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August  
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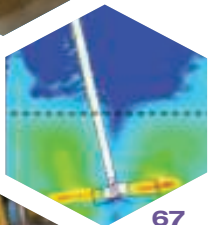
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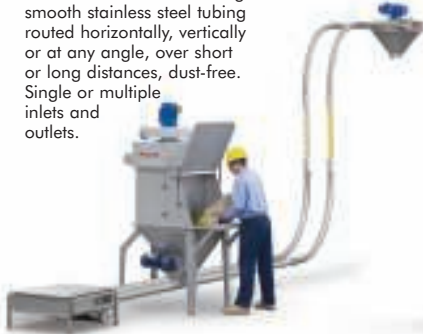
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Cover: Rob Hudgins

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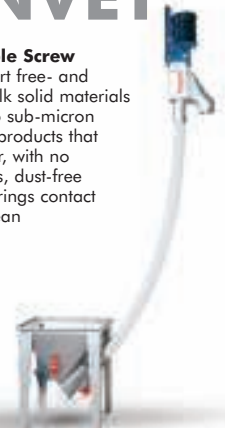
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## 2015 Kirkpatrick finalists

Five innovative technologies that were commercialized in the past two years have been selected as finalists for the 2015 Kirkpatrick Award for Chemical Engineering Achievement. The winner will be announced on November 18 at the Chem Show in the Jacob Javits Center in New York. Here is a brief summary of the finalists:

**AM Technology — Coflore reactor.** Flow, or continuous reactors have a number of advantages over batch reactors for certain applications, including a reduced size. For mass-transfer-limited reactions, however, good mixing is essential and relying on passive mixing, such as static mixers, can be insufficient. AM Technology has developed a flow reactor based on the same principle used to mix paint in aerosol cans. The mixer element in the Coflore reactor is loose, and mixing is generated by lateral vibration of the reactor body. This eliminates the need for rotating shafts and seals, and delivers better mixing than passively mixed systems.

**CB&I — CDAlky alkylation technology.** This sulfuric acid alkylation process is for the production of motor fuel alkylate. The novelty of the technology is the design of the reactor system, where proprietary static internals create the contact needed between the hydrocarbon and the acid phases at lower temperatures (below 0°C) than conventional technologies. Rotating mixers in conventional technologies typically cannot go lower than 7–8°C because of the acid phase's high viscosity. The lower reaction temperatures favor the formation of high-octane trimethylpentane and minimize unwanted side reactions.

**Clariant — HGM technology for propylene dehydrogenation.** Clariant has introduced an innovation to the Catofin process, used for producing isobutylene and propylene. The innovation is the addition of Heat Generating Material (HGM), which is a metal oxide on a proprietary carrier that is loaded into the catalyst bed. HGM is not a catalyst, and is inert to the feedstock. It undergoes oxidation and reduction, both of which generate heat inside the catalyst bed to drive the endothermic dehydrogenation reaction. The heat generated improves the catalyst-bed temperature profile to increase olefin selectivity and limit byproduct formation. The lifetime of the catalyst is also increased.

**DOW Performance Plastics — Intune olefin block copolymers.** Intune is a family of olefin block copolymers (OBCs) that incorporates both crystalline polyethylene and crystalline isotactic polypropylene — the two largest-volume thermoplastic polymers in production. Typically, blends of these two polymers exhibit poor physical properties because the polymers are immiscible, and multi-layer structures fail due to poor interlayer adhesion. Intune OBCs are produced through developments in catalyst design, the nature of the chain-shuttling agent and reactor control. The interplay of selective catalyst and chain-shuttling agents make the unique polymer structure possible.

**Newlight Technologies — AirCarbon Process.** Newlight has developed a new biocatalyst that allows thermoplastic polymers to be manufactured from methane more cost effectively than from oil-based raw materials. The new biocatalyst can produce nine times more product for the same amount of catalyst input as compared to earlier biocatalysts. In the AirCarbon process, air and methane are mixed with the biocatalyst to produce polymer at ambient operating conditions.

Dorothy Lozowski, Editor in Chief



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## Chopey scholarship awarded

The 2015 Nicholas P. Chopey Scholarship for Chemical Engineering Excellence has been awarded to Stephanie Kong, a student at the University of Buffalo ([www.buffalo.edu](http://www.buffalo.edu)), who is majoring in both chemical engineering and Spanish.



Kong is a member of Tau Beta Pi (National Engineering Honor Society) and Phi Beta Kappa, and is on the Engineering Dean's List. This year, she was selected as a Goldwater Scholar for her research in model surfactant systems that can be used to create eco-friendly dispersants for oil spills. Kong has also worked for Praxair's Research and Development Division. She plans to complete her double degree in May 2016 and then begin her studies toward a Ph.D. degree in chemical engineering the following fall.

### About the scholarship

*Chemical Engineering* has sought to bring recognition to the chemical engineering profession and to continually advance that profession, since the magazine's founding in 1902. To help advance those goals, *CE* established the annual Chopey Scholarship for Chemical Engineering Excellence in late 2007. The award is named after Nicholas P. Chopey, the magazine's former Editor-in-Chief, whose valuable contributions to *CE* over the 47 years that he devoted to it are long-lasting. To honor his contributions to the chemical engineering profession, *CE* established the scholarship in his name.

**Applicant qualifications.** The scholarship is awarded to current third-year students who are enrolled in a full-time undergraduate course of study in chemical engineering at one of the following four-year colleges or universities, which include Mr. Chopey's alma mater and those of our editorial staff:

- University of Buffalo
- University of Kansas
- Columbia University
- University of Virginia
- Rutgers University
- University of Oklahoma

The scholarship is a one-time award. The program utilizes standard Scholarship America recipient selection procedures, including the consideration of past academic performance and future potential, leadership and participation in school and community activities, work experience, and statement of career and educational goals.

More information about the award, including how to apply and how to contribute a donation, can be found at [www.chemengonline.com/npcscholarship](http://www.chemengonline.com/npcscholarship)

### Postscripts, corrections

June, 2015, "High-Performance Internals Boost Tower Capacities", p. 24 and p. 27. In the article and in the photo caption for Figure 1, NeXRing is incorrectly referred to as a structured packing — it should be described as a random packing. The corrected version of the full article can be found at [www.chemengonline.com](http://www.chemengonline.com).

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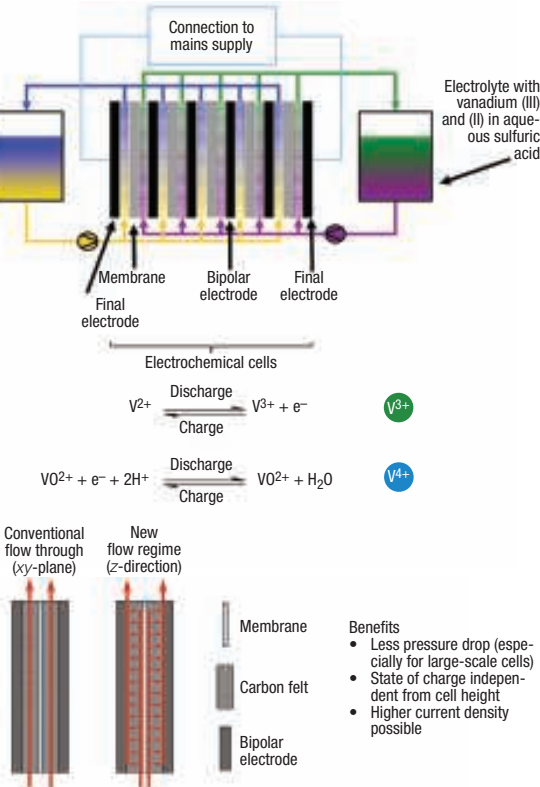
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## Commercial debut slated for redox flow batteries

A new concept for large-scale redox-flow batteries (RFB) for energy-storage applications has been developed by ThyssenKrupp Industrial Solutions AG (Dortmund, Germany; [www.thyssenkrupp-industrial-solutions.com](http://www.thyssenkrupp-industrial-solutions.com)). The system is based on the oxidation and reduction of vanadium ions (diagram, top) for either storing electrical energy (charging), which is produced by renewable sources (solar or wind), or utilizing this energy (discharging) during periods when power generation is interrupted (windless days or nighttime).

The all-vanadium RFB was designed in order to take advantage of economy-of-scale for large flow-through electrochemical cells, said Niels Bredemeyer, senior chemist at the company's Process Technology business unit, during a congress session at Achema (June 15–19; Frankfurt, Germany). Conventional flow-through cells suffer from large pressure drops, flow maldistribution and a state-of-charge (SOC); remaining capacity of a battery that depends on the cell height, explained Bredemeyer. These drawbacks are especially problematic when scaling up to large scale, he said.

