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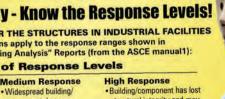
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Cover: David Whitcher

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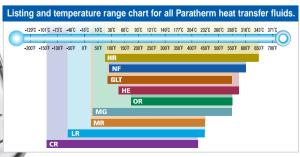
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### **Editor's Page**

### China's playing field levels

or a little over two decades now, China's rise in the chemical process industries (CPI) has often appeared to outsiders like a runaway reaction. Low wages, a very low-valued currency and certain government policies have provided the perfect migration spot for jobs, contracts and intellectual property that had previously been sheltered in developed nations. According to analysts, however, China's wage advantage — arguably one of the most crucial factors in the equation — could evaporate in less than five years.

In fact, according to a recent analysis by The Boston Consulting Group (BCG; Chicago, Ill.; www.bcg.com), the U.S. is expected to experience a manufacturing renaissance as the wage gap with China shrinks and certain U.S. states become some of the cheapest locations for manufacturing in the developed world. "All over China, wages are climbing at 15 to 20% a year because of the supply-and-demand imbalance for skilled labor," says Harold L. Sirkin, a BCG senior partner. "We expect net labor costs for manufacturing in China and the U.S. to converge by around 2015."

After adjustments are made to account for American workers' relatively higher productivity, wage rates in Chinese cities such as Shanghai and Tianjin are expected to be about only 30% cheaper than rates in low-cost U.S. states. And since wage rates account for 20 to 30% of a product's total cost, manufacturing in China will be only 10 to 15% cheaper than in the U.S. — even before inventory and shipping costs are considered. After those costs are factored in, the total cost advantage will drop to single digits or be erased entirely, Sirkin says.

Meanwhile, Chris Kuehl, economic analyst for the Fabricators & Manufacturers Association, Intl. (FMA; Rockford, Ill.) highlights another comparison that brings the issue into an even stronger light. In a recent economic update newsletter, Fabrinomics, he says, "If one looks at the managerial levels and among skilled workers, the rate of Chinese wage growth is about 135% per year; in the U.S., that same group is seeing wage growth of 3.7%. The Chinese pay scale is still far less than in the U.S., but that gap is closing very fast."

Kuehl admits China has made great strides in terms of productivity an improvement by a factor of ten in the last 20 years. Yet, he claims, this still leaves China at one-third of the productivity the U.S. boasts, and the U.S. is seeing productivity gains of almost 8% per year these days.

"The amazing observation from all this is that China is not going to have a cost advantage over the U.S. after 2015," he says. "If, as expected, the Chinese are forced by inflation threats to start pushing the value of their currency higher, the balance could shift pretty quickly. Then there is the potential for much higher transportation costs as the price of oil rises."

Kuehl believes that for both nations, future emphasis will be on the domestic market, and that could well be significant for the U.S. manufacturer in a variety of ways. "If China shifts its attention to its own domestic market and away from exports, it will allow U.S. producers to recapture domestic market share," he says. "As the U.S. manufacturing company looks

to its own market, it will be generally better positioned than the Chinese competitor, as the distribution infrastructure in the U.S. is better suited than China's."

According to Kuehl, most everything in China's transportation network is currently pointed out of the country to service export, and its internal transportation system is often inferior. China will need some infrastructure work to be able to service its domestic markets as effectively as U.S. suppliers are able to service American customers.







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### Letters

### **Controller-based batch systems**

Chris Morse's article in the March issue [Solving Common Batch Problems: Controller-Based Systems, pp. 39-43] makes an excellent case for embedding the batch execution engine in the DCS controller rather than the commonly adopted "legacy" approach of relying on a PC-based batch execution server. [...] As postulated by Mr. Morse, our customers have, in fact, benefitted from absolute batch execution security, scalability, availability, reduced cycle times, ease of engineering and maintenance, integrated operator interface, modularity, flexibility, and enhanced batch reporting and analysis.

On the other hand, I would dispute Mr. Morse's implication that batch manufacturers' recent competitive pressures have drastically changed to suddenly require this "new" embedded architecture. I offer a few examples to support my point:

- The stated benefits within the ISA S88 standard itself are unchanged between the 1995 and 2010 versions. These include reduced time to production, reduced batch automation cost, straightforward recipe development, and reduced lifecycle engineering efforts
- Presentations from the early years of WBF [World Batch Foruml (late 1990s) tout the same manufacturers' needs and automation benefits - reduction in implementation time and costs, reduced cycle time, less plant idle time, better data and understanding, predictable time to introduce new products, better schedule optimization, modularity, flexibility and so on
- · A review of Functional Design documents from our own group's late 1990s projects reiterates the same project objectives and economic justifications — secure recipe execution, reduced cycle time, centralized and integrated operator interface, reduced operating delays, flexibility, better utilization of process equipment, enhanced reporting, and batch data archiving, among others

All these are the same as the "new" requirements that Mr. Morse cites. Instead, I would argue that the needs have been there all along. [...]

**Dave Christie** 

Engineering Manager, Yokogawa Atlanta Engineering Center, Batch Center of Excellence, Newnan, Ga.

### **Correction**

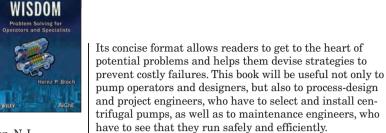
April 2011 Newsfront — Shedding Light on Microreactors, pp. 17-20: On p. 19, middle column, the dimensions given for Ehrfeld-BTS's Miprowa system are incorrect. Line 4 should read: "The laboratory unit has channel dimensions of 1.5 mm x 12 mm, which enable the kinetics of the reaction to be understood. In the pilot unit, the reactor has channel sizes (3 mm x 18 mm) that are small enough to maintain the advantages of microchannels, but sufficiently large for increased production volumes and reduce fouling problems." The online version of the article and the downloadable pdf file of the article have been corrected.

### **Bookshelf**

Pump Wisdom: Problem Solving for Operators and Specialists. By Heinz P. Bloch. John Wiley & Sons Inc., 111 River Street, Hoboken, NJ 07030. Web: wiley. com. 2011, 224 pages, \$49,95.

Reviewed by Stanley S. Grossel, Process safety and design consultant, Clifton, N.J.

espite their ubiquity in the chemical process industries (CPI), process pumps fail catastrophically each year in large numbers. The author, a world-recognized expert on pumps and other process equipment, has written this book to explain some elusive failure causes and to provide permanent remedial actions to avoid or minimize such failures. In the preface to this excellent book, the author states that he intended to write a book that would squeeze material to enhance both pump safety and reliability into 200 or so pages. To date, some of this material has been too widely dispersed to be readily accessible, and some important material has never been published before. In my opinion, the author has achieved his goals. The book provides valuable knowledge based on the author's years of experience in the petroleum and chemical industries on techniques for stabilizing centrifugal pump performance and maximizing pump efficiency.



Topics covered in the book's early chapters include fundamental principles of centrifugal process pumps, including pump performance, operation at zero flow, impellers and rotors, as well as centrifugal pump types and pump mechanical response to flow changes. Early on, the book also offers a succinct review of pump selection and industry standards, including ANSI and ISO versus API pumps, and includes a wealth of practical information designed to help minimize problems. Among the topics found are piping design and selection of stationary seals and gaskets.

Subsequent chapters describe practices to ensure reliable operation of pump bearings, lubricant application and cooling considerations in good detail. Topics considered include lubricant viscosities, bearing housing protection and cost justification. There is also a good overview of mechanical sealing options for long life.

Later chapters present pump startup, surveillance and shutdown procedures, as well as impeller modifications



### **Bookshelf**

and pump and lubricant maintenance. The book offers a good review of vibration, including its causes, monitoring methods, effect on bearing life, and acceptance limits, and also discusses often-overlooked pump issues, such as drivers, couplings and alignment.

For a complete list of topics in each of the book's 16 chapters, view the online version of this article (www.che.com).

Plasma-Assisted Surface Coating. By Georg Erkens and others. Sulzer Metaplas GmbH, Zentralniederlassung, Am Böttcherberg 30-38, 51427 Bergisch Gladbach, Ger-

many. Web: sulzermetco.com. 2011. 72

pages. \$12.42.

TRIZ for Engineers: Enabling Inventive Problem Solving. By Karen Gadd. John Wiley & Sons Inc., 111 River Street, Hoboken, NJ 07030. Web: wiley.com. 2011. 504 pages. \$75.00.

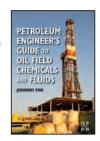
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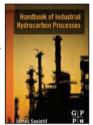
house Gas Mitigation. By Ian S.F. Jones. Cambridge University Press, The Edinburgh Building, Shaftesbury Road, Cambridge CB2 8RU, U.K. Web: cambridge.org. 2011. 184 pages. \$110.00

Petroleum Engineer's Guide to Oil Field Chemicals and Fluids. By Johannes Fink. Elsevier Inc., 30 Corporate Drive, 4th floor, Burlington, MA 01803. Web: elsevier.com, 2011, 808 pages. \$139.95.

Handbook of Industrial Hydrocarbon Processes. By James Speight. Elsevier Inc., 30 Corporate Drive, 4th floor, Burlington, MA 01803. Web: elsevier.com. 2010. 602 pages. \$150.00.

Renewable Raw Materials: New Feedstocks for the Chemical Industry. Edited by Roland Ulber, Deiter Sell and Thomas Hirth. John Wiley & Sons Inc., 111 River Street, Hoboken, NJ 07030. Web: wiley.com. 2011. 244 pages. \$180.00.





Membrane Process Design Using Residue Curve Maps. By Mark Peters and others. John Wiley & Sons Inc., 111 River Street, Hoboken, NJ 07030. Web: wiley.com. 2011. 264 pages. \$135.00. ■

Scott Jenkins

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### CHEMENTATOR

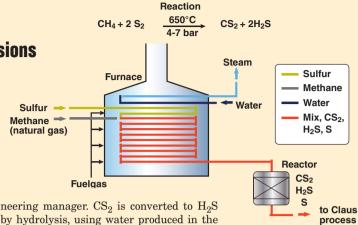
Edited by Gerald Ondrey July 2011

### An innovative way to recover elemental sulfur from SO<sub>2</sub> emissions

Sulfur dioxide emissions from metals smelters and other process plants are typically treated by converting the SO<sub>2</sub> to sulfuric acid, but this may not be convenient, because of the shipping cost for plants located far from an acid market. WorleyParsons (Monrovia, Calif.; www.worleyparsons. com) offers a solution to this problem with a process that converts SO<sub>2</sub> to elemental sulfur, which is easier and safer to ship.

WorleyParsons' process works with a pure  $SO_2$  stream that is extracted from the offgases by an amine-based concentrator. After that, the process has two steps, both of which use conventional technologies. In the first step, methane is reacted with liquid sulfur in an externally fired tube heater at about 650°C to obtain hydrogen sulfide and carbon disulfide (see figure). The  $H_2S$  is then reacted with the  $SO_2$  stream to produce elemental sulfur by the Claus process.

The Claus reaction takes place over two or three alumina-filled beds, says Helmy Andrawis, vice-president and process engi-



neering manager. CS<sub>2</sub> is converted to H<sub>2</sub>S by hydrolysis, using water produced in the process. "The only piece missing (compared to a standard Claus plant) is the reaction furnace to make SO<sub>2</sub>," says Andrawis. "We don't need that because we already have SO<sub>2</sub> from the smelter."

Andrawis adds that the process converts more than 95% of the total sulfur in the gas to 98%-pure sulfur. WorleyParsons has prepared designs for a commercial-sized plant under a contract with Norilsk Nickel (Moscow, Russia), he says, and is now bidding to build the plant.

### Process for renewably sourced polyols licensed

Aprocess for making polyols from soybean hoil and glycerin has been licensed by its developer, Battelle (Columbus, Ohio; www. battelle.org), to Emery Oleochemicals Americas (Cincinnati, Ohio; www.emeryoleo.com), who intends to produce the renewably sourced polyols at prices similar to those of petroleum-based polyols. As multifunctional alcohols, polyols are widely used in industry, especially for making polyurethane foams and coatings, as well as polyesters.

The Battelle-Emery process depends on an ozonolysis reaction of the carbon-carbon double bonds in soybean oil to produce a molozonide — a reactive, cyclic intermediate with three oxygen atoms and two carbons. Glycerin is then added to effect a ring-opening polymerization reaction that gives rise to polyols of various molecular weights. "We can control the molecular weights and —OH content of the polyols by adding different amounts of glycerin," says Battelle market manager Rick Heggs. The robust Battelle technology emerged out of efforts to find new uses for soybean-based products. The process also provides a way to use glycerin, an under-

utilized byproduct from biodiesel manufacturing. "It's more or less a one-pot reaction," says Heggs, and works without the need for pretreatment steps or product separation.

Emery's involvement in the project owes much to the fact that the company has had success handling ozone as a chemical reagent, and has learned how to generate the gas cost-effectively. With their renewable polyols, Emery will target the markets for rigid polyurethane insulation and liquid polyurethane coatings, where the renewably-sourced polyols can serve as drop-in replacements for petroleum-based products, as well as the market for flexible polyurethane foam, where the soybean-based polyols can replace about 25–40% of petroleum-derived materials.

Although the licensed technology was developed for soybean oil, Emery can potentially use other triglycerides as feedstocks. The company has been granted exclusive rights to the Battelle technology for North America, South America and Europe. Heggs said Battelle is working on a possible licensing agreement with another company for Asia.

### Waste-to-biofuels

Valero Energy Corp. (San Antonio, Tex.; www.valero. com) is one of five companies that have invested a total of \$60 million in new financing in Enerkem Inc. (Montreal. Que.; www.enerkem.com), which has developed a process to produce liquid fuels and chemicals from municipal solid waste. Valero, the largest independent petroleum refiner in the U.S., has made a number of investments in biofuels technology and operates ten corn ethanol plants, which make it the largest ethanol producer among U.S. refiners.

In Enerkem's process, shredded garbage is gasified and converted sequentially to methanol, acetate and ethanol. The company is building a commercial-scale plant in Edmonton, Alta. Scheduled for startup in 2012, it will convert 100,000 m.t./yr of garbage into 10 million gal/yr of methanol. A similar plant is being built in Pontotoc, Miss., with startup set for 2013. (For more details on Enerkem's process, see CE, October 2010, p. 14).

### Beet sugars yields PX

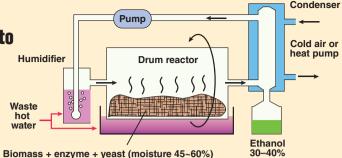
In an extension of its reforming process for producing liquid fuels from plant sugars, Virent Energy Systems, Inc. (Madison, Wisc.; www.Virent.

(Continues on p. 12)

### A continuous process that promises to slash the cost of making bioethanol

Acompact, inexpensive and energy-saving continuous process to make bioethanol is being developed by professor Yoshio Katakura at Kansai University (Osaka; http://microbial.life-bio.kansai-u.ac.jp), in collaboration with the Kinki Bio-industry Development Organization (Osaka), Kansai Chemical Engineering Co. (Amagasaki), and Akita National College of Industry (Akita, all Japan). The so-called CCSSF process (consolidated, continuous, solid-state fermentation) uses simple equipment that can easily be installed next to a biomass-treatment facility.

In the CCSSF system (diagram), a slurry (50 wt.% water) of biomass, diastatic enzymes and yeast is continuously fermented in a rotating drum. The ethanol vapor generated in the headspace of the drum is continuously removed and



condensed as a 30–40% aqueous solution. In laboratory trials, a 93% ethanol yield has been observed (87 g ethanol from 165 g of starch). The researchers have also demonstrated that cellulose-based raw materials, such as cotton fibers and recycled waste-paper, can be used as raw materials.

Unlike conventional processes, CCSSF does not require separate units for performing saccharification and fermentation, and only compostable residues are produced, thus eliminating the wastewater treatment unit. As a result, investment

costs for a CCSSF system are expected to be significantly lower than conventional ethanol plants. Katakura estimates that the system is capable of producing ethanol for less than \$100/L (\$1/L).

With support of about ¥200 million (\$2 million) from Japan's Ministry of the Environment, a 40-L demonstration unit is now being designed, with startup slated for 2013. This system will process five tons of raw material (dry) and be capable of producing 750,000 L of ethanol from 1,500 tons of starch-based raw materials.

### This one-step route to cellulosic ethanol features a multitasking yeast

The research group of professor Akihiko Kondo at Kobe University (Kobe City, http://www2.kobe-u. ac.jp/~akondo/index\_English.htm) has developed a one-step, pretreatment process for making bioethanol from cellulose and lignocellulose. The process uses an ionic liquid — which converts cellulose into a gel — and a genetically engineered yeast (arming yeast) that incorporates multiple enzymes at the cell surface for breaking down cellu-

lose into fermentable sugars.

The pretreatment process takes place at a lower (100°C) temperature than that of conventional hydrolysis (which uses dilute sulfuric acid at a temperature of around 200°C) and does not have to deal with waste associated with the acid treatment. When the arming yeast is added to the gel, the enzymes decompose the cellulose into sugars, which are then fermented — by the same yeast — into ethanol.

In laboratory trials, Kondo demonstrated that the arming yeast can survive in the presence of the ionic liquid, and that the method works for cellulose as well as bagasse, the lignin-containing waste from processing sugarcane. The researchers are now optimizing the process and seeking to lower the temperature even further with different ionic liquids. The one-step process is expected to "drastically" reduce the costs of ethanol production.

### Waelz slag used in commercial ceramic bricks

Researchers from the University of Cantabria (Santander, Spain; www. unican.es) have developed a process for incorporating Waelz slag into ceramic clay construction bricks, offering an alternative for disposal of the industrial byproduct. The engineers replaced between 20 and 30% of clay with slag, and found that the inclusion at that level did not have any significant detrimental effect on the physical, mechanical or chemical properties of the ceramic bricks. Incorporation of slag at levels higher than 30% begins to result in more significant effects on the brick properties.

The Waelz process is a commercial technology used to recover volatile metals from electric-arc-furnace (EAF) dust. It generates a product mixture of ZnO and PbO, as well as slag, the nonvolatile component of the dust, consisting mostly of iron oxide. Landfilling the slag can represent an environmental burden and can mean disposal costs in some areas. In addition to providing a way to manage waste material, the partial substitution of the clay with waste slag "also contributes to reducing the depletion of natural resources, minimizing production costs and providing a way to manage waste material," the research team says. The real push for incorporating industrial byproducts into construction materials would come with high disposal fees, adds researcher Guillermo San Miguel.

To produce the bricks, the engineers pre-treat the slag to reduce particle size and homogenize particle size distribution. Using industrial-scale equipment, the slag is then mixed with the clay mix and water, then extruded to its final brick shape. The encapsulation of heavy metals by the ceramic greatly reduces leaching, compared to using the slag as a filler material in civil engineering applications, such as roads.

### A catalyst line with low or zero rare-earth metals

Grace Davison (Columbia, Md.; www. grace.com) has commercialized a set of catalysts and additives for fluid catalytic cracking (FCC) that contain either zero or low rare-earth metal content. The new product family, known as Replacer, includes new catalysts for processing both hydrotreated and residual-fuel-oil (resid) feed, as well as environmental additives for sulfur treatment.

The product family employs the company's proprietary zeolites and incorporates new stabilization methods that allow the products to achieve similar performance to existing FCC technologies without the need for rare-earth metals, whose limited and uncertain supply makes them costly.

The new catalysts are the result of an effort over the past three years by Grace Davison to apply its experience in rareearth-free catalysts from the 1980s and 1990s to modern catalyst systems. "The overall R&D timeline was quick, and market willingness to trial new catalysts has also accelerated," said Shawn Abrams, vice president and general manager for Grace Davison Refining Technologies.

Most of the 25+ applications using

these new catalysts are in low-metals and hydrotreated feed applications, but Grace Davison has also developed a product for high-severity applications. called ResidUltra, that has 40% less rare-earth metal than benchmark resid catalysts. "The real challenge is to sustain activity and selectivity in resid processing," said Abrams.

The new catalysts have been tested by third parties and their performance has been confirmed by petroleum refiners in multiple full-scale commercial applications.

### A glass flow reactor is now available in ceramic

ast month, Corning SAS (Avon, France), a subsidiary of Corning Inc. (Corning, N.Y.; www.corning.com), introduced the Advanced-Flow G4 Ceramic Reactor — the latest addition to the company's line of glass flow reactors. The G4 is made of sintered-ceramic fluidic modules based on the design of the Advanced-Flow G1, G2 and G3 predecessors, which

are made of glass. The similar design of the fluidic modules eases scaleup, with a throughput factor increase of more than 60 when going from the Advanced-Flow G1 through the high-flow G4.

The G4 reactor has an internal volume of up to 6 L and has a processing capacity of more than 300 kg/h. The ceramic construction of the G4 - developed together with Mersen/Boostec (Paris; www.mersen.com) — has a "superior" thermal conductivity. Modules are connected together in a stainlesssteel frame with Perlast gaskets, and can operate at temperatures from -25 to 200°C, and pressures up to 18 barg in the process path (up to 6 barg in the heat exchange path).

### Finland becomes the home of a large biogasification plant

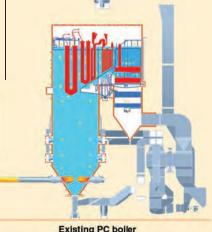
Metso (Helsinki, Finland; www.metso. com) is supplying a 140-MW<sub>th</sub> biogasification plant to Vaskiluodon Voima Oy in Vaasa, Finland. When it starts up in December 2012, the €40-million gasification plant, which will be located next to the existing 565-MW coalfired power plant, will convert biomass (mainly forest residues) into a so-called biogas that can be combusted with coal. The gasification facility will enable Vaskiluodon Voima to replace 25-40% of the coal it now uses with renewable energy, thereby reducing CO<sub>2</sub> emissions by about 230,000 ton/yr.

At the Vaasa plant, wet, pre-processed biomass is first dried on a large belt dryer and then fed to an air-blown, circulating-fluidized-bed (CFB) gasifier. The 140-MWth CFB gasifier - said to be the world's largest — operates at around 850°C and close to atmospheric pressure, and gasifies the dried biomass into biogas — a mixture of carbon monoxide, hydrogen, methane (and other hydrocarbons), carbon dioxide, water and nitrogen - with a heating value of around 6 MJ/Nm3.

"Metso's new biogasification technology, including biomass drying, offers a new, cost-effective alternative for large coal-fired plants to increase the share of biomass and reduce the proportion of coal and emissions," says Juhani Isaksson, product manager for Metso. Although coal plants can also directly

**Dried biomass** 

co-fire with some biomass, the incorporation of the drying step broadens the diversity of biomass that can be used. The net efficiency of the power plant is only reduced by a fraction of a percent due to the trace moisture remaining after drying, says Metso.



