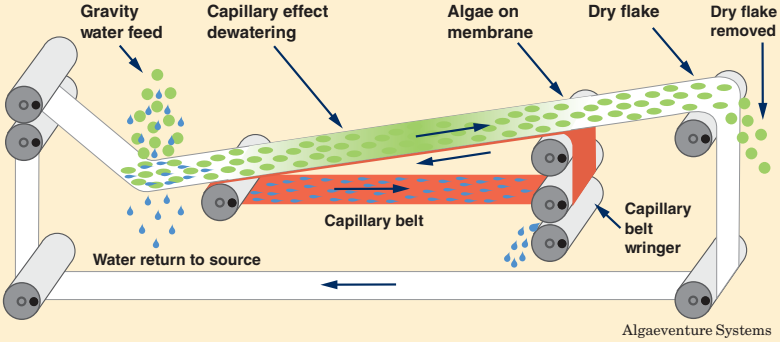
A low-energy, belt-based solid-liquid separation technology

Engineers at Algaeventure Systems (Marysville, Ohio; www.algaevs.com) have developed a system for separating liquids from solids and semisolids at very low energies. Originally targeted at harvesting and dewatering microalgae where the technology could remove a major cost barrier, the separation system can be used for a host of applications, including drying food-waste effluent, coal fines, hog manure, anaerobic digester effluent and others.

For example, Algaeventure's system is capable of producing 1.0 kg/h of product cake from a concentrated (11 g/L) algal feed solution at an energy cost of $2.76/ton. Company tests indicate that, using a centrifuge, the same feedstream produced only 0.05 kg/h of product cake at an energy cost of $2,718/ ton.

The technology consists of a polyester-based membrane screen moving on a conveyer belt system that comes in contact with a capillary belt moving in the opposite direction (diagram). "You're basically 'painting' a moving screen with the algae mixture," explains Ross Youngs, Algaeventure CEO, "and using a surface-tension differential to pull out the water."



Algaeventure Systems

After being gravity-fed onto the membrane, algae material passes over a capillary belt that pulls water from the screened algae by capillary action. The dried algae are then scraped off the membrane as a flaky solid. The capillary belt is made from a proprietary combination of natural and synthetic fibers, Youngs says.

Small-scale laboratory models have recently become commercially available, and slightly larger, prototype models are being used at partner sites. Algaeventure recently received a grant of close to $6 million from the U.S. Dept. of Energy's (Washington, D.C; www.energy.gov) ARPA-E (Advanced Research Projects Agency-Energy) program to develop the technology. Youngs says his company hopes to build production models with throughputs 300% higher than the prototype.

Recycling copper

Steinert Elektromagnetbau GmbH (Cologne, Germany; www.steinert.de) has recently installed its first XSS-F unit — said to be the world's first in-line system designed to extract copper from shredded ferrous scrap. Installed at a German recycling yard, the system is capable of processing high volumes of steel scrap directly out of the shredder.

The new sorting system features a high-speed X-ray fluorescence (XRF) sensor from Olympus Innov-X (Woburn, Mass.; www.innovx.com), which performs elemental analysis within milliseconds. The XXS has a throughput of 100 ton/h and has an average end-product output of more than 0.20% Cu.

Supercritical CO_2 Brayton-cycle system packs efficiency into small footprint

Electricity generation of 240 kW has been demonstrated in a Brayton-cycle system that uses supercritical carbon dioxide as the working fluid. Developed by researchers at Sandia National Laboratory (Albuquerque, N.M.; www.sandia.gov) and built by Barber Nichols Inc. (Arvada, Colo.; www.barbernichols.com), the prototype power-generation system, when scaled up, offers several advantages over steam-Rankine generators, including higher efficiencies at temperatures between 400 and 750°C, and several times smaller size than steam-Rankine-type generators of equivalent electricity generating capacity.

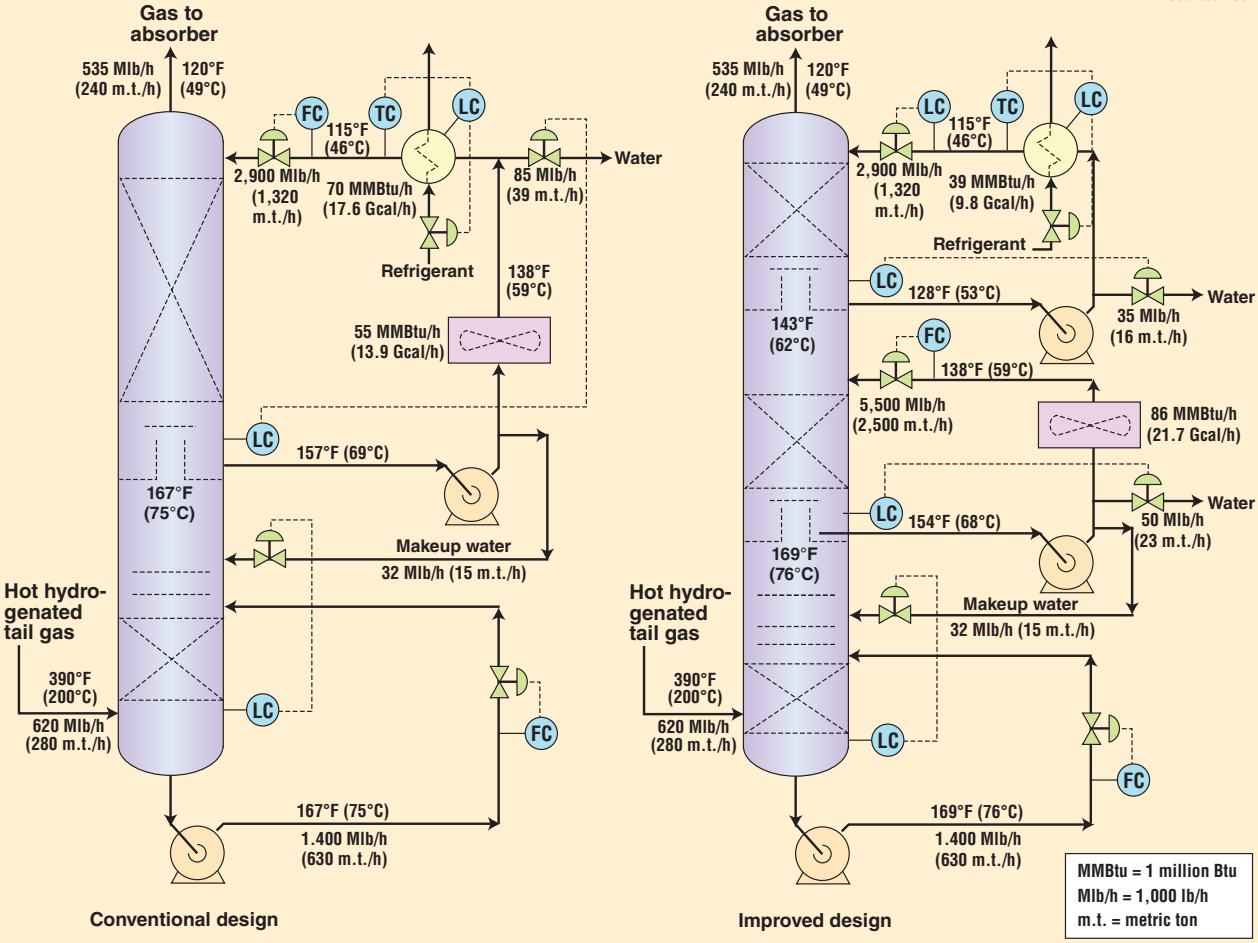
Sandia's Brayton-cycle system works by compressing and heating supercritical CO_2, then allowing the hot, high-pressure gas to expand in a turbine and extracting the work for electricity. Advantages of the system are largely due to the lower energy required for fluid compression, and the system's ability to reject heat at a nearly constant temperature during the cycle. "Near the critical point, CO_2 has a density 60% that of water, and is almost incompressible, which means the compressor acts more like a pump and requires less energy," explains Steve Wright, the Sandia scientist leading the project. "That, combined with a heat-rejection process that occurs at nearly constant temperature, accounts for the increased efficiency."

The properties of supercritical CO_2, including its high density and the fact that it remains in a single-phase throughout the cycle, means that even at large scales, the turbomachinery required for these Brayton-cycle systems would be small — 1-m dia. for CO_2 versus 5-m dia. for steam. Single-phase CO_2 also means the power-system design is relatively simple, reducing the need for ancillary equipment such as valves. Further, the low turbine-to-compressor pressure ratio means that only three or four stages of axial turbomachinery are needed, compared to 20–30 in a steam-plant turbine, says Wright. Sandia's system requires a heat recuperator, but those components can also be kept small using micro-channel, diffusion-bonded heat exchangers.

Having met the research goals with the prototype, the Sandia team is speaking with a number of potential scaleup partners to build a 10-MW_e system.

Note: For more information, circle the 3-digit number on p. 66, or use the website designation.

Source: Fluor



New condenser design for Claus tail gas slashes refrigeration duty

Last month, at the AIChE Spring Meeting in Chicago, Henry Kister, director of Fractionation Technology at Fluor Corp. (Aliso Viejo, Calif.; www.fluor.com) presented a patent pending process (U.S. Patent Application WO/2011/016797) that promises to cut energy demand and capital costs for direct contacting condenser (DCC) units that would typically require refrigeration or cooling water in the pretreatment of Claus sulfur-laden tail gas.

In a conventional process, tail gas from the Claus plant is hydrogenated to convert all the sulfur species to H_2S. The tail gas is then cooled in a DCC (flowsheet, left) to prepare it for absorption in an amine-based H_2S-removal unit. In many situations, DCC air coolers cannot sufficiently cool the H_2S-removal unit's feedgas stream. And in arid regions, cooling water is usually not available for trim cooling, so expensive refrigeration must be used.

Fluor's new process (flowsheet, right), developed and patented by Kister and Dick Nielson, vice president, of Process Technology at Fluor, splits the direct-contact tower's packed bed into two direct-contact packed beds, and the pumparound circuit into two separate circuits. The bottom pumparound circuit is all air-cooled, while the top pumparound circuit is all refrigerant or water-cooled. The new DCC design transfers as much as 50% of trim cooling duty to the air cooler, says Kister, and results in a significant net reduction in capital costs.

"You're no longer stuck with two pinches," says Kister. So, for each bed the temperature approach at the bottom can be larger (say 15°F or 8°C, compared to say 9°F or 5°C for the conventional process), which reduces required bed heights and renders the bed far less sensitive to liquid maldistribution and fouling, he explains.

Meanwhile, the higher temperature approach in the bottom section readily permits using a low (say 5°F or 3°C) temperature approach between the process water and gas at the top of each bed. This low temperature approach at the top of the bottom bed brings the refrigeration savings, allowing more heat to be removed in the air-cooler pumparound loop.

An evaluation of two recent large-scale Middle East projects indicated that this new design would result in about $13–20 million in power savings over a 20-year life of the plants and an additional $40–70 million in capital cost savings.

Ta heat exchangers

Last month, Tantaline (Waltham, Mass.; www.tantaline.com) launched the first fully welded plate-and-frame heat exchanger designed specifically for hot acids and corrosive materials. The tantalum-surface alloyed exchangers are said to have a heat-transfer efficiency 5–7 times higher than shell-and-tube designs, which

(*Continues on p. 14*)

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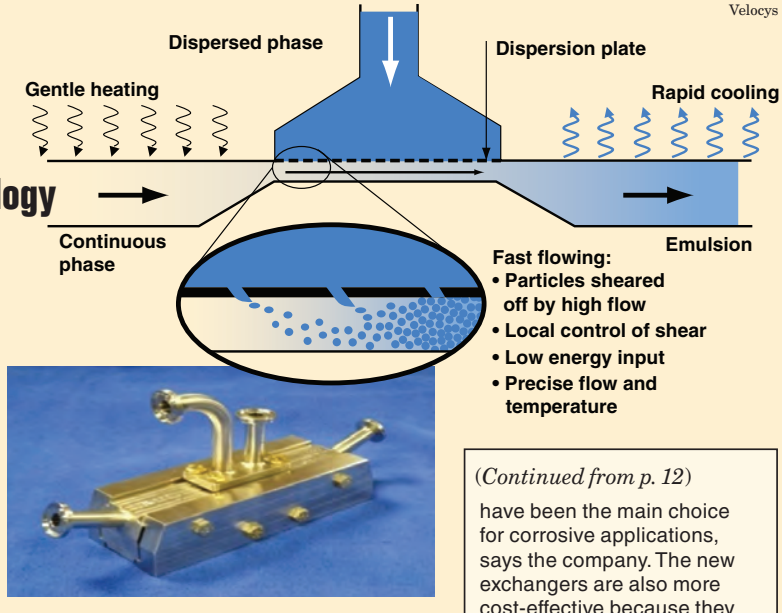
Commercial testing set for 'smooth' emulsification technology

A new emulsification process, known as Smooth, has been developed by Velocys, Inc. (Columbus, Ohio; www.velocys.com) — a member of the Oxford Catalyst Group Plc. (Abingdon, U.K.) — and is now ready for testing at the commercial scale. Smooth technology is based on microchannel devices, and has undergone successful pilot trials by a number of major companies in the cosmetic and personal care industries using a pilot device, which can produce up to 5 L/min of an emulsion. For commercial-scale production, the process can be scaled up readily by adding (numbering up) modules that retain the same process parameters proven in the small scale, says product manager Mark Grace.

In microchannel emulsification (diagram), droplets are formed one at a time by pumping the discontinuous phase through a porous dispersion plate into a crossflow of the continuous phase. This allows for high shear at the wall but low bulk shear to avoid damaging fragile emulsion components, explains Grace. The plates are stacked together to form a module, which can also incorporate microchannel plates for heating or cooling. Five modules can be combined inside a cradle for production capacities of up to 25 L/min.



The microchannel emulsifier can produce very small droplets (down to 1 µm and below) with a narrow droplet-size distribution, which leads to stable emulsions that reduce or eliminate the need for surfactants. Smooth technology has the advantage of precise mixing and control of mixing energy, which can "greatly" reduce the energy required to form emulsions, especially compared to rotor mixers, says Grace. The precise process control and very-low liquid inventories required by Smooth minimizes the amount of off-specification product and materials discarded during cleaning, he says.

A step toward industrial production of perillic acid

Last month, Brain AG (Zwingenberg; www.brain-biotech.de) and Dechema e.V. (Frankfurt am Main, both Germany; www.dechema.de) began a collaboration in the biotechnological production of perillic acid — a patented natural monoterpene owned by Brain, with promising applications as a bioactive compound in cosmetics products and as a natural preservative. The cooperation is aimed at further developing an integrated bioprocess — developed by Jens Schrader, head of the Biochemical Engineering Group of Dechema's Karl-Winnacker Institute (KWI; www.kwi.dechema.de) — for microbial (+)-perillic acid synthesis from the inexpensive precursor (+) limonene.

Up to now, there has been no industrial process for making perillic acid, and the laboratory synthesis involves four, low-yield steps, says Schrader. And natural sources for perillic acid, such as essential oils of lemon grass, are found in concentrations too low to be extracted economically, he says. In the Dechema process, limonene — which can be extracted in large quantities from orange peels — is oxidized by a strain of *Pseudomonas putida* (DSM 12264) into perillic acid in a batch fermentation reactor. In-situ recovery of the perillic acid is performed by continuous circulation of the culture with a peristaltic pump through a fluidized bed of anion exchange resin (Amberlite IRA 410 Cl), which enables continual removal of product. In laboratory trials, the process yields a high perillic acid concentration (31 g/L), thus reducing the downstream processing steps needed.

The collaboration between Brain and Dechema will combine the microbiological and molecular-biological methods of Brain with the strain and process optimization and scaleup know-how of KWI to develop an effective process for technical production of perillic acid.

(*Continued from p. 12*)

have been the main choice for corrosive applications, says the company. The new exchangers are also more cost-effective because they require one-seventh the footprint and use less material.

NH_3 catalyst

Scientists at QuantumSphere Inc. (Santa Ana, Calif.; www.qsinano.com) have developed a nanoscale iron coating for ammonia synthesis catalysts that can generate up to a 40% increase in catalyst activity. The 1% nano-Fe coating has the dual effect of increasing catalyst surface area and optimizing the crystal-structure geometry at the catalyst surface for the required Haber-Bosch ammonia synthesis chemistry. In company tests versus traditional, non-coated catalyst material, the coated catalyst also showed better durability, says Jason Norman, vice president for business development. The company's proprietary coating process is in the external validation stages, and Norman anticipates that the catalysts will be available for commercial use by the end of 2011.

Composite materials

Bayer MaterialScience AG (Leverkusen, Germany; www.bayerbms.com) has developed a new method for introducing fillers into reactive polyurethane (PU) blends. Normally, fillers are added to the polyol component before it is blended with the isocyanate component in the high-pressure mixing head and retracted. The disadvantages of this method are that

(*Continues on p. 16*)

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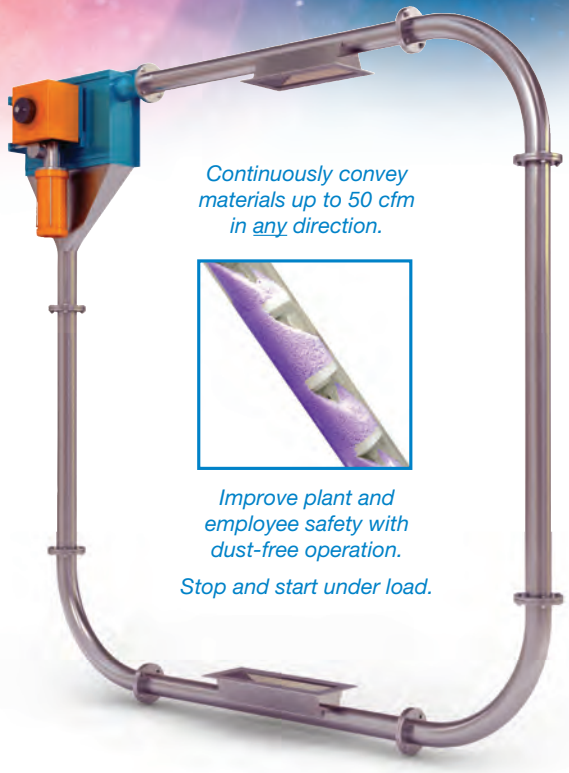

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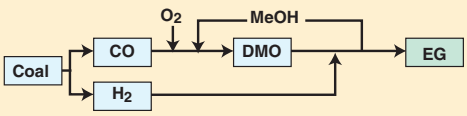
Making a polyester feedstock from coal



DMO process $2CO + {}^1/_2O_2 + 2CH_3OH \longrightarrow DMO + H_2O$

EG process $DMO + 4H_2 \longrightarrow EG + 2CH_3OH$

Overall $2CO + 4H_2 + {}^1/_2O_2 \longrightarrow EG + H_2O$

Ube Industries, Ltd. (UBE; www.ube-ind.co.jp) and HighChem Co. (both Tokyo; www.highchem.co.jp) will license their technologies for transforming coal-derived synthesis gas (syngas) into manufacturing ethylene glycol (MEG) — a polyester feedstock — to Qianxixian Qianxi Coal Chemical Investment Co. (Guizhou, Qianxixian, China). The license covers a process for manufacturing dimethyl oxalate (DMO) and a process that converts DMO into MEG. This will be the first commercial process for making MEG from coal, say the companies. Until now, MEG has been produced from ethane from gases associated with crude oil, or ethylene from naphtha.

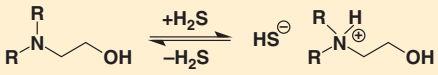
Qianxixian plans to build a coal-gasification facility and a 300,000-metric-tons (m.t.) per year MEG facility (720,000-m.t./yr DMO) in Guizhou Province, with plans to bring them online sometime around the end of 2012 or early 2013.

The companies did not disclose process details (flowsheet) except to say that the DMO process is based on a proprietary CO-coupling reaction from UBE that employs a palladium catalyst. UBE's proprietary nitrite technology is said to be highly selective for the formation of carbon-carbon bonds. The MEG process is now undergoing a HighChem-led pilot demonstration in China based on UBE technology.

A lower-energy approach to natural gas 'sweetening'

A new method for removing hydrogen sulfide from natural gas at ambient temperatures could allow significant energy savings compared to the current technique and increase the range of H_2S concentrations in natural gas that are economically viable to extract from sour gas wells.

In conventional sour-gas scrubbing, aqueous solutions of alkanolamines are used to selectively absorb H_2S, but due to water's high specific heat, temperatures over 100°C are required to regenerate the sorbent material. Scientists at Pacific Northwest National Laboratory (PNNL; Richland, Wash.; www.pnl.gov) have developed a new approach that uses anhydrous tertiary alkanolamines, such as dimethylethanolamine (DMEA), to react with H_2S in natural gas. The uptake of H_2S (which acts as a Brønsted-Lowry acid) forms a hydrosulfide salt in a reversible reaction (see equation). Then, instead of heating to



regenerate the DMEA, a nonpolar solvent is added to effect the rapid release of H_2S.

Known as antisolvent swing regeneration (ASSR), the technique could increase the efficiency of natural-gas sweetening by at least 10% — and more if the process uses an antisolvent that naturally phase-separates when reactivating the alkanolamine, explain David Heldebrant and Philip Koech, PNNL scientists who led the research project. Natural phase separation would avoid the need for a distillation step.

"We also suspect that avoiding the need for heat in regeneration will avoid thermal decomposition and lengthen the lifetime of the chemical sorbents," comments Heldebrant. The PNNL team is looking for industrial partners to help scale-up the process.

(*Continued from p. 14*)

the system components must be protected against wear caused by the filler, and large or mechanically sensitive filler particles limit the options.

With the new Solid Injection by Air Stream (SIA) method, polyol, isocyanate and filler are all added separately and not blended together until reaching the mixing head. The SIA method enables the use of very light or heavy fillers or a combination of different types — even reactive substances or particles with porous surfaces can be used. The company believes the technology will enable new possibilities for the use of composite materials, regardless of whether they are hard or soft, or if the PU is a solid or a foam. ❑

New propylene and butadiene process could be utilized next year

By the end of this year, Asahi Kasei Chemicals Corp. (Tokyo; www.asahi-kasei.co.jp) plans to complete development on two processes that utilize excess C_2 and C_4 fractions for producing propylene and butadiene, and if all goes smoothly, implement them by March 2012. The company has been developing its so-called E-Flex (for producing propylene) and BB-Flex (for producing butadiene) processes, and is now performing demonstration tests at its Mizushima Business Place.

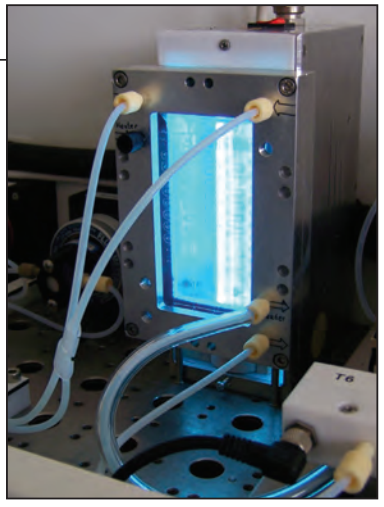
The E-Flex process converts C_2 fractions, such as ethane, ethanol and ethylene, into ethylene and propylene, with a high yield for propylene. The ethylene produced by E-Flex is recycled as feedstock to boost propylene production. Asahi Kasei says that E-Flex could be installed at its ethylene center where excess ethylene is produced, and also in the U.S. and the Middle East where ethane is available. E-Flex can also use bioethanol as raw material for making propylene.

The BB-Flex process produces butadiene from butane. After extracting butadiene and isobutylene, the raffinate fraction can be recycled as feedstock. The company expects that the BB-Flex process can be combined with a naphtha cracker or fluid catalytic cracking (FCC) unit.

Last year, Asahi Kasei invested ¥1 billion (about $10 million) in an E-Flex demonstration plant at Mizushima Business Place, and is testing the BB-Flex process in a bench-scale plant. The company is collaborating with Mitsubishi Chemicals Corp., with whom it operates the ethylene-production unit at Mizushima, and with JX Nippon Oil and Energy Corp., and plans further collaborations with other companies outside of Japan. Using excess C_2 and C_4 fractions enables downsizing of the two ethylene units at the site. ■

SHEDDING LIGHT ON MICROREACTORS

Small, intensified process equipment is quietly causing a paradigm shift in the way fine chemicals are produced, and the shift toward bulk chemicals is progressing



mikroglas chemtech

FIGURE 1. Microreactors are being developed for all kinds of chemical processes. This continuous photo reactor, developed by mikroglas chemtech (Mainz) is being illuminated by an excimer laser. Although mainly used for R&D, a reactor of this type is being used for the commercial production of a pharmaceutical product

Traditionally, most students have learned about chemical reactions in laboratories using test tubes, beakers and flasks. Those who have gone on to discover and develop new products have typically done so using these batchwise techniques. This mind-set has continued through the piloting and scaleup phase, so that today, many chemical products are produced batchwise in large, continuously stirred reactors.

This paradigm is now being challenged by microreactor technology, whereby a chemical reaction is performed continuously in tiny channels that have been machined, etched, burned or otherwise fabricated into glass, ceramics, stainless-steel and exotic-alloy plates that are stacked and bonded together to form modules. The small dimensions — with channel depths ranging from a few micrometers to several millimeters — result in extremely efficient heat and mass transfer, which enables improved reaction control with reduced side reactions and byproducts, and improved safety.

Since microreactor technology was first introduced in the mid 1990s (*CE*, March 1995, p. 52), it has quickly developed from a laboratory curiosity into commercial systems that incorporate correspondingly small static mixers, heat exchangers and other process equipment. Such devices are not only increasingly being used in laboratories of universities, research institutes and R&D departments of producers, but are slowly but surely being integrated into commercial production plants. Now the focus of R&D is toward "container plants" in which a complete chemical production plant can be assembled from standardized components, with mixers, reactors and downstream processing units that can be assembled onto mobile containers.