To overcome these issues, a new and unique flow regime was developed, based on the company's experience with large-scale electrolyzers for chlor-alkali and HCl pro-



Edited by:  
**Gerald Ondrey**

### FIRST FDI ADOPTER

With its Field Information Manager software, ABB (Zurich, Switzerland; [www.abb.com](http://www.abb.com)) has become the first host company to adopt new field-device integration (FDI) technology. The FDI platform offered by the FieldComm Group (Austin, Tex., [www.fieldcommgroup.com](http://www.fieldcommgroup.com)) brings together for the first time the HART and Fieldbus Foundation communication protocols to create industry-wide device-communication consistency. ABB Field Information Manager is designed for use on touchscreen tablets, and features pre-loaded device packages, customizable parameters and an archive for existing devices, so all equipment information is stored within the platform. It can be installed in just three

(Continues on p. 10)

duction. Instead of electrolyte flowing in the *xy*-plane as in conventional flow-through designs, the electrolyte is first fed perpendicular to the electrodes (*z*-direction) through a number of channels (diagram, bottom). This feature results in a lower pressure drop and an SOC that is independent of the cell height, as well as the ability to operate at higher current densities, says Bredemeyer.

Laboratory trials have been performed since 2012 in a test rack with two one-compartment testcells

(OCTs) with 0.16-m<sup>2</sup> of active area at the Niedersachsen Energy Research Center (EFZN; Goslar, Germany). Data from the test rig have been used as the basis for scaleup to a mid-sized OCT (about 0.7-m<sup>2</sup> area), and the company plans to commercialize the technology in 2016, targeting industrial-sized energy storage systems in the megawatt range. This size RFB (2.7-m<sup>2</sup> area, 20-MW power) is suitable for grid operators, integrated chemical plants and utility companies, says Bredemeyer.

## Generate steam by solar heating with nanoporous graphene

Researchers, led by professor Mingwei Chen at the Advanced Institute for Materials Research (AIMR) of Tohoku University (Sendai City, Japan; [www.wpi-aimr.tohoku.ac.jp](http://www.wpi-aimr.tohoku.ac.jp)), have developed a material that efficiently generates steam by solar heating. In laboratory trials using simulated solar radiation, the N-doped hydrophilic nanoporous graphene was

shown to generate steam with 80% conversion efficiency, which is well above the 56% efficiency achieved by graphite powders.

The heating rate with the new material was also found to be very fast, with an evaporation rate of 1.5 kg/h-cm, even though the graphene material is two orders of magnitude thinner than graphite materials. The researchers believe the material shows prom-

ise for low-cost, highly efficient solar steam generation because it uses environmentally friendly carbon materials with the lowest molar specific heat as opposed to using metals with high environmental burden. They also plan to develop a procedure to mass-produce the nanoporous graphene for industrial applications, including systems to generate water and to purify contaminated water.

Note: For more information, circle the 56-digit number on p. 74, or use the website designation.

minutes, says ABB. Furthermore, work processes that previously required multiple file downloads are now bundled so that only one file is required.

## OIL-FREE TURBINE

Siemens AG (Munich, Germany; [www.siemens.com](http://www.siemens.com)) has delivered for the first time, a steam turbine in the capacity range up to 10 MW that operates almost entirely without lubricants. The bearing systems consist of completely oil-free, air-cooled, electromagnetic bearings. The trial run of the first turbine equipped with magnetic bearings was successfully completed in Vattenfall's lignite-fired Jämschwalde steam power plant, located in southeastern Brandenburg. The turbine has been running reliably in regular full-load operation since February 2015, at speeds of up to 5,700 rpm. It is used as a feedwater pump drive.

Steam turbines equipped with magnetic bearings are more environmentally friendly, as they use nearly no oil, and also offer substantially better fire protection, says the company. Because the rotor makes no contact with the bearings and is not supported by a film of oil, there is no longer any wear or friction resistance present. As a result, the efficiency of the steam turbine can be increased by up to 1%, says Siemens. The first SST-600 steam turbine with magnetic bearings was developed in cooperation with the University of Zittau/Görlitz (Germany; [www.hszg.de](http://www.hszg.de)).

## RARE-EARTH METALS

A team of chemists at the University of Pennsylvania (Philadelphia; [www.upenn.edu](http://www.upenn.edu)) has developed a process for separating the rare-earth metals neodymium and dysprosium, which are used together in permanent magnets to provide the desired magnetic properties and allow the magnets to operate effectively over a range of temperatures.

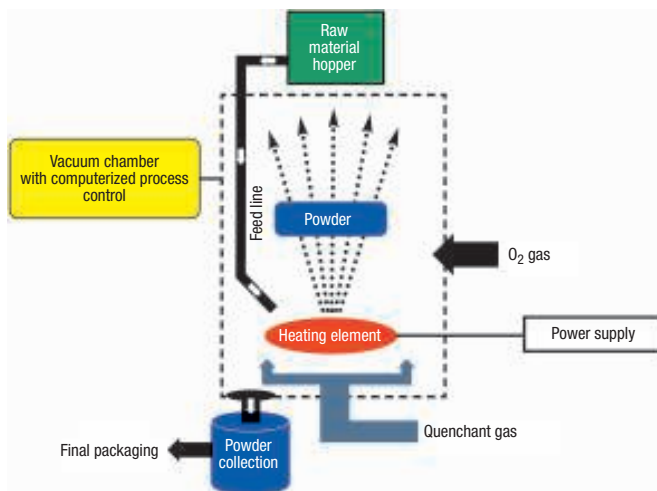
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# Iron nanocatalysts boost ammonia production

Nanoscale iron catalysts, designed for industrial chemical applications, have been validated in commercial ammonia-production reactors in China. According to manufacturer QuantumSphere, Inc. (QSI; Santa Ana, Calif.; [www.qsinano.com](http://www.qsinano.com)), incorporation of the nanocatalysts resulted in a 10–15% increase in ammonia production rate compared to standard ammonia catalysts. Iron nanocatalysts produced from QSI's manufacturing process (diagram) are highly uniform in size and have chemically active surfaces that allow reactions to occur at lower temperatures and pressures and at a higher rate.

To manufacture the nanocatalysts, QSI developed an automated gas-phase condensation process (*Chem. Eng.*, September 2004, p. 15) in which electric heating elements are used to melt and then vaporize iron in a vacuum chamber. A laminar flow of low-temperature helium condenses the metal vapor into nanoscale droplets, which then solidify into uniform spherical nanocatalysts. "By carefully controlling the vacuum level, metal flux, quench-gas flow and other process parameters, we are able to produce nanocatalysts with tailored particle sizes, tight size distributions and high surface energies," explains Kevin Maloney, QSI's CEO.

In a secondary operation, QSI employs a proprietary technique to partially passivate the nanocatalysts, creating a thin metal



oxide shell around the iron core which allows for safe handling and transportation of the nanocatalysts.

Alternative methods for creating nanocatalysts tend to be expensive and difficult to scale, Maloney says. After several years of process development, QSI has the capacity to produce iron nanocatalysts on the order of metric tons per year.

For ammonia production, iron nanocatalysts are coated onto conventional ammonia-production catalysts to increase product output without requiring capital expenditures or modifications to the existing production plant, Maloney says.

Although commercialization of nano-iron for the ammonia industry is the current focus, the QSI process can be used to make nanocatalysts of other metals, including copper, silver, nickel, cobalt, palladium and others. The company is working with partners to develop those products for additional industrial chemical applications.

## Conductive plastic reduces battery weight

Integral Technology (Canton, Mich.; [www.electriplast.com](http://www.electriplast.com)) has developed bipolar plates made from its electrically conductive plastic that can be used to reduce the space required for 12- and 24-V batteries, and virtually eliminate the need for lead metal in traditional lead-acid vehicle batteries. The bipolar plates, with positive and negative sides, are made from the company's conductive plastic, which consists of copper-coated carbon nanofibers inside conventional plastic resins (*Chem. Eng.*, June 2015, p. 11).

After the electrically conductive plastic plates are made, the company applies a patented process that renders them bipolar.

The plates are then stacked for use in battery applications. "The plates can be made in any shape, and they can greatly reduce the space required for the same power output," explains Doug Bathauer, CEO of Integral Technologies. Also, use of the bipolar plates, which contain no lead, allows a drastic reduction in the amount of lead compared to what would be found in conventional 12- and 24-V batteries.

The bipolar plates have been fully tested in batteries, and the company is now searching for a development partner to help commercialize the product for the automobile and other markets.

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## A faster way to determine $pK_a$ values

Instead of the currently used liquid-liquid extraction method, which can be expensive and energy-intensive, the UPenn team devised a ligand-based technique that works in a fraction of the time and energy required for liquid-liquid extraction, according to lead investigator Eric Schelter.

When the three-branched ligand is added to a mixed powder containing dysprosium and neodymium, its arms close around the metal atoms, leaving an aperture that is larger for neodymium than for dysprosium because of a slight difference in atomic size. The larger aperture allows the neodymium-ligand complex to form dimers, which can dissolve in aromatic solvents. The tighter dysprosium-ligand complex does not form dimers, and does not dissolve, allowing the metals to be separated. After stripping the ligand off with an acid, samples of both metals can be obtained with 95% purity, and the ligand can be recovered, the researchers say.

### METHANE TO METHANOL

A new zeolite catalyst for the partial oxidation of methane to methanol has

(Continues on p. 16)

Researchers from the University of Tasmania (Hobart, Australia; [www.utas.edu.au](http://www.utas.edu.au)) and the University of Barcelona (Spain; [www.ub.edu](http://www.ub.edu)) have found a method to drastically speed up capillary electrophoresis (CE) for measuring  $pK_a$  values (acid dissociation constants). This is especially valuable for pharmaceutical companies that have to test compounds' suitability for making drugs. One of the parameters those companies must measure is the  $pK_a$  which indicates how much a drug candidate will dissociate at a given pH.