Wet biomass

Biomass receiving

and pre-handling

Large-scale belt dryer wire drying plant

CFB gasifier 140 MW<sub>fuel</sub>

**Existing PC boiler** 

its commercial debut

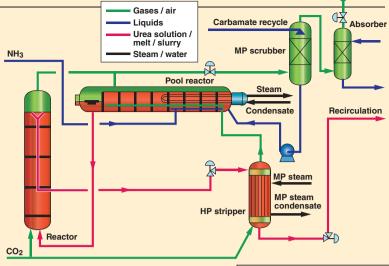
CHEMENTATOR

Stamicarbon B.V. (Sittard, the Netherlands; www.stamicarbon.com), the licensing and IP Center of Maire Tecnimont S.p.A. (www. mairetecnimont.it), has recently signed a license agreement with Tierra del Fuego Energia Y Quimica S.A., an Argentina based company controlled by shareholders from

China. The agreement concerns a urea synthesis plant and a urea granulation plant with a capacity of 2,700 metric tons per day to be built in southern Argentina.

The plants will be located near the city Rio Grande in Tierra del Fuego. Stamicarbon will deliver the process design package, the proprietary high-pressure equipment, and associated services for both the synthesis and the granulation plant. The plants will be built by Chengda Engineering Corp. of China (Chengda; www.chengda.com). Start up is planned for 2013.

The urea synthesis plant will use the Stamicarbon Avancore urea technology, marking the commercial debut for this process. Avancore (flowsheet) is a further development of Stamicarbon's Urea 2000plus technology, and reduces the required plant height to just 22 m — even for large-scale plants — thereby "considerably" reducing the investment costs. According to Stamicarbon, Avancore incorporates major improvements in the core urea technology that have



led to the elimination of several traditional equipment items (and the associated costs), including the following:

- Use of Safurex stainless steel an improved duplex steel developed with Sandvik Materials Technology (Sandviken, Sweden; www.smt.sandvik.com) the air supply for passivation is no longer required
- The absence of oxygen means there is no need to combust hydrogen, so the H<sub>2</sub> converter is eliminated and the synthesis section has become intrinsically safe with respect to explosion risks
- With the quantity of inert gas substantially reduced, less gas has to be vented from the urea synthesis section, so the high-pressure (HP) scrubber can be replaced by a medium-pressure (MP) scrubber

 $(Continued\ from\ p.\ 9)$ 

com) has produced *para*-xylene (PX) from beet sugar. Virent says the catalytic process converts plant-based sugars into PX molecules that are identical to those made from petroleum (For details on Virent's technology see *CE*, May 2010, p. 11).

### A new flowmeter

Fluid Components International LLC (FCI; San Marcos, Calif.; www.fluid-components. com) recently introduced its ST100 Series — the first thermal mass flowmeter to meet new Foundation Fieldbus (FF) H1 standards. Besides FF H1, the flowmeter can quickly be configured to other protocols, including Profibus PA and Modbus, thanks to a plug-in bus-communication card. The device also supports analog HART version 7.

Designed for monitoring gas flow in various applications, the ST100 features a backlighted readout that simultaneously displays flowrate, totalized flow, temperature and pressure. The display also has a writable field where operators can type in an identification term, and four through-glass, touch-activated buttons with extra functionality. The ST100 measures flow with an accuracy of ±0.75% of reading, and is the only flowmeter in its class to offer an option for a pressure transducer, allowing measurement of temperature, pressure and flow with the same probe.

### Magnetic-shape-memory alloy offers alternative to traditional actuators

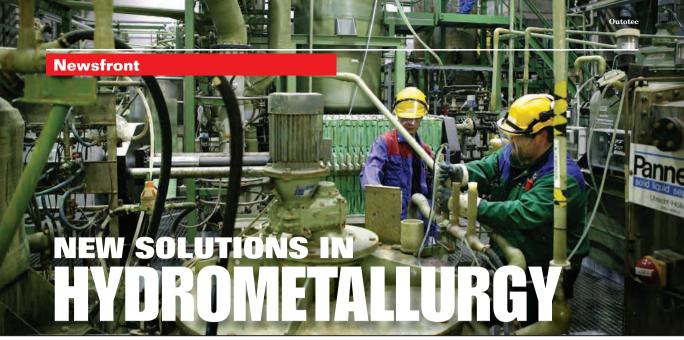
Anovel nickel-manganese-gallium alloy, introduced by Goodfellow Corp. (Oakdale, Pa.; www.goodfellowusa.com), elongates in a magnetic field, allowing the conversion of magnetic field energy into kinetic energy. The material offers an alternative to conventional actuators, and could find other uses in energy harvesting systems, breaker switches, sensors and others.

Grown as a single crystal using a modified Bridgman technique, the NiMnGa alloy has a crystal microstructure that realigns in a magnetic field of less than 0.8 T to produce growth in one direction. Proprietary modifications to the Bridgman method, which is used for single-crystal growth of some semiconductor materials, were necessary in order to maximize the material's performance.

The NiMnGa alloy exhibits greater strain outputs (magnitude of growth in the desired

direction) than piezoelectric and magnetostrictive materials, whose changes are typically in the range of 0.1–0.2% of their original length. The alloy typically lengthens between 3 and 5%, with up to 6% possible, and also changes with greater speed—cycle times of 1–2 kHz are observed. The magnetic field response "is faster and more efficient than a traditional temperature-induced response," says Goodfellow, and the elongation is "fully reversible," by applying a magnetic field at right angles to the original or by using a return spring.

The crystals, composed of 50% Ni, 28% Mn and 22% Ga, are offered in three standard sizes with a length of 20 mm, and varying thicknesses (1 or 2 mm) and widths (2.5 or 5 mm). The company, which has sold the material for research use already, also will make crystals with custom dimensions.



### Developing alternative process technology is not just nice, but necessary

FIGURE 1. Outotec's HydroCopper process is undergoing comprehensive testing at this plant at Pori, near Helsinki, Finland

s demand for basic metals grows, engineers and scientists are looking for new ways to extract these elements from ores. "The easy stuff has already been found and processed. One of the main challenges of mining and minerals processing companies is now the economical processing of low-grade ores", says Steve Rogers, managing director of the Parker Center for Integrated Metallurgy Solutions (Perth, Western Australia; www.parkercentre.com.au).

Rogers says other challenges include water and energy use, and the increasingly stringent requirements regarding the environmental performance of mining and processing activities.

Minerals processing companies are looking at flowsheets that can use poorer quality water, such as tailings wastewater or saline-process source waters, he says. For example, copper has been traditionally obtained from chalcopyrite (CuFeS $_2$ ) — the main copper ore — by smelting. Most copper is still recovered from chalcopyrite ores by smelting. However, smelting is energy-intensive and entails high capital costs and long construction times. Smelters can also emit considerable amounts of  $\mathrm{SO}_2$  gas — a main cause of acid rain.

Therefore, one of today's main challenges is to develop commercially

viable chalcopyrite-leaching technologies and this is a key objective of many mining and minerals processing companies.

Currently about 20% of world copper is produced from large-scale heap leaching of low-grade copper oxide ores followed by solvent extraction (SX) and electrowinning (EW). A small number of plants treat secondary copper sulfides via pressure leaching, followed by SX-EW.

To help meet the challenges of the minerals industry, the Parker Center undertakes research with the cooperation of CSIRO (Melbourne, Australia; www.csiro.au) and several universities. Its main projects include: developing a viable heap bioleaching process for treating low-grade chalcopyrite ores; controlling ferrihydrite precipitation in the zinc and nickel industries; and developing new technologies for the economic processing of nickel laterite ores.

### Tackling nickel laterite ores

Most nickel has been traditionally obtained from nickel sulfide ores, even though nickel laterite ores are the most abundant. However, technical challenges have so far delayed the exploitation of laterite ores. As the reserves of sulfide ores dwindled, companies have focused on developing

technologies for the economical exploitation of laterite ore reserves.

The companies involved with minerals processing have responded vigorously to those challenges and have developed many novel technologies, especially leaching technologies. Several of those technologies have undergone extensive testing and are now beginning commercial operation or are on the verge of commercialization.

A novel atmospheric leaching process for copper concentrates, called Galvanox, is offered by Bateman Engineering N.V. (Amsterdam, the Netherlands; www.bateman.com), which has its engineering center in Johannesburg, South Africa. Galvanox is a galvanically-assisted atmospheric leaching of primary copper concentrates originally developed by researchers David Dixon and Alain Tshilombo of the University of British Columbia. Canada. Bateman has obtained a license from the university to implement the technology (for flowsheet, see online version of this article).

Chalcopyrite is commonly associated with pyrite (FeS<sub>2</sub>). The enhancement of chalcopyrite leaching rate, when in contact with pyrite, has been attributed to galvanic interaction between chalcopyrite and pyrite.

Pyrite acts as the cathodic site for oxygen and ferric ion reduction reac-

tions, while chalcopyrite acts as the anode and is, therefore, preferentially dissolved.

Linus Sylwestrzak, leaching technology specialist with Bateman Engineering, says Galvanox does not require fine grinding, generates elemental sulfur and has low oxygen demand, requires no surfactants, can

cost-effectively treat low grade concentrates down to 9% copper, leads to complete copper recovery — typically in less than 12 h — and is fully compatible with conventional SX-EW.

While the Galvanox process has not yet been commercialized, a process involving high pressure and temperature autoclaves, known as Total Pressure Oxidation (TPOX), has been successfully commercialized by Free-port-McMoRan Copper & Gold Inc. (Phoenix, Ariz.; www.fmi.com).

The company's plant, at Bagdad, Ariz. — initially a chalcopyrite concentrate pressure demonstration plant capable of producing 40-million lb/yr of copper — has been converted into a molybdenum concentrate leaching plant able to produce high-grade molybdic oxide.

The company says the Bagdad plant was the first in the world to operate a medium-temperature pressure leaching process that minimizes acid production.

The TPOX process is followed by conventional SX-EW techniques. While the cost of TPOX is relatively high, high copper recoveries, better than 99%, are achievable.

### Different approaches

Quite a different technology, a chloride-based atmospheric leaching process called HydroCopper, is used by Outotec Oyj (Espoo, Finland; www. outotec.com) to leach the copper of chalcopyrite and other sulfides. The process has not yet been commercialized, but is undergoing comprehensive testing at the Pori site, a research center (Figure 1) and demonstration plant in Finland, says Outotec's metallurgist, Liisa Haavanlammi.

The HydroCopper process (Figure 2) has a flexible modular flowsheet that can be tailored to different kinds of copper concentrates. This allows optimizing the process from mine to metal and extending mine life to lower cut-off grades. When smelter concentrate cannot be cost-effectively produced, HydroCopper may prove a feasible alternative.

The process allows chalcopyrite and other copper sulfides to be effectively leached under atmospheric pressure at a temperature of 80–100°C in a strong, aggressive chloride solution using Cu<sup>+2</sup> ions as the oxidant.

Copper is precipitated from the purified leach solution as Cu<sup>+1</sup> oxide, which is reduced by hydrogen gas to metallic copper powder, and then melted and cast into copper product such as wire rod, bars or billets.

The process can effectively handle



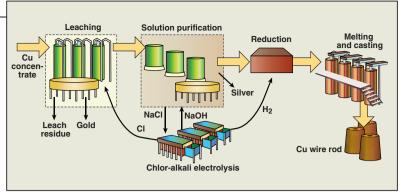


FIGURE 2. Outotec's HydroCopper process has a modular flowsheet that can be tailored to different kinds of copper concentrates

impurities, such as arsenic and mercury, which can be harmful in pyrometallurgical processes. According to Outotec, the process has been designed to meet the strictest directives for emissions.

Alternative technologies, which are said to be especially effective on high carbonate ores, have been developed by MetaLeach Ltd. (London, U.K.; www.metaleach.com), a wholly owned subsidiary of Alexander Mining plc. The company owns the intellectual property to two ambient temperature, ambient pressure, hydrometallurgical technologies, AmmLeach and HyperLeach.

The AmmLeach process utilizes ammonia-based chemistry for the extraction of base metals, especially copper, nickel, zinc and cobalt from ore deposits and concentrates.

The primary difference between AmmLeach and acid leaching is that the leaching is conducted in a moderately alkaline solution, which allows using AmmLeach on high carbonate ores where acid consumption would be prohibitive. Also, in the AmmLeach process, undesirable metals and other impurities are either insoluble or significantly suppressed, with considerable capital and operating cost savings, says the company.

The process has an extremely high selectivity for the target metal over iron and manganese, which are insoluble under AmmLeach conditions.

The company said it has developed a new process for the solvent extraction of zinc from ammoniacal solutions. It said testing has shown that zinc can be efficiently extracted using commercially available reagents in a single stage and stripped with acid solutions, with greater selectivity than has previously been reported.

Targets for the AmmLeach process include nickel laterite ores, gold/copper oxides and silver/zinc oxides, and molybdenum oxides.

Alexander Mining's CEO, Martin Rosser, says the company is conducting a demonstration project for the extraction of copper in the Argentine northwestern province of Salta. The company will now look at the next stage of commercialization of the process.

The company is also marketing its Hyperleach process, which is a chlorine based process that does not require  $\mathrm{Cl}_2$  gas to operate. The oxidant can be generated on-site via industry standard chlor-alkali technology. The process operates at ambient temperature and pressure and is suitable for heap leaching as well as tank leaching. It leaches base metal sulfides including chalcopyrite, bornite, chalcocite, millerite, and enargite.

The MetaLeach processes are still in the demonstration stage, but a copper and nickel extraction process, by CESL Ltd. (Richmond, B.C., Canada; www.cesl.com), part of the Technology Division of Teck Resources Co. (Vancouver, B.C.), is already in operation.

The first commercial hydrometallurgical facility using CESL technology is now operating in the Carajás region of Brazil (Figures 3 and 4). It is a 10,000 metric ton (m.t.) per year copper plant built by Vale S.A. (Rio de Janeiro, Brazil).

The CESL copper process consists of four main steps: copper mineral oxidation, copper leaching from the oxidation residue, solvent extraction to purify the copper leach liquor, and electrowinning to recover copper in a commercial product form.

The process uses existing technologies but combines them in a novel way. It involves oxidation of sulfide concen-

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trates at elevated pressure and temperature in the presence of catalytic chloride ions.

Impurity metals, such as nickel, cobalt and zinc are also oxidized during the process. The leach filter cake, containing oxidized copper, hematite and elemental sulfur, is repulped with recycled raffinate from solvent extrac-

tion. Impurities are removed from the copper-rich solution by solvent extraction. The purified solution is then electrowon, producing copper cathodes of LME Grade A standards.

The CESL nickel process also begins with a pressure oxidation step. Complete dissolution of the nickel, copper, cobalt and zinc occurs within the autoclave. Impurity metals are removed from the nickel solution by precipitation. The cobalt can be recovered from the solution through a purification stage. Nickel is precipitated as a hydroxide or sulfide, which may be processed further to metal or sold as an intermediate product. If metallic nickel is to be produced, ammonium sulfate is used to leach nickel from the intermediate product. The resulting nickel electrolyte is electrowon to produce nickel cathodes.

The company says the process produces no gaseous emissions and there are no significant liquid effluents. Solid byproducts are environmentally stable leach residues and gypsum, plus a minor amount of precipitated impurities such as magnesium and zinc, it says. A CESL plant can be located on-site.

Current work on the company's nickel processing technology focuses on the production of separate nickel and cobalt intermediate products using a novel solvent extraction process. Flowsheet enhancement work is underway to evaluate different processing methods for producing nickel metal from various intermediates.

### Simplicity

The many innovations that appeared during the past few years range from complex flowsheet development to simple, yet ingenious improvements, often resulting in lower energy requirements and cleaner operation.

An example is a simple, yet novel approach to pressure oxidation leaching called the Activox process, marketed by Norilsk Nickel (Moscow, Russia; www.nornik.ru). The process is a combination of fine grinding and pressure oxidation, resulting in milder operating conditions.

Conventional pressure oxidation operates at temperatures exceeding 200°C with overpressures of 2,200 kPa or more.

Activox is a form of pressure oxidation that operates at temperatures of about 100°C and pressures of 1,000 kPa. The milder operating conditions simplify the engineering requirements and reduce costs, yet maintain the advantages of pressure oxidation, says the company.



Vale S.A.



FIGURE 3. Shown here is the pressure oxidation autoclave at Vale SA's plant in the Carajás region of Brazil. The plant is the first commercial hydrometallurgical plant using CESL Ltd's technology

The process breaks down sulfide minerals by reacting them with oxygen to produce discharge slurry consisting of a solid residue and liquor. The company has developed downstream flowsheets for concentrates containing nickel, cobalt and copper, using well established solvent extraction technology.

The Activox process has been demonstrated in several pilot plants. The latest pilot plant was the Tati nickel plant in Botswana, in southern Africa.

Fine grinding is also at the basis of the Albion process, developed by MIM Holdings, now Xstrata Plc. (London, U.K.; www.xstratatech.com).

The process, intended to treat concentrates produced from refractory base and precious metal ores, consists of a hot oxidative leach of finely ground concentrates at atmospheric pressure. The company says the development of the process paralleled the development of new fine grinding mills. This has enabled leaching to be carried out under far less demanding conditions than previously required in pressure or bacterial leach plants.

The process was recently commissioned at Xstrata Zinc's Nordenham zinc demonstration plant in Germany, which commenced operation in January this year. Designed to treat 40,000 ton/yr of concentrate, it has

been able to treat up to 56,000 ton/yr of concentrate. The company said it is conducting a feasibility study to expand its Albion plant to produce 150,000 ton/yr of concentrate, with a view to commissioning the expansion in late 2013.

The company has also been operating a demonstration plant for the Albion process at the San Juan de Neiva zinc refinery in Spain, which started up in July of last year. It is designed to treat 9,000 ton/yr of concentrate to produce 4,000 ton/yr of metal. Xstrata Zinc is studying the option of treating 220,000 ton/yr of concentrate in an expanded Albion plant, to produce 100,000 ton/yr of zinc metal, with the intention of reducing operating costs and energy consumption. The expansion is planned to be commissioned in early 2014.

Both plants treat zinc/lead bulk concentrate from the McArthur River Mine in Australia. The concentrate is ground in a bank of M3000 IsaMills at the mine prior to transport.

A third Albion plant — for gold production — is scheduled to be commissioned in December this year at the Las Lagunas tailings-treatment project in the Dominican Republic. The plant is designed to produce 65,000 oz/yr of gold and 600,000 oz/yr of silver. An M3000 IsaMill is installed on gifts

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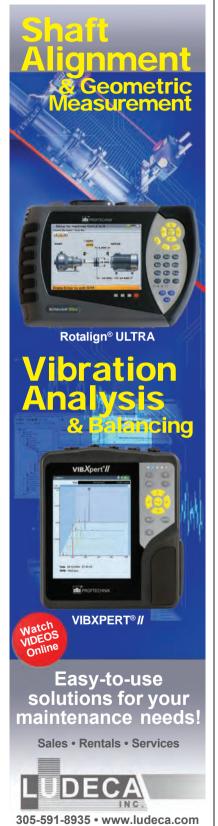
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FIGURE 4.
A view of the atmospheric I eaching process at Vale's plant at Caraiás



Tackling the hard stuff

Since the "easy stuff" has already been found and processed, the industry has been increasingly turning to the not-so-easy stuff, including nickel laterite ores. These have been the main target of Direct Nickel Pty Ltd (Sydney, Australia; www.directnickel. com). The company's hydrometallurgical process to treat nickel laterite ores will be tested at CSIRO's minerals processing facility at Waterford in Perth.

It involves tank leaching at atmospheric pressure and moderate temperature. The process uses nitric acid, instead of the sulfuric acid commonly employed for treating nickel laterite ores. The nitric acid is continuously recycled. Nitric acid consumption is about 30 kg/m.t. of feed material, versus 300–1,000 kg/m.t. for sulfuric acid-based leaching. Extraction efficiencies are about 95% of nickel and 85% of cobalt.

Direct Nickel says alternative processes are uneconomic when the magnesium content of the ore reaches 3%, whereas there is no upper limit for the its process. It says operating and capital costs are about half those of existing processes.

### Biohydrometallurgy

While all the technologies described above involve chemical and mechanical processes, a new field — biohydrometallurgy, including bioleaching — has opened up, involving the use of bacteria and other microorganisms to do part of the job.

For example, technologies for bioleaching sulfide ores in an engineering heap environment have been developed by GeoBiotics LLC (Lakewood, Colo.; www.geobiotics.com). The company's two main technologies are Geocat and Geoleach.

The company says Geocoat combines the low capital and operating costs of heap leaching with the high recoveries associated with other processes, such as roasting, pressure leaching, or stirred-tank biooxidation. The process is applicable to refractory sulfide gold concentrates and to sulfidic copper, nickel, cobalt, zinc, and polymetallic base metal concentrates.

The Geoleach process uses iron- and sulfur-oxidizing microorganisms to facilitate the oxidation and leaching of sulfide minerals. The organisms include mesophile bacteria (*Thiobacillus ferrooxidans*, *Thiobacillus thiooxidans*, and *Leptospirillum ferrooxidans*) and moderate and high temperature thermophillic bacteria, such as the *Archaea Sulfolobus* and *Acidianus*.

### The outlook

The present challenges faced by the minerals processing industry have stimulated the development of much good technology and science. Many companies and organizations around the world are doing research on issues including the following:

- Using microorganisms to develop a heap bioleaching process for treating low-grade chalcopyrite ores
- Controlling ferrihydrite precipitation in the zinc and nickel industries
- Developing solvent extraction systems for the recovery of copper, nickel, zinc and cobalt from leach solutions with a high chloride concentration
- Understanding the factors governing the rates of corrosion of lead-based alloys as anodes in the electrowinning of copper, nickel and zinc

These research efforts aim to help overcome current roadblocks in the minerals industry, and help exploit hitherto untapped mineral deposits.

Paul Grad

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# ABSORBENTS FOR SPILLS

While clay products have reliably been used to handle spills, modern absorbents often prove more labor-, time- and cost-efficient alternatives

or years, clay has been the go-to product for spill containment simply because it was cheap and did the job. While clay, also referred to as diatomaceous earth, sand or kitty litter, will always have a place in in spill containment, if it's the only product being used to clean up hazardous and other spills, it could be costing your facility more than you realize. With more modern options available including everything from polypropylene to super-absorbent polymers to corn cobs and cotton - experts suggest a clean-up plan including more than one type of absorbent is the most cost-effective and safest way to go.

"A lot of people in processing are old school and think clay is best way to clean up a spill. But anyone still exclusively using clay for spill containment is a fool," says Lenny Johnson, president of Chemtex, Inc. (Cumberland, R.I.) "Clay has a purpose and place in spill clean up, but it should be used in conjunction with other products to do the job in a way that cuts down on labor and disposal costs in cleaning up a spill — clay is not the only method in existence."