Moving into production

According to a recent survey conducted by Dechema e.V. (Frankfurt am Main, Germany), there are approximately 20–30 plants in operation worldwide using microstructured components, says Roland Dittmeyer, director of the Institute for Micro Process Engineering (IMVT), Karlsruhe Institute of Technology [KIT; formerly the Karlsruhe Research Center (FZK); Germany; www.imvt.kit.edu]. "The number of industrial pilot plants is believed to be much higher," he says.

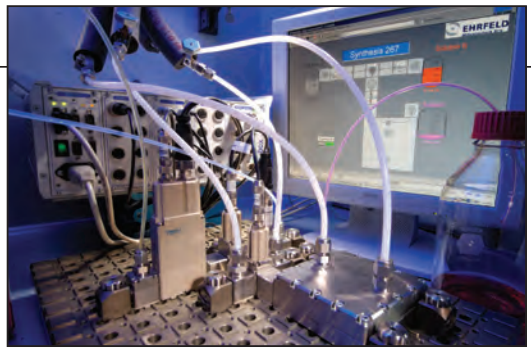
In one of the earliest, and still active commercial applications of microreactors, IMVT developed the mixer-reactor system for production of an amide by the so-called Ritter reaction at DSM Fine Chemicals (Linz, Austria). The system has a design throughput of 1.7 metric tons (m.t.) of liquid reactants per hour, and has been used for production campaigns — usually one per year lasting several weeks or months — at DSM Linz since 2005, says Dittmeyer. The system achieves a 15% higher yield than conventional technology, he says.

Although the system is not perfect — the mixer module has been further improved since the first campaign, and there have been problems with corrosion due to the use of hot sulfuric acid in the small channels — it has been used to produce well over 1,000 m.t. of product, says Dittmeyer.

Other commercial applications of microreactor technology that have been published and reported in recent years, include the production of nitro glycerine, azo pigments and liquid crystals as well as the industrial performance of Grignard, alkylation and other hazardous reactions. (A table of industrial applications is included in the online version of this article at www.che.com). The technology is even moving into the consumer sector, as a tiny liquid-propane gas reformer and fuel-cell system, developed at IMM Mainz GmbH (Mainz, Germany; www.imm-mainz.de), is being commercialized this year by Truma Gerätetechnik GmbH & Co. KG (Putzbrunn, Germany) for use in recreational vehicles.

From niche to bulk applications

While many of the demonstrations thus far have been in the fine chemicals business, Velocys, Inc. (Columbus, Ohio; www.velocys.com) is working toward applying microreactor technology for the production of liquid fuels and other chemicals. Velocys — a company of the Oxford Catalysts Group Plc. (Abingdon, U.K.; www.oxfordcatalysts.com) — is actively developing three applications: steam methane reforming (SMR), to convert methane into synthesis gas (syngas); Fischer-Tropsch (F-T) synthesis, to convert syngas into liquid hydrocarbons; and hydroprocessing, to



Ehrfeld Mikrotechnik BTS

FIGURE 2. An entire production process can be built on a table using equipment blocks connected together. The system shown here is primarily used for R&D purposes

process the F-T liquids into fuels and other products, says Jeff McDaniel, director of business development.

Furthest along Velocys' development pipeline is its F-T technology, with a 100-L/d demonstration unit operating in Güssing, Austria since last summer. The demonstration involves a coalition that includes SGC Energia (Alhandra, Portugal; www.sgc.pt), the Oxford Catalysts Group, Repotec GmbH (Vienna, Austria; www.repotec.at), the Technical University of Vienna (www.vt.tuwien.ac.at) and the gasification facility owner Biomass CHP Güssing. In Güssing, biomass is gasified (Repotec technology) into syngas, which is cleaned and fed to Velocys' microchannel F-T reactor. The reactor features a catalyst developed by Oxford Catalysts. The combination of this catalyst with microchannel reactors has achieved a productivity orders of magnitude greater than conventional F-T systems: 1,500 kg/m^3/h versus 100 kg/m^3/h for fixed-bed reactors and 200 kg/m^3/h for slurry reactors, says the company.

In addition to the Güssing plant, Velocys' SMR technology is to be demonstrated this summer along with the F-T technology in a 5–10-bbl/d integrated gas-to-liquids (GTL) pilot plant at Petrobras Brasileiro S.A. (Rio de Janeiro) petroleum refinery in Fortaleza, Brazil. Last April, Velocys entered into joint development agreement with Modec (Tokyo; www.modec.com), Toyo Engineering Corp. (Chiba, Japan; www.toyo-eng.co.jp), Kobe Steel, Ltd. (Kobe, Japan; www.kobelco.co.jp), and Petrobras designed to accelerate SMR by 200-fold and F-T reactions by 10–15 fold, says the company. The GTL demonstration in Brazil is scheduled to operate for at least 6 mo., and will include a microchannel SMR and a microchannel F-T reactor, which will be operated in an integrated manner, says McDaniel. Upon successful operation, Velocys will begin commercially licensing the microreactor GTL technology, he says.

Besides these syngas-based projects, Velocys is also working on commercializing a microchannel emulsifier for making cosmetics and other consumer products (see Chementator on p. 14).

Microreactor technology also played a key role in the development and scaleup of the hydrogen-peroxide-based process for producing propylene oxide (PO), which was recently commercialized by Uhde GmbH (Dortmund; www.uhde.eu) and Evonik Degussa GmbH (Essen, both Germany www.evonik.com). The complete process was demonstrated in a miniplant featuring all of the process steps, and described by means of a simulation model. This is particularly important in order to detect trace components in the closed recycle loops at an early stage and to permit a low-risk scaleup to commercial scale. The scaleup procedure — from miniplant to a world-scale PO facility with a capacity of 100,000 m.t./yr as a reference plant — was carried out in a single development step, says Uhde (for more, see *CE*, December 2009, pp. 17–21).

Scaleup with microreactors

Whereas ten years ago everyone thought reactors with tiny channels were going to solve every problem, some early adopters have been very disappointed, says Sergio Pissavini, business director, Reactor Technologies, Corning S.A.S. (Avon, France; www.corning.com). Back then, the idea was to make the channels as small as possible in order to obtain the theoretical advantages of heat-and-mass transfer. But clogging is a major problem for micrometer-sized channels.

Now, "the channels are as small as necessary instead of as small as possible," he says.

Last year, Corning launched the Gen 3 module for its Advanced-Flow LF reactors, which has a reactor volume of 70–90 mL compared to 45 mL for the Gen 2 module that was introduced at Achema 2009. When several units are run in parallel these reactors can handle volumes of 1,000 L and produce tons of product, says Pissavini. And the channels are sufficiently deep to allow handling of even slurries and precious-metal catalysts of 60–80 nm, Pissavini says.

At Ehrfeld Mikrotechnik GmbH BTS (Ehrfeld-BTS; Wendelsheim, Germany; www.ehrfeld.com), several standardized systems have been developed to enable process development from the laboratory through production. In the laboratory, a lot of information about a chemical reaction, such as the kinetics, can be learned faster in micrometer-sized channels than in batch studies in flasks, says managing director Joachim Heck. For this, the company offers the technology developed by Professor Wolfgang Ehrfeld and subsequently acquired by Bayer Technology Services GmbH (BTS; Leverkusen, Germany; www.bayertechnology.com) in 2004, when Ehrfeld-BTS was established. The Ehrfeld system is composed of block unit-operation components that can be quickly connected together — almost like Lego blocks — to perform and study the chemistry of a process (Figure 2).

The company also offers the Miprowa system, which was developed by BTS and commercialized in 2009–2010. Miprowa is specifically suited for process scaleup — to speed the time to market and reduce the scaleup risks. The technology was developed along the ideas used for scaling up tubular reactors, explains Heck. With tubular reactors, one first studies the chemistry in a small laboratory tube until the reaction chemistry is well understood. The next step is to pilot the reaction in a single tube having the same dimensions (diameter and length) as those used in a commercial installation. Once all the process conditions are understood, one goes to the production scale by simply numbering up the number of tubes.

With Miprowa, the same idea is used but instead of tubes, rectangular channels are involved. The laboratory unit has 1.5-µm deep channels, which enables the kinetics of the reaction to be understood. In the pilot unit, the reactor has channel sizes (3 mm by 80 mm) that are small enough to maintain the advantages of microchannels, but sufficiently large for increased production volumes and reduced fouling problems. Once the performance losses (due to changes in the reactor's surface-to-volume ratio) are well understood, scaleup to production involves "numbering up" the number of channels. This has the advantage that the principles are known in advance, so there will be no surprises during scaleup, says Heck.

Lonza Ltd. (Visp, Switzerland; www.lonza.com) used similar ideas when developing the so-called Lonza Reactor for its custom synthesis business. "This approach has proven successful and a number of flow processes already exist in the fine chemical industry, particularly for hazardous or even hardly controllable reactions," says Dominique Roberge, head of Continuous Flow, Microreactor Technology Business Development. "At Lonza, we have produced several tons of material based on micro-structured and milli-scale reactors. New applications are being investigated and further scaleups are planned for the future," he says.

"Our team specializes in the development of continuous flow processes for pharmaceutical and fine chemical applications," says Roberge. For some reactions in these fields, it is imperative to use a flow process to address scalability and safety of reactions. Thus flow processes are becoming more and more established and will play a significant role in the future in these fields, he says.

Last year, Lonza entered a sales and manufacturing agreement with Eh-

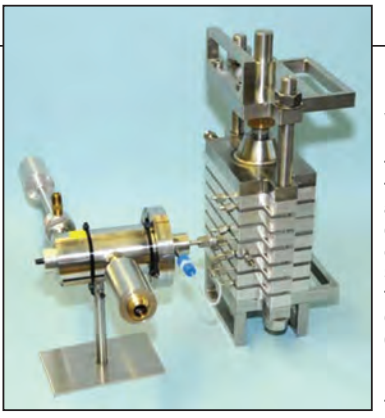

Lonza

FIGURE 3. While clogging is still a problem for microreactors, methods are being developed to overcome them. Shown here is the Lonza MicroReactor A6 (right) in combination with an ultrasonic system (left)

rfeld-BTS, which is manufacturing and selling Lonza's proprietary MicroReactor Technology on a global basis. This collaboration has enabled the Lonza prototypes to be further developed and commercialized as a complete reactor toolbox that is modular, robust and fully scalable up to industrial scales also under cGMP, says Roberge.

Where the research is

Although the once troublesome issue of scaleup is now well understood, plugging issues still remain. Lonza developed an ultrasonic solution to de-plug the microstructures in place without having to dismantle the reactor. This ultrasonic system works in combination with the Lonza MicroReactors (Figure 3), and permits deplugging of localized zones, such as the mixing zone. "Plugging problems are a main concern not only with microreactors, but also with continuous flow in general, and can occur in static mixers as well," says Roberge. "Thus, a microreactor is a useful tool to study this issue at the laboratory scale and solve the problem prior to scaleup," he says.

In Europe, several major research projects under the EU's 7th Framework Program are now underway. The biggest — a €30-million project called F3 Factory (www.f3factory.eu) that is coordinated by BTS — involves 25 partners from universities (13), equipment suppliers (5) and major industrial producers (7), including BASF, Evonik, Procter & Gamble and AstraZeneca. The goal of F3 Factory includes the development of standardized, modular, continuous production technology of which microreactors are a part.

KIT is involved in two of the many projects of F3 Factory: one to develop an onsite production process for the oxidation of SO_2 into SO_3 (used in manufacturing surfactants); and a modular system for performing three-phase hydrogenation reactions. The first involves a microreactor with internal walls coated with a catalyst, and the second is to develop a microreactor with the flexibility to handle different catalyst suspensions.

Another major EU project involving microreactors is the €17-million, four-year CoPiride (www.copiride.eu), which is being coordinated by IMM and involves 15 partners. Among the studies underway are the use of process intensification for ammonia production from biomass, and the development of modular, compact production plants based on a container approach. IMM and Evonik have already developed such a container platform that has normalized process control and safety infrastructures.

Among the investigations of both of these projects and other extensive research today is downstream processing. If you want a compact, skid-mounded production unit, it won't work if the reactor is small but you need a large column for separation of the product, says KIT's Dittmeyer.

Among the many efforts to develop small separation technology is mikroglas chemtech GmbH (Mainz, Germany; www.mikroglascom), which developed its first continuous separation modules in 2009. Together with German universities, the company is developing modules for: changing solvents during multi-step reactions; for separating and recycling expensive solvents; for two-phase, liquid-liquid separation based on the hydrophobicity of liquids and plasma-modified surfaces; for gas-liquid separation with a micro gravity settler; and a gas-liquid separator using capillary forces. ■

Gerald Ondrey

MIX IT UP!

Multi-function mixing equipment helps chemical processors gain a competitive edge

FIGURE 2. Designed for a range of services in the chemical process industries, Model 20 HT/GT agitators feature a high-efficiency gearbox designed for long service life

The chemical process industries (CPI) are looking for ways to decrease and control costs, and mixing operations are not exempt from this belt tightening. But because mixing is vital to most chemical processes, it can't be eliminated. As a result, processors are clamoring for mixing equipment that provides more functionality so they can perform more tasks with less equipment.

"Now more than ever, processors require versatility and flexibility from their mixing equipment. This may mean one of two things: either being able to run different products on the same piece of equipment or use one mixing vessel to perform different steps in the process," says Bjarne Darre, director of mixing systems with GEA Process Equipment (Skanderborg, Denmark). "It is a valid request, but this new trend means mixing equipment vendors will need to step up to the challenge. The ability to design equipment that can do more takes knowledge and know-how of both mixing and reactions."

Fortunately for the CPI, it would seem that many mixing equipment specialists are prepared to meet these demands.

One mixer, multiple products

Today's product lines have to be as versatile as possible, and this means chemical processors would like to have process lines where they can mix various kinds of products on the same mixing equipment.

To meet these needs, GEA has developed Batch Formula mixing systems, which feature interchangeable stator rings that allow end users to move from one product to another product using the same basic machine without a lot of changes. The dynamic stator system allows the mixing and homogenizing in one unit operation, going from low shear to high shear with a single output. As a result, very short mixing times are possible with a high degree of process flexibility.

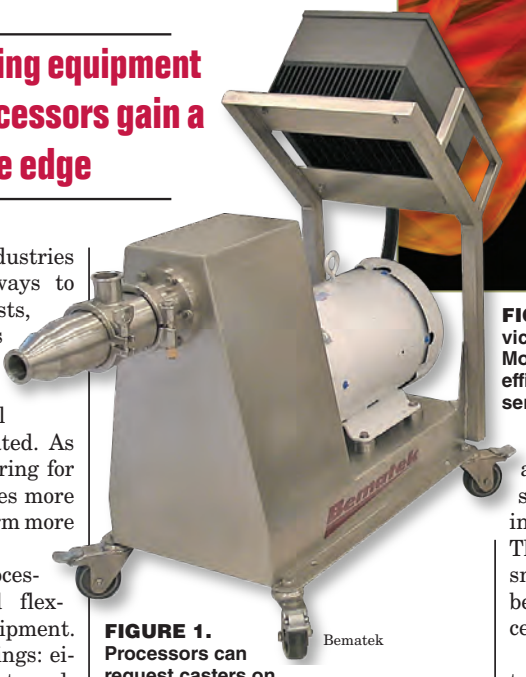


FIGURE 1. Processors can request casters on Bematek's Model 250 inline dynamic mixer if they require the flexibility to move the unit from process line to process line

Dave Ekstrom, president of Bematek (Salem, Mass.) agrees that flexibility is important. "We have one customer who changes products by adding different additives to the same base material, so by using an inline mixer, the processor can save material by only blending the additives into lower quantities of the base material right inline," he explains.

The benefits of this type of flexibility are many. Instead of having to make large batches in a large tank, the processor can run the basic ingredient made for every one of its customers and then add the various additives in smaller quantities through the mixing unit as needed for each customer. This saves time in that they can make smaller batches, reduces clean up time between batches and allows the processor to supply its customer faster.

"In a competitive market, they can tailor production without making big batches and warehousing those premixed materials for each customer and still be competitive," says Ekstrom.

Bematek's inline mixers have a Modular Engineered Design that provides the flexibility to instantly reconfigure the mixing chamber for processing multiple diverse products (Figure 1). The conversion is performed onsite with standard parts, using no special tools or alignment procedures. Optional casters and inlet/outlet adapters make the mixer mobile enough to be easily moved from one production line to another. "A single inline mixer can be purchased to process an entire line of products," says Ekstrom.

Shaun Mott, owner of Dynamix (British Columbia, Canada) concurs that smaller batches of multiple products is the more profitable way to go. "Many of our clients are looking to reduce their batch sizes in order to be more flexible with their end users and offer more products," he says "In the past this was restricted by the need to mix in large volumes in order to attain EOS [economies of scale]."

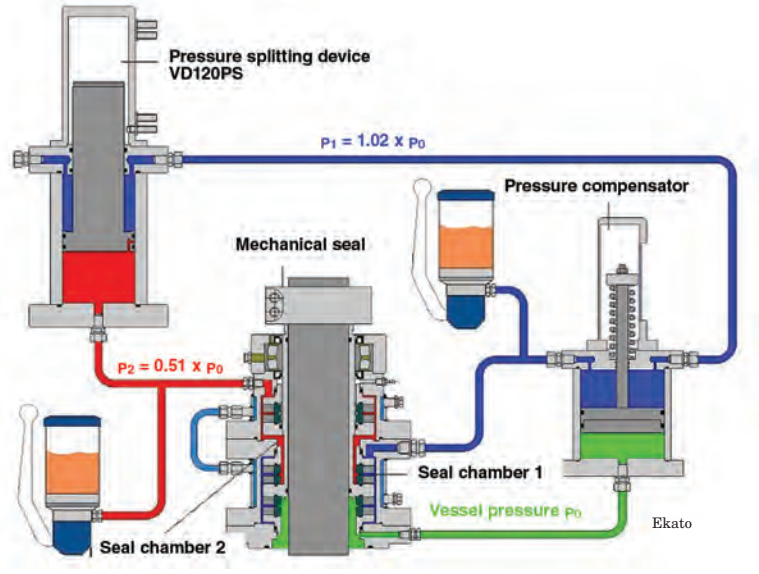


FIGURE 3. This triple-acting mechanical seal has two seal chambers and pressure splitters to increase the life of the seals in mixing and other applications

To help in this area, Dynamix has developed a line of Integrated Tote Mixers (ITM) that are designed to bring big tank mixing into the tote containers often used for transport. The 250–500-gal bulk containers are generally used for shipping, but with Dynamix's approach to mixing, these same containers can become process vessels.

"This increased mixing flexibility allows for smaller batch production," says Mott. "And in an interesting turn, some clients are also using the ITM to increase the large EOS as well. These customers are manufacturing in larger volumes and storing for longer periods of time. The totes allow them to blend and re-suspend just before shipping, bringing the product back into spec as if it had just been produced in the larger batch."

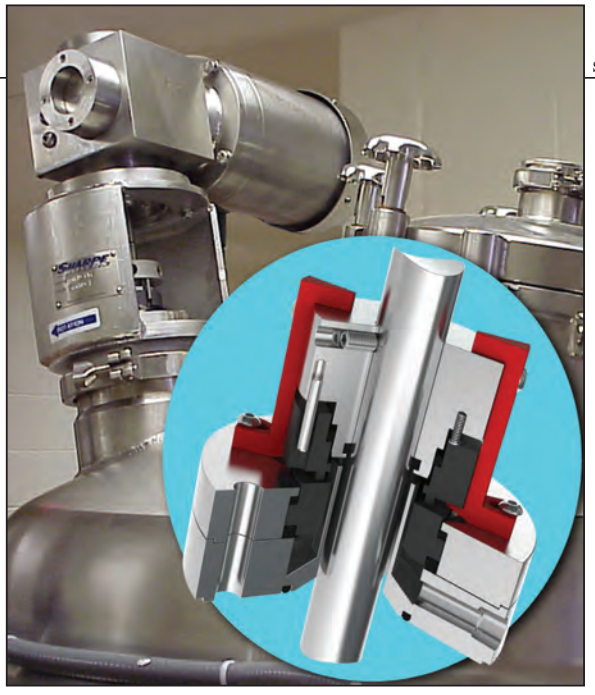
One mixer, multiple functions

In the CPI, folks are also looking to make a better product and make it with reduced mixing or transfer steps in an effort to increase efficiency and reduce costs, says Ken Langhorn, technical director with Ross Mixers (Hauppauge, N.Y.). What this means is that when processors have chemical reactions going on in a vessel and the next step of the process requires the mixing of two ingredients, they might look for a vessel that can handle the reaction and also the mixing. "They would be using the same piece of equipment for multiple steps or processes, which eliminates the need to transfer ingredients from one vessel to another," says Langhorn.

Multi-tasking equipment for mixing often results in a boat-load of benefits, he says, including less capital costs for equipment, reduced floor space in the facility, less labor, less product lost in the pipeline between two vessels, less risk of contamination in transfer if the materials are susceptible to contamination from air or particulates from the plant, and less risk of employee exposure if it's a hazardous material.

Doug Grunder, president of Marion Mixers (Marion, Iowa) agrees and says that every time a piece of equipment can handle another step of the process without transfer or human interaction, it is more cost effective. "It just doesn't make sense to have one piece of equipment that breaks up lumps and incorporates a liquid and then send that material along to another vessel that's dedicated to heating," he says. "But doing multiple steps with one piece of equipment can get tricky and often requires a customized solution."

For example, most mixers are ribbon-style blenders designed for flour or sugar. Those are simple applications, but when you get into chemical



Sharpe Mixers

FIGURE 4. Dry running mechanical seals meet ASME standards for cleanability

processing there are large amounts of material, different densities and different particle shapes. A paddle-style agitator may be recommended if it's a crystal shape, so it can be scooped rather than scrubbed.

Next, says Grunder, you would base the shape of the trough on the processing requirements. If the material needs to be heated, the trough will require a jacket. To put it under pressure will require a vacuum, which means the shape of the trough should be changed from the typical U-shape to a cylindrical shape. If there are agglomerates that need to be broken up or material that needs to be dispersed, choppers will come into play. If drying is another step, a minor vacuum may be added because material dries faster under vacuum.

Pulling it all together

Processors must have flexible equipment that allows them to make today's product in whatever batch size is needed, as well as tomorrow's product, notes Andy Stump, manager, Batch Solutions, with Rockwell Automation (Milwaukee, Wis.). And, if they don't, they are buying something that doesn't fit their longterm strategy. This means end processors require the ability to perform quick changeovers, try out new and different recipes without having to recode or rebuild the system and to allow the equipment's control system to stand alone or integrate to a larger scale as needed.

Rockwell Automation's PlantPAX Process Automation System with the new PlantPAX Logix Batch and Sequence Manager solves a range of local, controller-based batch and sequencing needs, allowing users to configure sequences directly in the controller through the human machine interface using a standard user interface. The solution is suitable for standalone units like mixers, blenders and reactors that require flexibility in the sequence of the process and the formulas for the recipe.

"Many applications require sequence management capabilities, but the complexity of the process may not be great enough to warrant a server-based batch software package," he says. "This solution allows the user to start small but if at any point the requirements grow, users can simply migrate the Logix Batch and Sequence Manager into a comprehensive software solution like FactoryTalk Batch without costly re-engineering and testing."

Most custom controller-based recipe management solutions only allow for setpoint downloads to a fixed sequence. As a result, when the sequence must change, users are forced to change the code. This adds risk to the process and can add significant cost to re-test and validate the system. Logix Batch and Sequence Manager give users the configuration tools needed to change both the sequence and formula through a standard tool.

MIXING EQUIPMENT SERVICE PROVIDERS

Bematek	www.bematek.com
Chemineer	www.chemineer.com
Dynamix	www.dynamixinc.com
Ekato	www.ekato.com
GEA Process Engineering	www.niroinc.com
Lightnin	www.lightninmixers.com
Marion Mixers	www.marionmixers.com
Munson Machinery	www.munsonmachinery.com
Philadelphia Mixing Solutions	www.philamixers.com
Rockwell Automation	www.rockwellautomation.com
Ross	www.mixers.com
Sharpe Mixers	www.sharpemixers.com

Bematek

FIGURE 5. Rotor/stator technology has some elements that rotate and some that are stationary to create mixing size reduction as product passes through. This is beneficial when it comes to clean up, saving time and labor

Reliability requirements

While flexible mixing equipment may help control costs, reliability still tops the list of requirements when seeking new equipment. "When it comes to mixing equipment, chemical processors say that finding reliable equipment is more important that finding the lowest-priced equipment, especially when it comes to rotating equipment, which takes more of a beating than static mixers," says Eric Janz, new product development and technology manager with Chemineer (Dayton, Ohio).

For this reason the company has developed Model 20 top-entering agitators with high-efficiency gearboxes designed specifically for agitator service (Figure 2). The agitators incorporate a modular design package that reduces the number of replacement parts that need to be carried in inventory by the customer. They offer quick and easy seal change capability, designed to save time and reduce maintenance costs; a standard cast dry well seal that eliminates lubrication oil leakage from the gearbox; and reliable performance due to the cast gearbox with heavy-duty output shaft and bearings.

Rotating equipment can also be trying on mechanical seals because there's a lot more shaft movement and lubrication is often a problem. To counter this issue, Ekato Group (Schopfheim, Germany) recently introduced high-pressure splitting devices that optimize high-pressure mechanical seals used in rotating equipment for the CPI. By using a pressure splitting device, it is possible to ensure a predefined pressure drop within the two seal chambers of a triple-acting mechanical seal (Figure 3).

The pressure from the vessel to the atmosphere decreases in steps, thus, reducing the pressure to which the individual seal-ring pairs are exposed. The pressure splitter automatically adjusts the pressure within the seal chambers, depending on the pressure in the vessel, by means of a hydraulic piston action. In this way, the pressure splitter extends the seal lifetime while offering relatively low investment costs.