Previously, scientists have streamlined the CE method by injecting analytes together with compounds with well-known  $pK_a$  values into the capillary tube. The reference compounds reduce the number of trials needed. However, this was still cumbersome due to the job of preparing the needed buffers, and at least 20 trials were needed over a range of pH levels.

Now, professor Michael C. Breadmore

of the University of Tasmania and coworkers made a microfluidic device that automatically mixes buffers of a specified pH in less than a minute. The device was built from commercially available parts with a 3D micromixer designed and printed to combine four different reagents into one single homogeneous flow.

The device automatically mixes, within a few seconds, four reagents in situ creation of the separation electrolyte with a pH range of 2–13, ionic strength of 10–100 mM and organic solvent content from 0 to 40%. According to the team, the combination of 1.2 kV/cm and a short effective length (15 cm to the ultraviolet detector) makes it possible to obtain electrophoretic separations in 20 s.

The team tested their method on a number of drugs, including ibuprofen, papaverine, clonidine and mefenamic acid, all with well-known  $pK_a$  values. The resulting  $pK_a$  values were within 1% of those obtained by a traditional method. The team's method could determine a  $pK_a$  value within 2 or 3 min.

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## Sensitive and rapid screening of low-mass hazardous compounds

Researchers from the Chinese Academy of Sciences (Beijing, China; [www.cas.cn](http://www.cas.cn)) and Fuzhou University (Fuzhou, China; [www.fzu.edu.cn](http://www.fzu.edu.cn)), led by the Academy's Prof Qian Liu, have reported a new method for high-throughput, sensitive, and rapid screening of low-mass hazardous compounds in complex media without the need for complicated sample preparation.

Current methods for screening blood, urine or river water for toxic substances involve multiple steps that require expensive equipment and may require days to complete. Traditionally, matrix-assisted laser desorption/ionization-time-of-flight mass spectrometry (MALDI-TOF MS) has provided a powerful tool for high-throughput analysis of large molecules such as proteins, peptides, and polymers. However, its application to small molecules is hindered

by insufficient sensitivity, ionization suppression effect, and matrix interferences in low-mass regions.

To speed up the process, the researchers tested four commercially available tubular mesoporous forms of carbon and silica for their ability to act as molecular sieves. The mesoporous materials are particles made of bundles of nanotubes with aligned channels, 4 to 7 nm in diameter. The researchers used those materials to concentrate small molecules such as bisphenol S and cetyltrimethylammonium bromide — out of a mixture containing large molecules. When they mixed these particles into a liquid sample, the small molecules entered and adsorbed to the tube walls while the larger molecules were excluded. They then recovered the mesoporous materials and extracted the compounds collected within, and deposited the compounds onto sheets of graphene for detection,

using MALDI-TOF MS analysis.

Ordered mesoporous carbon CMK-8 was used, which can exclude interference from large molecules in complex samples and efficiently enrich a wide variety of low-mass hazardous compounds. The researchers claim their method can work at concentrations down to part per trillion (ppt) levels, and is much faster than conventional methods. This new approach concentrates the toxic substances, improving sensitivity, while also reducing interference from salts and other contaminants. "We can solve two problems at once," says Liu.

They applied their method to rapidly screen and identify serum samples from 30 workers in a perfluorochemicals plant in Wuhan, and from five athletes in Beijing. They detected high levels of perfluorochemicals in the workers' blood, but not in the athletes'. The entire process took three to four hours.



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## Ethanol-based fuel cells get a boost with a new catalyst

**H**ideki Abe and colleagues at the National Institute for Materials Science (NIMS, Tsukuba City, Japan; [www.nims.go.jp](http://www.nims.go.jp)), in collaboration with the Global Research Center for Environment and Energy based on Nanomaterials Science (GREEN) and Tohoku University, have developed a new catalyst materials — intermetallic TaPt<sub>3</sub> nanoparticles — that can efficiently produce electric power from

ethanol fuel at ambient temperatures and pressures without generating toxic waste gases. The intermetallic TaPt<sub>3</sub> nanoparticles exhibit excellent catalytic performance for ethanol oxidation in polymer electrolyte membrane (PEM) fuel cells, producing a current density of about 4 mA/cm<sup>2</sup> at electrode voltages of 0.4 V, which is nearly ten times higher than that by the state-of-the-art Pt<sub>3</sub>Sn nanoparticles. In single-cell tests, the TaPt<sub>3</sub> NPs de-

livered output currents of more than twice that from Pt-based cells.

The TaPt<sub>3</sub>'s ability to cleave ethanol's C–C bond makes the catalyst a promising way to utilize ethanol as a fuel, without generating NOx or CO that are produced by ethanol combustion in a diesel engine.

The researchers plan to enhance the yield for TaPt<sub>3</sub> synthesis to produce several grams for operating a stack of PEM fuel cells.

## Mitigate NOx emissions and increase energy efficiency

**I**n an industrial-scale demonstration, a technology designed to lower NOx emissions has also boosted energy efficiency, according to developer ClearSign Combustion Corp. (Seattle, Wash.; [www.clearsign.com](http://www.clearsign.com)). In addition to keeping NOx emissions at 5 ppm or below in industrial burners, ClearSign's Duplex technology was found to increase thermal efficiency in a retrofitted once-through steam-generator (OTSG) system with a design capacity of 62.5

MMBtu/hr used for enhanced oil recovery (EOR) at Aera Energy's Belridge oil field, near Bakersfield, Calif.

The greater thermal efficiency can lead to overall energy savings of 3–4% when compared to systems operating with fluegas recirculation (FGR), which translates into yearly cost savings on the order of \$100,000 for OTSGs of this capacity, says Roberto Ruiz, chief operating officer at ClearSign. Conventional low-NOx burners operate with FGR

to keep NOx emissions down, but such systems penalize thermal efficiency, which leads to higher fuel costs, Ruiz explains.

The Duplex technology, first introduced in 2013, helps improve burner flame shape and heat transfer, while optimizing combustion chemistry (*Chem. Eng.*, July 2013, p. 13).

The company recently signed a separate agreement with another California-based company to retrofit another 25 million Btu/h OTSG system.

## Model relief devices in flow-balanced simulations.

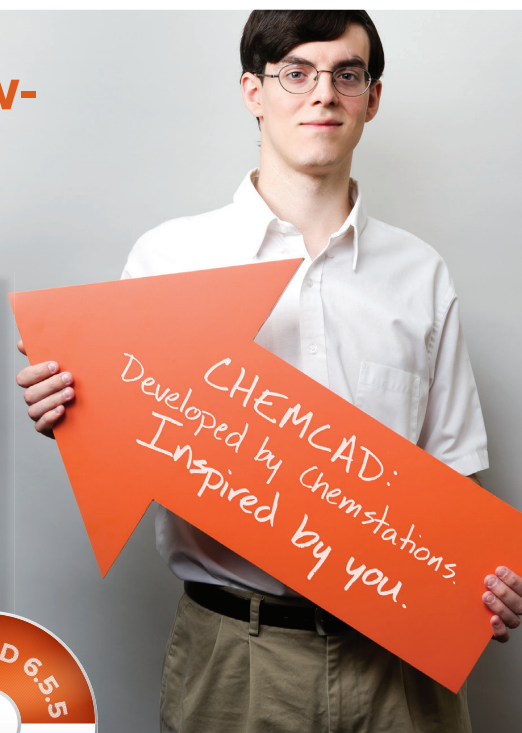
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been developed by a team of researchers from the Technical University of Munich (TUM; Germany; [www.tum.de](http://www.tum.de)), Eindhoven University of Technology ([www.tue.nl](http://www.tue.nl)) and University of Amsterdam (both the Netherlands; [www.uva.nl](http://www.uva.nl)). This copper-exchanged zeolite with mordenite structure mimicks the reactivity of methane monooxygenase (MMO) — an enzyme known to efficiently and selectively oxidize methane to methanol.

In a recent issue of *Nature Communications*, the researchers show that the micropores of the zeolite provide a perfect confined environment for the highly selective stabilization of an intermediate copper-containing trimer molecule. Trinuclear copper-oxo clusters were identified that exhibit a high reactivity towards activation of carbon-hydrogen bonds in methane and its subsequent transformation to methanol. "The developed zeolite is one of the few examples of a catalyst with well-defined active sites evenly distributed in the zeolite framework — a truly single-site heterogeneous catalyst," says TUM professor Johannes Lercher. "This allows for much higher efficiencies in conversion of methane to methanol than with zeolite catalysts previously reported."

The achievement has promising implications for small-scale, gas-to-liquids technologies for utilizing stranded natural gas.

## Making hydrogen from methanol

The research group of professor Ken-ichi Fujita at Kyoto University (Kyoto, [www.h.kyoto-u.ac.jp](http://www.h.kyoto-u.ac.jp)) has developed an efficient catalytic system for the production of hydrogen from an aqueous methanol solution. The process uses a new, anionic iridium complex bearing a functional bipyridonate ligand as a catalyst. This system operates in a weakly basic solution (0.046 mol/L of NaOH) and mild temperatures (below 100°C), which is far below the high temperatures (more than 700°C) needed to steam reform hydrocarbons into H<sub>2</sub>, as well as the 200°C needed to generate H<sub>2</sub> from methanol by other catalytic processes, says Fujita. Also, the process does not require an additional organic solvent, which makes it simpler and safer than the recently developed homogeneous transition-metal-complex catalyst system, which uses organic solvents such as tetrahydrofuran, toluene

and 2,5,8,11-tetraoxadodecane (triglyme) at rather strong basic conditions (8.0 mol/L of KOH).