### What's available

The spill containment industry is now host to numerous products, such as melt-blown polymer, recycled cotton and high-tech super-absorbent polymer products. While each material has its own set of characteristics and price range, almost all have advantages over clay-based products.

For example, polymeric products, like those available from New Pig (Tipton, Pa.) and Chemtex, are lighter in weight and more absorbent than clay. "Typical clay-based absorbents aren't really absorbent," says Chris Iuzzolino, product manager for absorbents and spill control product lines with New Pig. "Materials like oil will coat only the outer surface of the clay, which means you need more product to

clean up a spill, so they aren't really efficient. Polypropylene is really absorbent, so you use less product when cleaning a spill, which means there is less to dispose of on the back end."

Johnson of Chemtex believes that despite the lower upfront cost of clay, it actually costs two times more than polymeric products would in a real-life spill containment scenario. He points out that the main difference between clay and melt-blown polymer is that clay absorbs 0.5 to 1 times its weight, while the non-woven polymeric product his company offers absorbs between 12 to 25 times its weight, depending on the viscosity of the liquid. "If you have a chemical spill that requires 10 bags of clay, you would likely only need one bale of pads to absorb the same spill," he says.

Using this scenario, he explains that to move 10 bags of clay at 40 pounds each would require someone with a forklift. Conversely, one person could lift the 15-pound bale of pads and bring it to the spill site. In addi-







FIGURE 1. Hazardous lagoon sludge is treated with super-absorbent polymers, rendering it a solidified substance that is easier to remove from the site

tion, you would need multiple workers in protective gear with shovels and brooms to lay down the clay and then scoop and sweep it. One person could do the same job without the shovels and brooms, just by putting the pads on the spill, and disposing of them with a gloved hand into a drum. He adds that it is likely cheaper to dispose of the contaminated pads, as well. He estimates that 40 bags of contaminated clay would fill at least two drums, while the single bale of contaminated pads would require just one drum, meaning the facility would also save on disposal costs if those costs were incurred either by weight or number of drums.

Sellars Co. (Milwaukee, Wis.) offers absorbent products made from 80% recycled cellulose fibers that are suitable for cleaning up most leaks, drips and spills. Its Preferred pad has been proven in independent laboratory tests to absorb as much as two of the leading competitor's heavy-weight melt blown pads, which means it would

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### Newsfront

### **HOW TO CLEAN UP A SPILL**

- Assess the risk. Before you attempt to clean up a spill, make sure you are taking into
  account everything that might be affected by that spill. Look at the risks to human
  health, the environment and property. If possible, identify the spilled material and
  determine how much has spilled. Use the container's label or MSDS (material safety
  data sheet) to identify the liquid and the primary dangers posed to spill responders
  and the environment
- Select personal protective equipment (PPE). Choose the appropriate PPE to safely respond to the spill. This could include goggles, gloves or respirators. Check the MSDA and literature on the chemical spilled for the best PPE recommendations. If you're uncertain of the danger or the spilled material is unknown, assume the worst and use the highest level of protection
- Confine the spill. Limit the spill area by blocking, diverting or confining the spill. Stop
  the spread of liquid before it has a chance to contaminate a water source. Spill kits
  are designed to facilitate a quick, effective response. Non-absorbent barriers such
  as containment booms, drain covers and spill berms are available to confine liquid,
  minimize spill area and protect drains.
- Stop the source. After the spill is confined, stop the source of the spill. This may simply involve turning a container upright, closing a valve or plugging a leak from a damaged drum or container. Be sure to transfer liquids from the damaged container to a new one
- Evaluate the incident and implement clean up. Once the spill is confined and the source has been stopped, it is time to develop a plan of action for implementing the spill clean up. Place absorbents that are chemically compatible with the liquid spilled through the spill area. Once the absorbents are saturated, they should be properly disposed. Sorbents do not render liquid non-flammable, neutral or less hazardous and will take on the characteristics and properties of whatever liquid is absorbed. Therefore, all measures must be taken as if you were handling the liquid itself. Always refer to the MSDS for the chemical absorbed before proceeding
- Decontaminate. Decontaminate the site, personnel and equipment by removing or neutralizing the hazardous materials that have accumulated during the spill. This may involve removing and disposing the contaminated media, such as earth, that were exposed during the spill incident. An effective decontamination area ensures the health and safety of emergency responders.
- and safety of emergency responders

   Complete required reports. Complete all notifications and reporting required by local, state and Federal guidelines for reporting spill incidents. Failure to do so can result in severe penalties. Necessary reports can include: medical record reports, company incident reports, Local Emergency Planning Commission reports or National Responsible Center Reports

(Courtesy of Chemtex, Inc.)

pick up many times over what could be achieved using clay. "Users spend less time picking up saturated pads and rolls with the Preferred line of absorbents," says Mike Radovich, vice president of sales with Sellars. The products are made from recycled, 100% natural cotton grown in the U.S. and are "incredibly absorbent if left untreated." In addition, he says, "We are able to keep our costs down because these recycled paper products aren't part of the global market. Unlike oil-derived polymeric products, our pads haven't take a price increase in five years."

Then there are granular products such as Spill King (Palm Beach Gardens, Fla.) absorbent material, which is a non-toxic, non-flammable, environmentally safe and lightweight amorphous silica product. The substance can absorb all materials except hydrofluoric acid, leaving little or no residue. "Less than half a pound of Spill King can completely absorb one quart of motor oil without leaving any residue to re-clean," says Bob Irving, president. "Clay, earth and sand products require 15 lb of material to absorb the same amount and would leave a slippery residue."

Again, the benefits here include the upfront savings of using less product to capture a spill, which results in greatly reduced disposal costs on the back end. "This absorbent will save money on shipping, storing and disposal. Its light weight and high absorbency will make it easier to handle," notes Irving. "Clay, earth and sand products come in 40 to



FIGURE 2. Absorbent products made from 80% recycled cellulose fibers are suitable for cleaning up most leaks, drips and spills. The cotton-based products are capable of absorbing many times more than traditional materials



FIGURE 3. Polypropylene products are lighter in weight and more absorbent than clay-based materials, often making them easier and less expensive to use and dispose of in the long run

### **ABSORBENT AND SPILL KIT PROVIDERS**

Chemtex www.chemtexinc.com M2 Polymer Technologies

www.m2polymer.com

New Pig www.newpig.com Northwest HazMat

www.nwhazmet.com Sellars www.sellarscompany.com Spill Kina www.spillking.com

50 lb bags, while one Spill King bag at 10 lb equals 3 of bags of clay product."

And then there are the super absorbent polymers. M2 Polymer Technologies, Inc. (West Dundee, Ill.) offers Waste Lock products, which are a class of cross-linked, non-biodegradable polymers capable of absorbing and retaining up to 500 times their weight in water. "Waste Lock products are widely used in the environmental industry to treat many types of aqueous wastes," says Martin Matushek, owner of the company. "The advantage of these water-swellable super absorbent polymers is that they can absorb many times their weight in water with nominal or negligible increase in waste volume or weight.

"I've gone into meetings with this stuff with people who were dead set against spending what we charge for the absorbent, thrown a handful into their coffee cup and asked them to quickly calculate their labor savings and walked out with them buying it," says Matushek. "It has its advantages even though it seems expensive up front. Yes, it costs \$2/lb, but you may only need 10,000 lb of this, versus material that you could buy for \$0.40/ lb, but would need 80,000 lb. In addition, clay and mineral absorbents would take an hour to process a spill, but ours [super-absorbent polymers] takes 10 min. And then there are always the disposal cost savings you get when disposing of a fraction of what you would dispose of with clay."

### The right combination

While the proposed cost savings seem substantial, they shouldn't be the only determining factor in product selection. "The problem with absorbents is that there is no one-size-fits-all product," says Matushek. For this reason you need to consider several things when deciding what products to use in your facility.



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### **WHAT MAKES A SPILL HAZARDOUS?**

According to Spill King, a spill is generally considered hazardous waste if it contains any of the following characteristics:

- Ignitable: A flash point of less than 140°F (60°C)
- Corrosive: A pH of less than or equal to 2.0, or greater than or equal to 12.5, or corrodes steel at a rate greater than 6.35 mm per year at 55°C
- Reactive: Unstable, reacts violently with water; is sufficiently cyanide or sulfide bearing
  to produce toxic gas; is capable of detonations or forms potentially explosive mixtures
  with water
- Toxic: If the material tests positive and is above a certain concentration for 39 contaminants that the EPA has listed in Federal regulations (for example, certain pesticides, benzene, heavy metals, such as lead or mercury) and halogenated organic substances, such as pentrachloro phenol and trichloroethylene

New Pig



FIGURE 4. Knowing what types of liquids you're dealing with will clue you in to the types of absorbents or spill kits you need on hand

Iuzzolino suggests the first consideration is the liquids stored in the facility. "Knowing what types of liquids you're dealing with will clue you in to the types of absorbents or spill kits you need on hand. Many are designed to absorb certain types of liquids or might not be chemically compatible with what you have," he says.

The second criteria should be quantities. "We tell customers to plan for the worst case scenario," he notes. "If you have a 5,000-gal tank, but have a spill kit for 30 gal, you're not really prepared."

Finally, the chosen product must adhere to local, state and federal regulations regarding disposal. "If a customer sends waste to an incinerator, they want an absorbent with a high BTU value and low ash content," Iuzzolino says. "Or if you're sending material to a permitted, hazardous waste landfill that only accepts non-biodegradable absorbents, that is a very important consideration."

It's important to make the distinction between biodegradable or non-biodegradable absorbents. Although the absorbent materials themselves aren't hazardous, once they absorb something, they must be treated and disposed of based on the material they now contain. So depending on how the liquid is classified, the saturated

absorbent will have to go to either a hazardous or non-hazardous waste landfill, depending on regulatory classification. Usually hazardous waste landfills require non-biodegradable absorbents so they don't break down in the landfill and release the toxin, while biodegradable absorbents are preferable for non-hazardous material because they create less waste in the landfill.

Even with all this taken into consideration, it's important to note that nothing is perfect. As Jim Potts, director of operations with Northwest HazMat (Springfield, Or.), which both cleans up spills and sells spill kits, puts it, "There's always a trade off when selecting absorbents. Some materials are cheaper to buy, but are heavier to put down, pick up and dispose of. We generally use what works fastest and does the best job."

And often, a one-two punch of absorbents is needed, according to the experts. "There will always be a need for clay-based products because not all surfaces are perfectly flat," says Radovich. "There are cracks and crevices in most surfaces and pads tend to not get into those areas, so follow up cleaning may be needed, and that will likely include clay-type products. But still, using a more absorbent and lighter material on the majority of the spill will reduce labor and disposal costs, even if a follow up with clay products is required at the end."

Johnson agrees: "We are never going to do away with clay, but if the clean up uses pads or some other material for 90% of the spill and clay to get the rest, then instead of using and disposing of 50 bags of clay, you can get by with maybe five bags of clay and one bale of pads. Everyone concerned with limiting costs and having a proper spill plan should have multiple products on hand because you never know what a job will entail."

Joy LePree

### When troubleshooting the extractor

have an ongoing dispute with a longtime colleague, Bob Miller. I said he ruined my new safety boots. He said he didn't.

Bob was a young, fearless distillation engineer at a time when our extraction engineer refused to take a trip to Japan to inspect new trays going into an old extractor. I called Bob into my office and asked him to take the trip, even with zero extraction experience. Bob accepted the assignment, but worried that he had no safety footwear. I offered him my brand new safety boots. I would have given him all of my shoes if that is what it took to keep me out of that old, dirty extractor.

When Bob came back from the installation he handed me the boots without blinking an eye. They looked like they had been run over by an industrial lawnmower. He insisted that that is what they looked like when I loaned them to him. I disagreed. His tray installation had gone well — that is what mattered most.

About two weeks later, Bob gave a slide presentation to the entire distillation/extraction group, describing the installation. Half of the slides were photographs of the feet of a young, fearless distillation engineer standing on trays inside the column. The early photographs show bright and shiny boots; the later photographs showed the boots deteriorating quickly. Bob had given me the proof that I needed — I had indeed given him good-as-new boots.

Just a few days later, on a Friday afternoon, my supervisor came into my office stating, "Our Japanese sales office says that your extractor is not working. You need to catch a flight tomorrow." I looked high and low for Bob. He was nowhere to be found. I decided that I needed to take this trip myself. Troubleshoots sometimes last weeks, or years. My flight to Japan was fraught with anxiety. The chardonnay tasted like glycol. I arrived at the refinery on a Monday afternoon. The production engineer led me into the control room. The noise in there was almost deafening. It was the butterflies fluttering in my stomach.

I looked over the control board. The

unit was running very smoothly, but, it was running at the old feed and solvent rate. I asked their production engineer, "What happens when you increase the rates?" He said, "We don't know." I said, "You don't know?" Then he replied, "Column pressure drop is too high." To that, I said, "These trays are designed to operate at high pressure drops. Just go ahead and increase the rates." "Really?" he asked. "Really," I answered.

The next day when I arrived in the control room, the column was purring at the as-designed rates, which were about 20% higher than the old rates. All of the control-room engineers and operators were beaming. My review of the control board showed that all was well — very well. That night, the plant manager took us all to a long, expensive dinner, rife with sake, karaoke and toasts to the extractors.

The first moral of this story: We all get stuck in paradigms. The control room personnel had convinced themselves that the extractor was incapable of functioning above a certain pressure drop. In Bob's words, "Sometimes after a column revamp, the operators and technicians need to be retrained." The second moral of this story: Don't loan Miller your safety boots.

For an excellent how-to article on tower inspections, see pp. 44–48.

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





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### **FACTS AT YOUR FINGERTIPS**

Department Editor: Scott Jenkins

as-liquid reactors in the chemical process industries (CPI) have increasingly been designed to handle larger manufacturing scales. Since gas-liquid reactors can represent substantial capital and operating costs for the user, optimizing mixing and maximizing productivity are critical. The need for efficiency at larger scales places more importance on understanding the physical phenomena of mixing and more of a burden on equipment design.

### Physical demands of mixing

A number of complex physical phenomena must be considered to achieve optimal function of mixing equipment in cases where gaseous and liquid substances interact. For a gas-liquid reaction to occur, a low-density compressible gas must be dispersed into a much denser liquid with a reasonably long contact time. Usually, significant turbulence must be induced into the liquid phase to aid mass transfer and reaction. In addition, rapid movement of the liquid phase is often required at heat-transfer surfaces, which are often removed by some distance from mixing impellers. In some cases, the liquid phase can contain a significant level of solids, which must be kept suspended.

Gas-liquid reactors commonly consist of large pressure vessels with sophisticated internal components for gas feed and exhaust, liquid feed and outlet, heat-transfer and baf-

fling, as well as agitation.
The two major categories of gas-liquid reactions are those with a "pure" feed gas, and those in which the gas contains a significant fraction of inert gases in addition to the reactant. For a gas-liquid reaction to take place, a molecule of gas must dissolve in the liquid phase and then meet a molecule of the reactant. A catalyst material is often present, in which case both reactants must meet on the active site of a catalyst.

The catalyst's high specific surface area means that the reaction is usually limited by transport of the gas through the boundary layer around the gas bubbles and into the liquid phase (Figure 1). The specific rate of mass transfer through the boundary layer is governed by the standard mass-transfer equation:

$$m = k L a \left( c^* - c \right) \tag{1}$$

where c is the actual concentration of dissolved gas and  $c^*$  is the theoretical equilibrium concentration of dissolved gas.

The film mass transfer coefficient  $(k_l)$  is mainly a function of the physical properties of the reactants, and is less sensitive to mixing conditions. The specific surface area (a) also depends on material properties, but can be significantly increased by changes to process design. These two factors are usually expressed together as a specific mass-transfer capacity  $(k_l a)$ , since it is difficult to measure either one directly.

The term  $(c^* - c)$  in Equation (1) is sensitive to changes in process design in that influences can be designed to increase the theoretical equilibrium concentration of dissolved gas  $(c^*)$ .

According to Henry's law, the value of  $\dot{c}^*$  is proportional to the partial pressure of the reactive gas. So the use of a reactant gas in a pure form raises process efficiency, but the presence of volatile solvents reduces it. Increasing reaction pressure can boost productivity, but higher pressures also mean higher operating and capital investment costs. The target for the

design engineer is to optimize  $k_L a$  and operating pressure to achieve the required productivity at the lowest cost.

### Gases with inert components

Many industrial gas-liquid processes involve reactant gases diluted with significant amounts of inert gases. This includes all processes using air (21% oxygen in nitrogen) or fluegas (carbon dioxide or sulfur dioxide in nitrogen), in which the feed gases are used on a once-through basis and then discharged.

In cases where gases contain inerts, paying particular attention to the reaction stoichiometry is very important. Here is a list of considerations, using air as an example:

- The mass transfer rate is very sensitive to the concentration driving force (c\* c). The equilibrium concentration is proportional to the oxygen partial pressure, which is reduced by the dilution with nitrogen to about one-fifth
- 100% consumption of the oxygen from air is not possible, so reactors must operate with stochiometric excess. Exhaust from industrial oxidation processes, for example, typically contain around 4–15% residual O<sub>2</sub>
- O<sub>2</sub> partial pressure in the dispersed gas phase changes as the O<sub>2</sub> is consumed, and this must be taken into account in reactor design
- Mass transfer cannot be increased by recirculating gas from the headspace, since the headspace gas concentration is depleted
- Since large amounts of inert gas are present, a stoichiometric excess of reactant is required; often high gas rates result, and flooding of impellers is possible
- Practically, the loss in mass transfer capacity generally requires larger reactors, and large reactors require particular attention to maintaining the homogeneity of the mixture

Impellers for high gas rates

Traditionally, for reactors that have high gas rates, combinations of impellers, such as flat-blade disc turbines (FBDTs), pitch-blade turbines (PBTs) and wide-foil impellers are used. More recently, impellers with concave-shapes are increasingly being used (Figure 2). With these impellers, the gas feed can be dispersed into the liquid phase using a radially pumping primary disperser (PD). One or more secondary dispersers (SD) can also be used on the same shaft, but higher up in the liquid to provide a combination of axial blending of the liquid and redispersion of the gas (Figure 3). The left diagram shows a compartmentalized

### Gas-Liquid Mixing: Physical Considerations

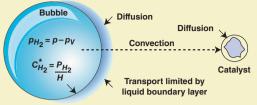


FIGURE 1. Gas-liquid reactions are limited by transport of the gas into the liquid phase

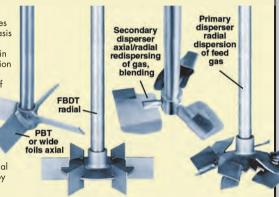


FIGURE 2. Impellers with concave shapes are used to help initiate movement of the liquid axially

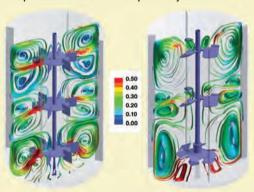


FIGURE 3. Flat-blade disc turbines produce compartmentalized flow (left), while primary and secondary dispersers redisperse the gas (right)

flow pattern with FBDTs. The right side shows axially extended vortices with the SD. The two vortices created by the FBDT, one above and one below the impeller, "roll" over one another, while The PD/SD system generates significant material exchange in the axial direction. This reduces the required blend time.

When there are significant quantities of dispersed gas present, traditional impellers rapidly lose power, whereas the concaveblade impellers of the PD/SD type are much less affected.

### Reference

 Himmelsbach, W., Kellar, W. Lovallo, M., Grebe, T. and Houlton, D. Increase Productivity Through Better Gas-Liquid Mixing. Chem. Eng., October 2007, pp. 50–58.

### **People**







Thibodeaux



Wakeham



Rucklow



Bloemhar

John Derbyshire becomes president of **KBR Technology** (Houston).

Doug Keith is promoted to president of the drive technologies division of **Siemens Industry** (Atlanta, Ga.).

Travis Thibodeaux becomes director of operations support for Multi-Chem (Houston), a provider of oilfield production and support chemicals.

ITT Corp. (White Plains, N.Y.) appoints *Colin Sabol* to lead strategy and business development for the standalone, publicly traded water company it will spin off later this year. He is currently responsible for strategy and business development for ITT's fluid and motion control group.

Robert Hansen becomes CEO of **Dow** Corning (Midland, Mich.).

Sir William Wakeham becomes president of **IChemE** (London). Wakeham was knighted in 2009 by Queen Elizabeth II in recognition of his service to the field of chemical engineering.

**Protexic Brands** (Arlington Heights, Ill.), a subsidiary of **Tegrant Corp.** (DeKalb, Ill.), promotes *Bill Bucklew* to vice president of engineering.

Jeroen Bloemhard is named business vice president and global executive director of **Dow Corning**'s Xiameter brand (Midland, Mich.).

PSE, Inc. (London), a provider of process modeling software and services, appoints *Dale Curtis Jr.* president of its Americas operation.

Suzanne Shelley



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### **FOCUS ON**

Valves and Actuators

### Modular valve construction allows for custom solutions

The Entech DRV-BN non-slam nozzle check valve (photo) has a modularity that allows for customized solutions, as well as in-field reconfiguration and a consistent spring force mechanism for quick and easy maintenance. The DRV-BN is a large-bore addition to this company's line of check valves for preventing backflow. The DRV-BN is suitable for applications where minimal pressure losses are essential, such as compressor stations and gas facilities, pump and wellhead injection plants, water distribution and treatment, and tank storage. — Cameron, Houston www.c-a-m.com

### An actuator for safety and emergency applications

The Skilmatic (photo) is a self-contained, electrohydraulic valve actuator that is designed for safety-related applications, including emergency shutdown duty. A major design feature of the actuator is its combination of electrical operation, hydraulic actuation and mechanical failsafe motion. The Skilmatic also has an advanced control and monitoring system. — Rotork Fluid Systems, Bath, U.K.

www.rotork.com

### A pressure-reducing valve for high-purity applications

Series PRH-U valves (photo) are new pressure-reducing valves that use thermoplastic materials that are selected specifically for industries where ultrahigh purity is required, such as the pharmaceutical and semiconductor industries. Constructed of a machined Kynar 740 PVDF (polyvinylidene fluoride) body, the PRH-U valves have metal-ion-free EPDM (ethylene propylene diene monomer) seals to achieve high purity in a fused piping system. In addition to using ultrapure materials, the PRH-U valves are prepared with a threestep cleaning process that includes a proprietary pre-assembly cleanroom treatment, a post-assembly hot-and-cold rinse with deionized water and a final class-100 Plast-O-Matic Valves

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Cedar Grove, N.J.