Because the system does not require circulation pumps or control valves, it is not susceptible to faults in the power supply or measurement and control equipment. Using a mechanical seal with a pressure splitting device increases operational safety and is suitable for use in toxic or highly hazardous processes with high temperatures and pressures.

And, a new line of dry-running mechanical seals (Figure 4) for mixers, agitators and reactors has been developed by Sharpe Mixers (Seattle, Wash.) for applications where product contamination from barrier fluids is unacceptable and where reduced cleaning time is required. The seals feature a carbon blend that eliminates the face squeal of other mechanical seals and can be supplied with or without a debris well. The sloped internal surfaces facilitate drainage and enhance cleanability, reducing required maintenance time. ■

Joy LePree

ERIC ABBEY / *OWNER*
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CHEMICAL ENGINEERING

‘I do not believe in entrainment’

Mike Resetarits is the technical director at FRI (Stillwater Okla.; www.fri.org), a distillation research consortium. Each month, Mike shares his first-hand experience with *CE* readers

It was the Dr. James R. Fair Heritage Distillation Symposium at the Chicago AIChE meeting last month. I had decided to finally give a presentation about something that has been bugging me since October 28, 1974, my first day as a practicing chemical engineer. “Regarding distillation trays, I do not believe in entrainment. I believe that I am at least 50% correct.” I had the audience’s attention, indeed.

Dr. James Fair’s 1961 article entitled “How to Predict Sieve Tray Entrainment and Flooding,” talked about sieve tray entrainment, including measurement difficulties. That article included the following thought-provoking comment, “Calculated entrainment values are as good or better than measured ones.” It also included a graph showing liquid entrainment as a function of percent flood and flow parameter (FP). That graph strongly implied that the flooding of distillation trays with high FP systems, such as high-pressure distillation columns and air-water test columns, is virtually unrelated to entrainment.

In retrospect, Dr. Fair’s contention could almost be regarded as prophecy. During my 37 years of experience, I looked through distillation column windows thousands of times. More often than not, tray flooding was caused by excessive froth heights rather than entrainment. For predicting flooding in high-pressure, trayed distillation columns, a reliable froth-height correlation is a better tool than an entrainment correlation.

My disenchantment with the entrainment variable did not end there. Subsequent to about 1985, modelers of distillation tray efficiencies have shied away from entrainment and have tended instead toward jetting. Most distillation trays do not function with identical bubbles rising upward through a cross-flowing liquid. Instead, vapor dispersion usually begins with a cylindrical jet of vapor. At the ends of these jets, small and large bubbles are formed. At increasing flowrates, these jets sometimes reach the tops of the froths.

A tray model that includes the entrainment variable will invariably exhibit decreasing efficiencies at increasing rates. A model that includes jetting is also capable of predicting such. In fact, a model that includes jetting will extrapolate better to the spray regime and will extrapolate better to near-flood conditions.

On trays, entrainment surely plays a larger role in the spray regime than in the froth regime. This was demonstrated at the Chicago meeting via video footage from one of FRI’s test columns. Roughly speaking, in 1980, half of the columns of the world were operating in the spray regime. At that time, however, structured packing entered the marketplace and random packing experienced a renaissance just a few years later. As a result, the majority of vacuum columns now contain packing not trays. Therefore in 2011, more trayed columns are functioning in the froth regime than in the spray regime.

My Chicago presentation raised eyebrows and created at least one homework assignment. I now owe Lowell Pless, business development manager at Tracerco Ltd., froth height data — observed versus gamma scan, because he wondered why gamma scans of trays functioning in the froth regime (high FP’s) do not exhibit sharp froth heights.

Even with a 50% confidence level expressed in my opening statement, the audience’s attention had surely been captured. And, either way, I stand by the following belief 100%: More attention should be given to jetting. ■

Mike Resetarits (resetarits@fri.org)

CHEMICAL ENGINEERING
FACTS AT YOUR FINGERTIPS
Department Editor: Scott Jenkins

Hopper inserts for improved solids flow

Storing solid materials in hoppers, bins and silos is ubiquitous in the chemical process industries (CPI), as are challenges associated with dispensing them. In many cases, using vessels with steep cones and smooth walls is enough to provide reliable flow. However, it is not always feasible to design and install hoppers with the optimal geometry, and many solids have difficult or variable flow properties. In those cases, a hopper insert may improve flow.

Inserts can be defined as any static fitting mounted onto the inside of a bulk storage container to alter the internal space of the vessel. The performance of inserts depends on hopper geometry, feeder type (or discharge control), physical properties of the particulate solids and on ambient and operating conditions, so insert selection requires an overall systems approach and should be based on the measured values of bulk solid properties.

When to consider inserts

Despite seeming somewhat counterintuitive, inserting an obstacle in the flow path or using a wall surface with higher resistance to slip can provide a crucial difference that allows reliable flow by influencing the local forces experienced by individual particles within bulk solids. Common reasons for installing inserts include the following:

- Reduce segregation or particle attrition at the inlet region
- Reduce inertial compaction by inflow and minimize dust generation at inlet
- Promote or sustain gravity flow at outlet
- Secure flow through smaller outlets
- Increase flowrates
- Secure mass flow with less-steep wall inclinations
- Reduce segregation
- Blend contents on discharge
- Reduce compaction pressures and accelerate de-aeration inside hopper
- Counteract "caking" tendency

Insert effects

Gravity flow occurs when the bulk material is deformed to the shape of the flow channel by stresses generated from the loss of potential energy of the system. Energy is lost in the form of friction, either by sliding on the container walls or by internal friction when the flow-channel boundary is within a bed of static product. Bulk solid will not flow unless the stresses generated in the flow channel due to gravity are greater than the local yield strength of the material.

Inserts act to improve the probability of gravity flow in one of three ways: by minimizing the development of strength in the bulk material; modifying the flow channel to generate stresses adequate to deform the bulk; and applying external forces. For example, impact of the fill stream onto a previously deposited bed of material can cause compaction. An insert to slow, diffuse, or deflect the flow path, in order to prevent the impact load on the sensitive flow region immediately above the outlet point, will reduce the forces that compact the material.

Insert types

Flow-channel modifiers. Modifying solids' flow channels can make the shape of convergence more favorable for flow. A cone-in-cone insert (Figure 1A, 1B) creates two flow channels — a central portion where steep walls promote mass flow, and an outer region that is an annular, V-shaped flow channel in which material deforms easier than in a cone. An inverted cone insert (Figure 1C) changes the flow channel from a radial flow form into a type of annular V-shaped form that has greater deforming capacity. Bullet-type inserts (Figure 1D) form a two-stage flow channel in the same form. All can increase the rate of discharge, expand the flow path or induce mass flow.

Inverted V-beam. Fitting a cross of inverted V-beam at the transition point between the parallel and converging sections of a vessel (Figure 2) usually reduces overpressures, provided that the remaining flow areas are large enough to prevent arching.

Slip-resisting fittings. Another approach to achieving the same transference of vertical stress is using slip-resistant fittings that ring the inside of the vessel at various elevations (Figure 3).

Layers of coarse grids. For some materials that are sensitive to compaction, such as soft, elastic or fibrous materials, layers of coarse grids can help avoid undue compaction forces (Figure 4). With the correct grid openness and intergrid spacing, the vessel contents exert only a small pressure on the layer below, but the granules dribble through each grid.

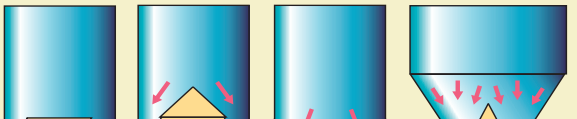
Inverted V-plates. In large hoppers storing pressure-sensitive granules, arrays of inverted V-shaped plates can be suspended from the roof of a silo or hopper. The flexibility of suspension avoids the formation of stable supports around a flow channel, as the insert will tend to move toward a region of differentially reduced pressure, as from a static bed to a live flow channel, thereby favoring flow to develop in the previously static region.

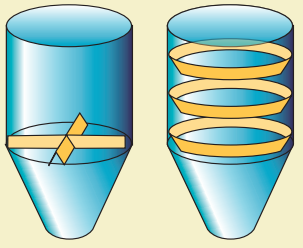
Tube inserts. The essential feature of tube inserts is that they weaken the circumferential stress in a conical hopper by redirecting the extraction of material from peripheral regions of the cross-section (Figure 5). Obstructing flow in the center favors flowdown from the shielded section under the insert, which allows the remaining cross-section to converge in a skewed manner.

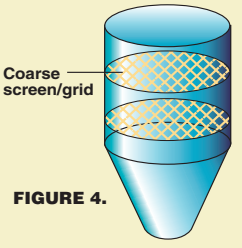
Wall liners. Liners with lower friction than the original walls provide a smoother transition from the vertical part of the hopper to the converging part.

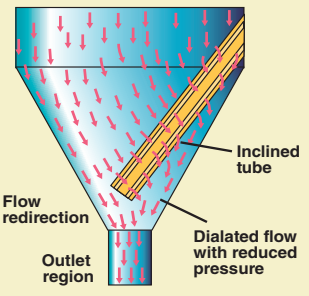
Inlet distributor. Diffusing the fill distribution of a hopper can reduce the effects of local concentration of the material (Figure 6).

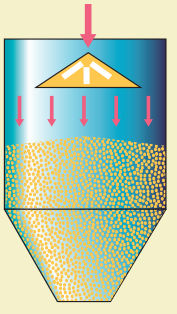
Vibrated inserts. A flat bar extended inward into the hopper from a wall-mounted vibrator that is tuned to resonate at the natural frequency of the applied vibration can transmit vibration to the sensitive flow regions. A rotary vibrator on a frame with hanging rods that vibrate at a natural frequency will accelerate de-aeration.











Material for this "Facts at your Fingertips" was adapted from the following article: Bates, L., Dhodapkar, S. and Klingzing, G., Using Inserts to Address Solids Flow Problems, *Chem. Eng.*, July 2010, pp. 32–37.


Liebelt


Bi


Watson


McLaughlin


Smylie

Uwe Liebelt becomes president of **BASF**'s Paper Chemicals Div. (Basel, Switzerland). The current president, *Ehrenfried Baumgartner*, is retiring.

Alchimer S.A. (Massy, France), a provider of nanometric deposition technology for semiconductor and electronic applications, names *Erik C. Smith* COO.

Jean-Marc Gilson becomes CEO of **Avantor Performance Materials** (Phillipsburg, N.J.; formerly Mallincrodt Baker), a manufacturer of high-performance materials for the microelectronics, photovoltaic, biotechnology, pharmaceutical and other industries.

Yunxia (Vivian) Bi is appointed technical director of the solubility initiative of **ISP Pharmaceuticals** (Wayne, N.J.), the company's portfolio of solubility-enhancing ingredients.

Mustang (Houston), a global engineering, project management, procurement and construction-operations company, appoints *Curt Watson* senior vice president of global business development and marketing.

Declan McLaughlin is named president and CEO of **CST Industries** (Lenexa, Kan.), a maker of factory-coated metal storage tanks, aluminum domes and specialty covers.

Jack Smylie becomes director of sales and marketing for **Magnetic Products, Inc.** (Highland, Mich.), a provider of magnetic and non-magnetic material-handling solutions. ■

Suzanne Shelley

PEMS: The Low-Cost Alternative To Emissions Monitoring

Real-world experience with installing and using both PEMS and CEMS at this methanol manufacturing facility is shared. The advantages of PEMS are many

FIGURE 1. This photo shows the methanol production facility of Arrazi in Saudi Arabia

Amer Khaqan
Saudi Methanol Co. (Arrazi)

Regulatory authorities around the world require continuous emissions monitoring of certain pollutants from large combustion sources. There are two main technologies for monitoring these emissions on a continuous basis — the more traditional one relies on sampling and analyzing exhaust gases from a continuous emissions monitoring system (CEMS); and the newer one relies on software that uses mathematical algorithms and equations to predict emissions levels from existing control-system data. This second system is called a predictive emissions monitoring system (PEMS).

In this article, practical experience and installation details of both CEMS and PEMS at the reformers and boilers of the Arrazi methanol manufacturing complex are presented. The real-world experience gained in about two-and-a-half years of operation is shared and can be used as a template to implement PEMS at any site.

THE MANUFACTURING SITE

The experiences described in this article are those of the Arrazi Saudi Methanol Co., an affiliate of Sabic (www.sabic.com), and the world's largest methanol manufacturing complex, located in Jubail, Saudi Arabia (Figure 1). Arrazi uses natural gas, both as fuel and as a raw material to produce grade AA methanol with a purity of more than 99.99%. The site has six reformers, ten boilers, two incinerators and two pre-heaters.

The environmental performance at Arrazi is regulated by the Royal Commission for Jubail and Yanbu (RCJY) and the Presidency for Meteorology & Environment (PME), which can be considered to be the equivalent to the U.S.'s Environmental Protection Agency (EPA). The Royal Commission Environmental Regulations (RCER) rely heavily on the EPA for guidelines. As per RCER, Arrazi is required to continuously monitor nitrogen oxides (NOx) emissions from combustion sources, namely reformers and boilers.

The reason why NOx is monitored on a continuous basis is a huge topic in itself and beyond the scope of this article. Here it will just be mentioned that NOx can cause or contribute to serious environmental problems (such as acid rain and smog), and regulatory authorities in almost all countries require around-the-clock monitoring of NOx from combustion sources.

CEMS VERSUS PEMS

As mentioned earlier, there are two basic ways to continuously monitor emissions: a hardware-based continuous emissions monitoring system (CEMS); and a software-based predictive emissions monitoring system (PEMS). The EPA and the RCJY approve both CEMS and PEMS.

CEMS

A CEMS consists of specific hardware — installed on combustion equipment stacks and in the field — that collects samples of exhaust gases and then analyses them to report the "real" emissions levels. A typical hardware-based CEMS consists of the following major parts: analyzer, sample handling system (which includes pumps, chillers, heated sample line and so on), flow-monitoring hardware in the stack, analyzer house, air conditioner, calibration gas cylinders and more. In addition to all of this, the CEMS also contains a data acquisition system (DAS), which stores the data gathered by the CEMS analyzer. A DAS is basically a computer running software that is specially designed for data acquisition and reporting.

PEMS

PEMS consists of software in a dedicated computer that collects data from the plant's existing control systems (for example a distributed control system; DCS) and uses mathematical algorithms and equations to "predict" emissions levels. The only piece of hardware that a PEMS requires is a dedicated computer. Details of how PEMS works can be found elsewhere, so here is just a short summary: Simply put, PEMS is a computer model that is capable of predicting the outcome of

FIGURE 2. PEMS was implemented on the boiler stacks at Arrazi, three of which are shown here

a "known" process (in other words if the input of a dynamic and live process is known). By using a sophisticated computer model, the dynamic and live output can be predicted fairly accurately. For emissions monitoring, this "known" process is combustion. (For more on PEMS, see The Maturation of a Technology: Predictive Emissions Monitoring, *Chem. Eng.*, July 2006, pp. 50–55.)

CEMS EXPERIENCE

In 2006, to meet the Royal Commission Environmental Regulations for continuous emissions monitoring, we installed traditional, extractive-type CEMS analyzers for NOx and CO on three of our reformers and one incinerator. This was Phase-1 of a CEMS installation at our site.

The hardware-based CEMS was supplied by a well-known German vendor, and was commissioned in 2007. Our experience with the CEMS installation and subsequent usage brought its strengths and weaknesses to light.

The biggest strength of the CEMS was its ability to report "actual" analyses, regardless of any upstream process changes (namely feed composition). If the analyzer was calibrated properly, those analytical results were guaranteed to be accurate to within ±2.5%.

The biggest weakness of the hardware-based CEMS turned out to be its low service factor and an average downtime of more than 40% during the initial months. The service factor improved over the next two years, but keeping the average cumulative service factor above 80% was a continuous struggle. (The regulations require an uptime of more than 95%.) With a dedicated, CEMS-analyzer-maintenance crew, the service factor could have been improved further. This reflects the simple logical conclusion that the more parts a system has, the more often it is prone to fail.

Furthermore, the running cost of extractive CEMS was high due to maintenance, manpower and energy requirements. Based on this experience, we knew that the total cost of operating the CEMS would grow higher as the number of CEMS installations at the site increased.

All of this inspired us to search for a more economical alternative for the CEMS. The answer to this quest came from EPA regulations, which discussed an alternative method to the CEMS. That alternative was the PEMS.

PEMS EXPERIENCE

The technology of PEMS has been around in one or other form since the 1980s. The first commercial installation was done in 1992 and was subsequently approved by the EPA in the same year. Since then, hundreds of PEMS have been installed around the world, and this technology has seen continuous growth and acceptance.

Installation

As our site approached the second phase of continuous-emissions-monitoring installations, our research led us to decide to install a PEMS instead of a hardware-based CEMS on our seven boilers (Figure 2).

FIGURE 3. One of the requirements of the PEMS was that it needed to be compatible with the DCS or PLC used for plant control and operation

Gaining acceptance. The chemical process industries (CPI) are typically cautious in embracing new technologies that have not been tried and tested for a long time — our site was no exception in this regard. We ran into some resistance at our plant about whether or not PEMS would work. Even after a couple of detailed presentations about PEMS, the skepticism didn't die completely. We knew that the technology had to be proven, just like in the old proverb "the proof of the pudding lies in eating".

The senior management of our company was convinced about the potential of PEMS and directed us to proceed with its installation at our site, hence we started the work on this project. We came across many vendors who could provide PEMS, and we wanted to be careful to choose the right vendor, since this was a new technology for us and the Gulf Cooperation Council (GCC) region as a whole.

Vendor selection criteria. The toughest part was to identify the vendor who was right for us. We thoroughly researched various existing PEMS installations worldwide, pinpointed the weaknesses that other PEMS had and then developed strict criteria for vendor selection. According to our guidelines, the PEMS to be selected needed to have the following characteristics:

- Installations in the U.S. that must be certified by the EPA
- At least five of the installations must each be certified as per 40CFR60 and 40CFR75
- There should be no recurring license fees for the PEMS and DAS software; and all licenses should be perpetual in nature
- The PEMS model should be open and fully configurable by the end

FIGURE 4. An onsite PEMS inspection and relative accuracy test audit (RATA) were conducted by regulatory agencies

FIGURE 5. This typical DAS reporting screen shows the status of all input parameters for one of the seven boilers. The green boxes at the bottom of the screen show that six of the seven boilers are running and one is shut down

user with no support required from the original vendor later on
- The PEMS model must not use any humidity sensors (these sensors are prone to drift and require frequent calibration)
- The PEMS must be able to perform accurately with a minimal amount of stack-testing data

The reason for developing these stringent guidelines was that we didn't want to be stuck with a PEMS that was vendor specific or proprietary, and we wanted a system that was very reliable and robust with no hidden costs. The above criteria ensured that we chose the best possible product. As a result of using these guidelines, we chose a statistical hybrid PEMS model provided by an American vendor.

The regulatory criteria of the EPA require an accuracy of 10%, whereas our chosen vendor promised an accuracy of 5–6%. After commissioning, our PEMS was giving accuracy in the range of 4–6%.

Implementation

For the PEMS to be successfully installed at the Arrazi site, the following were the mandatory pre-requisites:
- Compatibility with the plant's DCS or programmable logic control (PLC)
- The initiation of an analyzer maintenance program (if one did not already exist) at the site
- High speed (preferably > 500 kbps) internet access to the PEMS server for remote support from the vendor
- Availability of remote connection (such as a virtual private network; VPN) to the PEMS server

The project execution involved the following main steps:
1. Formation of a project team that included an environmental engineer, DCS control engineer, IT engineer, PEMS vendor specialist and DCS vendor specialist
2. Site survey by vendor to establish site-specific data
3. Installation of the PEMS server and connection to the open-connectivity (OPC) server. The DCS vendor supported this connection
4. Ensuring trouble free and reliable communication between the PEMS and the OPC server
5. Developing the PEMS model and selecting the input parameters for the PEMS model
6. Stacks testing to fine-tune the PEMS model. (We used a Horiba PG-250 for our stacks testing with a stand-by analyzer available all the time)
7. Pre-RATA (relative accuracy test audit) to verify the predictions of the PEMS model
8. RATA verification by a regulatory agency (Figure 4)
9. Report generation by the DAS (Figures 5 and 6)

The PEMS has been successfully operational at our site for about two-and-a-half years. It was certified and approved by the Royal Commission for Jubail and Yanbu.

Benefits

Once the project was completed and running for over a year, we realized the following main benefits by using a PEMS instead of a CEMS:
- Capital savings of more than 50%
- Almost all CEMS installations are hazardous locations requiring classified explosion-proof equipment (for example Class-1, Div-2) which increases the cost of the CEMS tremendously. By choosing a PEMS we avoided the use of any field-mounted hardware and saved a lot
- Operational cost savings of approximately 90%
- The air conditioning, heated sample line, analyzers, sample conditioning system and so on used in a CEMS are all energy intensive and increase operational costs. A PEMS uses only a computer for running, hence huge savings were also realized in this area
- Maintenance cost savings were approximately 90%
- With a complete absence of the hardware required for CEMS, there was no maintenance to be carried out for PEMS. Again it turned out to be an area of immense savings
- Manpower cost savings of approximately 90%

FREQUENTLY ASKED QUESTIONS ABOUT PEMS

During the implementation of the project and even now, the Arrazi team has received many inquiries about its PEMS installation. Some of the most commonly asked questions are shared here:

Q: Does the U.S. EPA approve PEMS?
A: Yes, PEMS is approved by the U.S. EPA.

Q: Can PEMS be used for every combustion application?
A: No, there will always be sources that can only have an extractive CEMS, such as incinerators burning hazardous waste of hugely varying composition.

Q: What is the accuracy of a PEMS?
A: Well-designed PEMS are accurate to within 5–6%. Their accuracy improves with time as more and more operating data are fed into the PEMS model, increasing accuracy to 2–3%.

Q: What are the limitations of a PEMS?
A: PEMS can only predict what it has been "trained" to do. It can't work outside of its "training" envelope. In other words, it can't accurately predict if you are operating outside of the operating parameters that were initially fed into the model.

Q: If PEMS is such a good technology, why isn't every site in the world replacing their CEMS with it?
A: CEMS has been around for over 30 years and so is well known. PEMS is a "relatively" new technology and is catching up fast. We expect to see it more once awareness about it increases.

FIGURE 6. The summary status of all crucial boiler parameters can be displayed on one page

- The hardware of a CEMS requires dedicated manpower and analyzer technicians. A PEMS requires no such dedicated support
- Uptime of more than 98%
- With no pumps, heated lines, air conditioners, sample conditioning system or analyzers, the PEMS had almost nothing that could go wrong
- Short project time. The whole PEMS project was completed in less than 6 months as opposed to over a year for the CEMS

Problems

During our almost two-and-a-half years of operation, we faced only the following two problems:

Problem 1: The PEMS server locked-up (hanging), requiring a reboot. This happened a few times during the project implementation and commissioning phase. Once the cause was addressed (remote VPN connection problem and configuration error), the problem never recurred. After the project execution was completed and handed over to us, we never experienced this problem, and the PEMS ran without a glitch thereafter.

Problem 2: There was communication failure between the PEMS server and the DCS/OPC server. Infrequently (approximately every two to three months), whenever our IT personnel uploaded any major updates to the OPC server (which required a reboot of the OPC server), the communication between the PEMS server and the OPC server failed. As a result, the PEMS server stopped reporting the emissions. This problem, though not caused by the failure of the PEMS server itself, still caused a loss of data-gathering capability until the OPC settings could be fixed. To address this, we have put procedural and software controls in place to ensure that whenever the OPC software is updated and the OPC server rebooted, the correct settings of the OPC server should be insured by IT to avoid the communications failure and subsequent data loss.

What we would have done differently. If given a chance to redo the whole project again, we would probably change only one thing: the DAS. The existing DAS we are using is not based on MS Windows. Almost all contemporary software packages are now Windows-based and hence offer many of the features and functionality that we take for granted. We had to work with our DAS supplier to have some functions specially provided for us, and this would have been easier to tailor with Windows-based software.

FUTURE OF PEMS

Our confidence in PEMS has found firm footing after almost two-and-a-half years of trouble-free operation. In contrast, the CEMS has been pretty demanding in terms of maintenance. We are already reviewing the proposals to replace the existing CEMS with a PEMS. ■

Edited by Dorothy Lozowski

Author

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Feature Report

Flexible Heat Exchanger Networks

When designing a network of integrated exchangers for optimized energy efficiency, don't tie your system to a fixed set of operating conditions

Seham A. EL-Temtamy and Eman M.Gabr
Egyptian Petroleum Research Institute (EPRI)

NOMENCLATURE

- A Heat transfer area
- B See Equation (6)
- C Controlled parameters (in the example, start temperatures are disturbed by D and all target temperatures are fixed, controlled C)
- CP Heat capacity flowrates
- CP_C Heat capacity flowrates for the cold stream
- CP_H Heat capacity flowrates for the hot stream
- D Disturbed parameters (in the example, start temperatures are disturbed by D and all target temperatures are fixed, controlled C)
- Q Exchanger heat load
- R See Equation (5)
- ΔT_{LM} Log mean temperature difference defined in Equation (4)
- T_i Internal temperature i
- ΔT_i Change in internal temperature T_i
- TS_i Supply temperature of stream i
- TT_i Target temperature of stream i
- U Overall heat transfer coefficient
- $\langle UA \rangle_j$ Contingency for exchanger j

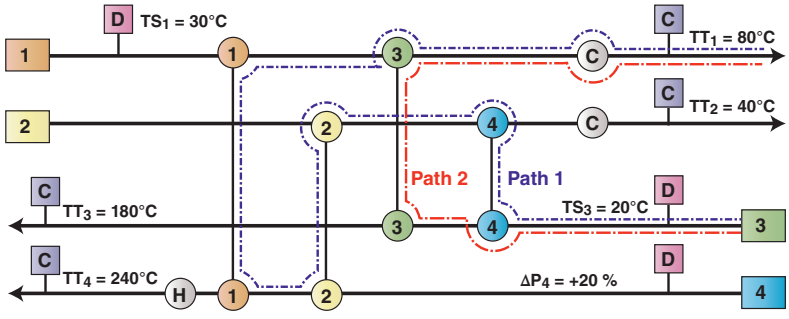

FIGURE 1. Heat exchanger networks should be designed for flexibility in operating parameters such as supply temperatures, heat capacity flowrates, and heat transfer coefficients, while maintaining controlled parameters such as target temperatures

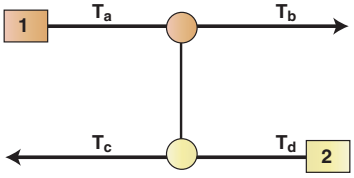

FIGURE 2. A single heat exchanger with hot Stream 1 and cold Stream 2

KEY FOR FIGURES

Stream numbers 1 2 3 4
Networked heat exchangers 1 2 3 4
Disturbed parameter D Cooler C
Controlled parameter C Heater H

The ever important aim of energy efficiency in the chemical process industries (CPI) is brought closer to its target by the practice of energy recovery in heat exchanger networks (HENs). When heat exchangers are designed to work together to exchange heat between hot and cold streams, the required utilities are effectively reduced. The HEN designer usually applies the assumptions of fixed operating parameters at nominal conditions for given specifications of a process. But practically, a HEN should remain operable under variations in operating conditions without losing stream temperature targets and, at the same time, maintain economically optimal energy integration.