The new, water-soluble iridium-complex catalyst is highly active for generating H<sub>2</sub> by dehydrogenation of methanol. In laboratory trials, an 80% yield for H<sub>2</sub> and CO<sub>2</sub> was observed with 0.5 mol% of the catalyst using a 1-to-4 mixture of methanol in water after refluxing for 20 h at 88°C. The chemists speculate (and experimentally confirmed) that this catalytic reaction is composed of four steps: (1) formation of formaldehyde by the dehydrogenation of methanol; (2) formation of methanediol by the hydration of formaldehyde; (3) formation of formic acid by the dehydrogenation of methanediol; and (4) formation of H<sub>2</sub> and CO<sub>2</sub> by the decomposition of formic acid.

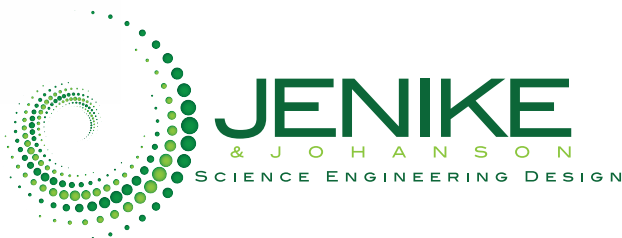
The researchers have also achieved long-term (150 h), continuous H<sub>2</sub> production with a turnover number (TON) of 10,000. ■

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## Plant Watch

### Asahi Kasei to construct production facility for cellulose nonwoven fibers

July 9, 2015 — Asahi Kasei Fibers Corp. (Osaka, Japan; [www.asahi-kasei.co.jp/fibers](http://www.asahi-kasei.co.jp/fibers)) will construct a new production facility for cellulose nonwoven fabric in Nobeoka, Miyazaki, Japan. The new plant's production capacity will be approximately 1,500 metric tons per year (m.t./yr). Groundbreaking will occur in late 2015, with startup scheduled for March 2017.

### CB&I awarded contract for new ethylbenzene/styrene plant in Egypt

July 9, 2015 — CB&I (The Woodlands, Tex.; [www.cbi.com](http://www.cbi.com)) has been awarded a contract by Carbon Holdings for the license and basic engineering design of an ethylbenzene/styrene plant to be built in Ain Sokhna, Egypt. The units will be a part of the Tahrir Petrochemicals complex expansion and will produce 400,000 m.t./yr of styrene monomer.

### Linde to install world's largest onsite fluorine plant in South Korea

July 8, 2015 — The Linde Group's (Munich, Germany; [www.linde.com](http://www.linde.com)) affiliate Linde Electronics and Specialty Gases announced the construction of a new onsite fluorine plant in Icheon, South Korea. The plant, which is said to be the world's largest onsite fluorine plant, will supply gas to SK Hynix Semiconductor, a manufacturer of flash memories.

### Total starts up lubricants plant in Singapore

July 6, 2015 — Total S.A. (Paris; [www.total.com](http://www.total.com)) has announced the startup of the company's largest lubricant-oil blending plant, located in Singapore. With a production capacity of 310,000 m.t./yr, this major hub will supply lubricants to a wide range of segments in the Asia-Pacific region, including automotives, industrials and marine.

### BP Zhuhai starts up world's largest PTA production unit

July 6, 2015 — BP Zhuhai, a joint venture (JV) in which BP plc (London; [www.bp.com](http://www.bp.com)) and Zhuhai Port Co. hold an 85% and 15% stake respectively, announced the startup of the new Phase 3 purified terephthalic acid (PTA) plant in Zhuhai, China. The Phase 3 plant was designed with a production capacity of 1.25 million m.t./yr, and is said to be the world's largest single-train PTA unit.

### Toyo wins contract for synthetic rubber plant in Indonesia

July 6, 2015 — Toyo Engineering Corp. (Toyo; Chiba, [www.toyo-eng.co.jp](http://www.toyo-eng.co.jp)) and PT Inti Karya

Persada Teknik (IKPT) have been awarded a contract for a new synthetic-rubber plant with a production capacity of 120,000 m.t./yr in Cilegon, Java, Indonesia from PT Synthetic Rubber Indonesia (SRI), a JV company of Compagnie Financiere Du Groupe Michelin and PT Chandra Asri Petrochemical Tbk. The target for completion of the plant is 2018.

### Sibur announces MTBE expansion project

July 1, 2015 — Sibur (Moscow, Russia; [www.sibur.com](http://www.sibur.com)) has started preparatory work to ramp up production capacity of methyl tertiary butyl ether (MTBE), a high-octane petroleum additive, at its production site in Togliatti, Russia. The project will see the construction of a new MTBE unit, with the site's capacity expected to grow from 75,000 to 135,000 m.t./yr.

### Lukoil launches operations at Volgograd refining unit

June 29, 2015 — Lukoil (Moscow; [www.lukoil.com](http://www.lukoil.com)) held a ceremony in Volgograd, Russia to celebrate the launch of operations of a new primary-oil refining installation, the largest installation among those active at the Volgograd refinery. Its refining capacity is a reported 6 million m.t./yr.

### Arkema to double production capacity for specialty molecular sieves

June 29, 2015 — Arkema (Colombes, France; [www.arkema.com](http://www.arkema.com)), through its subsidiary CECA, will double production capacity for specialty molecular sieves at the Honfleur, France site, which is dedicated to molecular sieves. This capacity extension, representing a capital expenditure of around €60 million, is expected to start up in summer 2016.

### BASF inaugurates world-scale acrylic acid complex in Brazil

June 20, 2015 — BASF SE (Ludwigshafen, Germany; [www.basf.com](http://www.basf.com)) has inaugurated a production complex for acrylic acid, butyl acrylate and superabsorbent polymers in Camaçari, Bahia, Brazil. The complex has a capacity of 160,000 m.t./yr of acrylic acid. The investment of more than €500 million is BASF's largest to date in South America.

### Axens selected to provide services for refinery upgrade in Egypt

June 17, 2015 — Egypt's Assiut Oil Refining Co. has selected Axens (Rueil-Malmaison, France; [www.axens.net](http://www.axens.net)) to supply technologies for its refinery-upgrade project. Axens will provide various process technologies, as well as catalysts, adsorbents and equipment for the project, which has a processing capacity of 660,000 m.t./yr of naphtha.

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## Mergers & Acquisitions

### Praxair acquires packaged gas distributors in California and Texas

July 9, 2015 — Praxair, Inc. (Danbury, Conn.; [www.praxair.com](http://www.praxair.com)) has acquired two independent distributors of packaged gases — Cryospec, Inc. (San Francisco, Calif.), and Garland Welding Supplies, Inc. (Garland, Tex.), with combined 2014 annual sales of about \$10 million. Financial terms were not disclosed.

### Evonik acquires share in nanotech startup JeNaCell

July 7, 2015 — Evonik Industries AG (Essen, Germany; [www.evonik.com](http://www.evonik.com)) has acquired shares in JeNaCell GmbH, and now holds a minority stake in the company, which is a spin-off of Friedrich Schiller University (Jena, Germany). JeNaCell is a specialist in nanocellulosic biotechnology. The parties agreed not to disclose the volume of the transaction.

### Umicore and Solvay sell SolviCore JV to Toray

July 1, 2015 — Umicore N.V. (Brussels, Belgium; [www.umicore.com](http://www.umicore.com)) and Solvay S.A. (Brussels, Belgium; [www.solvay.com](http://www.solvay.com)) have sold their respective 50% stakes in JV SolviCore to Toray (Tokyo; [www.toray.com](http://www.toray.com)). Since its inception in 2006, SolviCore has provided membrane-electrode assemblies for use in fuel cells and proton-exchange-membrane electrolysis.

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### BASF exits polyvinyl chloride joint venture

July 1, 2015 — BASF is selling its 25% share in the JV SolVin to Solvay. Financial details were not disclosed. SolVin was established in 1999 as a 75-25 JV between Solvay and BASF in the area of polyvinyl chloride (PVC).

### Thermo Fisher acquires Alfa Aesar from Johnson Matthey

June 29, 2015 — Thermo Fisher Scientific Inc. (Waltham, Mass.; [www.thermofisher.com](http://www.thermofisher.com)) has agreed to acquire Alfa Aesar, a manufacturer of research chemicals, from Johnson Matthey plc (London; [www.matthey.com](http://www.matthey.com)), for around \$405 million. In 2014, the business generated sales of approximately \$125 million.

### Huntsman acquires polyurethane elastomer manufacturer in Italy

June 23, 2015 — Huntsman Corp. (The Woodlands, Tex.; [www.huntsman.com](http://www.huntsman.com)) has acquired Tecnoelastomeri (Modena, Italy), a manufacturer and marketer of hot-cast elastomer systems and processing machines based on methylene diphenyl diisocyanate (MDI). Sales revenue for Tecnoelastomeri was around \$12 million in 2014.