### This ball valve is an alternative to metal

The PE100 ball valve (photo, p. 27) is suitable for gas, sewer and water applications where metal ball valves have typically been installed. The thermoplastic valve is 50% lighter than comparable metal valves, and is available in 1–6-in. sizes. The new PE100 features a two-step planetary gear system to ensure smooth opening and closing, and eliminate water hammer. The PE100 is suitable for belowground service, and can be installed using butt, socket or electro-fusion technologies. — Asahi America Inc., Malden, Mass.

procedure. The valves regu-

late downstream pressure from 10-125

psi, with a maximum inlet pressure of

150 psi. — Plast-O-Matic Valves Inc..

www.asahi-america.com

### Rotary airlocks that are built for long service life

These rotary airlocks (photo) are designed for extended life, with features that include heavy-duty casting, precisely machined rotors with larger shaft diameters to minimize deflection and ANSI-rated flanges to resist distortion. The airlocks are available in sizes from 4 to 24 in. and come in carbon steel and stainless steel. They can be equipped with various drive packages. — *Cyclonaire Corp.*, York, Neb.

www.cyclonaire.com

### Eliminate sewer backflow with this check valve

The CheckMate inline check valve (photo) is designed for backflow prevention and odor mitigation in combined-sewer and sanitary-sewer overflow and stormwater



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applications. The valve features an elastomer-fabric-reinforced design that allows maintenance-free performance and allows significant cost savings. — Red Valve Co., Tideflex Technologies Div., Carnegie, Pa.

www.redvalve.com

### This large ball valve is 'actuation-ready'

The TB Series True Union Ball Valve (photo) incorporates an actuation-mounting pad that meets the requirements of the ISO 5211 standard, and an integral panel mount foot pad. With sizes ranging from 2.5 to 6 in., the TB Series features pressure ratings to 235 psi, double O-ring stem seals, low torque ratings and reversible PTFE seals. The new version of the TB Series is compatible with earlier versions of TB valves of the same diameter

and end connections. — Hayward Flow Control, Clemmons, N.C.

www.haywardflowcontrol.com

### This valve is designed for dust collection

This company's new DDV Dust-Duty Valve (photo) comes with a robust cast housing designed for greater capacity, and is said to be an ideal solution for dust collection applications. It is suitable for applications such as baghouses, cyclones and dust collectors with a maximum pressure differential of 60 in. of water column. Available in cast iron or stainless steel with square or round flanges from 6 to 14 in., the DDV can be used in environments with temperatures up to 300°F. — Wm. W. Meyer & Sons Inc., Libertyville, Ill.

www.wmwmeyer.com

### A metered scale is on the valve body

This company's needle-pinch valve has a metered scale on the valve body. Marked with 1-mm divisions, the molded-in scale allows calibrations to be recorded and replicated. Valve installation does not require fittings, cutting or removal of existing tubing, the company says. Fluid contact with the valve has been eliminated in the newly updated design, so the valve can be used, reused and disposed of without contamination. Constructed of Delrin acetal resin, the needle-pinch valves are intended for soft-wall tubing with O.D. from 1.0 to 13.0 mm. — Flow-Rite Controls Ltd., Byron Center, Mich.

www.flow-rite.com

(Continues on p. 56)

# A Clearer View Of Crystallizers

# Improved understanding of crystallizer equipment and operation can aid purification efforts in the CPI

### Wayne Genck

Genck International

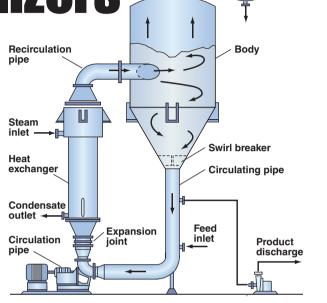
rystallization is a key purification technique for various sectors of the chemical process industries (CPI). Several approaches for industrial crystallization have evolved over time, and highly specialized crystallizer designs have been developed, especially in long-established industries. Solution crystallization is an important unit operation because the process can generate high-purity products from solutions containing significant levels of impurities with relatively low energy input.

This article is intended to expand knowledge of the various crystallizer configurations, including forced circulation (FC) and draft-tube-baffle (DTB) configurations, which will be discussed in detail.

### Classification of crystallizers

Two schemes have been employed to classify crystallization equipment. One uses the method of generating supersaturation, while the other scheme uses the method by which the growing crystals are suspended.

There are six primary methods for generating supersaturation: evaporation, cooling, adiabatic-flash (vacuum) cooling, chemical reaction, antisolvent addition and pH adjustment. However, classification according to the method of generating supersaturation is not entirely satisfactory, since different



water inlet

**FIGURE 1.** In a forced-circulation (FC) crystallizer, cooling at the liquid surface results in supersaturation that is relieved by crystal growth or the birth of new nuclei. High circulation rates enable evaporation of solutions with scaling solutes

means of generating supersaturation may be employed within the same type of equipment. When classifying according to the method of suspending the growing crystals, crystallizers fall into one of four basic types:

1) Mixed-suspension, mixed product removal (MSMPR) crystallizers. Also sometimes called circulating magna crystallizers, this type of equipment circulates the growing crystals through the zone of the crystallizer where the supersaturation conditions are generated. This may be accompanied by mixed- or classified-product removal, and with or without destruction of fines.

2) Circulating liquor/classifiedsuspension crystallizers. In this type, only the liquor or a weak slurry is circulated, while the bulk of the growing crystals are not circulated. Supersaturation is imparted to the liquor in one part of the equipment, whereupon this liquor is circulated to another

area where it relieves the supersaturation on growing crystals. This type of crystallizer is also available with or without fines destruction capabilities. The units are usually identified as Krystal- or Oslo-type crystallizers.

Non-condensable

gas outlet

Barometric condenser

3) Scraped-surface crystallizers. Crystallization is induced by indirect heat exchange with a cooling medium at the heat-transfer surface, which is continuously scraped and agitated to minimize fouling. This type of equipment employs vertical tanks with scrapers or horizontal pipes.

4) Tank crystallizers. Crystallization is produced by cooling the feed solution in either static or agitated tanks by natural convection and radiation, by surface cooling through coils or a jacket, programmed evaporative cooling, reaction or antisolvent methods.

### MSMPR crystallizers

The term MSMPR carries the assumption that a perfectly uniform product

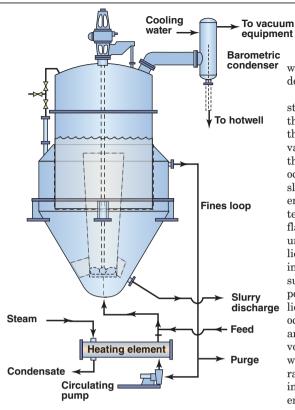


FIGURE 2. A draft tube is an efficient design for suspending solids with low energy input. In a draft-tube baffle crystallizer, slow-moving impellers move slurry upward to the boiling surface, where surface cooling and evaporation creates supersaturation, which is then relieved by crystal nucleation and growth

mixture exists in the mixed slurry of the active volume, where the percentage of suspended solids and crystal size distribution (CSD) are perfectly uniform. This uniformity is also present at the point of product discharge. In addition, it is assumed that the mother liquor has the same residence time as the solids. Thus, there is no deliberate attempt to control the residence time of the crystals of any size.

### Forced-circulation crystallizer

The FC crystallizer (Figure 1) is a type of MSMPR unit. This design usually has the lowest capital cost per pound of product generated. The feed usually enters the downpipe at a point following the discharge of the product slurry to the solid-liquid separation device. The combined stream, consisting of fresh feed and recirculated slurry, is pumped via an axial flow pump through the circulating pipe to a vertical or horizontal heat exchanger,

where it is heated by condensing steam.

This slurry-and-feed stream is introduced into the crystallizer below the liquid surface in the vapor body, the part of the FC where flashing occurs. It mixes with the slurry at the point of feed entry and raises the local temperature to cause flashing (evaporation under vacuum) at the liquid surface. The flashing causes cooling at the surface, resulting in supersaturation that is relieved as crystal growth occurs or as new nuclei are formed in the active volume. FC units operate with a high circulation rate, which limits scaling. The lowered scaling enables the unit to evaporate solutions that have scaling solutes, such as calcium sulfate.

At times, FC crystallizers can be designed for classified product discharge, usually by suspending an elutriation

step beneath the crystallizer body. Introduction of mother liquor to the lower portion of the leg fluidizes the particles prior to discharge, and selectively returns the smaller crystals to the body for future growth. Many NaCl plants have such a process step for both classified product discharge and for keeping a suspension of calcium sulfate in the unit to reduce scaling. The concept is that maintaining small  ${\rm CaSO_4}$  crystals in the vessel encourages growth on the crystal, rather than forming scale throughout the system.

Another modification of an FC unit involves a conical inlet for the slurry to flow into the crystallizer. The conical entrance improves mixing in the body, allowing better dispersion of the supersaturation and improving the uniformity of the slurry at the boiling surface. This installation may also be combined with baffling, in order to allow removal of fines and clear-liquor

advance (removing mother liquor without removing crystals). This builds the slurry density beyond its natural level.

An important point is that both classified product removal and fines destruction are not entirely consistent with the MSMPR properties regarding residence, because these practices are deliberate attempts to modify the residence time of the crystals based on their size.

The heat exchanger in an FC unit usually has a one- or two-pass configuration, often with 1.5-in., 12-gauge tubes. It is essential to limit the temperature differential between the steam and the slurry in the heat exchanger to avoid boiling in the tubes, which can lead to scaling, plugging and excessive nucleation. In addition, there must be adequate liquid head at the point of entry to the body (submergence of the slurry leaving the heat exchanger) to avoid flashing in the recirculation line. Doing this avoids premature flashing in the heat exchanger tubes and the inlet piping prior to entry into the crystallizer tank. Another aspect of the FC crystallizer is the use of an axial-flow pump to achieve high slurry flow with reduced crystal breakage and secondary nucleation.

In an adiabatic evaporative-cooling FC unit (where the heat exchanger is omitted), the feed is introduced to the body at a position adequately below the liquid-vapor surface to avoid flashing during the mixing process.

In both configurations, supersaturation is minimized by high circulation rates (typically about 7 ft/s) and by limiting the temperature drop at the liquid-vapor surface to  $3-8^{\circ}\mathrm{F}$ , with some inorganic salt slurries being limited to a  $1-5^{\circ}\mathrm{F}$  decrease.

For some applications, supersaturation is generated by indirect cooling, as opposed to evaporation. In this case, the process flowrate is designed to operate at a low temperature drop (0.5–5°F) and low log-mean-temperature-difference (LMTD) across the heat exchanger. The goal is to stay within the metastable zone to reduce fouling on the tubes. The end result is usually a large surface area, aimed at achieving the required heat transfer and improve on-stream time. The difference between an effective design

### **Cover Story**

versus a poor design could be a matter of running for weeks or months versus hours or a few days prior to washout.

Vortexing in the main body of the crystallizer can cause a supersaturated slurry to bypass the boiling surface, resulting in high levels of supersaturation within the body, variable nucleation rates and cycling of the CSD between fine particles and coarse crystals as the crystal surface area varies. A number of design features are used to minimize this problem, including the following:

- Installation of a vortex breaker in the cone of the crystallizer
- Maintaining the slurry inlet nearly tangential to the contour of the crystallizer body, with the angle depending on the vessel diameter
- Keeping sufficient submergence of the feed as it enters the crystallizer body, thereby preventing flashing and vortexing
- Adjusting slurry inlet velocity through the recirculation line to avoid a jet across the crystallizer tank, and unfavorable vortexing. An important parameter here is the Froude number, a dimensionless number used to evaluate the influence of gravity on flow

Froude number can be expressed as:

$$N_{Fr} = V^2/(D \cdot g) \tag{1}$$

Where V is slurry velocity in ft/s; D is the vessel diameter in feet and g is the gravitational constant (32.2 ft/s<sup>2</sup>). For example, a light organic slurry with a viscosity of 1–3 cP and a specific gravity of 1.2–1.3 would suggest a  $N_{Fr}$  value that is less than 0.14.

FC units typically range in diameter from 2–20 ft, with some units being as large as 40 ft. They are especially useful for high evaporation loads. A unit used for evaporating water at 380 mm Hg can typically be designed to handle 250–300 lb/h ft². The CSD is wide, with a theoretical coefficient of variation (CV) of 50%. In reality, the CV is often in the 20–40% range due to deviations from perfect mixing.

### Nucleation and crystal growth

The MSMPR configuration can be utilized to determine the kinetics of nucleation and growth for a system. Both growth and nucleation are de-

pendent on supersaturation, with nucleation often having a higher order of dependence on supersaturation versus growth.

Nucleation can be expressed as:

$$B^0 = k_I M \tau^j s^b \tag{2}$$

Where  $B^o$  is the number of nuclei formed per unit volume per unit time;  $k_l$  is the rate constant function of temperature;  $M_T$  is the slurry density; b and b are power functions; and b is supersaturation concentration.

Higher  $M_T$  values (slurry densities) promote secondary nucleation, but also lower the level of supersaturation. The goal is to find a balance with operating conditions to maximize size distribution. Other formulas include agitation speed raised to a power. Often b=1-3 and j=1.

Crystal growth can be expressed as:

$$G = k_2 s^g = dL/dt (3)$$

Where G is the growth rate of a characteristic crystal face chosen to represent crystal size. Change in characteristic length  $(k_2)$  is a function of temperature, agitation, impurities and the system; s is supersaturation concentration; g is system-specific.

Combining Equations (2) and (3) yields the following:

$$B^0 = k_3 G^i M_T{}^j \tag{4}$$

Where i = b/g. A critical kinetic parameter is the value of i, which determines the relative dependence on supersaturation of nucleation versus growth. The population-density model is used to determine the kinetics. An example of the determination of i is given in Ref. 3.

The traditional operation of an FC crystallizer affords limited flexibility in changing the size distribution. The following equation shows that the ratio of median crystal size  $(L_m)$  for two different conditions is:

$$L_{m2}/L_{m1} = (\tau_2/\tau_1)^{(i-1)/(i+3)}$$
 (5)

If one changes the residence time  $(\tau)$ , with everything else kept the same, the following is predicted: For i < 1 (which is rare), the median crystal size decreases slightly with an increase in residence time; for i = 1, theoretically,  $L_m$  remains the same, and for i > 1, the median particle size would increase.

The change is not great. For exam-

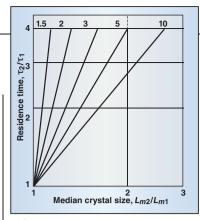


FIGURE 3. Lengthening residence times results in only modest improvements to mean crystal size

ple, when i=2,  $L_m$  increases only 15% when the residence time is doubled. This suggests that residence-time changes are not an effective means for increasing particle size.

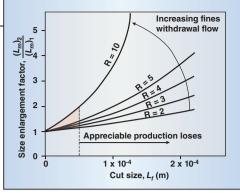
Figure 3 is a graphical presentation of Equation (5), which demonstrates limited improvement in particle size for i values in the 1–2 range, which is commonly seen. Other than adjusting the residence time or slurry density, the FC crystallizer offers little potential to change the CSD. Changing operating conditions, such as residence time for an MSMPR has a small effect on the mean particle size and CV.

Greater control of the CSD is possible by adjusting the residence time of the crystals in a certain size range, such that it differs from the residence time of the liquid phase. This can be accomplished by fines destruction and classified product removal, which are deviations from a true MSMPR machine. A cautionary note for classified product removal is that these units have a tendency to cycle (even with fines destruction) and while the CV is decreased, the mean particle size may decrease due to the cycling.

### **Draft-tube-baffle crystallizers**

Mechanical impellers and agitators can impact the level of secondary nucleation and breakage. For this reason, low-speed impellers in draft tubes are at times placed in the body to reduce the shear forces seen by the FC circulating pump. The draft tube is an efficient design to suspend solids with lower power input.

Figure 2 depicts a typical draft tube baffle (DTB) crystallizer. In this device, a slow-moving (60–125 rpm) impeller is installed in a draft tube, which circulates the slurry upward to the boil-



**FIGURE 4.** Increasing  $L_f$  yields moderate crystal size improvement, but also results in production loss due to dissolution of the fines. The plot is for an inorganic salt

ing surface where the supersaturation created by evaporation and cooling is relieved with nucleation and growth.

The high flowrates from the agitator are mixed with the feed stream to limit surface cooling to approximately 1–2°F during flashing. This limitation keeps the supersaturation at a low value. Cooled slurry is returned to the bottom of the vessel through the space between the outside of the draft tube and an annular baffle. It then mixes with the heated slurry returning from the heat exchanger or with an adiabatic feed.

The active volume includes volumes inside and outside the draft tube, but not areas behind the baffle. DTBs usually contain slurry equal to 25-50% of apparent settled volume. This active volume is circulated one to four times per minute, which limits nucleation and scaling on the vessel walls while promoting growth on the existing crystal surfaces. Crystal residence times are usually 4-6 h, with the production rates often ranging from 60-85 kg/m3 h. The period between washouts often increases with longer residence time and slurry density as a result of decreased supersaturation and scaling.

The annular baffle area functions as a settling zone through which a stream of mother liquor and fines is separated from the slurry in the active volume. The segmentation is due to differential gravitational settling. The result is a residence time for the fines that is less than the residence time for the product. The CSD in the body is controlled by adjusting the vertical velocity of the mother liquor in the baffle areas and controlling the maximum crystal size that will be removed and dissolved. This can be achieved either by changing the flowrate in the baffle area or the amount of area used for the baffles. The maximum size of the crystals  $(L_f)$  that is theoretically removed by the baffle can be used to modify the CSD of the product. The term  $L_f$  has a hindered settling velocity equal to the free vertical velocity. If  $L_f$  is too large, the increased supersaturation generated by recrystallizing the dissolved fines can cause nucleation in the unit, resulting in peri-

odic upsets in the system and cycling of the CSD.

Increasing the percent solids in the slurry to a concentration greater than the "natural make" (the percent solids without other means of building slurry density) is at times achieved by withdrawing a stream of mother liquor from the upper portion of the baffle zone. This procedure thickens the slurry in the body. This practice is referred to as clear liquor advance (CLA) or double-discharge operation (DDO). It is employed for systems that have low natural make. A larger crystal size may result due to lower levels of supersaturation (more crystal surface area in the slurry), increased residence time for the solids and removal of fines in the CLA. Potential downsides could include increases in secondary nucleation and crystal breakage. High slurry densities tend to reduce the baffle efficiency. To improve performance, a lamella plate is installed in the crystallizer body to direct flow vertically at the baffle entrance, or by the installation of altering doughnut baffles in the settling zone behind the regular baffles. These doughnuts dissipate large liquid eddies that can trap and carry out undesirable larger crystals.

A fines-destruction system produces larger crystals with a narrower size distribution. Ideally, the heat exchanger would supply enough heat to both satisfy the evaporation requirements and to raise the temperature of the low-solids slurry removed from the baffle to destroy unwanted fines. For applications where the only heat removed is that which is required for adiabatic cooling of the feed, fines destruction is achieved by selectively removing a weak slurry stream containing fines

and redissolving them via dilution prior to their return to the crystallizer, often in the region below the impeller.

This technique increases supersaturation in the crystallizer body, which increases both crystal growth and nucleation rates. The reason is that excess fines are destroyed by the heat of dilution and then the dissolved solute is recrystallized in the body. The result is a unit that operates at higher internal production rates, due to production of both product crystals and recrystallization of dissolved fines.

The fines-destruction stream, while low in percentage of suspended solids, can often represent a flow that is a multiple of the product underflow stream. Thus, the recrystallization of the dissolved fines can represent a significant percentage of the internal production of the unit. In comparison to the product stream, this configuration deliberately reduces the residence time of the crystals of size  $L_f$  or less. Some fines also migrate to the product stream. The ratio of the product crystal residence time to the fines residence time is referred to as R, or  $\tau_P/\tau_E$ .

Fines destruction, in the form of accelerated removal and destruction of fine crystals  $\leq L_f$ , can be used to increase the product size. With fines dissolution and recycling to the crystallizer, the supersaturation and production rate increase, thereby increasing the nucleation rate (fines are destroyed) and the growth rate, yielding a larger CSD. Under some conditions, a bimodal distribution with a small peak in the 0 to  $L_f$  size range is obtained. A first-order estimate of size improvement is:

$$\frac{(L_m)_2}{(L_m)_1} = \exp\left[\frac{L_f}{(i+3)G\tau_f}\right] = \left[\frac{RL_f}{(i+3)G\tau_p}\right]$$

This indicates that the size increase is dependent on R,  $L_f$  and i.  $L_m$  is the median crystal size.

One can then determine the impact of various combinations of  $L_f$  and R in order to yield the desired product size. Frequently,  $L_f$  is in the 20–80  $\mu$ m range, and there is a large impact of increasing R, which is often in the 4–10 range. Note that, as expected, the ability to improve the size distribution

### **Cover Story**

diminishes as the value of *i* increases.

Equation (6) indicates that the potential size enlargement is dependent on R,  $L_f$  and the relative kinetic order, i. This relationship is shown for a typical inorganic salt in Figure 4. Note that only increasing  $L_f$  yields moderate size improvement, with the negative impact of significant production loss due to dissolution of the fines. A more advantageous method is to increase the value of R, and remove the fines at a smaller size. The production loss is decreased while the circulation rate for fines removal is increased. The shadowed area in Figure 4 represents the approximate operating regime for most industrial crystallizers.

In a sense, a DTB is a hybrid MSMPR, with the active volume resembling the MSMPR mode, but coupled with a classification zone. The DTB has two distinct volumes at play, the crystallization active volume and the baffle volume used as a clarifier. From a kinetic standpoint, the crystallization zone may be referred to as an MSMPR unit. People may initially think that there are no fines in the product stream and that they all move to the baffles. This is not the case; there are fines in the product.

Further CSD improvements can be achieved with an elutriation device. This element will normally be coupled with a fines-destruction system to

avoid upsetting the percentage of solids and the dynamics in the unit, which can lead to cycling of the product CSD.

When destruction of fines is not desired, the baffles are eliminated and the internal circulation rate is designed to minimize nucleation in the active volume suspension. This configuration is called a draft tube (DT) mode of operation.

Edited by Scott Jenkins

### Author

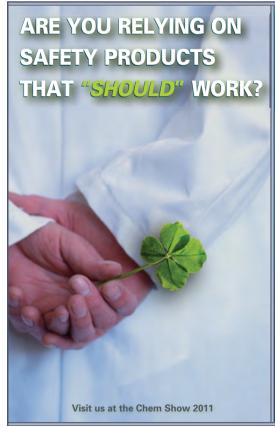


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specialty and commodity chemicals, and pharmaceutical sectors. He is a frequent lecturer at in-house seminars and public courses, and wrote the chapter on crystallization for the 8th ed. of Perry's [4]. Genck holds a Ph.D. in chemical engineering from Iowa State University.