This article illustrates how to use sensitivity tables to design an optimum, flexible HEN for a multi-period process that is feasible for N periods of operations. The sensitivity tables approach is developed, automated and illustrated on an example problem.

The approach is applied through two steps. The first one is developing a strategy for the choice of the HEN's base case, upon which sensitivity tables are generated. The second step is using sensitivity tables to reach target temperatures, and hence, target utilities of the alternative cases. In the end, we can have a HEN that realizes maximum energy savings under different operating conditions — in other words, a HEN with flexible and optimum properties.

Design of optimal, flexible HEN

The sensitivity tables approach. The use of sensitivity tables replaces rigorous simulation to study the passive response of internal and target network temperatures to changes in supply temperatures, heat capacity flowrates and effective values of heat transfer coefficients for the heat exchangers. Moreover, combined actions can be considered using sensitivity tables where tradeoff between energy, capital cost and flexibility can be established. The new mode of operation can be considered as a deviation from the base case, so it represents the disturbances in supply and target temperatures, and even heat capacity flowrates. Disturbances may occur for short or long periods where, certain parameters have to be fixed. These parameters are called controlled parameters (C) and disturbed parameters (D). The period for which the disturbances

HISTORY OF HEN SYNTHESIS AND SENSITIVITY TABLES

HEN synthesis is one of the most extensively studied problems in CPI process design, given the importance of determining the energy costs for a process and improving energy recovery. The first systematic method to consider targets for energy recovery was the thermodynamic approach of the pinch concept, introduced during the 1970s. Mathematical programming, stochastic optimization approaches and hybrid methods developed between the two have also been approached for optimal HEN design. Furman and Sahinidis [*1*] reported that over 400 papers have been published on the subject over the last 40 years. Gundersen and Naess [*2*] and Ježowski [*3, 4*] have also contributed thorough reviews on HEN synthesis.

Generally, HEN synthesis takes place under the assumptions of fixed operating parameters at nominal conditions for given specifications of a process. So, when operating conditions change, as they almost certainly do, stream temperature and energy efficiency targets can easily fall out of reach. Reviews of research into flexibility and operability can be found in Furman and Sahinidis [*1*]. Marselle and others [*5*] defined resilience for heat exchanger networks and stated other properties of resilience. They proposed a heuristic design method for structurally resilient networks with respect to inlet parameter variations. Swaney and Grossmann [*6*] introduced a flexibility index, which defines the maximum parameter range that can be achieved for a feasible operation. Grossmann and Floudas [*7–11*] introduced an active set strategy for the automated solution of the flexibility test and the flexibility index of Swaney and Grossmann [*6*] and introduced a systematic procedure for synthesizing flexible heat exchanger networks for multiperiod operation. It is assumed that, in general, different values are specified for the flowrates and inlet and outlet temperatures of the streams for *N* periods of operation. The objective is synthesizing a network that is feasible for the *N* periods of operation achieving minimum cost.

A systematic methodology in the design of HENs under multiple periods of operation is presented by Verheyen and Zhang [*12*]. The model presented a superstructure-based on mixed integer nonlinear programming (MINLP) model, which minimizes the total annualized cost containing heat exchanger area cost and utility costs. The model is based on the superstructure by Yee and Grossmann [*13, 14*], which was formulated for multiple periods by Aaltola [*15*].

Aguilera and Nasini [*17*] proposed a mixed-integer linear-programming (MILP) formulation for testing the flexibility of the HEN for flowrate variation, and later Aguilera and Nasini [*16*] introduced a flexibility test for the HEN with non-overlapping inlet temperature variations. Tantimuratha and others [*18*] proposed a screening and targeting process for the HEN design with flexibility consideration in both grassroots and retrofit cases. The screening stage is based on the screening models of Briones and Kokossis [*19–21*], and it considers both economic and flexibility aspects prior to network development. Konukman and others [*22*] introduced simultaneous flexibility targeting and the synthesis of the minimum utility HEN.

Kotjabasakis and Linnhoff [*23*] introduced "sensitivity tables", which simply correlate the response of the network temperatures to changes of input streams' temperatures or heat capacity flowrates and heat transfer rates (*UA*) of network exchangers. Sensitivity tables can be utilized to find the necessary corrections in order to make a nominal design sufficiently flexible, and for making decisions for the trade-offs between cost effectiveness and flexibility of the design. The aim is to design an HEN that is both optimized and flexible. This is the approach that has been automated and applied here for the purpose of designing a flexible HEN for multiperiod processes. ❑

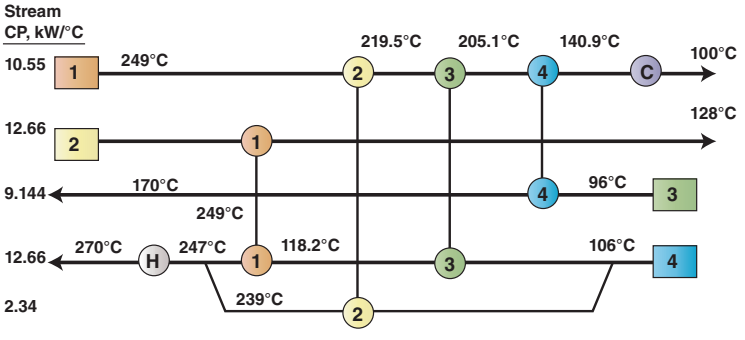

FIGURE 3. Generated heat exchangers network using PDM for Period 1

equal zero is called the "base case", such a case to be identified and presented on a grid diagram pointing out all the internal network temperatures before any analysis is performed.

Downstream paths. A path is an unbroken connection between any two points in the grid diagram (Figure 1). Propagation of a disturbance at point *D* on Stream 3 along the downstream path, Path 2, would affect the controlled target temperature of Stream 1 (TT_1). The propagation of the disturbance along the upstream path, Path1, would not influence TT_1. For corrective actions and to maintain a required *C* (in this case, TT_1), modifications of the heat exchangers by changing their effective *UA* will be considered as disturbances to counter balance the original disturbances. Consequently the exchanger having a downstream path into the controlled parameter is a candidate exchanger for contingency [*23*].

The sensitivity tables. The magnitude of the effect caused by *D* on *C* and the way to eliminate this effect are established by introducing the sensitivity tables approach [*23*]. The generation of sensitivity tables is based on the simple heat transfer equations of the heat exchanger. For a single heat exchanger shown in Figure 2, the heat balance equations are:

$$Q = CP_H\,(T_a - T_b) \quad (1)$$

$$= CP_C\,(T_d - T_c) \quad (2)$$

$$= UA\,(\Delta T_{LM}) \quad (3)$$

$$\Delta T_{LM} = \frac{(T_b - T_c) - (T_a - T_d)}{\ln\left[(T_b - T_c)/(T_a - T_d)\right]} \quad (4)$$

Where Q is the exchanger heat load, CP_H, CP_C are the hot and cold heat capacity flowrates for the hot and cold streams respectively, U is the overall heat transfer coefficient, A is the heat transfer area and ΔT_{LM} is the log mean temperature difference.

The following new variables (R and B) were introduced:

$$R = CP_C / CP_H \quad (5)$$

$$B = \text{Exp}\,[UA / CP_C\,(R-1)] \quad (6)$$

Using Equations (1–6) Kotjabasakis and Linnhoff [*23*] generated the following two equations:

$$(1-RB)T_b + (B-1)RT_c + (R-1)T_a = 0.0 \quad (7)$$

$$R(1-RB)T_d + (B-1)RT_a + (R-1)BRT_c = 0.0 \quad (8)$$

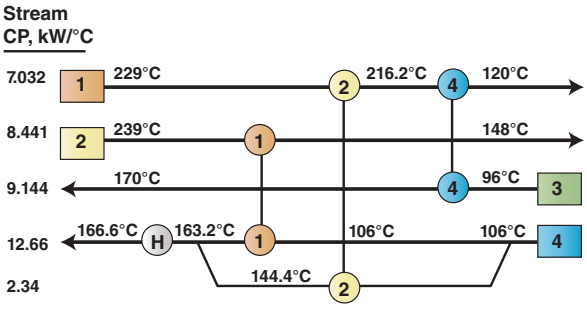

FIGURE 4. Generated heat exchangers network using PDM for Period 2

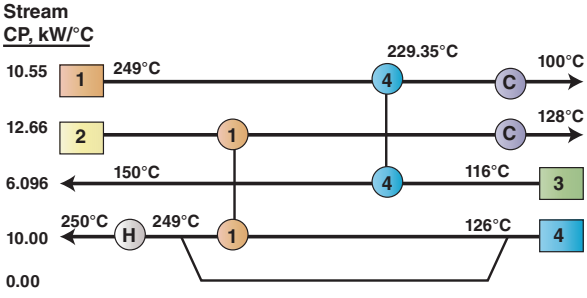

FIGURE 5. Generated heat exchangers network using PDM for Period 3

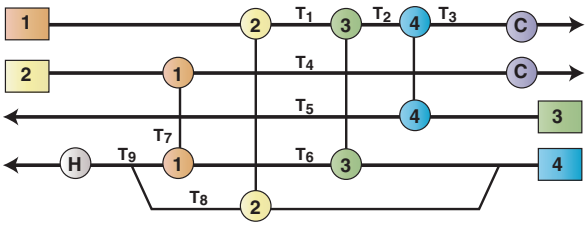

FIGURE 6. The base case network with internal temperature numbering system (T_1 through T_9)

TABLE 1. STREAM DATA FOR THE EXAMPLE PROBLEM

CP, kW / °C	TT_i, °C	T_{Si}, °C	Stream No.	Case No.
10.55	100	249	1	Base Case
12.66	128	259	2	
9.144	170	96	3	Period 1
15.00	270	106	4	
7.032	120	229	1	Period 2
8.44	148	239	2	
9.144	170	96	3	
15.00	270	106	4	
10.55	100	249	1	Period 3
12.66	128	259	2	
6.096	150	116	3	
10.00	250	126	4	

TABLE 2. MINIMUM UTILITIES REQUIREMENTS AND PINCH POINTS FOR THE EXAMPLE PROBLEM USING THE PDM METHOD

Period No.	Pinch point, °C	Minimum hot utility, kW	Minimum cold utility, kW
1	249 – 239	338.4	432.15
2	—	1,602.13	0.0
3	249 – 259	10.00	1,793.15

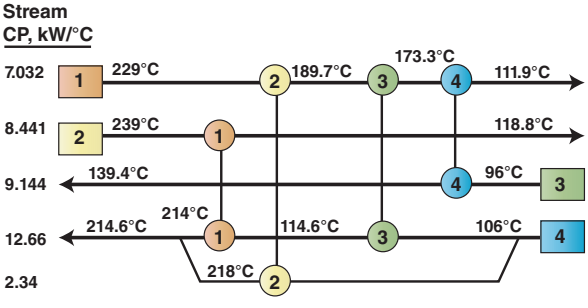

FIGURE 7. Network temperatures as a result of disturbance in Period 2, before corrections

Equations (7) and (8) are linear with respect to temperature and nonlinear with respect to both heat capacity flowrates and *UA*s. For the same heat exchanger in Figure 2, knowing two of the four temperatures (T_a, T_b, T_c, T_d) and knowing *UA*, CP_H and CP_C, the other two temperatures can be determined by solving Equations (7) and (8). This way of formulation is effective for each exchanger in any feasible network. Once the overall system of equations is solved for each exchanger in the base case, new network temperatures can be determined when changes occur in supply temperatures, heat capacity flowrates and effective *UA*s.

Constructing sensitivity tables. Three types of sensitivity tables can be constructed, where they simply correlate the response of the network temperatures to changes of input stream temperatures (T_S), heat capacity flowrates (*CP*) and the *UA* of the networked exchangers. The information needed are the base case stream data and the network structure. The different types of sensitivity tables are as follows:

1. T(T_S) sensitivity tables: These tables correlate the response of the network temperatures to unit variation of input stream temperatures. One table is enough to describe the responses to changes in all stream supply temperatures.
2. T(*CP*) sensitivity tables: These tables correlate the response of the network temperatures according to changes in heat capacity flowrate for a specific stream. Because Equations (7) and (8) are non linear with respect to *CP*, the *CP* sensitivity tables are constructed for different levels of *CP* variation in a specific stream. Interpolation between these values is possible.
3. T(*UA*) sensitivity tables: These tables need to be constructed for each individual heat exchanger in the network. These tables correlate the response of the network temperatures to changes of the effective exchanger (in terms of *UA*). Because Equations

TABLE 3. EXAMPLE T(T_S) SENSITIVITY TABLE (FOR EACH 1°C CHANGE IN T_S)

ΔT_i, °C	Stream 1	Stream 2	Stream 3	Stream 4
ΔT_1	.794	0.0	0.0	0.0
ΔT_2	.693	0.0	0.0	.127
ΔT_3	.283	0.0	.634	.098
ΔT_4	.077	.081	0.0	.841
ΔT_5	.469	0.0	.341	.106
ΔT_6	.094	0.0	0.0	.695
ΔT_7	.033	.38	0.0	.043
ΔT_8	.930	0.0	0.0	0.0

TABLE 4. THE T(CP) SENSITIVITY TABLE FOR THE EXAMPLE PROBLEM

ΔT_i, °C	ΔCP_1 −33.34 %	ΔCP_2 −33.34 %	ΔCP_3 −33.34 %	ΔCP_4 −21.01 %	ΔCP_5 −100 %
ΔT_1	−13.959	0.0	0.0	0.0	29.499
ΔT_2	−17.912	0.0	0.0	.19951	25.756
ΔT_3	−23.384	0.0	11.134	.128	10.656
ΔT_4	−1.667	−9.932	0.0	22.702	2.908
ΔT_5	−20.420	0.019	17.840	.1545	17.490
ΔT_6	−2.0051	0.199	0.0	2.782	2.920
ΔT_7	−.6772	−37.586	0.0	8.537	−.328
ΔT_8	−2.400	0.0	0.0	0.0	10

TABLE 5. T (UA) SENSITIVITY TABLE FOR EXCHANGER NO.1 OF THE EXAMPLE PROBLEM

	% Change in *UA* from the base case										
ΔT_i, °C	+ 100 %	+80 %	+60 %	+40 %	+20 %	0 %	−20%	−40 %	−60 %	−80 %	−100 %
ΔT_1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_4	−4.808	−4.25	−3.57	−2.7	−1.56	0.0	2.29	5.93	12.61	28.97	131
ΔT_5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_7	4.27	3.72	3.03	2.16	1.019	0.0	−2.83	−6.47	−13.15	−29.51	−131.6
ΔT_8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_9	4.07	3.601	3.023	2.288	1.324	0.0	−1.93	−4.993	−10.64	−24.44	−110.6

TABLE 6. T (UA) SENSITIVITY TABLE FOR EXCHANGER NO.2 OF THE EXAMPLE PROBLEM

	% Change in *UA* from the base case										
ΔT_i, °C	+ 100 %	+80 %	+60 %	+40 %	+20 %	0 %	−20%	−40 %	−60 %	−80 %	−100 %
ΔT_1	−2.026	−1.905	−1.709	−1.388	−0.863	0.0	1.433	3.842	7.979	15.366	29.5
ΔT_2	−1.769	−1.663	−1.492	−1.212	−0.754	0.0	1.252	3.354	6.967	13.416	25.76
ΔT_3	−0.683	−0.64	−0.569	−0.454	−0.265	0.0	.561	1.427	2.916	5.572	10.66
ΔT_4	−0.189	−0.177	−0.157	−0.126	−0.07	0.0	.151	.388	.794	1.52	2.91
ΔT_5	−1.181	−1.11	−0.993	−0.8033	−0.492	0.0	.868	2.295	4.746	9.12	17.49
ΔT_6	−0.413	−0.401	−0.38	−0.346	−0.290	0.0	−0.048	.2071	.645	1.426	2.92
ΔT_7	−0.564	−0.563	−0.562	−0.5596	−0.556	0.0	−0.538	−0.520	−0.489	−0.434	−0.328
ΔT_8	9.135	8.592	7.71	6.262	3.895	0.0	−6.460	−17.32	−35.97	−69.27	−133

(7) and (8) are non linear with respect to *UA*, the temperatures responses are evaluated at different values of *UA* percent of every effective heat exchanger of the base case network.

Using sensitivity tables. Once sensitivity tables have been constructed for the base case, the variation of any of the network temperatures can be calculated by simple summation of the variations resulting from individual disturbances (supply stream temperatures and stream heat-capacity flowrates). Sensitivity tables for *UA* can then be utilized for contingency-candidate exchangers in order to quantify the necessary change in exchangers' *UA* values that rectify the disturbances [*23*]. We assume that for each period, adjusting stream temperatures at the inlet of utilities can be used to nearly match target utility requirements determined via the pinch design method (PDM), which was developed by Linnhoff and his colleagues [*24*].

Conclusion

The sensitivity tables approach can be used to design optimal flexible HEN for multiperiods process. The tables are constructed by detecting the variations of the base case temperatures due to disturbances in streams supply temperatures, heat capacity flowrates and heat transfer coefficients (through *UA*s). Then, the sensitivity tables are used to adjust the streams' target temperatures to achieve target utility requirements as close as possible to PDM results for each period by adjusting candidate downstream heat exchangers' *UA* contingencies. Using PDM design at each period can evolve a flexible HEN design much easier than using sensitivity tables. However, sensitivity tables are more suitable to retrofit HEN to render it flexible for limited variations in process parameters.

Illustration of the approach

We have applied the sensitivity tables approach on a literature problem (Example 1 in Ref. 8, a process that has three modes of operation (defined as Periods 1 through 3). The conditions of the process changes periodically over the year. Each period differs from other periods in supply, target temperatures and in heat capacity flowrates. The problem data are given in Table 1.

Construction of sensitivity tables for the problem. In order to apply the sensitivity tables approach, we must define the base case for the HEN design upon which the sensitivity tables will be generated.

Utility targeting and selection of the HEN's base case. The PDM has been applied on the three periods of the process to locate the pinch points, minimum utilities consumption and minimum number of units and to establish matches for each period (Figures 3, 4 and 5). Table 2 displays minimum utilities consumption and pinch location for each period of the problem. The base case is chosen as the HEN containing the largest number of units (Period

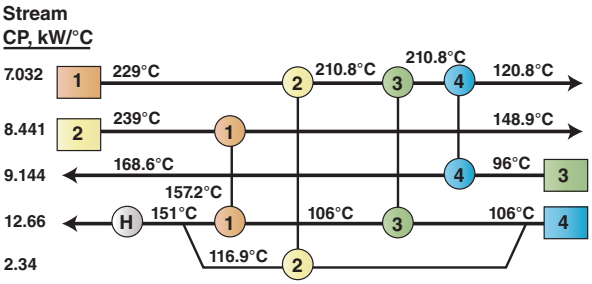

FIGURE 8. The final network temperatures resulting after performing *UA* contingencies in Period 2

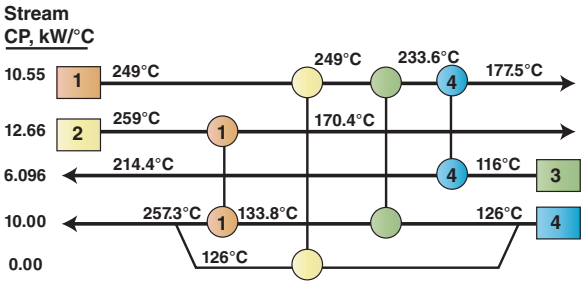

FIGURE 9. Network temperatures as a result of disturbances for Period 3

1). By inspection of the three periods' HEN, it can be concluded that almost all equipment required in Periods 2 and 3 can all be covered by matches in Period 1. Only the cooler of Stream 2 in Period 3 will be added to the HEN of Period 1. This will be the base case, and the temperature diagram of the problem is shown in Figure 6.

Generation of the sensitivity tables. Now, we have a HEN base-case design with four heat exchangers as shown in Figure 6. The internal network temperatures (T_i generically, or T_1 through T_9 in this case), heat exchanged and *UA* for each heat exchanger are calculated. Then, the variables R and B for each heat exchanger are determined using Equations (5) and (6). Then, Equations (7) and (8) are constructed for each heat exchanger (see Figure 2). Eight equations, two for each exchanger, were solved for the internal temperatures shown in Figure 6. A value for T_9 is obtained from T_7 and T_8.

We considered that all start temperatures are disturbed [D] and all target temperatures are fixed, controlled [C].

Keeping other conditions at the base case values, the equations have been solved for the following cases:

1. A positive unit change in the disturbed temperature, one at a time.
2. Variable percent change in *CP* from the base case for each stream.
3. Variable percent change in *UA* from the base case for each exchanger.

The sensitivity tables for the problem are listed in Tables 3–8. They represent the response of network temperatures resulting from the variation listed above.

Synthesizing an optimal flexible HEN for Period 2. The disturbances in Period 2 are due to variations in T_{S1}, T_{S2}, CP_1 and CP_2. The disturbance in the different network temperatures is calculated as follows:

$$\Delta T_i = \Delta T_i\langle T_{S1}\rangle + \Delta T_i\langle T_{S2}\rangle + \Delta T_i\langle CP_1\rangle + \Delta T_i\langle CP_2\rangle \quad (9)$$

Where

$\Delta T_i\langle T_{S1}\rangle$ is the change in T_i as a consequence of D in T_{S1}.

$\Delta T_i\langle T_{S2}\rangle$ is the change in T_i as a consequence of D in T_{S2}.

$\Delta T_i\langle CP_1\rangle$ is the change in T_i as a consequence of D in CP_1.

$\Delta T_i\langle CP_2\rangle$ is the change in T_i as a consequence of D in CP_2

ΔT_i is the change in T_i due to the combined disturbances.

By using the sensitivity tables (Tables 3, 4) and applying Equation (9) we can deduce the internal temperatures of the network as a result of combined disturbances for Period 2 (Figure 7).

Example calculation for ΔT_1: If T_{S1} in Period 2 is 20°C lower than in the base case, and the T_S sensitivity table (Table 3) shows that ΔT_1 is 0.794 for each 1°C temperature disturbance in T_{S1}, then:

$\Delta T_1 = (0.794)(-20) + (0.0)(-20) + (-13.95) + (0.0) = -29.83$°C

By comparing Figures 4 and 7, we find variations in target temperatures due to the combined effect of variations in target temperatures from the base case and Period 2 (Table 1) and the disturbances in the streams' inlet temperatures and heat capacity flowrates. The target temperatures in Figure 7 need to be adjusted to those shown in Figure 4 by an increase or decrease in each heat exchanger's *UA*, but at the same time utilities need to be kept as close as possible to the target minimum values predicted by PDM. By using the downstream paths,

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TABLE 7. T (UA) SENSITIVITY TABLE FOR EXCHANGER NO.3 OF THE EXAMPLE PROBLEM											
	% Change in *UA* from the base case										
ΔT_i, °C	+ 100 %	+80 %	+60 %	+40 %	+20 %	0 %	–20%	–40 %	–60 %	–80 %	–100 %
ΔT_1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_2	–11.4	–9.31	–7.14	–4.86	–2.49	0.0	2.61	5.34	8.21	11.22	14.4
ΔT_3	–4.65	–3.79	–2.89	–1.96	–0.98	0.0	1.12	2.24	3.43	4.67	5.98
ΔT_4	8.83	7.22	5.54	3.78	1.94	0.0	–2.01	–4.12	–6.34	–8.67	–11.14
ΔT_5	–7.71	–6.3	–4.82	–3.28	–1.67	0.0	1.79	3.64	5.59	7.63	9.79
ΔT_6	9.3	7.56	5.75	3.85	1.87	0.0	–2.37	–4.65	–7.04	–9.55	–12.2
ΔT_7	0.12	0.001	–0.13	–0.26	–0.40	0.0	–0.70	–0.86	–1.03	–1.21	–1.4
ΔT_8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

TABLE 8. T (UA) SENSITIVITY TABLE FOR EXCHANGER NO.4 OF THE EXAMPLE PROBLEM											
	% Change in *UA* from the base case										
ΔT_i, °C	+ 100 %	+80 %	+60 %	+40 %	+20 %	0 %	–20%	–40 %	–60 %	–80 %	–100 %
ΔT_1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_3	–14.1	–12.24	–10.03	–7.36	–4.08	0.0	5.39	12.57	22.71	38.1	64.2
ΔT_4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_5	16.3	14.19	11.64	8.57	4.78	0.0	–6.15	–14.43	–26.13	–43.9	–74
ΔT_6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ΔT_8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

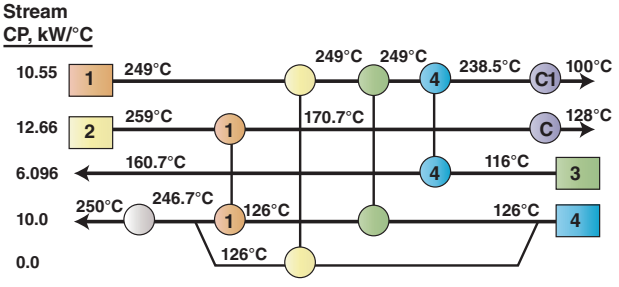

FIGURE 10. The final network temperatures as a result of *UA* contingencies for Period 3

TABLE 9. COMPARISON OF UA CONTINGENCES BETWEEN SENSITIVITY TABLE AND PDM FOR PERIOD 2 AND PERIOD 3				
	Period 2		Period 3	
UA_i	Sensitivity table	PDM	Sensitivity table	PDM
UA_1	–87%	– 92 %	–61 %	–63 %
UA_2	–90 %	–87 %	–100 %	–100%
UA_3	–100 %	–100 %	–100%	–100 %
UA_4	+27 %	+ 17 %	–92 %	–88%

we can detect the candidate heat exchangers for adjusting each *UA*. Using the *UA* sensitivity tables (Tables 5–8) and after several trials, the contingency for each exchanger's *UA* ($\langle UA_j \rangle$ for j=1 to 4) was found as follows: $\langle UA_1 \rangle = -87\%$, $\langle UA_2 \rangle = -90\%$, $\langle UA_3 \rangle = -100\%$, $\langle UA_4 \rangle = +27\%$

The final network with corrected internal and target temperatures is generated using the adjusted heat exchangers' *UA* through Equations (10) and (11), where the final disturbance in network temperatures is the summation of individual disturbances due to contingencies in the exchangers' *UA*s:

$$\Delta T_{i(overall)} = \sum_{j=1}^{n} \Delta T_i \langle UA_j \rangle \qquad (10)$$

Where $\Delta T_i \langle UA \rangle_j$ is the disturbance in network temperature resulting from using *UA* contingency in exchanger j. In this case, the number of exchangers (n) is 4.

$$T_{i\,(new)} = T_{i\,(disturbed)} + \Delta T_i \qquad (11)$$

Where:

ΔT_i is the final disturbance in internal network temperature resulting from using *UA* contingences.