### AkzoNobel and Evonik announce JV for chlorine and KOH production

June 16, 2015 — AkzoNobel (Amsterdam, the Netherlands; [www.akzonobel.com](http://www.akzonobel.com)) and Evonik have joined forces to build and operate a new membrane-electrolysis facility for potassium hydroxide (KOH) solution and chlorine production in Ibbenbüren, Germany. Startup is expected by the end of 2017. The 50-50 JV involves the construction and operation of a dedicated plant with a nameplate capacity of around 130,000 m.t./yr of KOH solution and 82,000 m.t./yr of chlorine. ■

Mary Page Bailey

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## Water Takes the Stage at Achema 2015

The emphasis on water management at Achema 2015 indicates the critical nature of water to all industry sectors as the CPI expand and evolve

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**FIGURE 1.** Over 166,000 visitors took part in Achema 2015's exhibition and congress sessions

In June, chemical engineers from around the world convened in Frankfurt am Main, Germany for Achema 2015 (Figure 1), the 31st world forum for the chemical process industries (CPI). The event assembled professionals from all sectors of the CPI to draw focus on new technologies and conduct in-depth discussions on some challenging topics the CPI will have to address in the future, with much emphasis placed on the sustainable utilization of resources — especially water.

The industries' continued growth was greatly evident at Achema 2015. This year, more than 166,000 attendees and over 3,800 exhibitors (up from 2012) from 56 different countries took part in Achema's exhibit halls and myriad technical lectures. Underlining the event's international reach, Dechema e.V. (Frankfurt; [www.dechema.de](http://www.dechema.de)), the event's organizer, noted that for the first time, more than half of the exhibitors came from outside Germany. The event also included region-specific panel discussions dedicated to the chemical industries of Saudi Arabia, Brazil and the U.S. Gulf Coast (box on p. 22).

While seemingly countless topics were

covered, the conference portion of Achema highlighted three areas with special focus: advances in bio-based processes; innovative process analytical technologies; and industrial water management. This article examines a selection of the water-related topics covered at Achema 2015.

### Collaborative water management

At Achema 2015's first plenary lecture, "Integrated industrial water management — Efficiency in the process industries," Nathalie Swinnen of Solvay S.A. (Brussels, Belgium; [www.solvay.com](http://www.solvay.com)) and Niels Groot of Dow Benelux B.V. (Terneuzen, the Netherlands; [www.dow.com/benelux](http://www.dow.com/benelux)) discussed the importance of industry collaboration for a successful water-management strategy (Figure 2). "Water is not just a raindrop, water means a lot. Water means something for the public, it also means something for the industry," said Swinnen during the lecture's introductory portion, indicating that industrial water management goes beyond the reach of the CPI. She also stressed that water scarcity is not only a concern in arid countries, and conservation must be addressed globally.

One area where water usage may go unnoticed to the public, explained Swinnen, is "virtual water," the water required to process or manufacture food and other goods. An awareness of the overall water footprint (including virtual water) is crucial for various parties to collaborate on large water-management projects. Another key point emphasized by Swinnen was that industrial water management — not simply wastewater treatment — is the all-encompassing level at which water stresses must be addressed, and options like recycling and recirculation, as well as the use of alternative materials, (such as air in place of water for heat-exchange purposes) should be considered.

In the second part of the lecture, Groot

### IN BRIEF

COLLABORATIVE WATER  
MANAGEMENT

NOVEL WATER  
CONCEPTS

ELECTROCHEMICAL  
TREATMENT

CONSERVATION IN  
AQUACULTURE



**FIGURE 2.** Nathalie Swinnen (left) and Niels Groot (right) examined some methods for industrial water management during one of Achema's two plenary lectures

and Swinnen detailed various case studies from the E4 water initiative ([www.e4water.eu](http://www.e4water.eu)), a consortium dedicated to promoting efficient water management in the European chemicals industry. Swinnen and Groot discussed how these studies' findings can be applied across the CPI to improve water management. "With 19 partners from the chemi-

cal industry, as well as a couple of research institutes and other parties, the project sets a new paradigm for integrated water management in the industry," said Groot of the E4 initiative.

In a case-study example from a Proctor & Gamble site in Belgium, he described how the plant achieved an economically feasible closed-

loop process by integrating certain wastewater-treatment technologies and avoiding non-critical heating processes. "The aim was to develop new treatment mechanisms to ensure that they can keep the water inside the process and recycle it to the process," explained Groot.

Swinnen emphasized that collaboration across many sectors, including municipal bodies and neighboring industrial sites, is necessary to implement water-management best practices. She cited an example from a Solvay site in the so-called "Chemical Cluster" in Belgium's Port of Antwerp that experienced issues with water scarcity, despite a large amount of nearby water — the problems stemmed from the quality of the water, not the quantity. In order to obtain water of adequate quality, the site was using drinking water for its industrial purposes. As part of the E4 initiative, and with close synergy with industry partners in the Chemical Cluster, the site was able to reuse and share a number of streams, en-

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## U.S. GULF COAST RESOURCES

Concerns about conservation of water and other resources are magnified on the U.S. Gulf Coast, where a recent burst of new-build activity (due in part to the relatively low price and high abundance of natural gas, alongside the decrease in crude-oil prices) has allowed for some new, more efficient technologies to be implemented. At Achema's Houston Business Forum, a panel of speakers from the U.S. and Germany discussed the petrochemicals business in Houston, as well as in the U.S. Gulf Coast region in general, and also presented some guidelines for European companies looking to establish operating bases in the U.S. One trend seen among new-build CPI projects is a proliferation of modular construction options, explained forum panelist Denis Menegaz of Fluor Corp. (Irving, Tex.; [www.fluor.com](http://www.fluor.com)). Repeatability is the most important factor in the modularization trend, said Menegaz, because costs and risks can be decreased, and project schedules have more certainty. Modular systems have a considerably smaller footprint, when compared to traditional "piece-by-piece" construction, and the requirements for cables, wiring and materials of construction, such as stainless steel and concrete, are reduced. Thus, resources can be utilized effectively, and unnecessary waste reduced.

Another panelist, Mark Eramo of IHS Global Chemical Insights elaborated on the new construction activity, focusing on the relationship with basic-chemical prices and production volumes. Eramo emphasized that in the wake of the natural-gas boom, the industry is moving away from ethylene-based chemistry, in favor of other molecules, including propylene and methanol, both of which will see considerable volume growth in the coming years, as new production plants come onstream. Still, ethylene volumes will remain high in the region — Eramo highlighted 14 new ethylene projects that are slated for completion by 2020. Many of these projects will be operated by international companies, such as Sasol, BASF and Formosa, indicating the increasingly global nature of the U.S. Gulf Coast region. □



Mark Eramo of IHS Global Chemical Insights (left) and other panelists (right) discuss the economy of the U.S. Gulf Coast at Achema's Houston Business Forum

abling an estimated 30% decrease in freshwater usage. The amount of effluent water released to the environment was also reduced. This project, says Swinnen "demonstrates the value of collaborating to maximize water savings."

***"Water means something for the public, it also means something for the industry," said Solvay's Nathalie Swinnen***

In another example, from Aretusa, Italy, a common groundwater source was being used for municipal, recreational and industrial purposes. Through an E4 water-management project, the site collected water from a local wastewater-treatment plant and recycled it as cooling-tower makeup water, freeing up additional groundwater for other uses. In addition to collaborative resource use, risk assessment, fund prioritization and identification of recycling opportunities were also discussed as means for properly integrating an efficient water-management plan.

### **Novel water concepts**

With nearly 300 exhibitors offering products and technologies dedicated to industrial water management, Achema's exhibit halls offered many solutions for water-related challenges. Ten institutes of the German

research conglomerate Fraunhofer-Gesellschaft (Munich, Germany; [www.fraunhofer.de](http://www.fraunhofer.de)) exhibited at Achema, with various water-related topics highlighted both in the exhibition halls and in conference sessions. Volker Heil, Ulrich Seifert and

Lukas Swiatek of Fraunhofer Umsicht ([www.umsicht.fraunhofer.de](http://www.umsicht.fraunhofer.de)) presented a conference session on an area of opportunity for water conservation in laboratories and process plants that may often go overlooked — safety showers. To determine the effect of water flowrate on the decontamination abilities of safety showers, the researchers used a full-scale, sensor-equipped test dummy. The comprehensive tests evaluated a variety of different shower heads, spray patterns and contaminants. They also took into account the typical movements a contaminated individual might exhibit.

Their findings — released publicly for the first time at Achema — determined that while higher water flowrates are necessary to extinguish large flames, for many cases of general first-aid provision, using too much water could be detrimental to

the decontamination effectiveness of the shower, depending on the type of equipment used. The team believes that this work could be advantageous in the development of improvements in shower-head construction, particularly in the area of choosing the optimal volumetric flowrate for a particular head. In this way, while improving the overall operation of a crucial piece of safety equipment, an opportunity to eliminate wasted water is also realized.

Fraunhofer Umsicht is also conducting work in the area of utilizing titanium-oxide-coated activated microsieves (with pore sizes between 1 and 20  $\mu\text{m}$ ) for precise wastewater treatment. The sieves' coating, when exposed to ultraviolet (UV) light, produces free radicals that can remove dissolved micro-pollutants, such as bisphenol A, from wastewater. In the municipal sector, Fraunhofer Umsicht is investigating ways in which buildings' wastewater can be used to supply agricultural plants with nutrients.