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# Heat Exchangers for Hot Acids: Material Selection

**Dean Gambale**Tantaline

o make the modern materials we use every day, such as plastics, metals, electronics, pharmaceuticals, fuels and so on, strong acids are used to convert the earth's raw materials into modern materials. In fact, so many tons of acid are used (200 million metric tones of sulfuric acid annually worldwide) that the gross domestic product (GDP) of most industrial nations are directly correlated to sulfuric acid usage.

In the chemical process industries (CPI), many kinds of acids are utilized as reactants, intermediates and key ingredients to create the chemical building blocks of materials we use in our daily lives. These acids — which include sulfuric, hydrochloric, nitric, phosphoric and acetic, to name a few popular ones — are often heated and cooled in heat exchangers.

Heat exchangers represent one of the most fundamental pieces of chemical processing equipment and are vital to heating-and-cooling process streams and reagents. In many cases, acids are heated in heat exchangers to increase their reactivity, improve yields and drive higher efficiencies. While a productivity gain can be realized by increasing an acid's temperature, so does its associated corrosiveness increase — and in an exponential manner. As a result, heat exchangers are subject to some of the most severe corrosive environments, because they are typically the point where the acids are the hottest and most aggressive. Therefore, to handle highly concentrated hot acids (greater than 100°F), specialty materials and metals are often utilized.

This article focuses on the specialty metal solutions for heat exchangers. These specialty metal solutions include titanium, nickel alloys, zirconium, and the exotic metal tantalum.

# As temperatures rise, corrosion rates increase exponentially, making these units extremely vulnerable to failure

This article will take a close look at and compare the different material options from the corrosion resistance, reliability, availability and material cost perspectives; and describe the advantages, shortcomings and potential possibilities each material option represents for heat exchangers.

A specialty-metal heat-exchanger solution serves a niche in the industry, delivering corrosion resistance and mechanical stability where no other materials can survive. Although usually found in the most severe applications, these materials have specific appeal in numerous industries including the chemical processing, oil and gas, specialty chemical, pharmaceutical, food and mining industries.

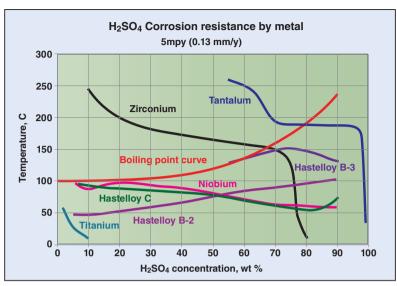
This "niche" however is growing as the next generation of chemical processes are being developed. Compared to the processes developed decades ago, newly developed chemical processes are utilizing higher temperatures, higher pressures and more aggressive chemistries in an effort to achieve greater efficiencies. Furthermore, existing processes are being pushed to achieve greater efficiencies. As a result, higher performing materials are needed to successfully run, contain and safely operate these processes. In these demanding applications, engineers are faced with critical decisions to specify heat exchangers that meet their organization's targets of performance, safety and costs while also taking into account a plethora of process related variables and application specific criteria. To make good decisions engineers must evaluate numerous factors, which include the following:

- Corrosion resistance that is needed for each unit's unique processing conditions
- Corrosion rates and how they will affect the heat exchanger's performance and useful life over time
- The combination of pressures, temperatures, concentration and flow and the effect they will have on the heat exchangers and their corrosion rates
- Abrasiveness of the processing fluid or slurry and the need to combat erosion and abrasion
- Need to accommodate for process swings and variability
- Heat exchanger design type that is most suitable for the process

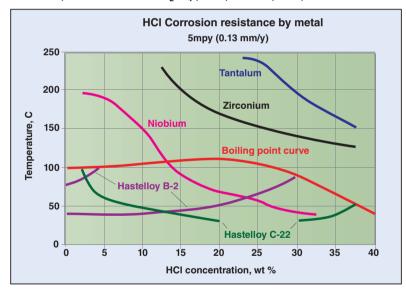
Only when these basics are understood can the engineer begin to make an informed decision on the best material of construction for heat exchangers that will ensure a high performing, safe and economical process.

Fortunately, there exist some high quality manufacturers of heat exchangers that can help guide engineers through the gauntlet of choices that exist. Nevertheless, it is essential for engineers to understand the specialty material options that are available — both traditional and cutting edge. This understanding will give the engineer the background he or she needs to make the best decisions.

With regard to heat exchangers, there are many different types and designs available that go beyond the scope of this article. However within the CPI, tube-and-shell, plate-and-frame heat exchangers represent the most popular types of designs.



**FIGURE 1 and 2.** As can be seen in these charts, tantalum's corrosion resistance is second to none, followed by zirconium, nickel alloys ("Hastelloy" is the common trade name) and titanium in both  $H_2SO_4$  (above) and HCI (below) solutions



Specialty metal materials are generally considered to be titanium, nickel alloys, zirconium and tantalum. Although tantalum is an exotic metal, this article will include tantalum when referring to specialty metals. Some things for the engineer to consider when selecting materials for heat exchangers include:

- Estimated service life of material
- Reliability (safety and economic consequences of failure)
- Material costs

#### Estimated service life

The most important thing to consider when evaluating a specialty metal solution is the corrosion resistance or corrosion rate of the metal in the target media, because it directly impacts the estimated service life of the heat exchanger. Each specialty metal has its own niche, and depending on the processing environment and the needs of the application, a metal's strengths and weaknesses may or may not be suitable. For example, to illustrate

how these materials compare, Figure 1 shows the relative corrosion resistances of the various specialty metals in sulfuric acid  $(\mathrm{H_2SO_4})$ , while Figure 2 compares the metals' performance in hydrochloric acid (HCl). Typically corrosion rates are shown in 5 mills  $(0.005~\mathrm{in.})$  per year, which for a heat exchanger would cause problems relatively quickly.

As can be seen in these charts, tantalum's corrosion resistance is second to none, followed by zirconium, nickel alloys ("Hastelloy" is the common trade name produced by Havnes International) and titanium in both solutions. Tantalum. From a corrosion perspective, tantalum is the most corrosion resistant metal that is in common use and gets its corrosion resistance from a tenacious oxide layer. Tantalum's superb corrosion resistance is comparable to glass and is practically inert to most oxidizing and reducing acids. except fuming sulfuric, hot alkalis and hydrofluoric acid. Taking no other factors into consideration, tantalum metal is an ideal choice from the corrosion resistant point of view, giving the engineer the best chances for success. However tantalum metal is also a very expensive material and is typically cost prohibitive even when cladded. Only in process conditions where no other material will perform adequately has tantalum been the material of choice for heat exchangers — at least in its traditional forms.

Typically, tantalum could be found in a variety applications and industries that deal with hot concentrated acids such as sulfuric acid, hydrochloric acid, and nitric acid in polymer production, metal pickling, acid production and specialty chemical production. Due to its negligible corrosion rate, tantalum also finds use in the pharmaceutical and food manufacturing industries where even the smallest amount of metallic impurity cannot be tolerated in many products [1].

Recently, however, there have been developments in processing tantalum metal to create a surface alloy on process engineering equipment (*CE*, November 2008, p. 16). This surface treatment, is based on a chemical vapor deposition process that grows tantalum metal into a base substrate, like

#### **Feature Report**

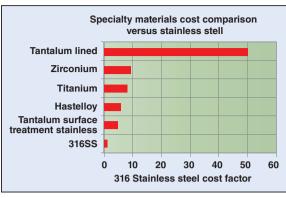
stainless steel, until a thin, uniform, rugged surface of pure tantalum metal is created, exhibiting all the chemical properties of tantalum, but at a much lower cost. This material option is an excellent choice if the engineer desires the corrosion resistance of tantalum. However this option should be avoided when considering slurries, as abrasion could lead to premature failures.

Zirconium alloys. Zirconium alloys exhibit excellent resistance to corrosive attack and work well in many organic and inorganic acids, salt solutions, strong alkalies and some molten salts. Zirconium is produced as two major alloys for CPI applications; grade 702 is considered "pure" zirconium, while grade 705 is zirconium alloyed with 2.0–3.0% niobium. Of the alloys, Zr 702 has better corrosion resistance than Zr 705, however Zr 705 has better strength properties due to the addition of niobium [2].

Zirconium owes its corrosion resistance to the natural formation of a dense, stable, self-healing oxide film on its surface. Unalloyed zirconium has excellent resistance to sulfuric acid up to 60% concentration at the boiling point and has excellent corrosion resistance in hydrochloric acid. Zirconium is also highly resistant to most alkali solutions up to their boiling point. Zirconium's corrosion resistance could be compared with titanium in many ways, however zirconium is much more robust than titanium in withstanding organic acids, such as acetic, citric and formic acids at various concentration and elevated temperatures. For all of zirconium's attributes, it still can be corrosively attacked by fluoride ions, wet chlorine, aqua regia, concentrated sulfuric acid above 80% concentration. and ferric or cupric chlorides [3]. Zirconium has found its way into many types of applications and industries, including hydrogen peroxide production, rayon manufacture and the handling of phosphoric, sulfuric acids and ethyl benzene.

**Titanium.** Titanium is an established metal when dealing with corrosive applications. Titanium is available in a range of different alloys with the most corrosion resistant grades being titanium 7, 11 (containing 0.15% palladium), and 12 (containing 0.3% Mo

FIGURE 2.
Although the costs of specialty metals are higher than for stainless steel, the need for corrosion protection in hot acid applications is often justified



and 0.8% Ni). In the CPI, titanium and its alloys offer good corrosion resistance in many process solutions and owe their corrosion resistance to the strong oxide film. The oxide film formed on titanium is more protective than on stainless steel, and it often performs well in media such as seawater, wet chlorine and organic chlorides. While titanium offers good corrosion resistance to these solutions, it certainly is not immune to them, especially at elevated temperatures such as seawater at temperatures greater than 110°C [3].

Titanium equipment can be found in a variety of industries, which include chemical-processing, pulp-and-paper, and marine applications. A major use for titanium is in seawater or brackish water applications. It is also used extensively in the production of chlorine. Nickel alloys. Nickel alloys are commonly used when typical steel materials don't offer the corrosion performance that is needed. When dealing with aqueous solutions to enhance the performance of nickel materials, the most important alloving elements are Fe, Cu, Si, Cr and Mo; with Cr and Mo playing a major role in nickel's corrosion resistance. By varying the concentrations of Cr and Mo in the nickel alloys, the corrosive environments in which nickel alloys can be successfully applied are varied; but they typically are found in a range of acid, salt and alkali applications. The addition of chromium (15-30%) improves the corrosion resistance to oxidizing solutions, while the addition of molybdenum (up to 28%) significantly improves the resistance to non-oxidizing acids [4].

Focusing on some of the more corrosion resistant nickel alloys, C-22, C-276, and B-2 all have good corro-

APPROXIMATE ALLOY CONCENTRATIONS				
	Cr	Мо		
C-22	22%	13%		
C-276	16%	16%		
B-2	1%	28%		

sion resistance in a variety of media. In the case of HCl, the corrosion resistance of these alloys depends greatly on the molybdenum content. The alloy with the highest concentration of molybdenum, B-2, exhibits the best corrosion resistance.

In other solutions, such as nitric acid (HNO<sub>3</sub>), chromium is an essential alloving element responsible for providing the necessary corrosion resistance. Nickel alloys' weaknesses evolve around their interaction with the media and their environment in the form of impurities. Under ideal testing conditions, for example B-2, the alloy works well in pure deaerated H<sub>2</sub>SO<sub>4</sub> and HCl, but deteriorates rapidly when oxidizing impurities, such as oxygen and ferric ions, are present. Another important consideration is the presence of chlorides (Cl<sup>-</sup>). Chlorides generally accelerate the corrosion attack, but the degree of acceleration differs for various alloys [3].

Having a wide range of applicability in acids, salt solutions and caustic environments, nickel alloys have found their way into a variety of industries such as the chemical, petrochemical, oil and gas, nuclear, conventional power generating, paper and marine.

#### Reliability

The role that heat exchangers play in a process is far from insignificant in that they provide the needed energy.

#### **HEAT EXCHANGER TYPES**

Tube-and-shell heat exchangers remain the most prevalent type in the CPI due to their rugged, welded design and full range of materials available for construction. Because they do not have gaskets, the potential leak paths are minimized. Tube-and-shell heat exchangers also are known for their low pressure drop and ability to handle a wide range of materials, from clean liquids and vapor streams to slurries and steams containing high levels of particulate matter.

Plate-and-frame heat exchangers are versatile and highly efficient. They can also be produced in a variety of different materials, including specialty alloys. However, they all utilize gaskets between each plate, which somewhat limits pressure and temperature. Despite the limitations, plate-and-frame heat exchangers can be up to 5–7 times more efficient than a tube-and-shell heat exchangers. This efficiency is a result of a more tortured path, which creates higher turbulence and consequently a better heat transfer coefficient (U-value).

Another variety of the plate-and-frame heat exchanger is the "fully welded" plate and frame. This variety does not use gaskets, but takes advantage of a special welding process to bond the plates together. The process provides the strength and durability of a tube-and-shell heat exchanger, while maintaining the high efficiency of a plate and frame. While these welded plate and frames are somewhat limited in materials options, when compared to the options available in a tube-and-shell exchanger, especially specialty materials, recent advances in this technology are making more corrosion-resistant materials available (other than stainless steel). One example is the use of tantalum surface alloys in welded plate and frames, where the product would have the efficiency of a plate-and-frame heat exchanger, the ruggedness of a tube-and-shell, and the corrosion resistance of tantalum.

Heat exchangers are typically a major piece of capital equipment, and most processes do not have redundancy of equipment at this scale. In most cases the failure of a heat exchanger would cause a processing line down to have an unexpected shutdown, costing time and money.

So what is the cost of a failed heat exchanger? The true costs of replacing or repairing a heat exchanger is not simply the equipment itself. Engineers must also consider product contamination, lost of production, safety and the high maintenance costs associated with premature failures. In many processing environments, the additional cost usually associated with materials that are more corrosion resistant is invariably less than the disruptions and distractions caused by failed capital equipment. This is especially true for large processing plants with several dependent operations.

#### Material and product costs

When looking at material costs it is far more advantageous to look at the cost of ownership versus the initial out-of-pocket costs. In most cases it is more cost effective to specify materials that will provide an extended exchanger life, and this is especially true in areas that are difficult to replace or are critical to the safety and perfor-

mance of the operation. Usually the increased costs of a specialty metal unit are insignificant when compared to the costs associated with loss of production time, out of specification products and maintenance.

Compared to 316 stainless steel or even polymer solutions, specialty metals are relatively expensive and are therefore reserved for applications that require specialty metal properties and characteristics. While metal prices are continuously fluctuating, the latest estimates for a solid specialty metal solution is anywhere from 4.5 to 10 times the cost of a 316 stainless steel solution, with the exception of a solid-tantalum unit, which would be significantly more. However, as a surface treatment, tantalum surface alloy actually costs less than other special metal solutions, since the tantalum metal is applied very efficiently.

#### Conclusion

When considering heat exchangers and specialty materials for your operation, there are always tradeoffs. Historically, the optimal properties of life, reliability and material costs are rarely realized in one single material set. In addition to the process requirements, organizational goals may influence your material selection by more strongly emphasizing different

aspects, such as safety, process reliability or cost reduction strategies. In any case, compromises need to be made, and it ultimately comes down to corrosion protection versus costs.

Advances in material science are opening up new possibilities in materials. Through material developments, combinations of materials are being engineered to minimize the tradeoffs we face in material selection for heat exchangers and other processing equipment. These new composites are taking materials like tantalum, which has superb corrosion resistance, and growing a durable, rugged surface on a standard material like stainless steel. The result is superb corrosion resistance, on readily available material at costs comparable to less corrosionresistant specialty metals. Is it the answer to all our corrosion materials problems? No. but with continual advances in materials the engineer will be armed with more tools in the battle between aggressive corrosive media and corrosion resistant materials for heat exchangers and other process equipment.

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# Liquid-Gas Coalescers: DEMYSTIFYING PERFORMANCE RATINGS

# Before selecting a coalescer, it's important to understand how they work and how they are rated

**Thomas H. Wines, Scott Whitney** and **Ali Arshad** Pall Corp.

ngineers working in the chemical process industries (CPI) sometimes have to deal with aerosol contamination issues in which liquid-gas coalescers are routinely employed. For example, liquid-gas coalescers are used to protect compressors, liquid-gas contactors, turbines, low-NOx burners, metering and instrumentation stations, and for many other applications.

Choosing the right coalescer type can be a confusing task as many of the equipment-supplier claims can be difficult to understand without more background information on how the products are rated. This article supplies this information and explains how the commonly used rating procedures can affect the performance claims output.

For evaluating a coalescer efficiency rating, it is important to have the test procedure specified and consider the different test options, as they will affect the rating. Furthermore, the same coalescer can give different performance ratings depending on the test method used.

This article compares the different test methods commonly used to rate liquid-gas coalescers, including the DOP [1], sodium chloride [2], ANSI/CAGI [3] and the liquid aerosol separation efficiency (LASE) test [4]. A review of how vertical liquid-gas coalescers operate is also presented, including key model features of media velocity and annular velocity as they pertain to test conditions.

Both the DOP and the sodium chloride methods provide information only on the media capture efficiency and do

not take into account many of the factors associated with how a liquid-gas coalescer operates. The ANSI/CAGI test is a marked improvement operating under oil-saturated conditions, with a poly disperse inlet particle size distribution. The LASE test takes the evaluation to a further degree by increasing the "challenge" load (inlet concentration) to > 1,000 ppm, and also taking into account the annular velocity and using a full flow sampler to eliminate any side stream bias.

#### **COALESCER BASICS**

As mentioned above, there are a number of methods that have been applied to evaluating liquid-gas coalescers in a laboratory setting. In order to understand how the test procedures affect the performance ratings, it is first necessary to have an understanding of how liquid coalescers operate.

#### Vertical liquid-gas coalescers

Figure 1 depicts a vertical high-efficiency liquid-gas coalescer system. Inlet gas with liquid aerosol contamination enters at the bottom of the housing into a first-stage knockout section. Here any slugs or large droplets (> 300 µm) are removed by gravitational settling. The gas then travels upward through a tube sheet and flows radially from the inside of the cartridges through the coalescer medium to the annulus. The inlet aerosol distribution ranges from 0.1 to 300 µm, and after passing through the coalescer medium, is transformed into enlarged coalesced droplets ranging from 0.5 to 2.2 mm. The advantage of flowing from the inside to outside of

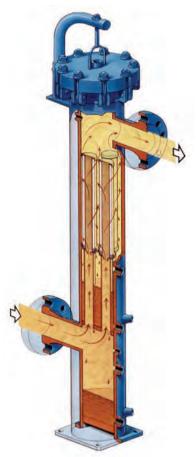


FIGURE 1. Shown here is a typical high-efficiency, liquid-gas coalescer

the coalescer cartridge is that the gas velocity can be more easily adjusted in the annulus by selecting the optimum housing diameter to prevent reentrainment of coalesced droplets.

Four steps have been identified with the mechanism of the formation and removal of droplets in the coalescer medium:

- 1) Capture
- 2) Coalescing
- 3) Release
- 4) Drainage and separation from media The formation of the coalesced droplets first involves the capture of the small aerosols onto the fibers of the co-

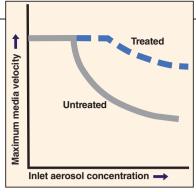


FIGURE 2. This graph shows the effect of surface treatment and liquid loading on media velocity

alescer medium. The actual coalescing or merging of the fine droplets is believed to take place on the fibers, and especially at fiber intersections. The coalesced droplets are then released from the fiber due to the drag force of the gas flow exceeding the adsorption energy. This process is repeated through the depth of the coalescer medium until the coalescing process is completed and the largest possible, stable droplet size is achieved. During the coalescing stages, the growing droplets are also draining downward inside the media pack due to the force of gravity.

Surface treatment. One way to improve the draining of the coalesced liquid drops in the medium is to apply a surface treatment that changes the medium's wetting properties by lowering the overall surface energy. This ensures that both oil and aqueous drops will not wet the surfaces and hence will have lower liquid-fiber attraction forces, thereby allowing better drainage.

#### Modeling the vertical coalescer

The modeling of the vertical liquid-gas coalescer system can be divided into two basic aspects for performance: media velocity and annular velocity. **Media velocity.** The media velocity  $(v_{med})$  is defined as the actual flowrate divided by the coalescer filter area:

$$v_{med} = Q_a / NA_{med} \tag{1}$$

Where:

 $Q_a$  = actual system flowrate (at system conditions)

N = number of coalescers

 $A_{med}$  = media area for one coalescer  $Q_a$  is obtained from the standard system flowrate,  $Q_a$ :

$$Q_a = Q_s Sg \rho_{air\,stn} / \rho_{\sigma} \tag{2}$$

Where:

Sg = gas specific gravity $\rho_{air,stp} = density \text{ of air at standard}$ 

temperature and pressure  $\rho_g$  = density of gas at system conditions

The media velocity is not the actual velocity through the open pores of the media, but rather an average by convention over the combined pore area and solid matrix area in the spatial plane normal to the flow direction. The maximum media velocity for a coalescer construction is related to a number of factors intrinsic to the particular coalescer design and to the physical properties of the system.

Effect of system conditions on media velocity. The ability of the coalescer medium to perform effectively will also depend on the system environment. While different coalescer constructions will exhibit quantitative differences, they will follow the same qualitative behavior. The media velocity has been determined to depend on system parameters such as inlet aerosol concentration, aerosol density, gas density and gas viscosity. An analysis of how the inlet liquid-aerosol concentration affects the maximum media velocity is presented in Figure 2 for surface treated and untreated coalescer media.

At low aerosol concentrations, the maximum media velocity is constant and is unaffected by aerosol levels. Under these conditions, the media is limited by the capture mechanism and is not affected by drainage. At higher levels of aerosol concentration, the coalescer medium becomes limited by drainage and is inversely proportional to the aerosol concentration. The effect of the surface treatment on this process is to enhance the drainage and allow for higher maximum media velocities under the same aerosol loading when limited by drainage. The plot of the surface-treated coalescer media is based on an increase in drainage ability of about threefold. The effect of the increased drainage of the surface treatment is to extend the constant portion of the plot and raise the drainage limited curve to three times the untreated value.