$T_{i\,(new)}$ is the new (or final) calculated internal network temperature.

$T_{i\,(disturbed)}$ is the old internal temperatures of the network as a result of the change in operating conditions.

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The final network for Period 2 that is both flexible and optimized is shown in Figure 8. It can be seen that streams' temperatures before utility use are very close to those of Figure 4.

Synthesizing an optimal, flexible HEN for Period 3. The steps applied to reach to a flexible, optimum HEN design for Period 2, can be applied again for Period 3. Sensitivity tables 3–8 are used to detect and correct disturbances of network, due to changes in supply temperatures, capacity flowrates and target temperatures. In this case since Stream 5, a branch of Stream 4, has been canceled (CP_5 = 0.0), then automatically Exchanger 2 is deleted. Only the disturbance resulting from Stream 5 cancellation is taken into consideration when calculating the overall disturbances' effect. HEN for Period 3 with disturbed temperatures is shown in Figure 9. The T (*UA*) tables are used to establish the exchangers *UA* contingencies to adjust the target temperatures of network. The best achieved results are as follows: <UA1> = –56 %, <UA2> = –100 %, <UA3> = –100%, <UA4> = –93 %.

The final Network for Period 3 is shown in Figure 10. In this case we were not able to achieve the required target temperatures using sensitivity tables; a deviation of 2.3–10°C from PDM results was noticed (Figure 5).

Comparison with PDM Design

It is interesting to find out whether PDM design can offer any insight to the flexibility problem. Indeed it does, as we mentioned before the matches for the three periods of operation are more or less identical, with one or more exchangers to be bypassed and the others to be adjusted for their *UA*s. Table 9 displays a comparison between *UA* contingencies as calculated from PDM and sensitivity tables for Periods 2 and 3 respectively. It is clear that there is fair agreement between the contingencies' values calculated by both methods. ■

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DISTILLATION BACK MIXING: Impact on Batch Yields

Steven D. Kohler,
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Renessenz LLC

Several case histories illustrate the benefits of identifying and correcting poorly designed reflux and product-handling systems

Manufacturers of specialty chemicals are routinely required to reduce costs and increase the production of their high-value products. Specifically, competitive pressures from the developing world, increases in raw material costs and volatility in energy costs are putting significant pressure on manufacturers to minimize processing costs and maximize product yields. The economic impact of optimization efforts can be magnified for temperature-sensitive products due to a decrease in their overall recovery rate with an increase in batch-distillation cycle time.

Process-improvement activities related to existing batch distillation columns have traditionally focused on maximizing the separation efficiency, debottlenecking operations, and developing optimal batch distillation strategies. This is typically accomplished by retrofitting equipment (such as column internals, heat exchangers and vacuum systems), following better operating strategies, and improving existing control systems (for instance, using batch-sequencing systems and procedures). In the process analysis phase, less attention is devoted to identifying and correcting poorly designed reflux and product-handling systems.

This article describes two different "back mixing" scenarios in the product-handling system and discusses their respective impact on batch yields and overall cycle time. Simple but effective solutions that have been implemented to solve the problems are presented for both cases.

In the specialty and fine chemicals sectors, batch distillation is widely used for purification of a variety of small-volume and high-value products. The multi-purpose batch-separation process still brings with it the operational flexibility that is needed to distill a variety of products. The flexibility of the production arrangements can also tolerate fluctuations or rapid changes in demand [*1*]. However, the operational flexibility afforded by this approach often requires additional energy and results in reduced yields compared to continuous distillation systems that are designed for a specific product.

TABLE 1. ABBREVIATED SEQUENCE FOR A HYPOTHETICAL BATCH DISTILLATION

Charge: 12,000 kg		
Operation step	**Process variable**	**Cut condition**
Lights removal	1. Set top column pressure: 50 mm Hg 2. Take-off rate: 500 kg/h 3. Lights cut to Tank #1	> 50% of desired component in distillate
Transitional removal	1. Distillate rate: Ramp to 250 kg/h in 1 hr 2. Transitional cut to Tank #2	> 99% of desired component in distillate
Heart cut removal	1. Distillate rate: Ramp to 1,200 kg/h in 1 h 2. Product cut to Tank #3	< 1% of heavy component in distillate

We define "back-mixing" as mixing between the column distillates at separate times due to the presence of material hold-up in an external vessel or material-handling system. The previously distilled material that is part of this material hold-up may come from the current or a previous batch distillation.

This phenomena is not considered to be an issue in continuous distillaton due to the steady-state nature of that unit operation. However, the inherently dynamic nature of batch distillation demands the consideration of back mixing as the distillate composition changes during the course of the batch. In addition to lowering yield and increasing recycle processing, back mixing also wastes the energy used in the distillation.

Common equipment-related causes of back mixing include the use of an oversized reflux-accumulation drum, but can be as simple as the use of a pipe with excessive inner diameter. Batch distillation under vacuum conditions (batch vacuum distillation) is especially prone to back mixing as the distillate-handling system must also function as a vacuum break between the column and product storage equipment.

Discussed below are the process for recognizing sources of back mixing, two specific cases, and three solutions for both cases. Actual production improvements that resulted from the implementation of two of the featured solutions are also discussed.

Problem identification

A comprehensive analysis of any batch distillation unit will identify locations for back mixing to occur. Any equipment that contacts the overhead distillate, condensed reflux or condensed

TABLE 2. MODEL 1 — BATCH DISTILLATION WITHOUT A REFLUX DRUM

Cut results	Lights	Transi-tional	Heartcut	Column, pot and reflux drum
Total holdup in kg	3,323.3	2,620.7	5,315.0	786.0
Composition in cut				
Desired component	3.31%	83.56%	99.97%	23.76%
Light component	72.07%	0.19%	0.00%	0.00%
Intermediate component	23.27%	16.25%	0.02%	0.00%
Heavy component	1.35%	0.00%	0.01%	76.24%

TABLE 3. MODEL 2 — BATCH DISTILLATION WITH REFLUX DRUM

Cut results	Lights	Transitional	Heartcut	Column, pot and reflux drum
Total holdup in kg	3,375.0	3,870.7	3,604.9	1,194.3
Composition in cut				
Desired component	6.24%	87.59%	99.98%	49.80%
Light component	70.22%	0.78%	0.00%	0.00%
Intermediate component	22.20%	11.63%	0.01%	0.00%
Heavy component	1.33%	0.00%	0.01%	50.20%

take-off is suspect. Proper design and operation will minimize the effect of back mixing on column performance.

The most obvious source of back mixing occurs with reflux drums. Reflux drums are commonly used in continuous distillation to provide surge capacity for the condensed reflux. When used in batch distillation, the residence time for reflux in the drum is critical to minimizing back mixing.

An often-overlooked source of back mixing is product piping. Often, the product piping is sized to manage the maximum product flow. However, the typical product flow can be much less than the maximum flow. In extreme cases, the oversized product piping can act as a poorly mixed tank. This can be especially true in batch vacuum distillation. In some applications, the product line serves as a vacuum break between the product tank and column. As a general rule, all line pockets should be avoided.

Even external condensers and product coolers can provide a location for back mixing. Special attention should be given to the installation of horizontal heat exchangers so that the application is self-draining. Condenser hold-up between batches provides a source of heavy components for back mixing.

Case studies

Two similar but specific case studies were examined for back mixing. The first involved the use of a reflux drum in batch distillation. The second case investigates back mixing resulting from a large-diameter product line.

Case 1: Back mixing due to a reflux drum. Reflux drums (overhead accumulators) are commonly used in continuous distillation columns for providing surge volumes, facilitating control of product or reflux streams, and providing settling time when separating two liquid phases (Figure 1). For a continuous distillation column at steady state, the composition of the condensed vapor stream from the column (and consequently, the holdup in the reflux drum) does not vary significantly and has no significant impact on the composition of the distillate and reflux streams. Batch distillation is, however, an inherently unsteady-state process with respect to compositions of the overhead condensate stream with time. Any back mixing in the reflux drum will have an impact on the compositions of the distillate and reflux streams. Back mixing results in reduced product yields and depressed overall batch cycle times. This is particularly true for "tough" separations (that is, those with low relative volatilities) and separations where the product specifications are very "tight" (for instance, those requiring high product purity or specific isomer ratios).

A batch distillation of a hypothetical four-component system is modeled using commercially available batch-distillation simulation software. The sequence of steps in the batch distillation (after total reflux) is outlined in Table 1.

Two batch-distillation models were developed — one with no reflux drum (an ideal case; shown in Table 2) and one with a reflux drum (Table 3). This example was developed to illustrate the effect of significant holdup in the reflux drum. The components in the system — light, intermediate, desired and heavy — are listed in order of their volatilities. Three distinct cuts are taken in the following order: Lights, transitional cuts (that is, a cut containing lesser amounts of the desired component than the heartcut but less than the lights), and the heartcut (the target product). At the conclusion of the heartcut, the contents of the pot, column and reflux drum are blended.

The hypothetical column modeled is assumed to have 20 separation stages. The level in the reflux drum was maintained at 50% by allowing the reflux flow to float (level control) and the product flow (flow control), was set by the operating steps. The net amount of holdup in the reflux drum was estimated by the model to be 3.5% of the total charge. The same heat input (and condenser duty) was fixed for both models for the entire length of operation. The top column pressure was maintained at 50 mm Hg, and the column differential pressure was estimated by the model based on the packing information provided by the vendor as an input.

The results of this modeling exercise indicate that a cycle-time reduction of 4 h (21 h versus 25 h) and a product yield increase of 14% (5,300 kg versus 3,300 kg) can be realized for a batch distillation with no overhead material hold-up. This is in addition to a reduction in amount of recycle cuts, energy and reduced loss of product.

From a thermodynamic standpoint, the energy that is used to separate two components in the column is wasted by the act of mixing them together in the reflux drum. The lighter components that are separated from the heavier components are subsequently refluxed back to the column (in addition to being take-off in the product stream). This results in lengthened batch cycle times, lowered yields and increased recycles.

Case 2: Back mixing due to take-off piping. Back mixing due to take-off piping is more of a problem in

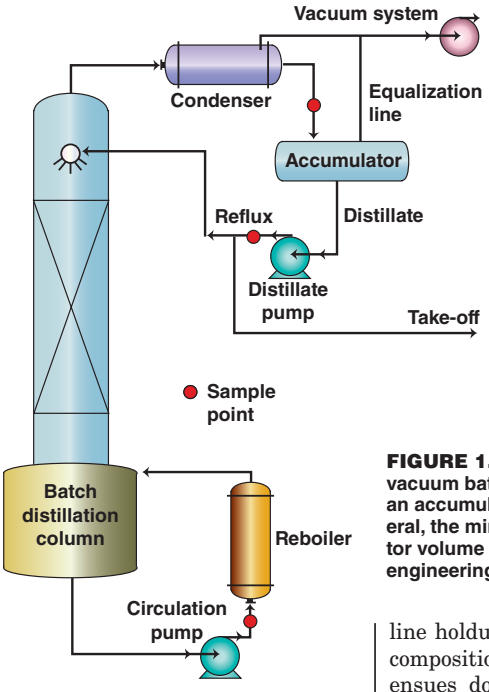

FIGURE 1. Shown here is a typical vacuum batch distillation system using an accumulator (reflux drum). In general, the minimization of the accumulator volume is considered to be good engineering practice

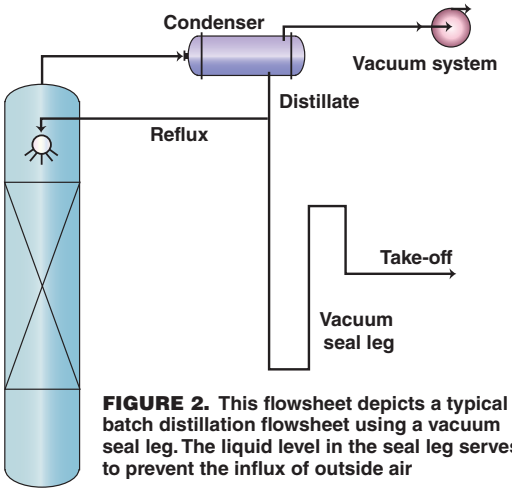

FIGURE 2. This flowsheet depicts a typical batch distillation flowsheet using a vacuum seal leg. The liquid level in the seal leg serves to prevent the influx of outside air

batch vacuum distillations. The piping used for the column product and take-off stream serves as the vacuum break between the column and the product storage equipment (such as tanks, totes and drums; Figure 2). The height of the column provides the motive force to transfer the column take-off to storage. This strategy typically eliminates the reflux drum and product pump.

To act as a vacuum break, the barometric leg must be full of liquid. However, when this occurs, the barometric leg may act as a poorly mixed, high-length/diameter tank. In extreme cases, this barometric leg may contain up to 5% of the batch charge. This line holdup does not affect the reflux composition, but the back mixing that ensues does impact the composition of the take-off stream. Diffusion and pipe vibration help to disturb the composition profile in the line so that the take-off stream is not truly plug flow.

In this case, a simpler binary system where the desired component has the lower volatility is considered. The take-off piping and associated equipment have a volume equivalent to 2% of the batch charge. Initially, the pipe is completely full of material at a mass fraction of 0.95 of the desired component. At time t=0, material enters the pipe at a constant rate with a mass fraction of 0.995 of the desired component. For this case, the time constant of the piping (vessel) is 0.7 h. Assuming that the piping is a well-mixed vessel, the following material balance is written around the mixing cell:

Accumulation = In – Out + Generation – Consumption

Solving the differential equation using the initial condition yields the following equation:

$$t=\frac{1}{\tau}\left[ln\left(\mathrm{m}\left(X_{Inlet}-X_0\right)\right)-ln\left(\mathrm{m}\left(X_{Inlet}-X_t\right)\right)\right] \quad (1)$$

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where:

t = time, h

τ = piping (vessel) time constant, h

m = flowrate of the take-off stream, mass / h

X_{inlet} = mass fraction of the desired component of material entering the take-off piping, 0.995

X_o = initial mass fraction of the desired component in the take-off piping, 0.95

X_t = mass fraction at the pipe exit

Evaluating the expression at t=2 h, the mass fraction of the take-off stream measured at the pipe exit is 0.984. It takes over three hours for the purity to reach 99% at the pipe exit. Depending on the product purity specifications, this lost production time lowers the product yield and increases the cycle time.

There is an additional incremental cost if the low-purity production is recycled to the batch distillation. The lower first pass yield requires the running of additional recycle batches. Typically, recycle batches have lower yields and longer cycle times than the first pass batches.

Proposed solutions

With high-value specialty chemicals purified by batch distillation, the ability to eliminate back mixing is worthwhile and, at times, even critical for profitability. There are many ways to eliminate back mixing; however, all methods have a common theme — the hold-up volume in the take-off system must be reduced or greatly eliminated.

In the first case, the obvious solution is to operate the reflux drum at the minimum safe level. Other options considered were the installation of a smaller reflux drum, or even reducing the volume of the existing drum. In addition to operating the drum at a lower level, we lowered the reflux ratio during the transitional cut. The resulting back-mixing reduction had a greater effect on yield and cycle time than the lower reflux ratio. This change, along with others, decreased the cycle time by 8–14 h and resulted in a 15% increase in product yield.

Our solution to Case 2 required the installation of a transfer pump to move the take-off to the associated tank. The pump substitutes for the barometric leg to act as the vacuum break. This installation greatly lowered the amount of product piping hold-up. A very small amount of hold-up is now needed to satisfy the net positive suction head required (NPSHr) of the product pump. This change increased the product yield by more than 3%. ■

Edited by Suzanne Shelley

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Designing SAFER Process Plants

Several often-overlooked strategies to increase inherent safety are discussed here

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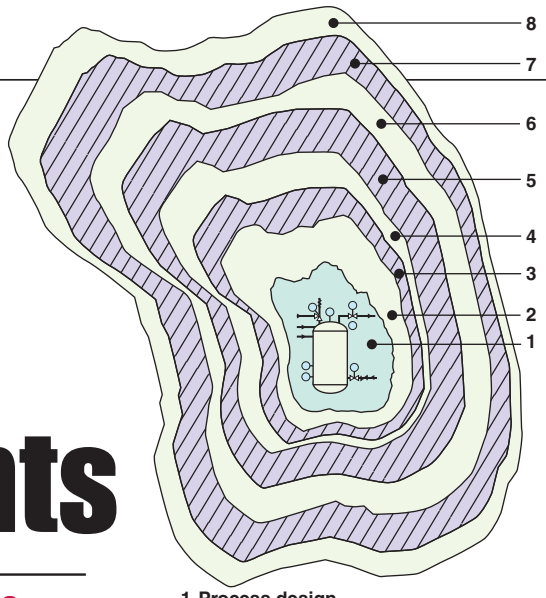

1. Process design
2. Basic controls, process alarms and operator supervision
3. Critical alarms, operator supervision and manual intervention
4. Automatic action safety-instrumented systems (SIS) or ESD
5. Physical protection (relief devices)
6. Physical protection (dikes)
7. Plant emergency response
8. Community emergency response

FIGURE 1. Shown here are some typical layers of protection that can be employed in a modern process plant [*4*]. At the core is an inherently safe process design. Moving outward from the core, the proposed options move through the spectrum from inherent to passive to active to procedural or administrative controls, which are considered to be progressively less reliable

Many individuals and organizations have made important contributions to the creation of inherently safer (IS) products, processes and process plants [*1–3*]. A brief survey of successful case histories shows that most reported applications relied on only a few of the core IS principles. This paper emphasizes the opportunities presented by three particular — and often-overlooked — possibilities for inherently safer processes.

The methods proposed here ensure integration of IS methods beginning with process conception and continuing through process plant engineering design. Particular emphasis is given to matching the IS principles with the state of the project. For example, substitution is best applied during product and process research, while limitation of effects is most effective during plot plan layout and equipment arrangement.

The chemical process industries (CPI) face the challenge of working with processes and products that present many hazards, such as the following:

- The manufacture of fuels uses and produces products that burn with significant energy release
- Certain basic chemicals, such as mineral acids and halogens are toxic and/or corrosive
- Many manufacturing processes either release or require significant energy transfer to achieve chemical transformation
- Some manufacturing processes produce benign products but require hazardous chemical intermediates in their manufacture

For these reasons, rigorous process and product safety practices must be used throughout the lifecycle of process plants and must be applied to their associated raw materials and products. In recent years, this has led to major efforts in green chemistry and engineering to develop products, manufacturing processes, and plants that are safer for both people and the environment.

Before green chemistry and engineering achieved prominence, there were pioneering insights in the design of safer process plants. Early approaches to safer processes often employed additional instrumentation and procedures. These measures were often helpful and necessary, but instrumentation and operators can fail, especially when faced with complexity.

Trevor Kletz [*1*] recognized that "What you don't have can't leak", when he first proposed the concept of the inherently safer chemical processes in 1977. His approach placed an emphasis on the inherent nature of the process. Since then, important related concepts such as product design for safety and safer products, process and plant lifecycles have also advanced.

Creation of IS processes has been the objectives of a number of creative individuals and organizations since Kletz's path finding proposal, with many notable successes.

Complete coverage of the entire product/process/plant lifecycle is needed to assure optimum health, safety and environmental performance of a chemical enterprise.

This article focuses on how to ensure maximum incorporation of IS processes into the creation of a process plant by beginning at the product and process research stages and concluding with the detailed design. No effort is made to address the application of inherently safer principles beyond plant design, although these are also important.

Layers of protection

The classical onion diagram (Figure 1) illustrates the safety layers that technical professionals throughout

FIGURE 2. The Flixborough tragedy ushered in a new era in process safety [6]

the CPI use to prevent process plant incidents. This diagram helps to explain the following four basic process risk-management strategies: Inherent, passive, active, and procedural or administrative

Inherent safety is at the core of the onion — the process design. A process that cannot have a major fire, explosion or toxic release is inherently safer than one that could if one or more layers of protection were to fail.

Passive safety layers represent the addition of such safety features as a dike or a blast wall. Because passive layers of protection require no active intervention by a human or by a machine, they are deemed more reliable than active layers of protection or procedural layers of protection. Nonetheless, the ability to make an explosion impossible — when possible — is clearly better than trying to mitigate the effects of a potential explosion by adding a blast wall.

Active layers of protection represent such features as the basic process control system, a safety-instrumented system, and mechanical interlocks.

Procedural or administrative safety layers are generally considered to be the least reliable and include operating procedures and operator intervention. Depending on the site-specific hazard, procedural or administrative controls may be entirely appropriate.

In general, the preferred ranking of methods to control process risks is shown below:

Inherent > passive > active > procedural or administrative

Basic concepts

Inherently safer process concepts are summarized below [*1*]:

- Substitution
- Minimization or intensification
- Moderation or attenuation
- Simplification
- Limitation of (hazardous) effects
- Avoiding knock-on effects
- Making incorrect assembly impossible
- Make status clear
- Tolerance of error
- Ease of control
- Administrative controls or procedures

In 2007, the Center for Chemical Process Safety (CCPS) of the American Institute of Chemical Engineers (AIChE) concluded that these eleven basic concepts could be reduced to the following four principles [*2*]:

- *Minimize*
- *Substitute*
- *Moderate*
- *Moderate and simplify*

This more concise set of principles makes IS practices simpler to understand and easier to apply. The excellent new CCPS book (2009) goes on to distinguish between first-order and second-order IS:

- First-order IS efforts change the chemistry of a process
- Second-order IS efforts change the process variables

As can be seen by a survey of the process safety literature, most published work has applied one or more of the first four concepts of the eleven cited by Kletz and Amyotte [*1*] For this reason, this article emphasizes three other promising concepts.

Often-overlooked IS concepts

Three underutilized IS concepts are presented here and illustrated with examples:

1. Hybridization or transformation. One relatively new IS concept is based on the recent innovative work by Chen [*5*] who reports an inherently safer process for the partial oxidation of cyclohexane. Partial oxidation processes often involve hazardous conditions, as illustrated by the Flixborough, England, tragedy in 1974 — which killed 28 people, destroyed a plant, led to new process safety regulations, and inspired Trevor Kletz to propose his inherently safer design concept. The Flixborough plant carried out liquid-phase oxidation of large inventories of hot cyclohexane in large pressurized vessels. When containment was lost, a large flammable vapor cloud formed, ignited, and exploded with devastating effect (Figure 2, from Mannan [*6*]).

The traditional cyclohexane-oxidation process to produce a mixture of cyclohexanone and cyclohexanol (K/A oil or ketone/alcohol oil) was operated at low conversion rates (typically 3–5%) to avoid formation of unwanted byproducts. The K/A oil was subsequently converted into adipic acid and caprolactam for the production of nylon.

Oxidation of cyclohexane with air instead of oxygen is common practice to reduce risks of transition from a partial oxidation reaction to an uncontrolled deflagration in bubbles or in the vapor space in the reactor. Low conversions and reaction rates led to large inventories of liquid cyclohexane.

During systematic research on the flammability and deflagration hazards of cyclohexane, air and oxygen mixtures, Chen [*5*] discovered that the addition of a small amount of water — which is inert and does not participate in the reaction — helped to inert the otherwise flammable vapors. Cyclohexane and water are known to form minimum-boiling azeotropes. The increase in the vapor pressure of the cyclohexane/water liquid results from the increased vapor pressure of the water. The water vapor inerts the vapor mixture by lowering the upper flammable limit of the vapor [*5*]. Chen's work suggests that it will be

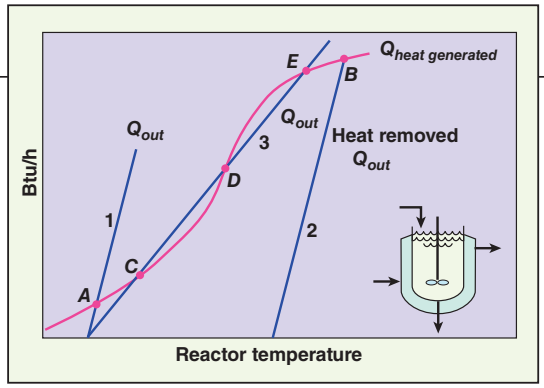

FIGURE 3. Heat-generation ($Q_{heat\ generated}$) and heat-removal (Q_{Out}) rates as a function of reactor temperature for three different heat-removal designs [9]. Heat generation is equal to heat removal at points *A, C, D, E,* and *B*, so steady state operation is possible. However, the reactor is not stable at point *D* without the addition of controls or a modification of the design

safe and practical to use pure oxygen for cyclohexane oxidation. Benefits include both IS operation and improved productivity. They also suggest that this approach could be extended to safer processes for partial oxidation of other liquid hydrocarbons using pure oxygen.