### **Electrochemical treatment**

Another Achema exhibitor presenting water-management solutions was VTU Engineering GmbH (Grambach, Austria; [www.vtu.com](http://www.vtu.com)). VTU's Dieter Woisetschläger led a congress session entitled "Electrochemical wastewater treatment for industrial





**FIGURE 3.** Reminders of the bioeconomy were spread throughout Achema via the Biobased World initiative

applications,” outlining the company’s Coolox process, which is an advanced oxidation process (AOP) that employs boron-doped diamond electrodes to generate hydroxyl radi-

cals ( $\cdot\text{OH}$ ) in wastewater. These  $\cdot\text{OH}$  radicals help to degrade organic water pollutants into carbon dioxide, water and organic salts. Traditionally,  $\cdot\text{OH}$  radicals are formed when an active oxygen component is introduced to the water in the presence of some form of excitation (such as UV lamps or catalysts). The Coolox process, due to its electrochemical nature, does not require the use of chemical additives, instead directly generating  $\cdot\text{OH}$  radicals through the application of an electric current. According to Woisetschläger, boron-doped electrodes are ideal for use in wastewater-treatment applications due to their inert surface, low absorption properties and high redox potential, as well as their ability to non-selectively react with organic pollutants. The process has successfully been expanded from laboratory scale, through pilot testing, to full-scale treatment cells. Various fully automated demonstration modules have already been deployed in Europe and China.

Woisetschläger presented several

case studies where Coolox systems were used to treat challenging wastewater streams, including one from a pharmaceutical manufacturing site in Zibo, China. The wastewater stream, which contained a mixture of streams from various irregular batch processes and multiple active pharmaceutical ingredients (APIs), had persistent toxic constituents and a very high conductivity. Other water-treatment methods, including filtration, reverse osmosis, coagulation and chemical oxidation were deemed ineffective. In this example, it was seen that through electrochemical treatment, complete oxidation was possible, and that the Coolox process was suitable for treatment with various APIs and mixed pharmaceutical process streams.

Another fully automated Coolox treatment plant was successfully commissioned by Siyao Pharmaceutical Co. in Changzhou, China, just two weeks prior to Achema, said Woisetschläger. Beyond pharmaceuticals, additional stud-

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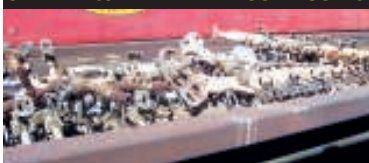
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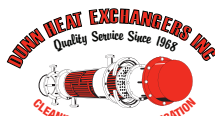
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ies showed that electrochemical wastewater treatment is also effective in the fine chemical and food sectors, as well as for treating phenol-containing streams.

### **Conservation in aquaculture**

Biotechnology was also a key focus at Achema 2015. The event's "Biobased World" platform (Figure 3) highlighted exhibitors, live demonstrations and technical sessions dedicated to the bioeconomy and its increasingly important role in the CPI. Special floor tiles in the exhibit halls led attendees to the over 200 exhibitors involved in Biobased World, and attendees with a particular area of interest (such as biotechnology equipment or biomaterials testing) could follow a "themed walk" guiding them directly to the most relevant areas.

Water management and bio-based technologies intersect in many ways, including aquaculture, the process of cultivating marine organisms, plants and chemicals, including fish oils and certain fatty acids. Ana Maria Otero of the University of Santiago de Compostela (Santiago de Compostela, Spain; [www.usc.es](http://www.usc.es)) led a conference session at Achema outlining the use of microalgae biomass in aquaculture applications, and the ways in which algae use can be made more effective and efficient. Particular focus was given to optimizing the use of microalgae in integrated multi-trophic aquaculture, where wastewater from one species can be re-used to provide nutrients for another species, greatly decreasing the amount of water effluent. Advanced photobioreactors that facilitate convenient onsite production of microalgae, along with improved algae-preservation methods and the identification of new species, contribute to the realization of new levels of efficiency in the use of microalgae in aquaculture.

The emphasis on industrial water management in all segments of the CPI at Achema demonstrated an industry-wide awareness of global water concerns and sustainability. Going forward, specialized solutions like those presented at Achema will be crucial to effectively managing resources. ■

Mary Page Bailey

# Smarter Sensors for a Smoother Process

Advanced sensors help reduce downtime, increase efficiency and safety

As more and more advanced electronics, mechanics and software are employed within sensors, tasks that were previously managed by control systems or other tools for data aggregation are now being handled, and handled well, by devices aptly dubbed “intelligent” or “smart” sensors. “Smart devices are changing the role of sensors in processing facilities because they are becoming more like data appliances,” notes Tracy Doane-Weideman, product marketing manager, analytics, for the U.S. with Endress+Hauser (E+H; Greenwood, Ind.; [www.us.endress.com](http://www.us.endress.com)). “Smart sensors not only have the ability to gather information, but also to put it into a format that processors can use to better manage equipment and processes.”

And, better management of the instrument lifecycle leads to more efficient, less costly and safer control of the process, as well as higher quality products with less waste. “At the end of the day, that’s what processors are truly after — gathering data and information to improve process, safety and quality,” notes Doane-Weideman.

Designed to do all of the above, E+H’s Memosens line of sensors (Figure 1) uses digital technology to make liquid analysis easier, more reliable and safer. The technology converts the measured value to a digital signal and transfers it inductively to the transmitter, eliminating the problems associated with moisture and corrosion. With signal alarms in the event of transmission disruption, Memosens offers safe data transfer for increased availability of the measuring point and safe, trouble-free processes.

The company’s Memosens CPS96D combined pH/ORP sensor for heavily soiled media in chemical processes, paper or paint production offers open aperture for reliable, simultaneous pH and oxidation-reduction potential (ORP) measurement for a better process overview. Additional platinum electrodes enable constant monitoring of sen-



Endress+Hauser

sor quality, and the device is not affected by fluctuations in pressure and temperature, allowing more reliable data for better, more informed decision making.

### More efficient maintenance

One of the biggest benefits of smart sensors is their ability to perform self-diagnostics. This capability allows processors to stop wondering if performance issues are related to malfunctioning sensors or whether the trouble exists within the process itself. “Ultimately we are trying to measure the process, not decide whether a sensor is working,” explains Doane-Weideman. “When we have better information, we can create predictive-maintenance schedules based upon that information, rather than running sensors to failure, which leads to less unscheduled maintenance.”

Along these lines, Electro-Chemical Devices (Irvine, Calif.; [www.ecdi.com](http://www.ecdi.com)) offers its Sentinel product (Figure 2), which helps users avoid unnecessary downtime by alerting

**FIGURE 1.** E+H’s Liquiline transmitter platform and Memosens sensors are smart devices intended to increase process efficiency and safety using digital technology

## IN BRIEF

MORE EFFICIENT  
MAINTENANCE

MORE RELIABLE  
CONTROL

SAFER FACILITIES

FINAL REMARKS



**FIGURE 2.** The Sentinel offers a “visual sensor life indicator” that allows users to appropriately schedule sensor maintenance, reducing unplanned downtime and increasing reliability of measurements

them to problems with the electrodes, according to Steve Rupert, product manager for the company. “In the past, pH and ORP sensors used ref-

erence electrodes to measure the primary parameter, and when there was a change in the voltage of the sensor, users assumed the pH or ORP

value was changing, when, in fact, it was often a drift in the reference electrode,” he explains. “This resulted in a lot of unnecessary downtime. To help determine whether the reference is valid, we incorporated a second reference electrode.”

Model S80 sensors for the measurement of pH, ORP and various ions use replaceable electrode cartridges specific for the measured parameter. These electrode cartridges have a measurement cell and a reference cell. The reference cell is designed to produce a standard potential independent of the solution it is immersed in. While this style of electrode is typically trouble-free, there are conditions that lead to failure. The Sentinel Pre-pHault Diagnostic addresses these issues by including an additional sleeved silver element into the reference cell. When the electrode cartridge is new, both silver elements are at the same potential, but as the electrode ages or becomes poisoned, the bare element changes its potential in re-



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**FIGURE 3.** GF Piping Systems offers new ultrasonic sensing instrumentation for precise level measurement in virtually any liquid

sponse to the electrolyte depletion or poisoning. The Sentinel monitors the potential difference between the two elements and displays the value as a gage of the electrode's remaining life. The protected silver element is still producing the correct potential but it is in danger of failing due to the changing environment inside the reference cell. This Pre-pHault indication notifies the user of the potential electrode failure before the measurement actually fails.

Trend monitoring is another smart-sensor feature intended to help users decide when maintenance might be necessary before failure occurs, according to Karmjit Sidhu, director of business development with American Sensor Technologies (Mt. Olive, N.J.; [www.astensors.com](http://www.astensors.com)). "Some sensors have the ability to look at trends in the process and, from those trends, help processors decide when maintenance will be in order," he says. "For example, if you have a pump with sensors that detect vibration, the sensors can tell maintenance technicians that, over time, the pump or the bearings are aging prematurely, and they can, based on the trending information they collect, let them know when maintenance will be needed before the pump breaks down."