**Annular velocity.** The annular velocity  $(v_{ann})$  is defined as the actual flow-

rate divided by the annulus area:

$$v_{ann} = Q_a / A_{ann} \tag{3}$$

Where  $A_{ann}$  is the cross-sectional annular area defined as the cross-sectional area of the housing without coalescers minus the area of the coalescer end-caps:

$$A_{ann} = \pi R_h^2 - N\pi R_c^2 \tag{4}$$

Where:

 $R_h$  = radius of the housing

 $R_c$  = radius of coalescer end-cap

N =number of coalescers

The enlarged droplets leaving the coalescer media pack can be assumed to be as large as possible for the given flow conditions when complete coalescence has occurred. Therefore, the coalesced droplet diameter will be the same for any specific design of the coalescer cartridge as long as complete coalescence has been achieved. If complete coalescence is not achieved, the calculation of the coalesced droplets must take into account the degree of coalescence.

In most industrial applications, the coalesced droplets will range in size from 0.5 to 2.2 mm and will be mostly influenced by the interfacial tension, which is significantly affected by the liquid-gas types, liquid density, system temperature and system pressure. As the pressure is increased, the gas density will increase, while the liquid density is only slightly affected. The solubility of the gas in the liquid is enhanced with increasing pressure. This leads to a substantial decrease in interfacial tension with increasing pressure and consequently to significantly smaller coalesced droplets at the higher pressures.

Once the coalesced droplet size has been estimated, the next step is to determine the maximum annular velocity that can be sustained without reentrainment. In general, the coalesced droplets will produce Reynolds numbers (Re) outside of the creeping flow regime (< 0.1) and Stokes law. Instead, a force balance is used between the liquid droplets settling by gravity and the drag force of the gas flowing upward in the opposite direction.

As the gas leaves the coalescer cartridge and travels upward in the annulus, it contributes to the total flow,

#### **Engineering Practice**

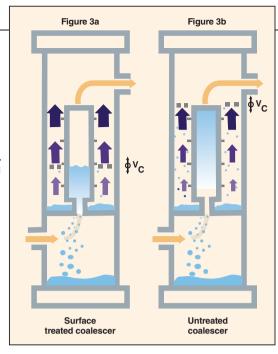
thereby increasing the annular velocity. The annular velocity is modeled as a linear function with vertical distance, and the annular velocity is zero at the bottom of the cartridge and increases to a maximum value at the top of the cartridge.

Once the coalesced droplets are formed, they immediately drain vertically downward in the coalescer-medium pack. As a direct consequence of the treatment, the coalesced droplets are shielded from the upward gas flow in the annulus in most of the length of the coalescer cartridge. The coalesced droplets are first exposed to the annular gas flow when they appear on the external face of the coalescer medium pack at the bottom third of the coalescer cartridge (Figure 3a). Once the coalesced droplets are released to the annular space they are subjected to the force of the upward flowing gas. The trajectory of the coalesced droplets is modeled on a force balance between gravity settling and the drag force created by the gas flow past the droplets. This analysis leads to the calculation of a critical annular velocity for re-entrainment  $(v_c)$ .

The use of a surface treatment on high-performance vertical liquid-gas coalescer cartridge systems has been proven to significantly enhance performance by allowing higher flowrates or smaller housing diameters compared to untreated coalescers [5].

Due to the surface treatment, there are minimal coalesced droplets present in the annulus above the drainage point at the bottom third of the coalescer cartridge. For a coalescer cartridge that is not specially surface treated, the coalesced liquids are present throughout the length of the coalescer in the annulus space, and the critical annular velocity for re-entrainment is given for the top of the element (Figure 3b). For the treated coalescer, it is allowable to have annular velocities greater than the critical value for re-entrainment in the portion of the annulus space where there are no liquids present. This permits the maximum annular velocity at the top of the coalescer cartridge to be about three times the critical reentrainment value needed at the vertical position of the lower one third

FIGURE 3. Surface treatment of the media reduces the coalesced droplets present in the annulus above the drainage point at the bottom-third of the coalescer cartridge. As a result, treated coalescers (a) can have annular velocities greater than the critical value for re-entrainment than for untreated coalescers (b)



of the cartridge height where liquids are present.

Determination of minimum housing diameter. The housing diameter is determined from the area of the annulus and the area of the coalescer end-caps. The maximum annular velocity at the top of the coalescer cartridges is used to determine the annular area required. The value of the maximum annular velocity  $[v_{ann}]$ (max)], at the top of the coalescer cartridges is dependent on the critical annular velocity for re-entrainment  $(v_c)$ and the vertical location at which the coalesced droplets are present in the free annulus space. This relationship can be described as follows:

$$v_{ann}\left(\max\right) = k_{a}v_{c} \tag{5}$$

where  $k_a$  is the annular velocity enhancement factor due to drainage.

For the untreated coalescer medium, the coalescer cartridge is completely wetted and coalesced droplets are present in the annulus space up to the top of the annulus where the annular velocity is highest. There is no drainage enhancement, and  $k_a=1$ . The maximum annular velocity to prevent re-entrainment is then equal to the critical value for re-entrainment:

$$v_{ann}$$
 (max) =  $v_c$  (6)

The effect of the surface treatment is to greatly increase the drainage, and the annular velocity at the top of the coalescer cartridge can now be significantly higher than the critical value since there are no coalesced droplets present in the annulus except in the bottom third of the cartridge. The maximum annular velocity is now determined, with  $k_a = 3.1$ , as follows:

Surface treated coalescer:  

$$v_{ann}$$
 (max) =  $3.1v_c$  (7)

Convincing evidence for the enhanced maximum annular velocity given by Equation (5) has been demonstrated by laboratory tests [6, 7, 8] and is presented in Figure 3. Visual observations during these tests also confirm that liquids are present on the outside of the coalescer pack only at the bottom third for the surface treated coalescer and are present throughout the length of the wetted untreated coalescer.

#### LABORATORY TEST PROCEDURES

A description of laboratory methods that have been reported by a number of gas-filter and coalescer manufacturers and their relevance to actual field operation is provided below.

#### **DOP** test

An organic liquid, dioctyl phthalate (DOP), is first vaporized by heating and then cooled down, allowing the DOP to condense and create a nearly mono-disperse drop-size distribution at 0.3 µm. A portion of the aerosol mist created is mixed with carrier air and flowed through a filter disc used

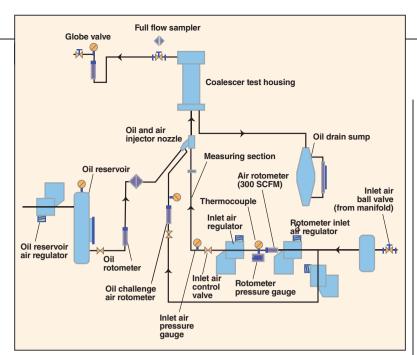


FIGURE 4. The schematic of the liquid aerosol separation efficiency (LASE) test

as the test sample at a controlled flow-rate with an aerosol concentration of  $100 \pm 20$  µg/L (~ 77 ppm). Typically, the test sample is a filter disc with an area of  $100~\rm cm^2$  and is challenged at a flowrate of 32 L/min. The inlet and outlet of the test sample is analyzed for aerosol content using a forward light-scattering photometer.

The test is run on clean, dry filter samples and at minimal pressure to assure sufficient flow with the outlet at atmospheric pressure. Results are measured as percent penetration on a scale setting of down to 0.001% or even to 0.0001%. Values are commonly reported as percent removal at 0.3 µm, with percent removal equal to one minus the percent penetration.

Advantages. The DOP test is an industry standard used for rating high-efficiency particulate air (HEPA) filters, and standard test equipment is readily available. The test is a reliable and useful way to evaluate the capture efficiency of a filter media under initial use conditions.

Disadvantages. The test conditions are not representative of field conditions. The aerosol pressure is very low, and the challenge aerosol concentration is below that of many typical field applications. The aerosol challenge is also nearly mono disperse and uses a different liquid than would be encountered in actual service. The test is run with a clean and dry filter in service. Also the test sample is a fil-

ter disc, and this is not always a good simulation of a coalescer cartridge that can contain pleated media and outer wrap materials. Lastly, the test is not measuring a saturated media that would be expected for a liquid-gas coalescer in service.

#### Sodium chloride test

An aerosol challenge is created by atomizing a sodium chloride solution into a clean, dry filtered air stream. The water carrying the sodium chloride is vaporized, leaving behind solid salt crystals. The salt particle-size distribution can be varied in a controlled manner by adjusting the sodium-chloride solution strength, the pressure and the air flowrate. The aerosol challenge is passed through a test filter disc (typically 90-mm dia.) used as the test sample.

The test flowrate is adjustable and field-service gas fluxrates are typically used. An isokinetic probe is used to draw off a controlled portion of the aerosol stream and pass it to a laser particle counter. The concentration of the aerosol stream is maintained above 106 particles per cubic meter and both inlet and outlet air streams are evaluated for particle counts.

**Advantages.** The sodium chloride test allows for the use of an aerosol challenge that has a varied particle-size-distribution range similar to that encountered under field conditions. The flow per filter area is adjustable

and is typically run at conditions similar to actual field use. The test apparatus includes laser particle counters that have improved accuracy over the light scattering methods used in the DOP test. This method has found wide acceptance in many industries, including the microelectronics field, and is a reliable and useful way to evaluate the capture efficiency of a filter media under initial use conditions.

Disadvantages. The test conditions are not representative of field conditions. The aerosol pressure is very low, and the challenge aerosol is made up of only solid particles. The test is also run with a clean and dry filter in service. Also, the test sample is a filter disc, and this is not always a good simulation of a coalescer cartridge that can contain pleated media and outer wrap materials. Lastly, the test is not measuring a saturated media that would be expected for a liquid-gas coalescer in service.

#### LASE test

The efficiency of liquid-gas coalescers is measured using a test stand configured as shown in Figure 4. The test stand utilizes an assembly consisting of a standard size element installed in a housing of a standard inside diameter. An oil aerosol challenge is generated upstream of the element using an ultrasonic spray nozzle. Performance measurements are taken only after the coalescer assembly differential pressure and sump drainage rate have stabilized, that is, reached equilibrium. The test flowrate is adjusted up to the rated flow of the test coalescer and the annular velocity is also adjusted to representative field conditions by adjusting the test housing diameter.

The removal efficiency of the coalescer is determined by installing a full flow sampler at the outlet of the coalescer assembly. The reason for employing the full flow sampler is to eliminate sampling biases and ensure that all of the downstream oil, both entrained and wall flow, is captured and accounted for. An extraction and analytical analysis are then performed on the full flow sampler to determine the amount of oil that was collected during the test.

General description of the test

#### **Engineering Practice**

stand. The test stand is supplied with dry air that is prefiltered and coalesced to eliminate any background dirt or liquid aerosols. The oil is supplied to the atomizing nozzle via a pressurized oil reservoir, and the coalesced oil is collected in a sump and measured. It is important to measure the incremental amount of oil that is drained from the coalescer housing throughout the duration of the test. These data are used to determine the actual liquid challenge. Actual system flowrates are monitored and controlled by a regulating valve, and the flowrate is measured with a calibrated rotometer. System pressure, temperature, and differential pressure across the coalescer assembly are also measured.

The inlet to the housing is through the bottom center of the housing. No settling chambers, inertial separators or other attempts to precondition or remove oil challenge before contact with the element is permitted. The minimum air velocity between the atomizing nozzle and the test element inlet, including all parts of the housing, is 80 ft/s minimum (24.38 m/s).

**Aerosol generation.** A liquid loading system utilizing an ultrasonic spray nozzle is used to generate the aerosols. The oil used for this evaluation is Mobil Corp.'s DTE – 24 lube oil.

The quantity of aerosol (by mass) and size distribution produced by the nozzle depends on the flowrate through the nozzle and the physical properties of the medium being sparged. The varying sized aerosols generated by this system (0.1–1.0 µm) are considered to be representative of what would be typically found in the aftercooler exhaust air from a reciprocating compressor.

Full flow sampler. The full flow sampler is an inline design and contains a flat sheet, non-corrugated Teflon membrane having a removal rating of at least 0.45 µm and a minimum effective area of 0.26 ft² (0.025 m²). The sampler has a baffle plate to guard against direct impingement of oil droplets onto the medium and to provide a uniform flow across the membrane disc. It also has a surface finish, material of construction and design that allow proper extraction

TABLE 1: COMPARISON OF DIFFERENT LABORATORY TEST METHODS FOR RATING LIQUID-GAS COALESCERS

RATINO LIGOID-OAS COALLSCERS						
Test method	Aerosol type	Inlet aerosol challenge	Test run at saturated conditions	Test run at max. loading	Test run at maximum annular velocity	Outlet sampling method
DOP	Liquid (dioctyl phthalate)	100 ± 20 μg/L of Air (~ 77 ppm)	No	No	No	Full flow
NaCl	Solid (salt)	> 106 par- ticles per cubic foot greater than 0.003 µm	No	No	No	Isokinetic probe
CAGI	Liquid (lube oil)	40 ppm	Yes	No	No	Isokinetic probe
LASE	Liquid (lube oil)	1,112 ppm	Yes	Yes	Yes	Full flow

and clean up. The inlet and outlet are equipped with Triclover fittings to facilitate installation and demounting. These fittings are sealed when not in use and during extraction procedures to avoid contamination.

Sampler extraction and analysis method. After a test run, the downstream sampling membranes are extracted by laboratory-grade hexane. A pre-weighed quantity of each solvent is introduced into the sampler and allowed to mix for a known period of time. An aliquot of the mixture is removed and analyzed by either an infrared spectrophotometer when Freon is used, or gas chromatography mass spectrometer (GCMS) method when hexane is used as the extracting solvent. The minimum detectable oil level is 0.001 ppmw, based on air at 100°F and 100 psig. The upstream or challenge aerosol concentration is determined by direct gravimetric measurement following the saturation of the test element and stabilization of the saturated assembly differential pressure by measuring the sump drainage oil from the coalescer housing during the sampling period. Subsequently, the actual upstream-liquid-challenge concentration is determined by adding the downstream aerosol concentration to the sump concentration.

Advantages. The LASE test allows for the use of an aerosol challenge that has a varied particle-size-distribution range similar to that encountered under field conditions. The flow per filter area is specified at 100% rated flow. The test conditions are under pressure and the test coalescer is tested for efficiency after it has become saturated with oil. The aerosol concentration in the challenge feed is 1,112 ppm and represents a realistic and difficult field application. The an-

nular velocity is controlled at conditions expected under field conditions also at the maximum velocity per the LASE sizing method as calculated for the test conditions. The sampling technique used is a full flow sampler, and this method allows for more accurate results than sidestream evaluation.

**Disadvantages.** The test conditions are not completely representative of field conditions, as the test pressure is still lower than typical operating conditions and uses air as the carrier gas and oil as the liquid aerosol.

#### ANSI/CAGI

The ANSI/CAGI method follows a similar procedure and uses similar equipment to the LASE test as described above, with a few exceptions that are shown here:

- 1) The aerosol challenge is much lower at  $40~\mathrm{ppm}$
- 2) The annular velocity is not adjusted to the expected industrial use
- 3) The downstream sampling uses a sidestream method that first catches wall flow in a small vessel separator followed by a membrane sampler

Advantages. The ANSI/CAGI test allows for the use of an aerosol challenge that has a varied particle-size distribution range similar to that encountered under field conditions. The flow per filter area is specified at 100% rated flow. The test conditions are under pressure and the test coalescer is tested for efficiency after it has become saturated with oil.

**Disadvantages.** The test conditions are not representative of field conditions. The aerosol pressure is lower than typical operating conditions and uses air as the carrier gas. The aerosol oil challenge is set to 40 ppm, which is lower than many field applications and makes this an easier test to get

#### **TABLE 2: TEST RESULTS FOR A HIGH-EFFICIENCY LIQUID-GAS** COALESCER Test **Performance** Method Rating DOP 99.999% at 0.3 µm NaCl 99.7% ≥ 0.3 µm ANSI/CAGI 0.001 ppmw oil downstream LASE 0.01 ppmw oil downstream

high-efficiency results. The test protocol does not specify the annular velocity, and this will also enable the test to provide high-efficiency results as well. The downstream sampling method uses a wall flow collector and iso-kinetic probe that is not as accurate as using a full flow sampling membrane.

#### **Concluding observations**

Depending on the test method used, varying efficiency ratings can be obtained for the same test coalescer. A comparison of the test methods discussed here are presented in Table 1 along with the actual test results in Table 2 obtained using the SepraSol Plus liquid-gas coalescer.

The DOP and sodium chloride tests were found to provide information only on the media capture efficiency and were not taking into account many of the factors associated with

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how a liquid-gas coalescer operates.

The ANSI/CAGI test is a marked improvement operating under oil-saturated conditions, with a poly disperse inlet particle-size distribution.

The LASE test takes the evaluation to a further degree by increasing the challenge load to > 1,000 ppm, and also taking into account the annular velocity and using a full flow sampler to eliminate any sidestream bias.

So for evaluating a coalescer efficiency rating, it is important to have the test procedure specified and consider the different options, as they will affect the rating. As seen in Table 2, the same coalescer gave quite different readings such as a 0.001 ppm outlet using the ANSI/CAGI test, and a magnitude higher outlet of 0.01 ppm when tested under more severe conditions using the LASE test.

Edited by Gerald Ondrey

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# Inspecting Fractionation Towers

While physically inspecting the interior of a distillation column can be a tedious, and often dirty task, it is also a great opportunity to identify and avert potential process problems

Juan Manuel Sanchez, Álvaro Valverde, Carlos Di Marco and Eduardo Carosio YPF S.A.

istillation columns are often workhorses in the chemical process industries (CPI), and at times, it is necessary to physically inspect the interior of a column. This article demonstrates what an inspection can reveal, by sharing the experience of actual fractionation-tower inspections in a petroleum refinery. The points mentioned are intended to provide guidance to future tower inspectors, so that they can focus on extremely important and relevant points that will directly impact the operation of distillation columns.

#### **Inspection basics**

Who inspects? Whenever a fractionation tower is opened for inspection, it is important that it be inspected and analyzed by a qualified process engineer, who has been trained in what to look for.

Typically, inspections are carried out by a number of different departments, each one focused on its particular area of concern. Column components are usually inspected by the equipment inspection department. The production department, besides checking if it



FIGURE 1. Check nozzles for plugs. This one is blocked with a piece of packing



FIGURE 2. Water testing quickly identifies nozzle plugging

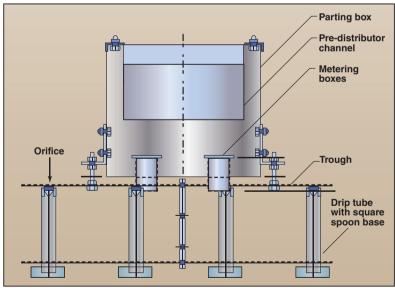


FIGURE 3. This schematic shows the configuration of the gravitational distributor

is necessary to replace or repair internals, inspects if the tower is clean or if it has accumulated large quantities of coke, salt or corrosion products during operation. Cleaning, repairs and mountings are the responsibility of the maintenance department.

The process department inspector is

usually responsible for critically analyzing changes made in a column and for identifying potential operational problems. Something as simple as the replacement of a downcomer can generate a significant fractionation-efficiency loss if it is not properly mounted. For example, if the clearance under a

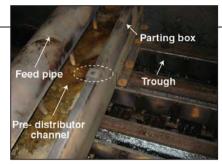


FIGURE 4. Inspection uncovered a missing bolt



FIGURE 5. Some drip tubes were bent, causing uneven liquid distribution



FIGURE 6. Even though dirt was found in the distributor, the design compensated for the fouling

downcomer is too small, the column could suffer from flooding because of the flow restriction. The construction crew, however, does not always understand how a column works, and might therefore make decisions that are not the best. It is essential to have people who are knowledgeable about the distillation process to critically analyze and address these types of details in the column. In addition to the critical analysis, it is imperative that the process inspector avoid repeating old practices and errors that can be dragged on for years and become part of the way of working.

**Preparing for inspection.** A critical inspection is a tedious and often dirty task. It is necessary to remain inside the tower for hours. A job welldone, however, can identify potential problems and will save headaches



FIGURE 7. This blockage required that the distributor be dismounted and cleaned





FIGURE 8A (top) and 8B (bottom). This design mistake caused a pipe to be wetted from a nozzle, which could potentially cause corrosion

when the tower is back in operation. It is important to identify problems before opening the fractionation tower for inspection. Monitoring the variables in the column will allow the inspector to focus on sectors that have been identified as problematic in advance. For example, if the column is flooded at the top, between the kerosene sump and the top of the tower (identified by the differential pressure and gamma scanning), that section should be inspected immediately when the column is opened. In our particular case, we intended to replace the distributors and the wash bed of the vacuum tower, because we knew from monitoring pressure drop during operation that it was filled with a large quantity of coke.

This article covers these examples, and more from the point of view of

the process. To put this type of inspection into practice, it is necessary to prepare a detailed program to know what to inspect and how to look for the potential problems. An experienced process engineer once said that a good observer and detector of potential problems inside the column turns himself into the liquid that flows through the downcomers and into vapor, which rises through the trays.

#### The manufacturing site

This article shares experiences from inspections performed in the vacuum and atmospheric-pressure fractionation towers in our refinery, during routine maintenance shutdowns of the units in November 2008. The refinery, owned by YPF S.A. is called Luján de Cuyo, and is located in the province of Mendoza in Argentina.

Our vacuum tower, CV10, is 37 m long with a diameter of 8.2 m in its largest section. It processes around 9,000 m<sup>3</sup>/d of atmospheric residue. Our crude distillation atmospheric tower, CV1, is 39.6 m long and 4 m in diameter. It processes around 11,000 m<sup>3</sup>/d of crude oil.

Full inspections for each of the towers had been planned. Previous planning was necessary to avoid delays and time extensions of the scheduled shutdown. Although a complete inspection of each one of the towers was conducted, this article presents only the points of major interest, which will be useful for process inspectors. We focus on the following points of concern:

#### Vacuum tower

- Inspection and testing of distributors
- Inspection and testing of sumps and chimney trays
- Inspection of dual-element filter screens
- Design problems

#### Atmospheric distillation tower

 Inspection of trays (valves, holes, measurements of weirs and clearances under the downcomers)

#### **Engineering Practice**





FIGURE 9A and B. A longer nozzle might have caused a worse corrosion problem to the pipe weld

- Inspection and testing of sumps
- Design problems
- Test of gravity-flow distributor

Vacuum distillation tower Light vacuum gas oil (LVGO) pump around distributor. Among a total of 19 spray nozzles, only one was found to be plugged. It was agreed with the maintenance staff to randomly select 20% of all nozzles, dismount and check them. If 50% of these nozzles were plugged or dirty, all nozzles would be dismounted for cleaning and inspection.