Chen's approach is a first-order IS process innovation because it changes the chemistry of the gas phase in a gas-liquid reaction and prevents the unwanted side reaction of combustion from occurring in the gas phase.

Although reference [5] did not claim to have demonstrated a new IS concept, Chen's work is different from the classical definition of the *Substitute* principle because the same reactants, chemical reactions, and products are involved. If the name *Substitute* were broadened to names such as *Change in Chemistry* or *Hybridize*, then it could be lumped in with the many successful applications that are possible when using the *Substitute* concept.

Chen's innovation permits rapid cyclohexane oxidation at lower temperatures and pressures, and could thus be said to be an example of the inherently safer principle *Moderate*. However, Chen's approach enables more moderate conditions by narrowing the flammability limits through the addition of a new component, water. It is thus an example of supplementation or hybridization.

Although not proposed by Chen [5] himself, his work suggests that there may be many other opportunities for transformation or hybridization of other potentially hazardous reactions to make them inherently safer. Although water would be high on anyone's list as a potentially transforming additive, it probably will not help many potentially hazardous reactions. However, there are many other chemicals that may be inert to the reaction and thus also be capable of inerting the vapor phase involved in an otherwise reactive liquid-vapor reaction. For instance, there are many examples of azeotropic mixtures in the literature and there are many compounds that could prove inert to oxidation reactions (such as, certain halocarbons).

Applications are not limited to partial oxidation with air or oxygen; other oxidations include chlorination and bromination reactions, for example. And there may be other examples of vapor-liquid reactions, such as hydrogenation reactions, where addition of a new chemical could improve the safety of the process.

Addition of an additional compound to a reaction mixture to minimize hazardous reactions may add complexity to the purification process, but it may be justified by the increased safety.

Chen's [5] paper on cyclohexane oxidation illustrates transformation or hybridization, in which the basic chemistry is maintained, but the addition of another chemical component transforms a potentially hazardous reaction process into a much safer one.

2. Create a robust process to stabilize or ensure dynamic stability. Not all process designs are inherently stable, and if the process design is to be safe, the process engineer must ensure dynamic stability as well as ensuring that the steady-state mass and energy balances are achieved. A number of processes exist that have narrow safe-operating limits but have been made stable by the addition of control systems. Dynamic stability and control of chemical processes has been extensively studied [7].

Designing the process to be more inherently stable to process upsets with and without control systems is clearly inherently safer, although this principle is not addressed in most discussions of IS. The IS principle *Ease of Control* has usually been interpreted to mean a process with a control system that the operator can understand clearly and manage effectively.

CCPS briefly mentions the advantages of designing processes that are inherently more stable or robust [2]:

"It is inherently safer to develop processes with wide operating limits that are less sensitive to variations in the operating parameters...Sometimes this type of process is referred to as a forgiving or robust process."

Designing a robust process increases inherent safety by imposing a change in the process variables and is a form of *Moderate*, a second-order inherently safer design.

CCPS [2] also cites the work of Luyben and Hendershot [8] that highlights how minimization or intensification in a reaction system that is intended to improve process safety may lead to less robust processes with the opposite effect.

I propose here that *Stabilize* or *Ensure Dynamic Stability* be added to the list of IS concepts to be sure that it is not overlooked in the quest for inherently safer processes.

Application of some of the other IS principles can adversely affect the dynamic stability of a process. For example, reduced liquid inventories (*Minimize*) in a distillation train make the process inherently safer from one perspective because the smaller process inventory decreases the consequences of loss of containment. However, the smaller inventory also shortens the response time of the distillation system to process upsets, increasing the risk that the basic control system will not be able to restore the distillation system to the desired operating conditions and avoid a potentially unsafe operating condition and/or an unscheduled process shutdown [2].

Chemical reactors carrying out exothermic chemical reactions are perhaps the best known examples of processes that can be dynamically unstable. Harriott [9] provides the illustration of an irreversible first-order chemical reaction being conducted in a continuous-flow, stirred-tank reactor (CSTR). Figure 3 shows the heat-generation rate by the chemical reaction as a function of reactor temperature. Heat-generation rates are low at low

TOOLS FOR INHERENTLY SAFER PROCESS PLANT DESIGN

- Process hazards reviews
- Chemical interaction matrices
- Dow Fire and Explosion Index and Chemical Exposure Index
- Fire, explosion and toxic-release consequence modeling and risk assessments
- Layer of protection analysis
- Spacing tables for units and for process equipment
- Dynamic process simulation
- Inherent safety analysis
- Periodic design reviews during product and process research, development and design
- Reviews of plant siting, plot plan, equipment arrangement and 3-D computer models
- Occupied building evaluation and design
- Area electrical classification
- Safety integrity level assessments and safety instrumented systems
- Human factors reviews
- Ergonomics reviews
- Safety case development
- The design process itself

temperatures, but as temperature increases, the reaction rate increases rapidly because of the exponential dependence of the reaction rate coefficient on temperature. At higher reactor temperatures, the shrinking concentration of reactant (due to conversion to product) reduces the reaction rate and partially overcomes the still-increasing reaction-rate coefficient. The heat-generation rate eventually reaches a constant maximum value when the reaction has reached complete conversion.

Figure 3 also shows three different straight lines for the heat-removal rate from the reactor for three different reactor-cooling-system designs. To achieve a steady-state energy balance, the rate of heat generation ($Q_{heat\ generated}$) by the chemical reaction must equal the rate of heat removal (Q_{out}) by the reactor cooling system. That energy balance occurs when the heat generation curve intersects the heat removal curve (where $Q_{heat\ generated} = Q_{out}$). In Figure 3, the three different heat-removal-rate lines intersect the reactor heat generation rate curve at five points. At four of these points (*A, B, C, E*), the steady-state energy balance solution is stable. At each of these points, if there is an increase in temperature, the rate of heat removal increases more rapidly than the rate of heat generation by the reaction and the reactor temperature tends to return to the desired operating point. Similarly, if the temperature drops slightly at one of these four operating conditions, the rate of heat removal decreases more than the rate of heat generation by the reactor and the temperature trends back up to the desired operating condition.

In contrast, point *D* in Figure 3 is an inherently unstable operating condition even though the steady state rate of heat generation by the reactor equals the rate of heat removal by the reactor cooling system. At point *D*, an increase in reactor temperature increases the rate of heat generation by the reactor more than it increases the rate of heat removal by the reactor cooling system, so the reactor temperature increases more instead of cooling back to the desired operating point.

This further increase in reactor temperature then leads to an even larger rate of heat generation rate by the reactor and additional heating of the reactor. Without any effective control actions, the reactor temperature will tend to increase to point *E* in Figure 3 before it stabilizes.

Similarly, in Figure 3 a decrease in reactor temperature at point *D* could eventually lead to the reactor temperature and conversion dropping back to point *C*.

Clearly, of the three reactor cooling-system designs represented by the three straight lines in Figure 3, the reactor cooling system represented by line *CDE* is the least desirable from a dynamic-stability perspective. Addition of an effective control system might be able to provide dynamic stability — but at the cost of installation and maintenance of the control system and at the cost of residual risk if the control system fails.

Another example of potential sources of process instability results from efforts to improve energy efficiencies in distillation trains through heat integration. In these cases, the feed to a column may be preheated by the bottoms product of a second downstream column. This may increase the risk of process upsets due to increased interactions between the two columns.

While avoidance of add-on controls has always been a goal of inherently safer design, achievement of that goal has seldom mentioned the concepts of Ensure dynamic stability or Stabilize as tools of the process engineer. It should be considered when considering other means to assure inherently safer processes during process design. The process engineer should work closely with the control systems engineer to address the dynamic stability of both the uncontrolled process and the controlled process to ensure a robust process.

3. Limit hazardous effects during conceptual and detailed engineering. David Clark published a seminal paper [*10*] on the limitation of effects when siting and designing process plants. He reminds us that there is a strong, non-linear decrease of fire, explosion, and toxic effects with separation distance. Comparatively small decreases in separation distance have a major effect, while larger increases in separation offer diminishing returns.

Methods, such as the Dow Fire and Explosion Index [*11*] and the Dow Chemical Exposure Index [*12, 13*], provide quantitative screening estimates of the hazards from various parts of a chemical process. Other indices have been developed and evaluated to perform a similar objective to the Dow indices [*1, 2, 14*]. These screening tools can identify those parts of a process where increased separation distances are needed to limit potential escalation of an incident.

In one typical plant design, a 10% increase in separation distances for all units increases total plant investment cost by only 3%. Similarly, doubling the separation distance for a hazardous unit representing 10% of the investment cost of the plant would cost only 3% more. Because of the nonlinear effect of separation distance, doubling the separation distance for a hazardous unit could reduce explosion overpressures on the adjacent units by a factor of four or more.

The strong decrease in hazardous effects with modest increases in separation distances will often more than justify increased capital cost.

Spacing also offers important benefits in crane and other maintenance access, ergonomic advantages and decreased risk of incident escalation. Future plant expansions or process improvements are also facilitated, although expansions that decrease spacing may increase hazardous effects.

Applying different IS principles

As discussed, the different IS principles are best applied at different stages of the process plant timeline. Although IS checklists are often used at the screening process hazards analysis (PHA) level, much more is needed throughout the development and design of a process plant.

For example, *Substitute* is best done during the product and process research phases before significant investments of time and resources in a particular product and process are made. *Hybridize* or *Transform* is best done during process research and development, as is *Moderate*.

Minimize, *Simplify*, and *Error tolerance* have the best result when applied during the process development, conceptual design and detail design phases. *Stabilize* or *Ensure Dynamic Stability* is also best done during design development.

Limitation of effects, which is closely related to passive protection, has its greatest impact during development of the plot plan and equipment arrangement.

IS processes and plants

As mentioned previously, the CCPS [2] defines two levels of inherent safety:

- *First-order inherent safety* results from changes in the chemistry of a process that reduces the hazards of the chemicals used or produced. *Substitute or Hybridize* efforts lead to first-order inherent safety
- *Second-order inherent safety* results from changes in the process variables. Examples include *Minimize*, *Simplify* and *Stabilize* the operations.

It is also helpful to distinguish between IS processes and IS plants. Even when hazards cannot be eliminated from the chemistry of the process, the plant using the potentially hazardous process can be made inherently safer through judicious design.

Note also that even with IS process chemistry, it is essential to employ IS principles during the process and plant design to ensure an IS plant.

Tools for IS plant design

There are a number of tools available to aid in designing process plants that are inherently safer (Box, p. 18). Although inherently safer reviews are a valuable tool for identifying opportunities for improvement, it is important to keep the principles of inherently safer in mind throughout the design process. ■

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Biodegradation and Testing of Scale Inhibitors

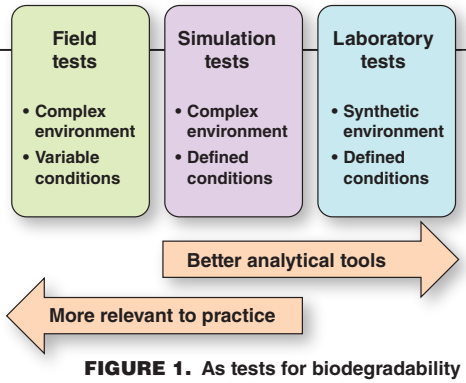

FIGURE 1. As tests for biodegradability more closely mimic the environment, the less control can be exerted, and the less reliable the data tend to be

Progress is being made toward better-performing and more-biodegradable scale inhibitors for water treatment

Kelly Harris
BWA Water Additives Ltd.

TABLE 1. INHERENT BIODEGRADABILITY OF COMMONLY USED SCALE INHIBITORS AND THE NEW 'GREEN' INHIBITORS

Inhibitor Type	Acronym	Inherent biodegradability result*
Phosphonates	PBTC	17% in 28 days
	ATMP	23% in 28 days
	HEDP	33% in 28 days
Polyacrylic acid	PAA	10% in 35 days
Phosphinopolyacrylates	PPCA	0% in 35 days
Polymaleic acid	PMA	18% in 35 days
Maleic acid terpolymer	MAT	35% in 35 days
Sulfonic acid co-polymers	SPOCA	7% in 28 days (OECD 306)
Polyaspartate	PASP	83–87% in 28 days
Carboxy methyl inulin	CMI	>20% (OECD 306)
Polycarboxylic acid	PCA	68.6% in 28 days (OECD 306)
Maleic acid polymer	MAP	54.9% in 35 days

* OECD 302B test unless otherwise stated.

Water systems are common in the chemical process industries (CPI). Mixing, heating, concentrating and evaporating water in these systems will often result in an accumulation of inorganic scale, which can markedly decrease process-system efficiency.

Scale inhibitors are chemical substances that, when added to industrial water at very low levels, act to reduce or prevent the formation of scale. Over the past several decades, the water treatment industry, as well as those applying its products, have been increasingly concerned about the environmental impact of scale inhibitors, along with that of other chemicals. The long list of available scale inhibitors includes several newer chemicals that are designed for biodegradability.

As the water treatment industry focuses on producing efficient scale-prevention products that minimize harm to the environment, practitioners in the CPI face questions about which scale inhibitors to use. As scale-inhibition products continue to improve in both areas, water users in the CPI must take into consideration the lowered environmental persistence of scale-inhibiting substances as well as their scale-inhibiting performance. Questions still remain about whether the new class of "green" products are effective scale inhibitors.

In hunting for biodegradable inhibitors, sometimes less-effective products are selected due to their perceived green qualities, despite the fact that lower efficacy may actually result in increased chemical discharge back to the environment. A poor inhibitor could potentially do more damage in the long run as larger volumes of additive are required to control the scale and, therefore, much larger volumes are discharged. A high degree of biodegradability is a worthy aim, but it should not be at the sacrifice of overall performance. In an ideal world, a very small amount of chemical would be used, and it would then disappear altogether. Although that target has not yet been reached, the industry is moving in the right direction. Tests are established for evaluating both inhibitor effectiveness and biodegradability, and they can help evaluate which available products reach closest to the ideal of high effectiveness and high biodegradability.

Scale inhibitors

Most common industrial processes, including petroleum production, mining, cooling water, desalination, reverse osmosis, pulp and paper manufacture, geothermal power production and sugar refining, operate using water systems. For various reasons, scale forms during operations, such as mixing, heating, concentrating and evaporating water. Scale accumulation can cause huge losses in production, increasing costs both financially and to the environment as systems become inefficient. For example, heat exchangers can become insulated by the scale and therefore cannot efficiently cool or heat.

Scale is formed by the increasing concentration of scaling cations, such as calcium and barium, as well as scaling anions, such as carbonate and sulfate. Once the concentration of ions exceeds super-saturation levels, nucleation will occur. Over time, nucleation leads to precipitation and the development of scale at the macroscopic level.

What happens at the surface of nascent crystals depends upon the relative rates of formation and dissolution of the scale. Generally the rate of formation is greater, thus leading to growth of the crystal. These small crystals can then clump together to form larger crystals, which will eventually block the system. Inhibitors can work to prevent the catastrophic buildup of scale at three separate stages: the nucleation stage, the growth stage and the deposition stage.

At the nucleation stage, threshhold inhibitors bind with scale-forming ions, but unlike chelants, the bound ions must be available to interact with their counterions. This disrupts the ion cluster at the early equilibrium stages of crystal formation, disrupting them before they reach the critical size for nucleation. As a result, the ions dissociate, releasing the inhibitor to repeat the process.

At the growth stage, growth inhibitors slow the growth of the scale by blocking the active edges of the crystal. Once the inhibitor has bound to the lattice, the crystal will form much more slowly and be distorted. Often they are more rounded in shape, which makes them less likely to adhere to surfaces and more easily be dispersed throughout the system.

At the deposition stage, dispersants prevent new crystals from coming together to form a large body of scale material. Dispersant-type inhibitors interact with the surface and repulse other charged particles to prevent binding.

A vast array of scale inhibitors is available today (Table 1), including phosphate esters and phosphonates, such as PBTC (phosphonobutane-1,2,4-tricarboxylic acid), ATMP (amino-trimethylene phosphonic acid) and HEDP (1-hydroxyethylidene-1,1-diphosphonic acid), polyacrylic acid (PAA), phosphinopolyacrylates (such as PPCA), polymaleic acids (PMA), maleic acid terpolymers (MAT), sulfonic acid copolymers, such as SPOCA (sulfonated phosphonocarboxylic acid), polyvinyl sulfonates. More recently, the so-called green inhibitors — polyaspartic acid (PASP), carboxy methyl inulins (CMI), polycarboxylic acids (PCA) and maleic acid polymers (MAP) — have become players.

TABLE 2. SUMMARY OF OECD BIODEGRADATION TESTS AND HOW THEY RELATE TO THE NATURAL ENVIRONMENT

	Soil	Aquatic sediments	Sewage treatment plants	Fresh water	Seawater
Ultimate					306
Ready				301 A-E	306
Inherent	304 A		302 A	302 B	
Simulation	307 B	308 B	303 A		

TABLE 3. CALCIUM CARBONATE JAR TEST WATER CHEMISTRY

Ion	Concentration, mg/L
Calcium	150
Magnesium	45
Sodium	211
Carbonate	51
Bicarbonate	269
Chloride	397
Total dissolved solids	1,084

TABLE 4. ICW EVAPORATIVE RIG TEST WATER CHEMISTRY

Ion	Concentration, mg/L
Calcium	250
Magnesium	120
Sodium	94
Carbonate	57
Bicarbonate	148
Chloride	262
pH	8.8

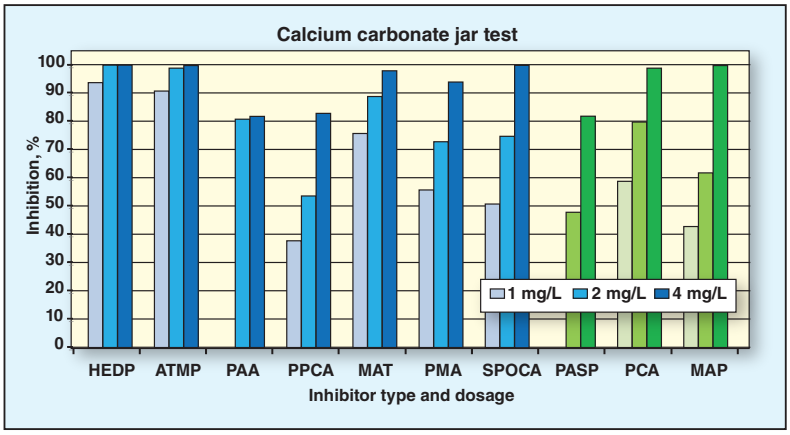

FIGURE 2. The calcium carbonate jar test assesses the ability of additives to inhibit $CaCO_3$ precipitation, and its water chemistry simulates cooling water

Biodegradation

Since the 1972 United Nations Conference on the Human Environment in Stockholm, Sweden, environmental pollution has been a major concern for all industries. Across the globe, a number of governments and regional economic integration organizations have since established programs for identifying and assessing substances that could cause longterm harm. *Harm* is defined as the undesirable effects resulting from the accumulation in living organisms of degradation-resistant substances above certain concentrations. These persistent organic pollutants (POPs) or persistent, bioaccumulating, toxic substances (PBTs) are identified, characterized and classified using a variety of tests and are subject to regulations concerning their use. These tests are dependent on the final destination of the chemical, and knowledge of how the environment will be impacted by its presence is paramount. The most harmful chemicals are generally those that remain within the environment, building up in the tissues of the biological organisms that inhabit the area until a toxic level is reached.

Biodegradation can be defined as the natural process by which organic substances are decomposed by microorganisms (mainly aerobic bacteria) into simpler substances, such as carbon dioxide, water and ammonia. Defining the degree of biodegradation is a consideration — complete degradation into these final simple components should be distinguished from partial degradation into a different related compound. Moving forward, industry will likely need an increasingly comprehensive body of knowledge about the biodegradation products resulting

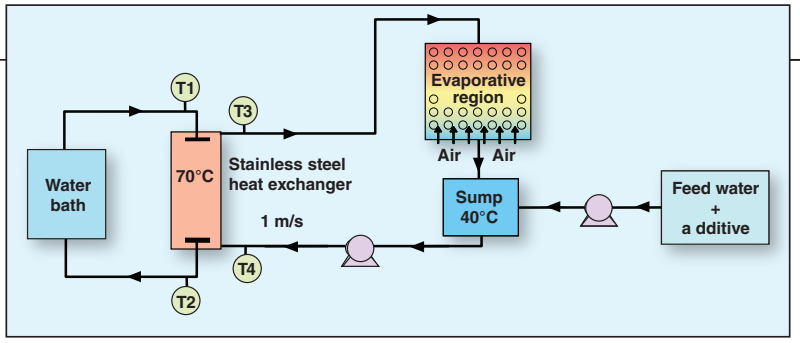

FIGURE 3. The apparatus of the pilot cooling-tower evaporative test is designed to measure an additive's ability to inhibit calcium-carbonate deposition

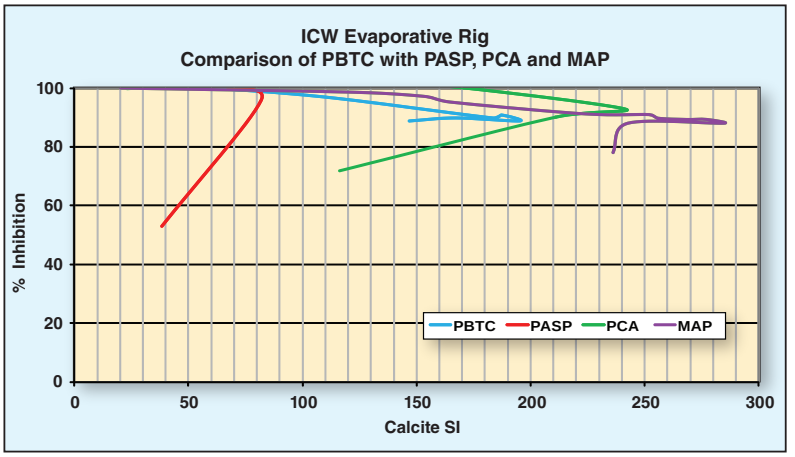

FIGURE 4. The higher a saturation index (SI) that can be reached indicates a more efficient inhibitor

from a substance, its ability to bio-accumulate and its toxicity.

The biodegradability of some currently available inhibitors is shown in Table 1. Before the push for 'green' products, very few, if any, were actually biodegradable. With biodegradability above 30%, HEDP and MAT are just barely considered inherently biodegradable (will degrade eventually). Looking at the new generation of 'green' inhibitors, it is clear to see the difference. PASP is the most biodegradable, but all four products are well above the level required for the inherently biodegradable classification, as well as what is required to be considered a non-persistent chemical. Tests conducted by the author suggest that PCA and MAP offer a significant improvement over other biodegradable products such as PASP, and are also more efficient than their non-biodegradable counterparts against calcium carbonate scale.

OECD test categories

For measuring biodegradability, the most recognized tests are the Organization for Economic Cooperation and Development (OECD) series, which demonstrate the susceptibility of a substance to microbial degradation under environmental conditions. The OECD biodegradation test series represents a variety of methods that include both purely laboratory-based tests, as well as simulation and field-based tests.

Since biodegradation is affected by many factors, each environment (such as seawater or freshwater) is very different. This causes a number of issues for method development. A trade-off exists between the closeness with which a test mimics the environment, and the reliability of the resulting data. As the tests more closely mimic the environment, the less control can be exerted, and the less reliable the data are. Figure 1 illustrates the continuum of laboratory tests versus those that simulate the actual environment.

In laboratory tests, biodegradation is encouraged with high levels of the test substance or a low substance-to-biomass ratio, and a simplified environment. Simulation tests are a good middle ground with regard to controlling external factors such as temperature and pH, but in a more realistic environment. This includes using a concentration of substrate that reflects its likely level when in use, as well as the use of indigenous biomass material to allow adsorption of the chemical onto the biomass. The test should also be conducted at a temperature typical for the environment it is designed to simulate. These are summarized in Table 2 and can be broken down into categories for the specific environments in which the substrate will be located. A series of tests can then be undertaken as follows:

Ready/ultimate tests. These are rigid screening tests with a high level of test substance (2–100 mg/L). Although they are laboratory tests, a positive result means that ultimate biodegradation in the environment will occur. A failure does not necessarily mean that the chemical will not biodegrade at all, so inherent biodegradability tests may be performed.

Inherent tests. These tests have a high capacity for degradation with long exposure times and a high biomass-to-substance ratio, thus giving the substrate the best chance to break down. Again, this is a laboratory test with a controlled and synthetic environment. A positive result will demonstrate that the substrate is inherently biodegradable, but a negative result can still not rule out eventual degradation in its final environment.

Simulation tests. These tests use a low concentration of the chemical and are performed in an environment that closely mimics the real world. A positive result here strongly suggests that a chemical will biodegrade in the natural environment. A negative result will give an indication that the chemical is likely to be persistent.

By following this process of beginning with the ready biodegradability tests and moving down the chain, a good understanding of how a substance will behave in the environment can be obtained. When this information is used in combination with the toxicity and bio-accumulation data, the impact of releasing this chemical into the environment can be assessed with a high degree of confidence. However, determining if a chemical biodegrades is only half the story, since all of this is futile if it is a poor inhibitor.