His company offers trend monitoring on some sensors, such as its AST5400 differential pressure

transducers, which measure the differential pressure of liquids or gases across filters or calculate flow across orifice plates. They offer digital compensation, electronics with fail-safe condition on the output signal and trends monitoring for improved maintenance scheduling, says Sidhu.

And, some vendors are adding features that permit "plug-and-measure" functionality, meaning the sensors can be pre-calibrated offline,

away from the measurement point, and then exchanged in the field. "In the past, analog sensors needed to be calibrated at the measurement point in the field, under a variety of conditions, including being outside, exposed to the weather. However, today's digital sensors allow you to calibrate them under controlled conditions," explains Stefan van der Wal, industry manager for the chemical segment of Mettler-Toledo (Urdorf,

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Switzerland; [www.mt.com/pro](http://www.mt.com/pro)). “And, in the case of pH sensors, this also ensures that buffer solutions are always at a stable temperature, which is important for proper calibration, and often a problem in the field. Now, you can calibrate in the workshop and then swap the pre-calibrated sensor out with the one at the measurement point.”

Van der Wal says this leads to more efficient calibration routines and more reliable calibrations, which result in more reliable measurements. Having sensors with a digital output increases measurement reliability as the signal is not prone to interference as analog signals can be. ISM (Intelligent Sensor Management), Mettler-Toledo’s intelligent sensor platform, offers several digital sensors, among them pH and dissolved-O<sub>2</sub> devices, which can be pre-calibrated in the workshop and swapped out in the field. They also monitor the aging of the sensor in real-time with the dynamic lifetime indicator, check the impedance of the pH reference electrode for early detection of diaphragm clogging and determine when the next calibration is due with the adaptive calibration timer. The ease of use and digital sensor-to-transmitter communications allow more efficient calibration and maintenance, notes Van der Wal.

### More reliable control

Similarly, Krohne Messtechnik GmbH’s (Duisburg, Germany; [www.krohne.com](http://www.krohne.com)) Smartsens analytical sensor family also offers offline calibration in smart sensors. However, this line does not require the use of transmitters. Typically sensors require an external transmitter on site that translates the sensor signals to the process control system. But, false installation, cabling or configuration of the transmitter sometimes causes errors. To eliminate these risks, Krohne miniaturized the transmitter and fitted it into the sensor head. As a result, any Smartsens sensor can be connected directly to the process control system, featuring direct communication via 4–20-mA HART communication, reducing the risk of failures along the chain from the sensor to the process control system and in-

General Monitors



**FIGURE 4.** Ultrasonic gas leak detectors use acoustic technology to swiftly detect outdoor gas leaks by sensing the distinct high-frequency ultrasound emitted by high-pressure gas leaks, allowing faster response times

creasing the reliability of information. The product line includes sensors for pH, ORP and conductivity.

Also intended to reduce error, thus improving reliability of measurements and process operation, is the new ultrasonic line of sensing instrumentation for level measurement (Figure 3) introduced by GF Piping Systems (Irvine, Calif.; [www.gfps.com](http://www.gfps.com)). “The new ultrasonic products offer more intelligence,” says Dave Vollaire, instrumentation product manager for GF Piping Systems. “They can differentiate between substances like condensation or agitation and the true, actual level of the medium, thereby avoiding inaccurate measurements.”

The new level transmitter and sensor operate on the measurement principle of ultrasonic time-of-flight, and feature  $\pm 0.05\%$  accuracies of the measurement range. The sensor can recognize foreign matter and disregard it to ensure reliable level measurement, even in tanks where there are heavy vapors, condensation and turbulence.

Smart sensors are also enabling measurements to be taken in places that were previously too difficult to monitor, which leads to better control over the process, as well as increased safety, says Melissa Stiegler, global product manager, wireless, at Emerson Process Management (Shakopee, Minn; [www.emersonprocess.com](http://www.emersonprocess.com)).

“In a chemical plant, the more you

can sense, the more you can solve,” says Stiegler. “Adding more sensors provides the ability to gather more data and easily transform it into actionable information to help you take better control of your plant. Think about equipment that, up until now, hasn’t been monitored due to the expense of instrumentation and monitoring or due to physical limitations. Anything that wasn’t previously deemed a critical asset can now be cost-effectively monitored, thanks to intelligent wireless instrumentation. Improving visibility into the operation of assets, such as heat exchangers, blowers, process pumps, valves and others, allows operators to take action before equipment fails, reducing downtime and process upsets, as well as providing needed maintenance to extend equipment life.”

The return on investment of monitoring these expensive, but less critical assets, is often not sufficient when using wired instruments and sensors, but the limitations are now eliminated with Emerson Process Management’s family of IP 67-rated wireless products. These wireless sensors are also intelligent, as they can communicate information in addition to the measured process variable.

### Safer facilities

Smarter sensors used in flame and gas leak detectors also have the ability to increase the level of safety in process facilities, says Ardem Antabian, senior strategic technology and product manager with General Monitors (Lake Forest, Calif.; [www.generalmonitors.com](http://www.generalmonitors.com)). “In the past, detectors were plagued with false alarms that could shut down processes or whole plants, adding up to a lot of unnecessary downtime and costs associated with restarting the plant,” notes Antabian.

He says these detectors have been improved through the use of added intelligence in the sensors using microprocessors that have the ability to determine an actual alarm from a false alarm.

For example, General Monitor’s FL4000H Multi-Spectral Infrared (MSIR)/Neural Network Technology (NNT) Flame Detector reliably dis-

criminate between actual flames and nuisance false alarm sources, such as arc welding or hot objects. The flame detector's advanced technology makes use of a multiple infrared (IR) sensing array that samples different IR spectrum wavelengths. The analog sensor signals are sampled and converted into digital format for signal pre-processing to extract time and frequency data. The time and frequency information are used by the detector's neural network classification algorithm to identify whether the IR signals are emitted from a flame or non-flame source. The flame or non-flame decision is then reported as an output via LEDs, relays, analog, HART or Modbus.

With the NNT flame discrimination algorithm, the FL4000H is highly immune to false alarms. The NNT signal processing model also offers the ability to adapt to customer application conditions, resulting in reliable flame protection with superior false-alarm immunity.

Intelligent sensors can help users detect gas leaks faster, says Antabian. Technologies that have traditionally been used to detect hydrocarbon gas leaks, have all had one limitation: in order for a leak to be detected, the gas itself must either be in close proximity to the detector or within a pre-defined area. However, in outdoor conditions, traditional gas-detection systems sometimes fail because environmental conditions prevent the gas from reaching the detector. In response, General Monitors released the Observer-I ultrasonic gas leak detectors (UGLD; Figure 4), which are based on acoustic technology, so they detect outdoor leaks by sensing the distinct high frequency ultrasound emitted by high-pressure gas leaks. With the ultrasonic detecting technology, leaking gas does not have to reach the sensor, just the sound of the gas leaking, so by adding UGLD detectors, faster response times and lower operation costs can be obtained.

### Final remarks

Whether you are monitoring pH, a heat exchanger or the safety of the facility, there is a smart sensor out

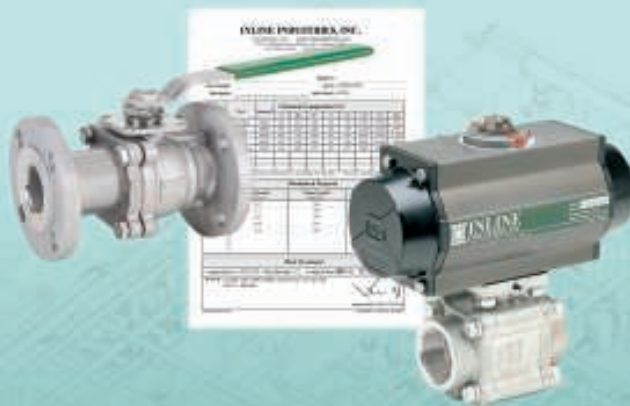
there that can help you see deeper into the process, which provides benefits in the form of reduced maintenance costs, improved process efficiency and safer facilities.

"The world of instruments used to be totally under the world of control systems, but today our sensors and devices are reliable data sources that can connect everything in the plant to the local control system, al-

lowing processors to really rely upon collected information to monitor equipment that previously couldn't be monitored," summarizes Craig McIntyre, chemical industry manager with Endress+Hauser. "Usually the benefits of this ability are not associated with the devices themselves, but in the value that is radiated and generated by them." ■

Joy LePree

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## Rigid container available for the transport of bulk solids

The P-433 Ship Shape bulk forklift container (photo) features a standard 44-by-44-in. footprint that seamlessly fits into many bulk-bag fillers. This rigid, reusable container can be used to move, handle and store bulk solids and other granular, free-flowing dry products. The P-433 shipping container handles payloads of 1,200 lb within a heavy-duty, one-piece bin. It eliminates the potential for sidewall collapse during filling (and the associated product waste, cleanup effort and downtime), which is a common problem for flexible bulk bags, says the company. It can be transported directly via a forklift truck without the need for a pallet, improving safety (since bagged payloads often shift and slip when transported via forklift on a pallet). Rotationally molded in a single piece from 100% waterproof polyethylene, the lidded bulk container is designed for four-way lift access. Options include permanent, molded-in logos, numbers and graphics, plus industrial-strength casters, fork safety tubes and tamper-evident closures. — MODRoto, Ashtabula, Ohio  
[www.modroto.com](http://www.modroto.com)

pany. — KLV Plastics, Monroe, Ohio  
[www.klvplastics.com](http://www.klvplastics.com)

## Rugged plastic film provides packaging, unitization support

The recently reformulated Cor-Pak VpCI Stretch Film (photo) provides cost-effective corrosion protection for primary packaging and unitization (when loading pallets). It offers both vapor-phase and contact protection for carbon and galvanized steels, aluminum, copper, brass, zinc and other metal containers. The film provides puncture resistance (under maximum ASTM D1709-04 dart-drop load) and load holding to ensure packaging integrity. The film is made from a blend of polyethylene resins and the company's patented VpCI technology. It does not contain polyisobutylene (PIB) or other tackifiers in the cling layer, so it does not leave residue on parts. The self-adhering film bonds to each layer for added strength. It is FDA-approved for use on food-handling equipment, says the company. — Cortec Corp., St. Paul, Minn.  
[www.cortecvci.com](http://www.cortecvci.com)



Cortec Corp.