The nozzle that was found to be plugged (Figure 1), was blocked with a piece of packing (Flexiring). To shed light on our question of how it could have gotten there, we went to inspect the dual-element filter screens that are external to the tower. We found two different possible explanations: 1) We found no meshes inside the filters, and this would allow the packing piece to bypass the filters, and 2) One of the dual-element filters was dismounted to connect a segment of piping that bypassed the dual filters. Someone had decided to dismount it to give operational flexibility to the unit.

**Distributor testing.** To verify that no spray nozzles had been plugged, they were tested with water. Figure 2 shows what happens when the flow through the nozzles is not adequate.



FIGURE 10. The "Y" filters were inspected



FIGURE 11. This deformed mesh taken from the Y filter would allow solids to pass to the spray nozzles and potentially cause plugging

This is due mainly to operational failures, or in the worst case, plugging of the nozzles. A plugged nozzle produces a poor flow for washing on a portion of the bed.

LVGO/heavy vacuum gas oil(HVGO) fractionation bed distributor. We have a gravitational distributor in the LVGO/HVGO fractionation bed (Figure 3). The LVGO fills the pre-distributor channel through a feed pipe. The liquid overflows into a parting box. Then, troughs are filled. At the top of each trough there are orifices, where the liquid flows to drip tubes. The liquid arrives to spoons bases (the liquid drains through the corners of a base, called the spoon base — see Figure 3) and distributes liquid to the bed.

The first thing that was observed during the inspection of this distributor was that the central parting box was uneven. Figure 4 shows a missing bolt necessary to hold the box.

Unevenness in the central parting box will affect liquid distribution to the bed below, since the liquid will flow preferentially to some troughs. A section of the packing will be dry due to reduced irrigation, and a poor end point (the highest temperature recorded during distillation) or cloud point (the temperature at which the liquid has a cloudy appearance signifying that wax is beginning to crystallize) would result.

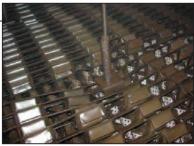


FIGURE 12. Inspection uncovered deformed column internals, such as this grid



FIGURE 13. Modifications need to be made according to design. A correct grid arrangement is shown here

In certain sections, drip tubes with square spoons bases were not in place, but were instead on the bed. Also, as shown in Figure 5, some were bent so that the liquid distribution to the bed was not good.

Even though to enter the column it was (and should be) washed for hours, there was dirt inside the side troughs of the distributor (Figure 6). As the holes from where the liquid enters are above where the dirt was in the troughs, there should have been no obstruction to the passage of liquid. This shows the effectiveness of this design in terms of antifouling.

Even with the advantages of this technology, however, the inspection uncovered a clear blockage in the center parting box (Figure 7), which hindered the passage of liquid to the lateral troughs. This was discovered through a water test of the distributor. As a result, the distributor was dismounted and cleaned.

HVGO pump around. We found a design mistake in this section. Figures 8a and 8b show how a branch pipe was wetted by liquid issuing from the spray nozzle. This occurred because the pipe was inclined. Given that the values of total acid number (TAN) and sulfur content of the current HVGO are 1.3 kg (mg KOH per gram of hydrocarbon) and 0.67 wt.% respectively, and the operating temperature exceeds 200°C, the jet flow from the nozzle could neg-





FIGURE 14A (top) and B (bottom). Some valves on the travs were found to be missing or corroded



FIGURE 15. Improper assembly of column internals can lower fractionation efficiency



FIGURE 16. This panel was rotated about its normal position, rendering the inlet weirs inoperable



FIGURE 17. Check that downcomer clearances are within specifications

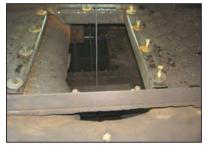


FIGURE 18. During installation of the distributors in the column, downcomers were damaged

atively impact and contribute to corrosion of the feed piping.

To resolve this issue, we tried placing a longer nozzle to stop the liquid from wetting the branch pipe. The tube placed was as long as we could make it, taking the inclination of the pipe into account. Figures 9a and 9b show how the spray distribution would have wetted the branch-pipe weld if we decided to carry out the modification. Once we saw this, however, we became concerned about corroding the weld. Any leak in branch pipe would cause poor distribution over the entire bed, leading to poor heat transfer and possibly coking on the packing. Therefore, we decided to go back to the original arrangement, and we mounted the shorter pipe back, with the compromise to replace it during the next shutdown of the unit.

While no nozzles were plugged, it is appropriate to comment on another design error as to the dual filters of the HVGO pump-around loop. They are located upstream of the control valve. Dual filter screens should be downstream of any equipment before entering the column, to avoid plugging in the nozzles. This anomaly dates from the construction of the plant in 1970. We will fix them during the next shutdown of the unit.

Wash bed distributor. Before entering the tower for inspection, we observed from the manway that an arm of the distributor was broken, so liquid came out between the two sections. The visual inspection, inside the column, showed that eight out of 61 nozzles were plugged. In this particular case, all the spray nozzles were replaced by new ones, so all of them were dismounted. When we inspected the dismounted nozzles, we were surprised to find that more than eight nozzles were actually plugged. About 50% of the nozzles were plugged internally. This would have probably appeared during the water test of the spray nozzles. The lesson in this case is the importance of the spray-distributor water test.

Each flowstream that circulates to the column contains two "Y" filters (Figure 10). These dual filters are meant to stop any solids that could plug the column internals. We found only one filter whose mesh was deformed (Figure 11), which allowed the passage of dirt to the spray nozzles.

In inspecting the upper grid of the bed, which was raised to the height of the manway, we found a broken tie rod. Figure 12 shows how the grids were deformed. One bed was entirely replaced as planned. One thing to consider is that when the maintenance staff mounted this new bed, they were doing so without drawings. According to design drawings, each bed layer should be at a 45-deg. angle

to the layer below, and this was not being respected in the installation. So, we had to intervene to properly reinstall the layers. The lesson here is to try to keep abreast of all changes made. Figure 13 shows a correct flow grid layer arrangement.

#### Atmospheric distillation tower

This column is composed of 39 valve trays and structured packing. From top to bottom, the configuration is the following:

- Trays 1 to 13
- Tray 14 (kerosene sump)
- Trays 15 to 23
- Tray 24 (atmospheric light-gas-oil sump)
- Trays 25 to 33
- Tray 34 (atmospheric heavy-gas-oil sump)

Bottom trays 35 to 39 (stripping section between petroleum feed and stripping steam inlet)

Trays 1 to 10, are two-pass, and 11 to 39 are four-pass trays. The inspection was conducted in detail for each of the trays and structured packing. This article includes only the highlights.

Travs inspection. We found missing valves in some cases along the column, and some valves were found to be corroded (Figures 14a and 14b).

Improper assembly of the panels that compose the trays, produced holes (Figure 15) that allowed weeping, which worsened the quality of

#### TIPS FOR TOWER INSPECTIONS

Foremost, it is important that an inspection is carried out by a team of people that includes a qualified process engineer who is familiar with the fractionation column and has been trained in what to look for. Here are some additional tips gained from experience in tower inspections:

- 1. Identify problems before opening the fractionation tower for inspection. Monitoring pressure drop and other variables can pinpoint problem areas in advance of the inspection
- 2. Prepare a detailed schedule for inspection. Keep in close contact with the maintenance personnel
- 3. It is important to have adequate tools that will be needed during the inspection
- 4. "Think like the liquid" when inspecting and closely observe where the liquid will flow through the downcomers and where the vapors will rise up, looking for obstructions or design
- 5. Test spray nozzles with water to check for plugging
- 6. Monitor any changes made to the column, making sure installations are done according to design
- 7. Check that clearances under the downcomers are within specifications. Improper clearances can cause flooding or entrain-
- 8. Before closing the column at the end of an inspection, make sure all tools or other items brought in have been removed
- 9. Inspect and maintain the filters periodically

fractionation. Besides the formation of holes, improperly placing a panel may negate the functionality for which it was built. Figure 16 shows a panel rotated about its normal position. As can be seen, if the panel is rotated, the inlet weir will not work. The inlet weir function is to prevent the liquid from the downcomer from lifting the valves closest to the inlet downcomer and weeping through the inlet row of valves.

In each of the trays we took measurements of the weirs, and the clearances under the downcomers to ascertain a liquid seal on the tray. If there is no liquid seal, gas may rise up the downcomers, producing liquid entrainment and flooding. Conversely, if the clearance under the downcomer is too small, it will increase the height of liquid in the downcomer. When the downcomer backup reaches the tray above, flooding will occur. It is sufficient to have one undersized clearance to bottleneck an entire fractionator.

In the particular case shown in Figure 17, the clearance under the downcomer was 29 mm. According to the design drawing, however, this clearance should be  $43.5 \pm 3$  mm. This problem was solved by cutting the bottom of the downcomer to acceptable values.

Between trays 19 and 20 are the atmospheric light-gas-oil (ALGO) pumparound inlet pipe distributors. It was noted during the inspection that during the installation of the distributors in the column, downcomers were damaged (Figure 18). This caused significant losses of fractionation capacity.



FIGURE 19. When flow is increased above design values, poor distribution of liquid results in the gravity flow distributor

Sumps. Kerosene, ALGO and atmospheric heavy-gas-oil (AHGO) sumps were tested with water. They were filled with water to a certain level. Then we cut off the water and waited about 20 minutes to look at the fall of the water level. The conclusion of the test was that no problems were observed with sump leakage.

Anecdotally, during the final visual inspection of the tower, just before being closed, we found a pipe scaffold in the AHGO sump. It was hiding, so that if we had not properly performed the final inspection, the scaffold debris may have generated significant damage in the area.

Gravity flow distributor. The gravity flow distributor, located in the wash section, was tested with water to see what happens when the flow increases. Figure 19 shows how it fills the central parting box, and the poor distribution of liquid that results when the flow is increased above design values.

Edited by Dorothy Lozowski

#### Authors



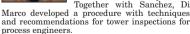
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# New Piping Code for High-Purity Processes

It is essential that industry codes, standards and regulations keep up with evolving technology and changing demands of the chemical process industries

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n the emerging and ever-expanding areas of bioprocessing, where maintaining hygienic designs and practices is of paramount importance, and semiconductor manufacturing, which has its own stringent purity requirements, there is a need to standardize the essential codes and standards that are available. The goal is to consistently achieve process systems that meet the highly refined cleanliness and cleanability requirements that these industries demand. In addition to cleanliness and cleanability requirements, process operators must integrate safety into all high-puritydesign philosophies and standardization efforts.

This article discusses the importance of, and need for, engineering codes and standards that govern the design of high-purity process piping systems. The focus of this article is the new Chapter X (High Purity Piping) that is found in the 2010 issue of the American Society of Mechanical Engineers (ASME) B31.3 Process Piping code. This chapter deals mainly with the bioprocessing and semiconductor industries, but also includes a sub-

Simplified bioethanol process diagram

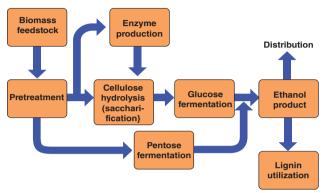


FIGURE 1. CPI manufacturing involving biological or biochemical processes requires high-purity system design that provides an environment that is conducive to desired bacteria while preventing, through its integrated cleanability, any unwanted bacterial contamination

set of bioprocessing-related industries, such as pharmaceuticals manufacturing, biofuels production, food-and-dairy production and others.

#### **Evolving purity requirements**

Early on (in the 1920s), the food-anddairy industry, through the cooperative effort of the International Association of Food Industry Suppliers (IAFIS; now the Food Processing Suppliers Assn.), the International Assn. for Food Protection (IAFP), and the Milk Industry Foundation (MIF) formed the 3-A Sanitary Standards organization, or simply "3-A SSI." 3-A SSI was instrumental in establishing the first set of standards, protocols and methodologies to ensure that this industry could produce food products on a repeatable basis that were free from pathogenic bacteria. Such bacteria are potentially derived from contaminated piping systems as a result of an inadequate cleanability design, an insufficient cleaning regimen, or cross contamination of dissimilar products.

Until the late 1990s, the food-industry standards that were initiated by

3-A SSI were widely utilized by two other industry sectors — pharmaceuticals and semiconductors — that both require a particularly high degree of purity throughout their processes and utility systems, but for very different reasons.

The pharmaceutical industry, like the food-and-dairy industry, expends great effort to design, install and maintain its process systems to ensure a high degree of hygienic purity. In general, process systems used by pharmaceutical manufacturers require added care and documentation during both the manufacture of individual components that make up these systems, and the fabrication and installation of the complete systems. While the pharmaceutical and food-and-dairy industries both require high degrees of cleanliness, they each have their own differing set of guidelines on how to achieve and maintain the desired cleanliness.

Piping used throughout the semiconductor industry, on the other hand, requires a degree of purity that is even higher than that required of the pharmaceutical, food-and-dairy

#### **Pristine Processing**

industries, but for altogether different reasons. During semiconductor manufacturing, bacterial contamination is not the driving consideration that it is in the food-and-dairy, and pharmaceutical industries. Rather, semiconductor operations have a critical need to mitigate the potential for particulate contamination, which can be devastating to today's highly miniaturized electronic components. Microscopic particles in semiconductor facilities, whether coming from equipment, tubing, or the various fluids used during the manufacture of silicon chips, can render the chip useless, or at the very least, out of specification.

In the face of such exacting purity requirements, the widely used ASME B31.3 Process Piping code proves its adaptability, in terms of keeping pace with changing technology demands across these varied industry segments. Three primary segments — food-anddairy, semiconductor and pharmaceutical - have served as initiators and proponents during the development of standards to meet the needs of their respective industries. In particular, the industry-specific standards developed by 3-A SSI, Semiconductor Equipments and Materials International (SEMI), and ASME's Bioprocessing Equipment (BPE) Committee led the way in establishing criteria for the high-purity component design, system design, fabrication and installation requirements to meet the needs of these specific industry sectors. While individual, industry-specific codes are in place, the ASME B31.3 piping code is also relevant to all of these industries, as it establishes engineering practices to ensure piping system integrity and safety.

While the industry-specific standards define the particular degree of purity and cleanability required in those sectors, and establish the component and system designs needed to meet those requirements, ASME B31.3 has recently expanded its content to incorporate requirements that establish structural integrity and safety parameters for high-purity applications. In order to meet these integrity and safety requirements, the reader or user of an industry-specific standard can now be referred to the ASME

B31.3 Process Piping Code, and more specifically to its latest Chapter X.

In an effort to harmonize its efforts and dovetail seamlessly with the prevailing codes and standards mentioned above, ASME recognized the fact that while many of the B31.3 sections and paragraphs referenced by 3-A, SEMI, and BPE could be applied appropriately as written, there was concern that B31.3 did not meet all of the needs of the bioprocessing and semiconductor industries, especially when it comes to high-purity fabrication, examination, testing and inspection. This was the impetus for the development of the new Chapter X addition to the ASME B31.3 Process Piping code.

#### Chapter X: High Purity Piping

The ASME B31.3 Process Piping code has developed over time to become the preeminent piping code for the chemical process industries (CPI). The 2008 issue of B31.3 consisted of nine chapters. Chapters I through VI are considered to be the base code. These chapters are essentially written for metallic piping that is intended for fluid services that can be categorized according to what B31.3 defines as normal and Category D fluid services. [Author's note: Shortly after the writing of this article, the 2010 issue of the ASME B31.3 Process Piping code was published in March 2011.]

The requirements for nonmetallic piping and piping lined with nonmetallic materials can be found in Chapter VII, and are supplemental to the base code. Nonmetals were initially introduced to the code in its 1976 publication, but not given their own chapter until the 1980 publication. The paragraphs in Chapter VII are numbered with respect to the paragraphs in the base code with the added prefix A.

Requirements associated with handling toxic fluids, defined by ASME B31.3 as Category M fluid services in Chapter VIII, were first added in the 1976 publication. This chapter establishes more-stringent requirements for toxic fluid services, and was also developed to supplement the base code. The paragraphs in Chapter VIII are numbered with respect to the paragraphs in the base code with the added prefix M.

Chapter IX, added in the 1984 publication, provides supplemental requirements for operations involving high-pressure fluids. The paragraphs in Chapter IX are numbered with respect to the paragraphs in the base code with the added prefix K.

Adding to those supplemental chapters is the latest Chapter X High Purity Piping, which is included in the 2010 issue of the ASME B31.3 code (as noted, the latest issue was published in March 2011). As in Chapters VII, VIII, and IX, Chapter X is supplemental to the base code, so that the respective base code paragraphs included in Chapter X carry the added prefix U, to establish their connection with the high-purity piping requirements detailed in Chapter X.

#### Application of Chapter X

As noted, Chapter X is a supplement to the base code of B31.3. It provides supplemental recommendations to augment those paragraphs in the base code where additional requirements are needed for high-purity applications. However, readers should note that while ASME B31.3 is considered by many to be the preeminent piping code, it is not a design guide. Specifically, as stated in its introduction: "The designer is cautioned that the code is not a design handbook; it does not do away with the need for the designer or for competent engineering judgment."

High-purity fluid service is defined in B31.3 as "A fluid service that requires alternative methods of fabrication, inspection, examination, and testing not covered elsewhere in the code with the intent to produce a controlled level of cleanliness. The term thus applies to piping systems defined for other purposes as high purity, ultra high purity, hygienic, or aseptic."

This definition touches on the relevant points in which the requirements that are spelled out in the supplemental B31.3 Chapter X are needed—specifically during the fabrication, inspection, examination and testing of high-purity piping systems. However, depending on the industry- or case-specific requirements related to material attributes and specific installation requirements, the designer or engineer may need to go beyond

#### ORGANIZATIONS AND STANDARDS RELATED TO HIGH-PURITY PIPING

3-A Sanitary Standards, Inc. (3-A SSI; 6888 Elm St., Suite 2D, McLean, VA 22101; 3-a.org)

- P3-A 002:2008 Pharmaceutical 3-A Sanitary/Hygienic Standards for Materials for Use in Process Equipment and Systems
- P3-A 003:2008 P3-A End Suction Centrifugal Pumps for Active Pharmaceutical Ingredients

American Society of Mechanical Engineers (ASME; Three Park Ave., New York, NY 10016-5990; asme.org)

- ASME B31.3 2008 and 2010 editions
- ASME-Bioprocessing Equipment (BPE) Standard 2009

American Welding Society (AWS; 550 N.W. LeJeune Rd., Miami, FL 33126; aws.org)

 AWS D18.1 Specification for Welding of Austenitic Stainless Steel Tube and Pipe Systems in Sanitary (Hygienic) Applications

International Society of Pharmaceutical Engineers (ISPE; 3109 W. Dr. Martin Luther King, Jr. Blvd., Suite 250, Tampa, FL 33607-6240; ispe.org)

• ISPE Baseline Pharmaceutical & Engineering Baseline Guide

Semiconductor Equipment and Materials International (SEMI; 805 East Middlefield Road, Mountain View, CA 94043; semi.ora)

- SEMI E49.8-2003 Guide for High-Purity and Ultrahigh Purity Gas Distribution Systems in Semiconductor Manufacturing
- SEMI F1-96 Specification for Leak Integrity of High-Purity Gas Piping Systems and Components
- SEMI F19-0310 Specification for the Surface Condition of the Wetted Surfaces of Stainless Steel Components
- SEMI F20-0706E Specification for 316L Stainless Steel Bar, Forgings, Extruded Shapes, Plate, and Tubing for Components Used in General Purpose, High Purity and UltraHigh Purity Semiconductor Manufacturing Applications
- SEMI F22-1102 Guide for Ğas Distribution Systems
- SEMI F78-0703 Practice for Gas Tungsten Arc (GTA) Welding of Fluid Distribution Systems in Semiconductor Manufacturing **Applications**
- SEMI F81-1103 Specification for Visual Inspection and Acceptance of Gas Tungsten Arc (GTA) Welds in Fluid Distribution Systems in Semiconductor Manufacturing Applications

B31.3 Chapter X and refer to the other industry-specific design requirements, as mentioned earlier.

#### Safety considerations

Chapter X in B31.3 also integrates safety into high-purity piping systems, by adapting the B31.3 code to incorporate some preferential, safety-related

nuances that are associated with those industries that utilize high-purity piping systems. It does so by adapting its basic philosophy for safety to that of the industry-specific components, material joining methods, and purity requirements.

For example, achieving acceptable, repeatable welds is a key element during the fabrication of high-purity piping systems. These high-purity welds are accomplished most efficiently by means of a certified welding operator using an orbital welder. In addition to the requirements for acceptable gas tungsten arc (GTA) welds listed in B31.3, the user will need to refer to the criteria for acceptability of these types



#### **Pristine Processing**

of welds as defined in ASME-BPE, or the respective SEMI standards.

Biochemical processes. The addition of Chapter X could not be more timely. Over the past few decades, the breadth and depth of bioprocessing operations has continued to grow. For instance, the growth of the biofuels industry, coupled with the introduction of many new and evolving biotechnology-based processes throughout the CPI, has increased demand for process systems that are able to reliably handle biologics, both refined while controlling and industrial. the risk of contamination. This has pushed the demand for high-puritysystem design beyond the boundaries of the pharmaceutical industry, and has cascaded into industries that are typically unfamiliar with the need for system cleanability.

Biochemical processes utilizing hybrid cellulase enzymes and bacteria as catalysts demand a very different set of design guidelines compared to chemical processes that do not use living organisms. During biochemical processing, operators must maintain an environment that ensures that the specialized enzymes and bacteria can thrive and perform their consumption and processing of the pretreated feedstock.

However, efforts to maintain an environment that is conducive to the survival of the intended bacteria and enzymes also creates a suitable environment for unwanted bacteria to thrive. If the proper steps are not taken throughout the process, a proliferation of unwanted bacteria can devastate colonies of the desired bacteria, ruining the process. Specifically, efforts to prevent the contamination of a biochemical process, such as the one shown in Figure 1, requires a system design that is conducive to clean-inplace (CIP) or steam-in-place (SIP) capabilities (Note: SIP systems are often also defined as sterilize-in-place or sanitize-in-place systems; the terms are considered to be synonymous).

Figure 1 shows the key stages in the biochemical manufacture of ethanol. The only segments of this process that would require high-purity-piping design concepts are those that handle the enzymes (the primary catalyst enzymes for the process), namely in the enzyme production (if the enzymes are produced onsite rather than outsourced), saccharification and fermentation steps of the process.

Readers should note that the term high-purity, in the case of Figure 1, should not be misconstrued as an application for hygienic piping. During the production of ethanol, for example, the process system does not need to achieve a hygienic-level of cleanliness. But, it does have to be cleanable from an engineering standpoint.

This is where ASME B31.3 Chapter X and BPE work well together, by establishing acceptable design and fabrication requirements that are needed to achieve a cleanable system. These include criteria that define acceptable welds, surface finishes, mechanical joint connections, required slope, examination requirements and more.