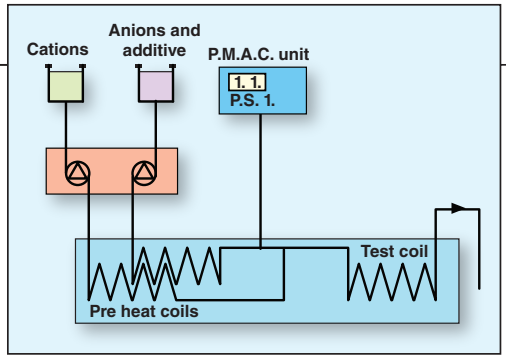

FIGURE 5. In the dynamic scale loop test, calcium carbonate deposition reduces the bore size of the test coil, causing an increase in pumping pressure

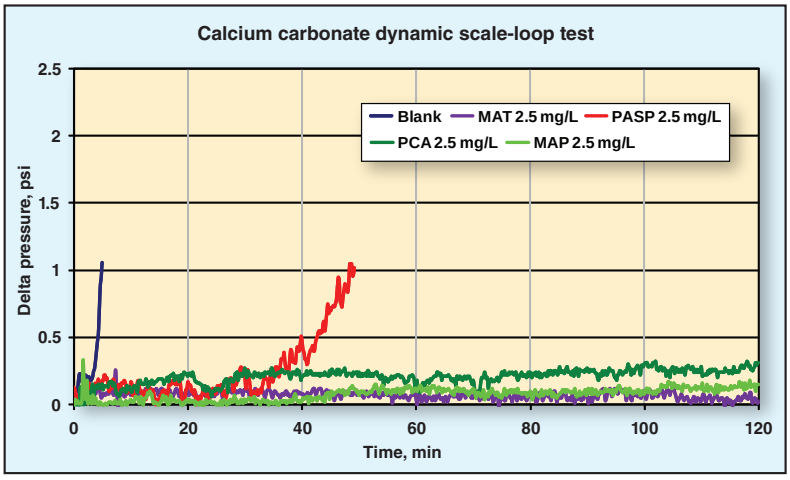

FIGURE 6. Biodegradable inhibitors performed as well as commonly used ones in the dynamic scale loop test

Testing inhibitor efficacy

In the industrial water treatment (IWT) area, the most commonly encountered type of scale is calcium carbonate, which may occur in three possible crystal forms — aragonite, calcite and vaterite. When testing for the efficiency of a scale inhibitor against calcium carbonate scale, the following tests can be performed.

1. The calcium carbonate jar test is a 30-min homogeneous test that demonstrates the threshold inhibitor ability of a product.
2. The pilot cooling-tower evaporative unit test is designed to test both the threshold and dynamic inhibitor mechanisms against calcium carbonate under heat transfer conditions.

Calcium carbonate jar test. This test is designed to assess the ability of additives to inhibit the precipitation of calcium carbonate. The water chemistry simulates cooling water, and the high temperature represents heat-exchanger surface conditions. Air bubbling is used to facilitate carbon dioxide removal, which shifts the equilibrium toward carbonate formation. This shift increases the test severity by raising the pH of the test solution.

To carry out the test, a solution containing calcium chloride and magnesium chloride is mixed with an equal volume of a solution containing sodium carbonate and sodium bicarbonate and that already contains the additive to be tested. The air-bubbled solution is heated at 70°C (158°F) for 30 min, after which time the solution is filtered and the calcium remaining in solution is determined by EDTA (ethylenediaminetetraacetic acid) titration. The water chemistry for this test is presented in Table 3. The higher the amount of calcium retained in solution, the greater the scale inhibition ability of the product.

Figure 2 represents sample tests conducted by the author's employer. The results are expressed as percentage inhibiton against dose level. At dose levels of 1 and 2 mg/L, HEDP and ATMP are clearly the most effective, with PCA and MAP being the best among the green scale inhibitors. Once a 4-mg/L dose level has been reached, a number of inhibitors are capable of 100% inhibition of calcium carbonate (PCA and MAP included), but PASP only reaches an 80% inhibition level. This may seem like a high figure, but unless 100% is reached, calcium carbonate will continue to form and ultimately reduce the efficiency of the plant significantly.

Pilot cooling-tower evaporative unit test. The evaporative unit test is a dynamic test designed to provide a realistic measure of an additive's ability to control calcium carbonate deposition. The pilot cooling-tower evaporative unit has constant make-up but has no blowdown, so the system water concentration increases with time as evaporation occurs. The system water is circulated over a 316 stainless-steel heat exchanger. The heat exchanger is heated by passing hot water through the tube. The surface temperature of the heat exchanger is approximately 70°C (158°F). The evaporative region maintains bulk water temperature at 40°C (104°F) by passing air countercurrently to the water flow in the cooling tower. The higher the calcite saturation index (SI) that can be reached, the more efficient the inhibitor. The initial water chemistry for this test is given in Table 4 and a schematic diagram of the equipment used is given in Figure 3. Initial dose level of additives is 10 mg/L as solids.

In Figure 4, PBTC shows what level a good calcium carbonate inhibitor can achieve in this test. Its failure point occurs at a calcite SI of approximately 200. According to data from the author's company, MAP exhibited the best calcium carbonate control of the green inhibitors, reaching a calcite SI of 285. PCA also fared well, with a failure point at 240 calcite SI. Both of these results are a significant increase over that reached by PBTC. PASP, however, gave a rather poor result, failing at a calcite SI of approximately 80. This is less than one third of the level reached by MAP and PCA.

Both tests show that some green inhibitors can replace existing effective non-green inhibitors such as PBTC.

Petroleum-industry scale tests

When considering application scale inhibitor tests in petroleum fields, performing both the calcium carbonate and the barium-sulfate dynamic scale-loop tests is required to provide a good indication of inhibitor performance in the reservoir.

TABLE 5. CALCIUM CARBONATE DYNAMIC SCALE-LOOP TEST WATER CHEMISTRY

Ion	Concentration, mg/L
Calcium	350
Magnesium	56
Sodium	10,077
Potassium	283
Barium	50
Strontium	50
Bicarbonate	1,000
Chloride	16,058
Sulfate	0
Total dissolved solids	27,924
pH	7.8

TABLE 6. BARIUM SULFATE TEST WATER CHEMISTRY

Ion	Concentration, mg/L
Calcium	636
Magnesium	634
Sodium	14,760
Potassium	446
Barium	120
Strontium	190
Bicarbonate	0
Chloride	26,930
Sulfate	530
Total dissolved solids	44,246
pH	5.5

Calcium carbonate dynamic scale-loop test. In some ways, the dynamic scale-loop test is less severe than the threshold static jar test, because the inhibitor is replenished, so it maintains constant concentration. In the jar test, when a crystal is formed, some of the inhibitor is consumed as it binds onto the crystal surface. Because inhibitor levels are not replenished, concentration will drop over time. Having a constant inhibitor level throughout the dynamic test ensures that it is the growth-inhibition mechanism that is being studied, with metal surfaces acting as growth sites.

This test is conducted using synthetic water with the chemistry shown in Table 5. Separate solutions containing the anions and the cations are pumped through pre-heat coils at 90°C (194°F) and mixed in a T-piece prior to the 0.1-mm I.D., 1-m-long 316 stainless-steel test coil. A schematic representation of this apparatus is shown in Figure 5. During the test, calcium carbonate deposition reduces the bore of the test coil, causing an increase in pumping pressure. The rate of change in pressure across the coil is monitored with a pressure transducer and data captured. The test is considered successful if the change in pressure remains below 1 psi (6.9 kPa) over a two-hour period.

MAT, a commonly used inhibitor, demonstrates that a 2.5 mg/L dose level is sufficient to completely inhibit calcium carbonate scale formation. The green inhibitors PCA and MAP also display excellent scale inhibition at 2.5 mg/L. PASP is unable to prevent scale formation at this dose, reaching 1 psi (6.9 kPa) in only 50 min.

Barium sulfate dynamic scale-loop test. The water chemistry for this dynamic scale-loop test is given in Table 6 and is equivalent to a 80:20 troll-to-seawater mixture. The anion and cation solutions, this time with with no inhibitor present, are pumped through preheat coils at 90°C (194°F) and mixed in a T-piece prior to the 316 stainless-steel test coil, which has a 0.1 mm I.D. and is 1-m long. Barium sulfate deposition reduces the bore of the test coil, causing an increase in pumping pressure. Once a 1 psi (6.9 kPa) change in pressure has been achieved, a third solution containing anions plus inhibitor replaces the anion solution. The test is run for 2 h unless the additive fails to prevent further barium sulfate scale.

Figure 6 illustrates the data for MAT and the three green inhibitors PASP, PCA and MAP. At a 4 mg/L dose level, MAT was able to stop deposition completely, thus leading to no further increase in pressure. PASP, PCA and MAP were equally efficient at this dose level, demonstrating that in this test, the green inhibitors are as efficient as those already in common use. ■

Edited by Scott Jenkins

Author

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REFINERY OPERATORS AND MAINTENANCE TECHNICIANS:

Mapping Competencies

Use this new visual mapping method to assess functional qualifications

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A variety of factors — including a shift in the median age of workers worldwide and the impending skills shortage due to attrition and early retirements — have created a need to find fast and reliable methods and tools for mapping the technical competencies of professionals in the chemical process industries (CPI). Typically competency-mapping projects in the CPI begin with some form of task or hierarchical job analysis. However, historically many of these initiatives have been slowed down or have even failed due to incomplete or excessive lists of competencies, a lack of sense of ownership among the workers and a lack of fit between generic competency maps and project-specific requirements. Some competency map designs capture "what needs to be done" with "how it is done and who does it." Such maps have a short shelf life because they have to be recreated every time there is a change either in the reporting structure or in the tools being used by the enterprise.

The main objective of this article is to introduce a new framework for mapping functional competencies and to share new insights gained by applying the framework to petroleum-refinery operators and maintenance technicians.

The new framework

The new competency-mapping framework (U.S. Patent No. 61/358,262 pending) is used to map technical competencies and it employs the model described below.

Certain minimum levels of cognitive competencies (or knowledge) and functional competencies (or skills) are required for a person to qualify for a task. Additional metacognitive, social and context-specific competencies enable a qualified person to excel at his or her job.

As shown in Figure 1, the framework consists of three grids or maps. The first map shows "what needs to be done." This map is called the outcome/experience (O/E) map because it shows the value-added outcomes and can also be used to capture workers' experience. A "value-added outcome" is something for which someone is willing to pay an employee. Monitor a process line, diagnose a compressor problem, write a plan, analyze a sample, teach mathematics, escalate a distillation unit problem, optimize a process unit — these are examples of value-added outcomes. A blank O/E map can also be used by an employee to mark

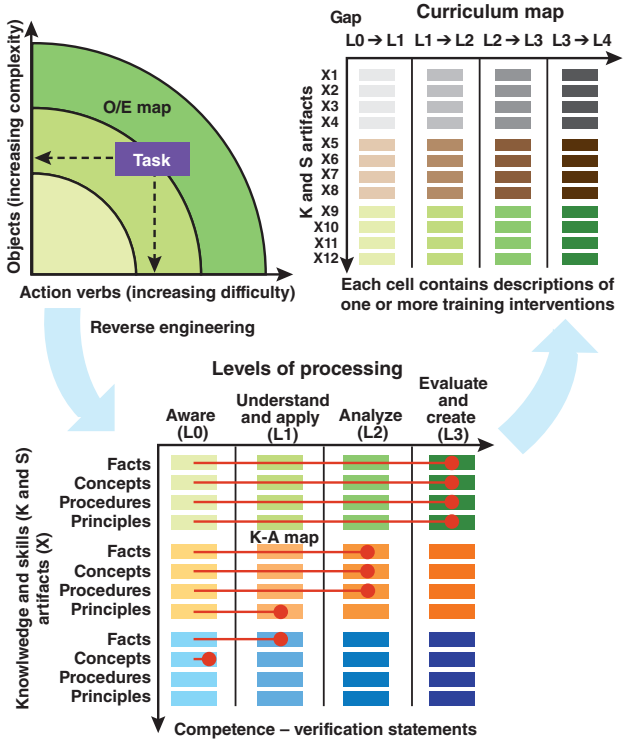

FIGURE 1. Shown here are the three types of maps and two conceptual relationships; between the value-added task and the required qualifications (K-A map) and between competency gaps and the required training interventions

all the value-added tasks he or she has done in the past.

The second grid or map in Figure 1 shows "what one needs to know and be able to do" in order to qualify for each task shown in the first map. This second map is thus called the knowledge-ability (K-A) map and is derived from the first map by reverse engineering each task. The rows of the K-A map contain artifacts divided by types of content. This division of artifacts into facts, concepts, principles and procedures is based on the Component Display Theory (CDT) developed by Merrill [*1*].

- *Facts (sometimes called vocabulary)* include names, labels, values and designations that are associated with objects, places and events. Facts are usually time-bound or ephemeral. Facts are easier to identify than concepts
- *Concepts* are concrete or abstract categories that enable grouping of several facts. Concepts are generally not time-bound and are considered to be universal
- *Principles* are rules or relationships that connect two or more concepts
- *Procedures* consist of sets of steps that must be executed in a specific sequence to accomplish specific tasks

TABLE 1. TYPICAL ARTIFACTS	
Typical resources for knowledge and skills for each process unit	Common to all process units
Plots and diagrams	
Large control diagrams	
Process and instrumentation diagrams	
Process startup and shutdown sequence diagrams	
Plot plan	
Electrical line diagrams	
Manuals	
Unit process description manual	X
Shutdown, startup and emergency procedures	X
Field training guides	X
Board training guides	X
RWD exercises and analysis strategies	X
Distributed control system	X
Advanced process control	X
System isolation	
Fundamentals	
Fundamentals of regulatory control	X
Fundamentals of advanced control	X
Refinery process fundamentals	X
Procedures	
Process description manual methodology	

TABLE 1. This list of typical artifacts contains explicit knowledge that operators need to interact with in order to improve their competencies in a process plant environment

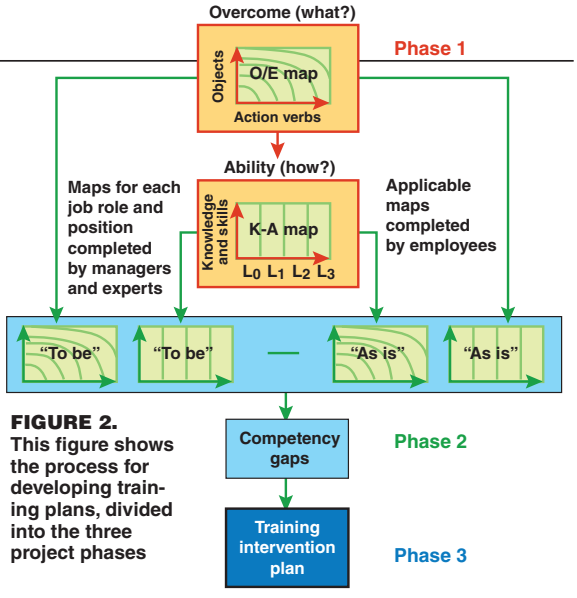

FIGURE 2. This figure shows the process for developing training plans, divided into the three project phases

Each of the columns of the K-A grid represents a level of cognitive, affective or psychomotor outcome. Cognitive domain involves knowledge and development of intellectual skills. Bloom [*2*] originally proposed a taxonomy to assess outcomes of learning. Bloom's initial work focused on the cognitive domain. He divided a learner's cognitive development into six levels. He labeled each level by a specific mode of cognitive processing used by the learner.

Bloom's taxonomy is used extensively in the field of instructional design and development. His work was later extended by other researchers to include taxonomies for outcomes in the affective [*3*] and the psychomotor domains [*4*]. Affective domain focuses on attitudes, emotions and feelings. Skills in psychomotor domain describe ability to physically manipulate a tool or an instrument.

The last map (top-right) in Figure 1 is a curriculum map that lists all training interventions recommended for bridging all possible competency gaps. Here is an example of a competency gap: If for a specific artifact (say, X5), an individual needs to be at L2 to qualify for a task but is at level L1, there is a competency gap (defined as L2–L1).

The individual needs to participate in a specific training intervention outlined in the curriculum map corresponding to the artifact X5 and in the column that shows L1–L2 that will move that individual to level L2. The K-A grid illustrated in Figure 1 has 12 artifacts shown in column one and has four levels (L0, L1, L2 and L3).The maximum number of possible qualifications gaps for that specific grid is 48.The curriculum map for that situation shows 48 cells. Each cell contains at least one training intervention. Additional details on the framework and the maps are available in the literature [*5–7*].

The process outline

As shown in Figure 2, the process for developing training plans using the new framework consists of three phases: Create competency maps and a curriculum map (Phase I), identify competency gaps (Phase II), and develop a training-intervention plan (Phase III). The recommended process for the first phase includes the following steps:

Step 1. Start with the creation of O/E map. Break down each task description into an action verb and an object, such as "Diagnose a problem." Show the verb on the *x*-axis and the object on the *y*-axis.

Step 2. Identify the "most relevant" cycles or processes. Objects shown on the *y*-axis typically follow a life-cycle consisting of different states. Each function, discipline or group performs work using one or more core processes that distinguish it from other functions, disciplines or groups For example, engineers often rely on a problem-solving cycle, software developers use a development cycle, and project teams use PDCA (for "plan, do, check, adjust") or some other variation of the Demming cycle [*8*]. The core work processes performed by a function or a group may vary depending on the state of the object. Use of cycles ensures that the list of verbs is a closed list.

Step 3. Position verbs and objects on the two axes. Arrange all of the verbs, such as monitor, optimize, plan, verify, design, commission, and so on, in an order of increasing difficulty on the *x*-axis (columns). Rearrange all the objects (components, functional units, process units and so on) vertically in an increasing order of complexity. This ensures no overlap between tasks (shown in cells) and also permits linking of different job levels to different sets of tasks. The job levels increase diagonally as one moves away from the origin.

Step 4. Derive the K-A map. From the O/E map, derive the K-A map by reverse engineering each cell on the O/E map. For example, if the task is "Monitor process lines" ask the questions: How do you define "monitor"? What specific steps are involved? Define "process lines" as explicitly as possible. Break down all the answers for a given task into facts, procedures, concepts and principles and then list them on the *y*-axis (rows) of the K-A grid. Use Bloom's and other taxonomies [*2–4*] to define the relevant levels (three

Objects ▲											
Process units ▲	U-1500 (fractionator)	E	E	E	E	E	E	E	E	E	
	U-1400 (sulfolane)	E	E	E	E	E	E	E	E	E	
	U-1300 (unifining-platforming)	E	E	E	E	E	E	E	E	E	
	Compressors & blowers	D	D	D	D	D	D	D	D	D	
	Ionic exchangers	D	D	D	D	D	D	D	D	D	
	Heat exchanger systems	C	C	C	C	C	C	C	C	C	
	Distillation systems	C	C	C	C	C	C	C	C	C	
Equipment items ▲	Ejectors	B	B	B	B	B	B	B	B	B	
	Blenders and stirrers	B	B	B	B	B	B	B	B	B	
	Tanks	B	B	B	B	B	B	B	B	B	
	Process lines	B	B	B	B	B	B	B	B	B	
	Fire protection equipment	B	B	B	B	B	B	B	B	B	
Operational state ▶		Normal/steady state									
Action verbs ▶		Monitor	Detect	Analyze	Diagnose	Determine the solution: Execute	Determine the solution: Escalate	Verify	Document	Optimize	

FIGURE 3. Each cell in the grid represents a task, and the grid shows how tasks of operators change with their job levels (B, C, D, and so on)

Objects ▲																
Equipment items ▲	Motors of the polyethylene reactors	D	D	D	B	D	D	D	D	D	D	D	D	D	F	E
	Actuators of motorized valves	D	D	D	B	D	D	D	D	D	D	D	D	D	F	E
	Emergency diesel generators	D	D	D	B	D	D	D	D	D	D	D	D	D	F	E
	Battery bundles	C	C	C	B	C	C	C	C	C	C	C	C	C	E	D
	Average (balanced) voltage motors	C	C	C	B	C	C	C	C	C	C	C	C	C	E	E
	Desalters	C	C	C	B	C	C	C	C	C	C	C	C	C	E	E
	Low distribution boards	B	B	B	B	B	B	B	B	C	C	B	B	B	E	C
	Lightning and electrical outlet	B	B	B	B	B	B	B	B	C	C	B	B	B	E	C
Operational state ▶		Normal operation and transition														
Action verbs ▶		Prepare			Execute					Document		Improve			Support	
		1	2	3	1	2	3	4	5	1	2	1	2	4	1	2

FIGURE 4. Each cell in the grid represents a task, and the grid shows how the tasks conducted by electrical maintenance technicians change as their job levels advance (B, C, D, and so on)

or four) of cognitive, affective and psychomotor processing. Write competency statements for each cell in the K-A grid. One competency statement is written for each artifact for each level. A competency statement includes objective and observable attitude expressed, product created or behavior demonstrated by a person at the selected level of development for the selected artifact.

Case study. Discussed below is a case history that illustrates how to map the competencies of petroleum-refinery operators and maintenance technicians. There are around 717 oil refineries worldwide of which about 132 are located in the U.S. [9]. Operators are the "eyes and ears" of the enterprise closest to the unit. They play a significant role in ensuring safe operation, regulatory compliance and high uptime for petroleum-refinery units. Maintenance technicians ensure integrity, reliability and safe operation of all the assets. Operators and maintenance technicians work in round-the-clock in shifts. A typical refinery with ten process units employs about 300 operators and about 100 maintenance technicians.

Acknowledging the potential threat created when "knowledge walks out the door" over the next five years, Ecopetrol, S.A.'s petroleum-refining business unit, which operates two major refineries in Colombia, set the following strategic goal for its refining business unit: "By the end of 2011, Ecopetrol will have 80% of all its professionals working at 100% of their competency level." In 2007, Ecopetrol began developing competency maps for refinery engineers. The mapping process was based on the new visual framework that resolved many of the issues with the traditional approaches. The maps for engineers were completed in 2008 and a training program was launched in 2009 to bridge the identified competency gaps.

In 2009, Ecopetrol decided to extend the same competency-mapping approach used by engineers to all other professionals at the petroleum refineries in Barrancabermeja, and Cartagena, Colombia. As of this writing, competency maps have been created for operators and maintenance technicians, and a training program is underway at those facilities.

Figure 3 shows a section of an O/E map for process plant operators. The complexity of objects increases as one travels up the *y*-axis (rows). Individual components appear at the bottom, and large integrated systems are at the top of the *y*-axis. For plant operators, the object cycle of relevance is the plant life cycle.

The plant life cycle states are shown on the *x*-axis. One of the states of this plant lifecycle is the "normal operations state." Under this state, the process executed by the operators is the "problem identification and reporting process." The *x*-axis (columns) in Figure 3 shows the action verbs arranged by ascending degree of difficulty of execution as one moves away from the origin. The letters B, C, D and so on that are shown in the cells of Figures 3 and 4 represent different job levels for plant operators. Levels B, C and D are for field operators. Levels E and F are job-levels of board or panel operators responsible for monitoring entire process units.

Figure 4 shows an O/E map for maintenance technicians specializing in electrical equipment items. The representa-

TABLE 2. EXAMPLES OF COMPETENCY STATEMENTS			
	L1: Remember and understand	**L2: Apply and analyze**	**L3: Evaluate and create**
Verbs indicating the cognitive processing levels —>	Explain, retrieve, list, tabulate, interpret, describe, summarize, classify, compare, execute, forecast	Implement, use, predict, illustrate, calculate, prepare, produce, executve, classify, experiment, build, graph, organize, differentiate, achieve	Generate, evaluate, critique, plan, handle, review, recognize, train, audit, conclude, act, combine, repair, update, organize
Artifacts			
Professionalism, ethics and integrity guidelines	Describe rules and behavior that demand corporate professionalism, ethics and integrity. Explain these values from the company policies.	Illustrate the professionalism, ethics, and integrity topics through an example or any concrete situation. Differentiate the principles and ethical values of responsibility and integrity	Recognize the behavior contrary to corporate professionalism, ethics and integrity. Train on Ecopetrole ethics code
Process and instrumentation diagrams (P&IDs)	Describe the content and application of the information included in the P&IDs	Relate and use the information derived from the P&IDs to his or her activities	Guide the maintenance function according to the information and sequence of P&IDs. Identify critical zones and critical equipment in the plants. Train on the interpretation and application of the P&IDs
Software for capturing real-time plant data	Describe the data generated by instruments and equipment in realtime in the process plants, including the PI software in the distributed control system (DCS)	Interpret the data generated by instruments and equipment in realtime in the process plants to determine the operation condition of the equipment	Train on the use of data generated by instruments and equipment in realtime in the process plants
IEEE 1184 guide for Batteries for Uninterruptible Power Supply Systems	Describe the content of the IEEE 1184 standard guide	Relate the IEEE 1184 standard guide to his or her activities	Develop and modify the content of the training materials based on the standard IEEE 1184 materials. Train on the use and application of the IEEE 1184 guide

TABLE 2. Shown here are examples of competency statements written in a style and format that can be used to objectively assess the qualifications of electrical maintenance technicians

tive activity cycle for maintenance technicians is the repair order cycle. It is nested under the equipment or plant lifecycle. The *x*-axis (columns) in Figure 4 shows the action verbs in the repair order cycle arranged by degree of difficulty as one goes from left to right. The letters B, C, D and so on that are shown in cells are job levels for maintenance technicians.

Table 1 shows a list of typical artifacts — divided into drawings, manuals, fundamentals and procedures — that occupy the vertical axis of the K-A map for the board operators. Table 2 shows a section of the competency map for the maintenance technicians. Note that the competency statements shown in the cells of the grid in Table 2 are written as externally observable actions or outcomes that can be used to verify the level of each learner's development.