Semiconductor manufacturing. Unlike bioprocessing operations, the semiconductor industry has a relatively narrow bandwidth of technological requirements that are used by other industries. Specifically, the high degree of purity, testing and the extremely sensitive instrumentation required by semiconductor manufacturers do not readily translate into practical use by many other industries. Thus, the semiconductor industry is relatively autonomous in that respect.

With some semiconductor manufacturers producing chips with dimensions at the 32-nanometer (nm) level, and research going on at the 15-nm level, it is easy to see why the design, fabrication, and maintenance practices required to ensure exacting purity requirements of their process fluid distribution systems are of paramount importance.

During semiconductor device fabrication, a variety of ultrahigh purity gases and chemicals are used during many of the processing steps, such as dry etching, wet etching, plasma etching, chemical vapor deposition, physical vapor deposition, and chemical-mechanical planarization. Engineering steps must be taken to ensure that these fluids be of ultrahigh purity, and must ensure that all associated tubing and components that distribute these fluids be maintained in an ultra-high-purity mode, as well.

To meet these demands, semiconductor manufacturers can now use B31.3 Chapter X in conjunction with the prevailing SEMI standards, as these two documents bring together the necessary criteria to establish acceptable design attributes, acceptable materials of construction, fabrication quality, testing protocols, validation, examination and inspection requirements.

#### The impact of Chapter X

As mentioned earlier, the addition of Chapter X to the content of B31.3 could not be timelier. Chapter X augments not only the B31.3 base code, but the ASME-BPE, ISPE baseline guide, and SEMI standards, as well, at a time when all of these high-purity industries are undergoing significant changes and facing more-rigorous purity requirements than ever before.

This preliminary movement of the ASME B31.3 piping code into the realm of high-purity process requirements is just an initial step. Once a segment of industry is adopted by ASME in such a manner, it adds a whole new level of thinking and evaluation to the standardization of that high-purity industry. The American National Standards Institute (ANSI) accreditation program. to which ASME adheres, legitimizes the standardization process and institutes an ongoing review process, which brings fresh new insights and technological advances to the continuing evolution of the industries it touches.

In general, many of the standards that have been developed specifically for high-purity industries have been driven and guided by the participation of active standards-development committee members, who are directly associated with the pharmaceutical and semiconductor industries. The addition of Chapter X invites the involvement of a more-diverse array of experts from a broader group of industries (for instance, the biofuels industry and other CPI sectors) that also have demanding purity and cleanability requirements. This promises to bring new vision and cross-industry collaboration when it comes to the ongoing evolution of high-purity pipingsystem standardization.

Edited by Suzanne Shelley

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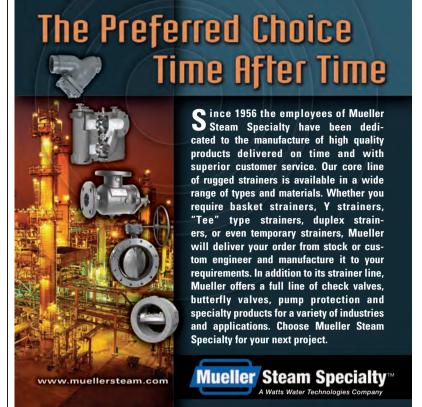
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Subgroup on High Purity Piping.



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New Products

#### Evaporators designed for mining and water treatment applications

DriBoss evaporation equipment (photo) is designed for mining, water-treatment and oil-and-gas applications. The efficient DriBoss wastewater evaporator is supported by company-provided services, as well as by ancillary components such as pumps, valves, controls and sensors, which are also supplied by this company. DriBoss equipment is available with both water-fracturing and atomized-mist designs, and can withstand caustic and corrosive environments. — Dust Control Technology, Peoria, Ill.

www.dustboss.com

#### A pump that is effective at low flowrates

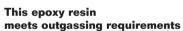
The STX1220A Sliding Vane line (photo) performs effectively and efficiently at lower flowrates (from 42–92 gal/min), and is ideal for use in handling low-viscosity chemicals, even those up to 22 centistokes. Constructed of stainless steel with PTFE mechanical seals, the pump is compatible with corrosive chemicals. The STX1220A's pushrod-free design allows higher pump speeds, from 700 to 1,200 rpm. — Blackmer, Grand Rapids, Mich.

www.blackmer.com

#### These floor decals withstand forklifts and pallets

Designed specifically for industrial facilities, DuraMarker decals can be used as floor signs and markers that stand up to turning forklift tires and sliding pallets. Made of a tough polycarbonate material, the DuraMarker signs have a smooth surface to resist torquing by forklift tires. Aside from durability, DuraMarker signs and floor striping are also designed for easy installation — just clean the surface, remove the backing and press down. Also, the floor decals can be easily removed if the facility layout changes they will not leave adhesive residue when peeled off. — FloorSignage LLC, Columbus, Ohio

www.floorsignage.com



A new expoxy resin formulation from this company, identified as 20-3652 Epoxy Potting and Encapsulating Compound, meets NASA outgassing requirements. The 20-3652 product is a filled epoxy casting, potting and encapsulating resin system. This is a dielectric-grade epoxy that exhibits excellent physical, thermal and electrical insulation properties. The 20-3652 resin shows excellent adhesion to most substrates. — *Epoxies, Etc., Cranston, R.I.* 

www.epoxies.com

#### Use this vacuum for 110-gal drums

The Reversible Drum Vac liquid vacuum (photo) can empty 110-gal drums containing coolant, hydraulic oils, wastewater and other materials. It can also be used for filling drums after a floor spill or transferring contaminated liquids to a filtration tank. Powered by compressed air, the Drum

Vac does not require electricity and has no moving parts. The two-way pump assembly comes with a number of accessories, such as a shutoff valve, a 10-ft flexible vacuum hose and a 110-gal drum with a bolt ring and lid. — Exair Corp., Cincinnati, Ohio

www.exair.com

### A modular version of wastewater treatment system is introduced

This company has introduced a modular version of its biological watertreatment system, known as ABMet. The patented system uses naturally occurring microbes to reduce levels of selenium and other metals in water from coal mines and power plants. The ABMet process involves running wastewater through a biologically active filter, which is "seeded" with microbes that target selenium and other potentially toxic metals. The initial ABMet systems were customized designs specific to a particular site, but the company is now offering its bioreactor water treatment product in





Microno



Kin-Tek Laboratories

a scaleable, modular format that reduces installation and overall project costs. The company says the modular version of ABMet is particularly wellsuited to meet the operational, regulatory and economic priorities of the coal mining industry. — GE Power & Water, Trevose, Pa.

www.ge.com

#### Bulk bag equipment that can empty partially

With a slide-gate system, the Material Master Bulk Bag Discharging equipment (photo) can halt material flow to allow partial bag discharge. The modularly constructed discharging apparatus is constructed from 304 stainless steel with continuous TIG (tungsten inert gas) welds, and features bag massaging to promote consistent material flow. The system's design ensures dust-tight operation, and easy operator access to the bag spout. — Material Transfer & Storage, Allegan, Mich.

www.materialtransfer.com

#### An industrial gas-standards generator for sulfur impurities

The Span Pac 261-I (photo) is an industrial gas-standards generator that is specifically configured to make standards for trace concentration sulfur impurities in an olefin matrix. The company's Trace Source permeation tubes add known concentra-



Material Transfer & Storage

tions of key sulfur species to a flow of high-purity ethylene or propylene gas. Dual temperature zones allow simultaneous addition of common sulfur contaminants at typical ppm and ppb concentrations. Rugged and easy to use, the Span Pac 261-I is designed for field mounting with process analyzers, and can be operated remotely. — Kin-Tek Laboratories Inc., La Marque, Tex. www.kin-tek.com

#### **New CFD software allows** increased functionality

A new post-processing tool for computational fluid dynamics (CFD) analysis allows engineers to more easily manipulate and manage data from multiple simulations. Known as Chorus, the software package can quickly analyze up to thousands of computer simulation cases simultaneously, enabling new insights. The Chorus software package features an integrated data management system. and is designed to allow engineers to collaborate and share results. The company says that the software's core benefit is bringing together an underlying understanding of the physics of fluid dynamics with statistical analysis abilities. Among the key features provided by the Chorus software are a unified environment for enhanced metadata and field-flow analysis, rapid visualization and exploration of 3D surfaces, and automated plot and data extraction. — Tecplot Inc.. Bellevue, Wash.

www.tecplot.com

#### Protect concrete structures with this compound

MCI POWR is a water-based alkylalkoxy silane that contains this company's migrating corrosion-inhibitor technology. Specifically designed to protect new and existing concrete structures from moisture, salt intrusion and freeze/thaw scaling. MCI POWR can extend the life of reinforced concrete bridges, highways, parking decks and concrete floors. According to the company, the small molecular size of MCI POWR allows deep penetration into concrete and provides repellency by

chemically reacting with the cement. Treated structures are hydrophobic. but retain their original appearance. MCI POWR can be applied by spray, brush, roller or squeegee. — Cortec Corp., St. Paul, Minn.

www.cortecvci.com

#### Electromagnetic interference won't affect this position sensor

Billed as the world's first commercially available fiber-optic position sensor (FOPS), the MR330 Series position sensor (photo) has an all-optical design that is not affected by electromagnetic interference such as lightning, radiation, magnetic fields and other harsh environmental conditions. The company says the product's fiber optics make it well suited to long-distance position sensing (up to hundreds of meters) without being affected by ground loop problems. The new FOPS employs a novel optical technique inside a passive sensor, and has an active controller connected to a duplex fiber-optic link. The MR330 works well in areas that require precise motion control and position measurement in challenging environments, such as oil rigs, satellite antennas, solar panel arrays, actuator systems, steel mills, transportation systems and medical instrumentation. — Micronor Inc., Newbury Park, Calif.

www.micronor.com

Scott Jenkins

#### **Focus**

(Continued from p. 27)

#### Globe valves that are available in many configurations

This company's Clampseal Y-angle and T-pattern globe valves are available in over 12,000 configurations, including sizes from 0.5 to 4 in. In addition, they come with socket welds, butt welds, clamp connectors, flanged ends or a variety of specialized arrangements. The Clampseal globe valves are also available in a variety of materials, including carbon steel (SA 105). low-alloy SA 182-F22 and F91, as well as 316 stainless steel and more. Design features also include a pressure-seal bonnet and a quickreplacement packing chamber. — Conval Inc., Somers, Conn.

www.conval.com

#### A butterfly valve with high strength and chemical resistance

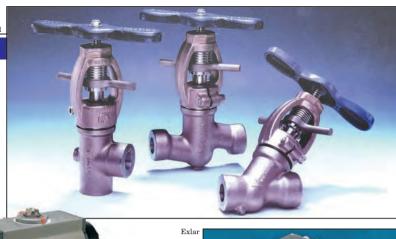
The FK Polypropylene Butterfly Valve Series is designed for highly corrosive environments. The FK valve's design features a special trapezoid shape in the liner, and a serrated body cavity that guarantees a bubble tight seal while keeping breakaway torque at a minimum. The versatile valve has double self-lubricating seals and direct-mount actuator capability. — Assured Automation. Clark. N.J.

www.assuredautomation.com

#### These actuators now have a UL listing

The Tritex II electric actuators are now listed by UL (Underwriter's Laboratories Inc.: Camas, Wash.: www. ul.com), with file number E325731. The a.c.-powered Tritex II combines a brushless servomotor with either rotary or linear actuation and the company's digital position-control system. The actuators' built-in mechanical converters eliminate the need for traditional ball-screw or gear-reducer mechanisms. Incorporating all required position control and power electronics into the actuator also eliminates the external servo amplifier and the cables associated with a typical servo system. — Exlar Corp., Chanhassan, Minn.

www.exlar.com







#### These ball valves have live-loaded stem packing

This company's 334 Series ball valve incorporates several innovative design features for improved process performance. The valve features high-cycle, live-loaded stem packing and direct-mount actuation along with a variety of seat options. The spring-energized stem seals self-adjust to compensate for wear, pressure and temperature changes, ensuring a leak-tight seal and a long service life. — Inline Industries, Baldwin Park, Calif.

www.ballvalve.com

#### This line of cast-bronze valves is American-made

Valves in a new line of cast-bronze products from this company are cast, machined and tested in the U.S. Gate, globe and check valves are available. The multiturn valves are suitable for applications such as heating, ventilation and air conditioning (HVAC) systems, potable water, irrigation and OEM. The valves are also available in a lead-free option. — Conbraco Industries Inc., Matthews, N.C.

www.apollovalves.com

#### This valve lock has universal compatibility

The NL-H Hybrid lock provides secure lockout for all sizes of manual valves, and has universal compatibility, the

company says, eliminating the need for maintaining an inventory of assorted lockout products for each valve type. The universal NL-H uses a flexible steel cable to immobilize a valve's handwheel or lever. The unit secures to the valve with a padlock. Constructed of 316 stainless steel, the NL-H valve lock is suitable for use in harsh environments, such as in offshore drilling or chemical applications. — Netherlocks Safety Systems, Houston

www.netherlocks.com

### Valves for proportional pressure relief

The RHPS Series PRV model pressure relief valves provide proportional venting of overpressures for piping systems up to 1 in. The valves are designed to operate smoothly, opening gradually and reseating accurately in proportion to the increase and decrease in pressure over the set opening pressure. The PRV's balanced design is unaffected by system backpressures of up to 50% of the valve's set pressure. They are constructed of 316L stainless steel, and have fluorocarbon or nitrile seats and seals. The PRV valves are available in a wide variety of set pressures and can operate in temperatures from -4 to 176°F. -Swagelok Co., Solon, Ohio

www.swagelok.com

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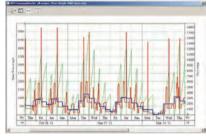
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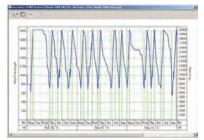
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#### **PLANT WATCH**

#### BASF to build world's largest single-train TDI plant in Europe

May 26,2011 — BASFSE (Ludwigshafen, Germany; www.basf.com) will build the world's largest single-train toluene diisocyanate (TDI) plant in Europe. The plant will have a capacity of 300,000 metric tons (m.t.) per year and will be fully integrated with precursor production. The TDI plant will be located at one of the company's integrated Verbund sites in Antwerp, Belgium or Ludwigshafen, Germany and will start production in 2014.

#### UOP technology selected to expand Iragi fuels production

May 25, 2011 — The State Company for Oil Projects (SCOP), under the Ministry of Oil for Iraq, has selected UOP LLC (Des Plaines, III; www.uop.com), a Honeywell Company (www.uop.com), to provide key technologies to process 300,000 barrels per day (bpd) of domestic crude oil into gasoline and diesel fuel at a new facility in Nassiriya, Iraq. UOP will provide reforming, isomerization, fluid-catalytic-cracking (FCC) and selective hydrotreating technologies. Design will begin in the 2nd Q of 2011.

#### M&G to launch new PET and PTA plants in the U.S. Gulf Coast region

May 12,2011—The M&G Group (Tortona, Italy; www.gruppomg.com), has announced that it will build a new polyethylene terephthalate (PET) plant in the U.S., co-located and integrated with a new purified terephthalic acid (PTA) plant. The new PET single-line plant will have a capacity of 1-million m.t./yr. The new PTA unit will have a capacity of 1.2-million m.t./yr and will provide full upstream integration for all of M&G's U.S. PET capacity. Two technologies are at the final evaluation stage for this PTA plant, which will be the largest in the Americas and among the largest in the world, says the company.

#### DuPont to expand titanium dioxide capabilities

May 11, 2011 — DuPont (Wilmington, Del.; www. dupont.com) has announced a comprehensive titanium dioxide expansion plan that will add about 350,000 m.t./yr of global capacity. A new line at the Altamira, Mexico site is scheduled for completion by year-end 2014, and will provide approximately 200,000 m.t./yr of new capacity. Facility upgrades underway at DuPont's five TiO2 manufacturing sites will continue over the next three years, yielding an additional 150,000 m.t. of capacity.

#### DSM and Roquette to build commercialscale bio-based succinic acid plant

May 9, 2011 — Royal DSM (Heerlen, the Netherlands; www.dsm.com) and Roquette Frères (Lestrem, France; www. roquette.com) will build a plant for the production of bio-based succinic acid. With a capacity of about 10,000 ton/yr, the plant will be Europe's largest bio-based succinic acid facility. It is expected to come on stream in 2012 and will be built on the premises of Roquette in Cassano Spinola, Italy. Financial details of the investment will not be disclosed.

#### Lanxess expands capacity for polychloroprene solid rubber

May 6, 2011 — Lanxess AG (Leverkusen, Germany; www.lanxess.com) is investing €17 million in the expansion of its production capacities for polychloroprene solid rubber. The expansion at the Dormagen, Germany site is scheduled for completion by the end of 2012 and will increase production by 10% to 63,000 m.t./yr.Lanxess produces and markets polychloroprene solid rubber under the brand name Baypren. Uses for this product range include the production of cable sheathing, hoses, belts, seals and air springs.

#### The Linde Group to build a hydrogen and synthesis gas plant in China

May 4, 2011 — The Linde Group (Munich, Germany; www.linde.com) plans to build a large hydrogen and synthesis gas plant in the Chongqing Chemical Park in Western China in a joint enterprise with Chongqing Chemical & Pharmaceutical Holding Co. (CCPHC). The project has a total investment value of around €200 million. Linde holds 60% of the shares in the joint enterprise with CCPHC. The plant, which will be supplied by Linde's Engineering Division, is expected to come on stream in the 3rd Q of 2014.

#### **MERGERS AND ACQUISITIONS**

#### Ashland plans to acquire International Specialty Products

May 31, 2011 — Ashland Inc. (Covington, Ky.; www.ashland.com) has agreed to acquire International Specialty Products Inc. (ISP; Wayne, N.J.), a global specialty chemical manufacturer of functional ingredients and technologies. Under the terms of the stock purchase agreement, Ashland will pay approximately \$3.2 billion for the business in an all-cash transaction.

#### BASF and Ineos sign joint-venture contract for Styrolution

May 30, 2011 — BASF SE and Ineos Industries Holdings Ltd. (Lyndhurst, U.K.; www.ineos.com) have taken an important step toward the establishment of the joint venture (JV) company Styrolution. On May 27, the companies signed a contract, which regulates the formation of Styrolution. The establishment of the JV is subject to approval by the appropriate antitrust authorities. The company headquarters will be located in Frankfurt am Main, Germany and 50% of the shares will be owned by BASF and 50% by Ineos. (For more, see CE, January 2011, p. 59)

#### Huber renames business unit to reflect broader base of fire retardant additives

May 11, 2011 - Huber Engineered Materials (HEM; Atlanta, Ga.; www.hubermaterials. com) is renaming its Alumina Trihydrate (ATH) business unit Fire Retardant Additives. The new name is said to more accurately reflect the variety of non-halogen fire-retardant and smoke-suppressant technologies, brands and products now offered for a variety of end-use applications

#### AkzoNobel plans to restructure ICI Pakistan

May 2, 2011 — Akzo Nobel N.V. (AkzoNobel; Amsterdam, the Netherlands; www.akzonobel.com) has announced its intention to seek agreement from the Board and shareholders of ICI Pakistan Ltd. (ICI Pakistan) to separate the organization's paints and chemicals businesses. Under the terms of the proposal, AkzoNobel would retain direct majority control of the paints business by separating it into a new legal entity (AkzoNobel Pakistan Ltd.) through a de-merger process approved by the Pakistani courts. Subsequently, AkzoNobel would dispose of its entire shareholding in the remainder of ICI Pakistan.

#### Clariant closes its acquisition of Süd-Chemie

April 26, 2011 — Clariant AG (Muttenz, Switzerland; www.clariant.com) has completed the purchase of 96.15% of the shares in Süd-Chemie from One Equity Partners and the family shareholders. Clariant will make a public offer to acquire the outstanding shares from Süd-Chemie minority shareholders. The overall transaction value amounts to approximately €1.9 billion (CHF 2.5 billion).

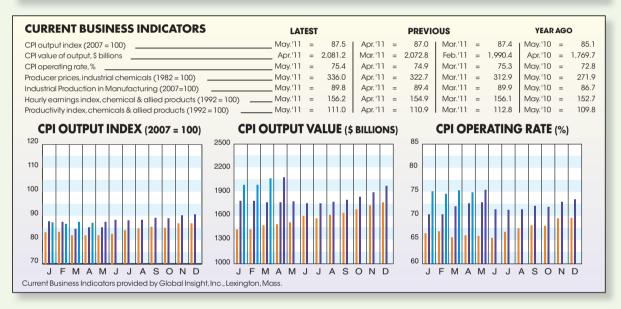
Dorothy Lozowski

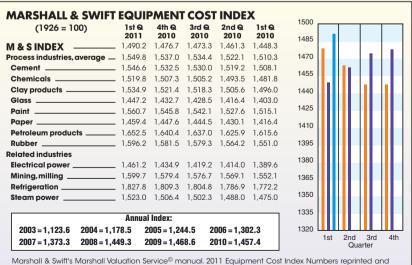
#### FOR ADDITIONAL NEWS AS IT DEVELOPS, PLEASE VISIT WWW.CHE.COM

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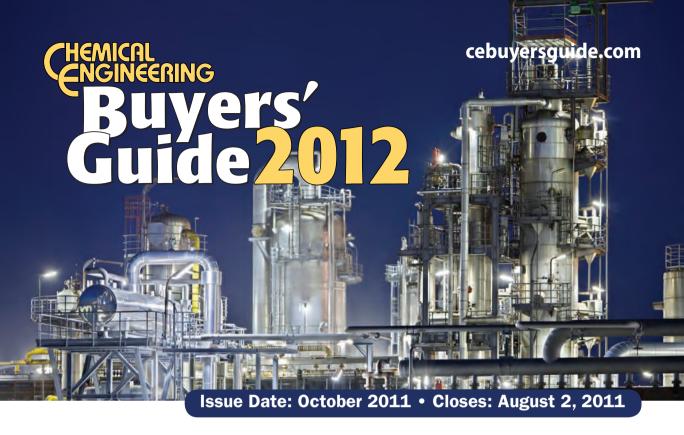
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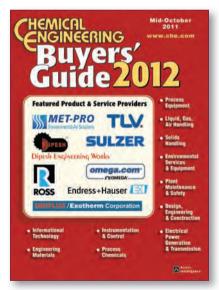
#### **CURRENT TRENDS**

Capital equipment prices, as reflected in the CE Plant Cost Index (CEPCI), increased approximately 1.14% on average from March to April.

Meanwhile, according to the American Chemistry Council's (Washington, D.C.; www. americanchemistry.com) most recent weekly economic report at CE press time, industrial production increases in May were led by pharmaceuticals and specialty chemicals, while production actually fell in basic chemicals.

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