Findings

We gained new insights from the process of applying this new framework and from the deliverables created by the process. It is important to make the competency-mapping process visual and develop it from the bottom up. The main success factor for the project was that the operators and maintenance technicians quickly recognized the value of the approach. This removed the cognitive barriers to its implementation. The visual nature of the process made it easy to explain to both the company executives and the employees. The managers saw how the maps can be used to identify strategic gaps, develop hiring plans and provide career guidance to new operators and maintenance technicians. The hierarchical breakdown of competencies allowed for highly pinpointed training interventions to be selected to bridge specific competency gaps.

Operators and maintenance technicians in the refining business share many common traits. The study showed that the following common competencies — problem-solving, troubleshooting, and systems-thinking capabilities — are required to move up in the job levels for both. The study also showed that these two types of professionals — operators and maintenance technicians — share over 30 competencies that are related to topics such as company vision, mission and values, organization structures, Hazop analysis and more. Operators and maintenance technicians also use many common software tools for such activities as data gathering, documentation and communication. Since operators and maintenance technicians are expected to work together, the identification of many common competencies creates an opportunity to conduct joint training sessions, which in turn will save costs and will help improve the teamwork between the two groups.

However, there are overt differences in how the two jobs are organized, and there are subtle differences in the mindsets that are required to be good at each job. Operators generally start as field operators. The main focus of field operators is equipment items such as pumps, tur-

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You & Your Job

bines, tanks and so on. When they become board or panel operators their emphasis shifts to process units such as an aromatics unit, ethylene unit, steam system and so on. This parallels the career-development path of process engineers. Maintenance technicians start by focusing on equipment components (rotors, shafts, welds and so on) and eventually shift to equipment systems. But unlike operators, the maintenance technicians are routinely organized by specialty areas such as mechanical, electrical and so on. Thus, their career progression often parallels those of mechanical and electrical engineers

For plant operators, the core process is problem identification and solution. By comparison, the core process for maintenance technicians is the repair-order cycle. The work of maintenance technicians is more structured and regulated than that of operators. This means that operators have to cultivate the ability to "improvise on demand" without risking the safety of the plant. Meanwhile, maintenance technicians have to be more cognizant of newer techniques, regulations and budgets relative to the operators. ■

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Kaeser Compressors

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Dryers and Evaporators

The Witte Co.

This salt-drying system recycles hot exhaust air

Among a host of advantages, the bulk salt-dryer system (photo) from this company captures half of its hot exhaust air for recycling, which both reduces energy requirements and cuts the volume of exhausted air. Designed for drying road salt, rock salt, sea salt, food-grade table salt and other salt products, the system combines a vibrating bed dryer with an integral baghouse collector. The unit captures up to 100% of fine salt particles entrained in the airstream. The fines are returned to the process for recovery, effectively eliminating product loss. The need for waste disposal is also eliminated, says the company. — *The Witte Company Inc., Washington, N.J.*
www.witte.com

The Grieve Corp.

This spray-drying system is designed for Li-ion batteries

New spray-drying technology from this company is specifically designed for use in manufacturing cathode and anode materials in high-performance lithium-ion batteries. With the company's rotary and nozzle atomization systems, the spray-drying technology can help produce Li-ion powders. — *GEA Niro, Søborg, Denmark*
www.niro.com

An RF drying system with high efficiency

The Macrowave radio-frequency (RF) drying system offers greater efficiency than conventional web dryers. The system enables the high-speed drying of water-based patterned glue and coatings at line speeds previously reached only with solvent-based coatings. The Macrowave is designed to selectively heat only the coated portions of the web, leaving the bound moisture in the substrate intact, preventing overdrying, discoloration and shrinkage. Capable of operating at web speeds of 1,500 ft/min, the system requires only one-third of the floor space required for hot-air and infrared dryers. — *Radio Frequency Co., Inc., Millis, Mass.*
www.radiofrequency.com

Material is redistributed for uniform drying

In this company's Turbo Dryer, material is intermittently redistributed to allow uniform drying. The unit consists of stacked circular trays that slowly rotate inside an enclosure where heated air or gas is circulated with internal fans. The Turbo Dryer handles temperatures from 60 to 1,200°F and features precisely controllable temperature settings that can be easily adjusted and automatically maintained. It is available in a range of sizes and materials. — *Wyssmont Inc., Fort Lee, N.J.*
www.wyssmont.com

Walk into this gas-fired drying oven

With physical dimensions of 72-in. high, 72-in. deep and 60-in. wide, the No. 948 walk-in oven (photo) is heated by a natural-gas burner, and heats to 650°F. The oven is equipped with a 1,500 ft^3/min powered exhauster with motorized dampers in the intake and exhaust for accelerated cooling. The No. 948 has a top-mounted heat chamber, an interior of stainless steel with type 304, 2B finish. Currently used to evaporate water from plastic, the oven has 5-in.-thick insulated walls and floor, an aluminized steel exterior and comes with safety protection equipment. — *The Grieve Corp., Round Lake, Ill.*
www.grievecorp.com

This compressed air dryer combines advantages

The Hybritec combination compressed-air dryer (photo) combines the energy savings of a refrigerated dryer with the very low dewpoints of a desiccant dryer. The air is first treated by a refrigerated dryer to remove most of the air's water vapor, then treated by a desiccant dryer to further reduce the dewpoint. The air is finally returned to a refrigerated dryer to be reheated and recycled to the air system. Hybritec dryers offer consistent outlet dewpoints, lower operating costs and longer desiccant life. — *Kaeser Compressor Inc., Fredericksburg, Va.*
www.kaeser.com

Recover solvents with this laboratory evaporator

The Centrifan PE is a laboratory evaporator that contains the gas in a

Note: For more information, circle the 3-digit number on p. 66, or use the website designation.

closed system, enabling high solvent recovery. The device is ideal for rapidly evaporating chemical reaction mixtures without the need for a vacuum pump or external gas supply. It can perform oxygen-free evaporations with either nitrogen or argon feeds, and temperature-controlled evaporations at setpoints from 10 to 60ºC. — *Modular SFC, Franklin, Mass.*
www.modularsfc.com

This dryer enables the use of low-temperature waste heat

For the drying of organic waste products, the Rolling Bed Dryer from this company overcomes a number of disadvantages often observed with alternative drying equipment, such as insufficient product residence times, partial overheating of the solid, poor solid mixing and the inability to use low-temperature secondary heat. The Rolling Bed Dryer combines the advantages of a drum dryer with those of a fluidized-bed dryer in a design that allows gentle and homogeneous drying at low temperatures. It allows waste heat from power stations to be used for drying such materials as wood chips, cropped biomass, sugar beet pulp, trimmings and green waste. — *Almo Process Technology Inc., Liberty Township, Ohio*
www.almoprocess.com

This desiccant is designed for power grid transformers

Drysphere is an activated alumina desiccant designed to remove moisture from transformers in the electrical transmission industry. The company says its Drysphere product absorbs three times more water than any other alumina desiccant product available, promoting efficient operation and extending overall transformer lifetimes. — *Dynamic Adsorbents Inc., Norcross, Ga.*
www.dynamicadsorbents.com

Spraying Systems

Hand-tighten these spray-drying nozzles

The SV Series SprayDry nozzles (photo) eliminates the need for special tools, with a hand-tight design. The company says the nozzles offer comparable performance to more costly swirlchamber-style nozzles. The SV Series nozzles produce a hollow cone spray with uniform drop-size distribution. Designed to resist clogging and improve wear resistance, the SV nozzles can extend production runs. Flowrates from 15.6 to 968 gal/h at 1,000 psi and spray angles from 55 to 90 deg are available. — *Spraying Systems Co., Wheaton, Ill.*
www.spray.com ■

Scott Jenkins

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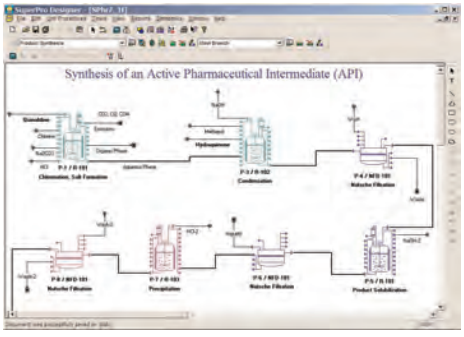

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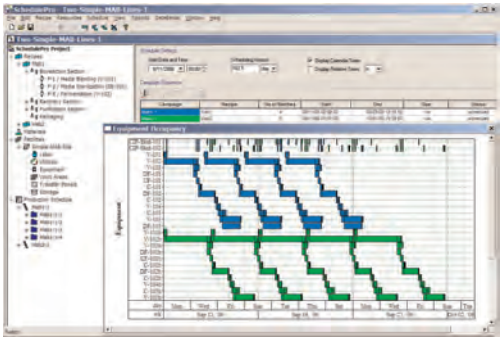

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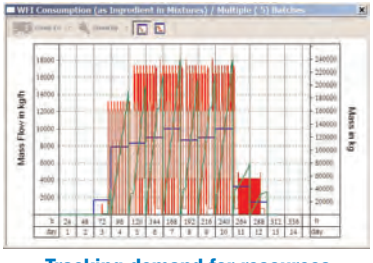
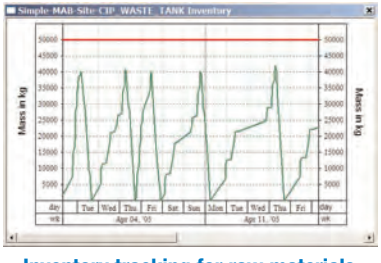
Tracking of equipment occupancy in multi-product facilities



Tracking demand for resources (e.g., labor, materials, utilities, etc.)



Inventory tracking for raw materials, intermediates, products, and wastes

SuperPro Designer is a comprehensive process simulator that facilitates modeling, cost analysis, debottlenecking, cycle time reduction, and environmental impact assessment of biochemical, specialty chemical, pharmaceutical (bulk & fine), food, consumer product, mineral processing, water purification, wastewater treatment, and related processes. Its development was initiated at the Massachusetts Institute of Technology (MIT). SuperPro is already in use at more than 400 companies and 500 universities around the world (including 18 of the top 20 pharmaceutical companies and 9 of the top 10 biopharmaceutical companies).

SchedulePro is a versatile finite capacity scheduling tool that generates feasible production schedules for multi-product facilities that do not violate constraints related to the limited availability of facilities, equipment, resources and work areas. It can be used in conjunction with SuperPro (by importing its recipes) or independently (by creating recipes directly in SchedulePro). Any industry that manufactures multiple products by sharing production lines and resources can benefit from the use of SchedulePro. Engineering companies use it as a modeling tool to size utilities for batch plants, identify equipment requirements, reduce cycle times, and debottleneck facilities.

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58 Materials of Construction
59 Compressors

1	16	31	46	61	76	91	106	121	136	151	166	181	196	211	226	241	256	271	286	301	316	331	346	361	376	391	406	421	436	451	466	481	496	511	526	541	556	571	586
2	17	32	47	62	77	92	107	122	137	152	167	182	197	212	227	242	257	272	287	302	317	332	347	362	377	392	407	422	437	452	467	482	497	512	527	542	557	572	587
3	18	33	48	63	78	93	108	123	138	153	168	183	198	213	228	243	258	273	288	303	318	333	348	363	378	393	408	423	438	453	468	483	498	513	528	543	558	573	588
4	19	34	49	64	79	94	109	124	139	154	169	184	199	214	229	244	259	274	289	304	319	334	349	364	379	394	409	424	439	454	469	484	499	514	529	544	559	574	589
5	20	35	50	65	80	95	110	125	140	155	170	185	200	215	230	245	260	275	290	305	320	335	350	365	380	395	410	425	440	455	470	485	500	515	530	545	560	575	590
6	21	36	51	66	81	96	111	126	141	156	171	186	201	216	231	246	261	276	291	306	321	336	351	366	381	396	411	426	441	456	471	486	501	516	531	546	561	576	591
7	22	37	52	67	82	97	112	127	142	157	172	187	202	217	232	247	262	277	292	307	322	337	352	367	382	397	412	427	442	457	472	487	502	517	532	547	562	577	592
8	23	38	53	68	83	98	113	128	143	158	173	188	203	218	233	248	263	278	293	308	323	338	353	368	383	398	413	428	443	458	473	488	503	518	533	548	563	578	593
9	24	39	54	69	84	99	114	129	144	159	174	189	204	219	234	249	264	279	294	309	324	339	354	369	384	399	414	429	444	459	474	489	504	519	534	549	564	579	594
10	25	40	55	70	85	100	115	130	145	160	175	190	205	220	235	250	265	280	295	310	325	340	355	370	385	400	415	430	445	460	475	490	505	520	535	550	565	580	595
11	26	41	56	71	86	101	116	131	146	161	176	191	206	221	236	251	266	281	296	311	326	341	356	371	386	401	416	431	446	461	476	491	506	521	536	551	566	581	596
12	27	42	57	72	87	102	117	132	147	162	177	192	207	222	237	252	267	282	297	312	327	342	357	372	387	402	417	432	447	462	477	492	507	522	537	552	567	582	597
13	28	43	58	73	88	103	118	133	148	163	178	193	208	223	238	253	268	283	298	313	328	343	358	373	388	403	418	433	448	463	478	493	508	523	538	553	568	583	598
14	29	44	59	74	89	104	119	134	149	164	179	194	209	224	239	254	269	284	299	314	329	344	359	374	389	404	419	434	449	464	479	494	509	524	539	554	569	584	599
15	30	45	60	75	90	105	120	135	150	165	180	195	210	225	240	255	270	285	300	315	330	345	360	375	390	405	420	435	450	465	480	495	510	525	540	555	570	585	600

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BUSINESS NEWS

PLANT WATCH

BASF to explore new production investment in Brazil

March 10, 2011 — BASF is exploring opportunities for a new investment in Brazil for the production of acrylic acid, butyl acrylate and superabsorbent polymers (SAP).The feasibility study will be completed in 2011, after which decisions regarding the plants to be built will be made. In preparation, BASF and Braskem S.A., a major chemical company in Brazil, have signed a memorandum of understanding (MoU) that defines the longterm supply conditions for propylene, which is used as feedstock for the acrylic acid production, as well as the supply of utilities by Braskem to BASF.

Lanxess expands global production for high-tech plastics

March 9, 2011 — Lanxess AG (Leverkusen, Germany; www.lanxess.com) has broken ground on a new compounding facility in Gastonia, N.C., where polymers, such as polyamide and polybutylene terephthalate (PBT), will be mixed and refined with additives.The initial capacity will be 20,000 metric tons (m.t.) per year.The facility represents an initial investment of €10 million. Construction is expected to commence in the 2nd Q of 2011, and production is scheduled to begin in 2012. Additionally, Lanxess and DuPont (Wilmington, Del.; www.dupont.com) will invest an extra €10 million to double the capacity of their joint compounding facility for PBT in Hamm-Uentrop, Germany. Production is scheduled to begin in 2012.

Solvay to double Solef PVDF production capacity

March 3, 2011 — Solvay S.A. (Brussels, Belgium; www.solvay.com) has decided to increase its polyvinylidene fluoride (PVDF) production capacity by 50% in response to the strong growing demand for this fluorinated specialty polymer, which is marketed under the name Solef. The capacity increase at Solvay's Tavaux plant, France, requires an investment of €26 million and will become operational in the 2nd half of 2012.

Oxea extends specialty ester capacity to keep up with growing demand

February 24, 2011 — Oxea GmbH (Oberhausen, Germany; www.oxea-chemicals.com) is expanding its capacity for specialty esters. The capacity of the existing ester unit in Oberhausen will be extended by 40%, effective the 2nd half of 2011. Additionally, Oxea plans to build a new unit in Oberhausen to come online in the 2nd half of 2012.

Outotec to supply oil-shale preparation plant in Estonia

February 18, 2011 — Outotec Oyj (Espoo, Finland; www.outotec.com) has agreed with Eesti Energia, an oil-shale-to-energy company, to design and deliver a new oil-shale preparation plant to be built in Narva, Estonia.The contract value exceeds €20 million. The new shale-oil production plant in Narva is scheduled for completion in April 2012. In the first stage, the oil-shale preparation facility will produce over 4.5 million m.t./yr of crushed oil shale, and it will have the capability for increased capacity in the future.

Chevron invests in lubricants plant in Mississippi

February 2, 2011 — Chevron Corp. (San Ramon, Calif.; www.chevron.com) has announced that Chevron Lubricants will commence construction of a lubricants manufacturing facility at the company's Pascagoula, Miss. location.The facility will manufacture 25,000 bbl/d of premium base oil. Construction is scheduled to be completed by year-end 2013.

AkzoNobel invests €90 million in pulp mill in Brazil

January 24, 2011 — AkzoNobel (Amsterdam, the Netherlands; www.akzonobel.com) is investing close to €90 million in a new facility being built in Brazil.The plant, operated by the company's pulp and paper chemicals business, Eka Chemicals, will supply the world's largest pulp mill.The agreement — with Eldorado Celulose e Papel — represents AkzoNobel's biggest investment ever in Latin America.The 1.5-million ton/yr greenfield mill is expected to come on stream in September 2012.

MERGERS AND ACQUISITIONS

Solazyme and Dow join forces to advance bio-based dielectric insulating fluids

March 10, 2011 — Solazyme, Inc. (San Francisco, Calif.; www.solazyme.com), a renewable-oils and bioproducts company, has executed both a joint development agreement (JDA) and a letter of intent (LOI) with The Dow Chemical Co. (Midland, Mich.; www.dow.com) to advance the development of algal oils for use in next-generation, bio-based dielectric insulating fluids. Under the terms of the JDA, Dow and Solazyme will combine their knowledge and capabilities to develop a new class of algal oils.The non–binding LOI provides that Dow may obtain up to 20-million gal of Solazyme's oils in 2013 and up to 60-million gal in 2015.

BASF plans to sell major parts of its fertilizer activities

March 1, 2011 — BASF SE plans to sell parts of its fertilizers activities, including production plants in Antwerp, Belgium and BASF's 50% share of the joint venture (JV) PEC-Rhin in Ottmarsheim, France.The activities have a total capacity of approximately 2.5 million m.t./yr of fertilizer. BASF plans to complete the transaction by the 1st Q of 2012.

Solvay acquires a Bulgarian fluorspar mine

February 22, 2011 — Solvay SA has acquired a fluorspar mine in Chiprovtsi, Bulgaria from the N&N Group.This acquisition reinforces the integration of Solvay's fluorinated specialty polymers and fluorinated specialty-chemicals production at a competitive cost. The acquisition has been approved by the Bulgarian government.

Altana acquires and integrates Kometra, a polymer modifier producer

January 20, 2011 — The specialty chemicals Group Altana AG (Wesel, Germany; www.altana.com) has signed an agreement to acquire Kometra Kunststoff-Modifikatoren und -Additiv GmbH, which produces polymer modifiers in Schkopau, Saxony-Anhalt, Germany. The company will be integrated into Altana's BYK Additives & Instruments Div.

GE and Shenhua take a step forward for coal-gasification technology

January 19, 2011 — GE (Atlanta, Ga.; www.ge.com) and Shenhua (Beijing, China) have agreed to form an industrial coal-gasification JV in which GE and Shenhua would sell industrial coal-gasification technology licenses, jointly develop integrated gasification combined cycle (IGCC) facilities and conduct R&D to improve commercial-scale gasification and IGCC solutions.This includes industrial coal-gasification applications in China as well as jointly pursuing the deployment of commercial scale IGCC plants.The JV company would be established and commence operation later this year, subject to regulatory approvals. ■

Dorothy Lozowski

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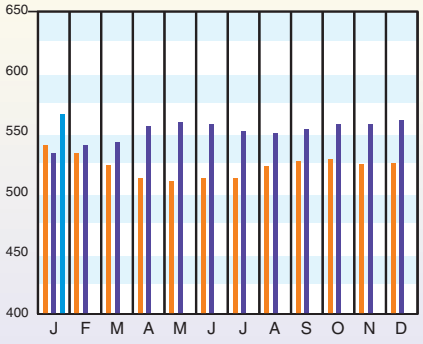
CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	Jan.'11 Prelim.	Dec.'10 Final	Jan.'10 Final
***CE* Index**	564.8	560.3	532.9
Equipment	681.7	674.6	631.8
Heat exchangers & tanks	635.8	627.1	571.9
Process machinery	642.5	627.6	601.9
Pipe, valves & fittings	859.2	854.3	794.5
Process instruments	431.0	426.2	419.8
Pumps & compressors	876.5	903.6	903.0
Electrical equipment	495.2	488.4	469.2
Structural supports & misc	707.4	696.3	640.2
Construction labor	326.7	328.1	331.0
Buildings	505.6	503.3	494.8
Engineering & supervision	334.8	335.6	342.4

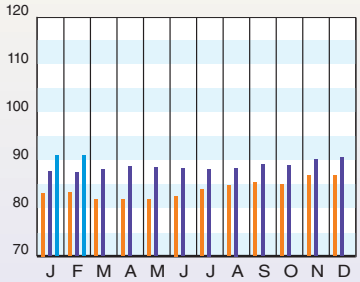
Annual Index:
2003 = 402.0
2004 = 444.2
2005 = 468.2
2006 = 499.6
2007 = 525.4
2008 = 575.4
2009 = 521.9
2010 = 550.8



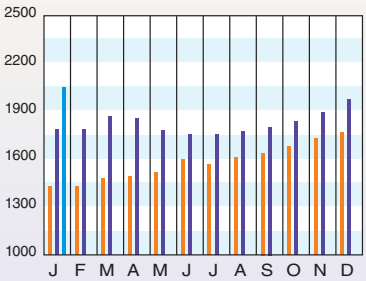
CURRENT BUSINESS INDICATORS

	LATEST			PREVIOUS			YEAR AGO		
CPI output index (2007 = 100)	Feb.'11 =	91.0		Jan.'11 =	91.1		Dec.'10 =	90.6		Feb.'10 =	87.4	
CPI value of output, $ billions	Jan.'11 =	2,050.1		Dec.'10 =	1,974.3		Nov.'10 =	1,893.4		Jan.'10 =	1,786.8	
CPI operating rate, %	Feb.'11 =	73.7		Jan.'11 =	73.8		Dec.'10 =	73.4		Feb.'10 =	70.3	
Producer prices, industrial chemicals (1982 = 100)	Feb.'11 =	304.2		Jan.'11 =	291.4		Dec.'10 =	282.8		Feb.'10 =	269.4	
Industrial Production in Manufacturing (2007=100)	Feb.'11 =	93.5		Jan.'11 =	93.1		Dec.'10 =	92.3		Feb.'10 =	87.5	
Hourly earnings index, chemical & allied products (1992 = 100)	Feb.'11 =	154.8		Jan.'11 =	156.3		Dec.'10 =	154.9		Feb.'10 =	150.4	
Productivity index, chemicals & allied products (1992 = 100)	Feb.'11 =	124.2		Jan.'11 =	124.1		Dec.'10 =	123.4		Feb.'10 =	121.0	

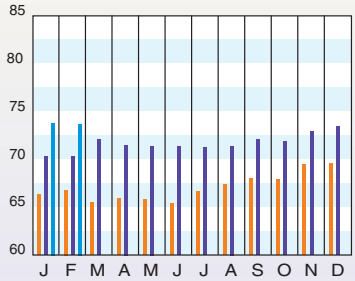
CPI OUTPUT INDEX (2007 = 100)



CPI OUTPUT VALUE ($ BILLIONS)



CPI OPERATING RATE (%)

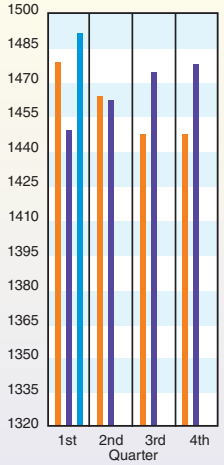


Current Business Indicators provided by Global Insight, Inc., Lexington, Mass.

MARSHALL & SWIFT EQUIPMENT COST INDEX

(1926 = 100)	1st Q 2011	4th Q 2010	3rd Q 2010	2nd Q 2010	1st Q 2010
M & S INDEX	1,490.2	1,476.7	1,473.3	1,461.3	1,448.3
Process industries, average	1,549.8	1,537.0	1,534.4	1,522.1	1,510.3
Cement	1,546.6	1,532.5	1,530.0	1,519.2	1,508.1
Chemicals	1,519.8	1,507.3	1,505.2	1,493.5	1,481.8
Clay products	1,534.9	1,521.4	1,518.3	1,505.6	1,496.0
Glass	1,447.2	1,432.7	1,428.5	1,416.4	1,403.0
Paint	1,560.7	1,545.8	1,542.1	1,527.6	1,515.1
Paper	1,459.4	1,447.6	1,444.5	1,430.1	1,416.4
Petroleum products	1,652.5	1,640.4	1,637.0	1,625.9	1,615.6
Rubber	1,596.2	1,581.5	1,579.3	1,564.2	1,551.0
Related industries					
Electrical power	1,461.2	1,434.9	1,419.2	1,414.0	1,389.6
Mining, milling	1,599.7	1,579.4	1,576.7	1,569.1	1,552.1
Refrigeration	1,827.8	1,809.3	1,804.8	1,786.9	1,772.2
Steam power	1,523.0	1,506.4	1,502.3	1,488.0	1,475.0

Annual Index:
2003 = 1,123.6 **2004 = 1,178.5** **2005 = 1,244.5** **2006 = 1,302.3**
2007 = 1,373.3 **2008 = 1,449.3** **2009 = 1,468.6** **2010 = 1,457.4**



CURRENT TRENDS

Capital equipment prices (as reflected in the *CE* Plant Cost Index; CEPCI) continued their increase from December 2010 to January 2011. With the December numbers now finalized, the Annual CEPCI for 2010 has been calculated at 550.8. The following lists the percent change in the Annual CEPCI over the past eight years, to help put the trends in perspective:

2003–2004	10.5%
2004–2005	5.4%
2005–2006	6.7%
2006–2007	5.2%
2007–2008	9.5%
2008–2009	–9.3%
2009–2010	5.5%

Visit www.che.com/pci for historical data and more on capital cost trends and methodology. ■

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