## Automated bag-filling machine gets the job done

The 3CM-P single spout, fully automatic bag filler (photo) handles 1,200 bags per hour. It can handle a variety of empty, pre-made bag types, including those made from multi-wall paper, polyethylene and woven poly with inner liners. An operator fills the bag magazine with premade empty bags, and the bag filler maintains bag control throughout the packaging and closing process. Its automated features and control system ensure smooth bag filling and closing, says the company. — American-Newlong, Inc., Indianapolis, Ind.  
[www.american-newlong.com](http://www.american-newlong.com)



American-Newlong

## Lightweight packaging options use less plastic

The E-Tainer line of products focuses on source reduction, increased use of post-consumer resin, multi-layer manufacturing technology, and combined hybrid technology through the use of precision mandrel-formed Meta corrugated cases. This line of packaging solutions includes four new container configurations, including a tight-head container, a lightweight container combined with a Meta corrugated case, a collapsible, semi-rigid bladder-in-a-box product, and a bag-in-box flexible packaging solution. These packaging solutions use up to 80% less plastic compared to standard, tight-head containers, and the corrugated designs weigh up to 30% less while maintaining container integrity, according to the com-

## Integrated system creates and fills carton containers onsite

The conventional transport of empty, pre-formed carton containers to a location for filling and further pro-

Note: For more information, circle the 3-digit number on p. 74, or use the website designation.



cessing is costly and inefficient. The InlineCan system (photo) lets users produce, fill and close cartons for powders and bulk solids in one continuous process. Onsite production of the carton-cans provides cost-saving efficiencies and reduces warehousing and logistics requirements for the process. A warehouse that formerly stored 1,000 conventional pre-made cans can now store the raw materials needed to produce 30,000 InlineCans of identical size and storage capacity onsite, says the company. The user can choose from a wide variety of possible shapes, from rounded square to octagonal, ovals and combined round/square forms. Depending on the product, the appropriate dosing systems are integrated into the machines. — *Optima Packaging GmbH, Schwaebisch Hall, Germany*  
[www.optima-ger.com](http://www.optima-ger.com)

### Streamline pharmaceutical-filling operations

The FXS Combi system (photo) is a flexible system for filling and closing pharmaceutical liquids into pre-sterilized, nested syringes, vials and cartridges (small glass containers), as well as all common filling technologies. The machine offers several positions, and two output rates (providing for slow- and medium-speed operation). It features an integrated capping station for vials and cartridges. By combining the machine with proven tub and bag openers, as well as different barrier systems, the FXS Combi can be flexibly integrated into complete line concepts, allowing for significant space savings, says the company. — *Bosch Packaging Technology, Stuttgart, Germany*  
[www.bosch.com](http://www.bosch.com)

### Robotic fulfillment system is designed for mixed-load orders

This company offers a suite of robotic solutions to handle mixed-load order fulfillment (photo). Key enhancements include adjustable end-of-arm tooling that can handle up to four cases simultaneously, and an integrated stack-and-wrap system. The system combines robotic integration, software, controls and vision technology to palletize and depalletize partial and mixed-load

pallets, enabling automated operations to replace manual processes with greater efficiency and accuracy. — *Intelligrated, Chicago, Ill.*  
[www.intelligrated.com](http://www.intelligrated.com)

### Thermal printer produces a range of packaging labels

The new H-Class H-8308p Thermal Printer (photo) is a highly adaptive 8-in./A4 thermal printing system that features an embedded, open-source Linux operating system, standard HP-compatible language and expanded management capabilities for operators. The machine produces a wide range of labels, tags and other documents, at lower cost than comparable laser printers, says the company. — *Datamax-O'Neil, Orlando, Fla.*  
[www.datamax-oneil.com](http://www.datamax-oneil.com)

### Packaging eliminates air and moisture to protect contents

Having recently acquired the Xtend product line from StePac, this company manufactures and supplies high-quality, precision-engineered, modified-atmosphere packaging and modified-humidity packaging options, to extend the life of products that are susceptible to damage from exposure to air and humidity. — *Johnson Matthey, Royston, U.K.*  
[www.matthey.com](http://www.matthey.com)

### Rugged drum labels meet industrial challenges

Industrial drums carrying chemicals present a range of labeling challenges. These include the container's need to feature proper identification of the materials inside and the label's need to withstand exposure to extreme temperatures and weather conditions. This company's Select Solutions Drum product line (photo) include a range of film and heavy-weight paper materials, which, when paired with best-in-class adhesives can withstand harsh environments and long periods of outdoor exposure. These drum labels can be used on a variety of surfaces, including painted steel, polyethylene drums and supersacks. They meet a variety of international standards and codes, says the company. — *Avery-Dennison, Mentor, Ohio*  
[www.averydennison.com](http://www.averydennison.com)

Suzanne Shelley

Optima Packaging GmbH



Bosch Packaging Technology



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# New Products

## A new mass flowtube compatible with many media types

The CFS25 mass flowtube (photo) provides direct measurements of both mass flowrate and density, eliminating the problems generally associated with multiple process measurements using volumetric flow devices, according to the company. With no sensitivity to varying process conditions, this Coriolis flowmeter, in addition to accurately measuring liquids, can also be used in applications with entrained air, non-Newtonian fluids, viscous and abrasive fluids, slurries and gases. — *Invensys Foxboro, Foxboro, Mass.*

[www.fielddevices.foxboro.com](http://www.fielddevices.foxboro.com)

## Use these steel grades with extremely low temperatures

The SZ-Steel (photo) family of steel grades features low impurity levels and controlled grain sizes, and is proven to retain its properties in temperatures down to  $-40^{\circ}\text{C}$ , and in extreme cases, has been tested to withstand temperatures as low as  $-101^{\circ}\text{C}$ , according to the manufacturer. The specific design properties of the steel help to reduce risks of embrittlement and fracturing. SZ-Steel grades meet key international safety standards, and their capabilities are proven in many applications, including stud bolts, hydraulic equipment, drilling tools, axles and valves that are used in the oil-and-gas industry, mining and energy generation. — *Ovako AB, Stockholm, Sweden*

[www.ovako.com](http://www.ovako.com)

## Polypropylene pumps with patented dry-run design

The MX-505 pump (photo) is constructed of glass-fiber reinforced polypropylene, and is capable of flowrates up to 225 gal/min and a total dynamic head up to 86 ft. Additionally, the MX-505 boasts a patented dry-run design and standard NPT sizes for suction (2.5 in.) and discharge (2 in.), making them simple to install and operate. With a patented self-radiating structure, heat-dispersion holes force the liquid to circulate around the spindle and bearing. — *Iwaki America, Inc., Holliston, Mass.*

[www.iwakiamerica.com](http://www.iwakiamerica.com)

## A heater technology for material lamination and food processing

The Mica-Foil high-temperature heater technology (photo) provides uniform heating with fast response up to  $600^{\circ}\text{C}$ . The Mica-Foil technology was designed for use with food-processing equipment and material-laminating applications where temperature profile and thermal response are critical to the manufacturing process. Consisting of a distributed-wattage etch-foil heater element bonded between high-temperature mica sheath material, the heaters can be customized to match the working surface of a particular application. — *Durex Industries, Cary, Ill.*

[www.durexindustries.com](http://www.durexindustries.com)

## New repeater-node technology extends range, redundancy

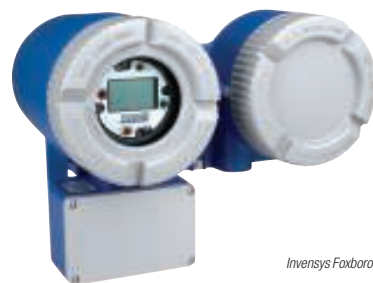
This company has developed a new repeater-node technology that, according to company, will provide both redundancy and additional reach not currently available in a standard point-to-multipoint systems. The repeater-node system will help extend range by connecting several repeaters, enabling switching from device to device. The repeater-node system also provides carrier-strength redundancy through multiple networking paths. If one connection goes down, it will be instantly rerouted. — *AvaLAN Wireless Systems, Inc., Madison, Ala.*

[www.avalanwireless.com](http://www.avalanwireless.com)

## This bag dump station has self-contained filtration

This company's new bag dump station (photo) retains all product material by preventing dust from escaping into the plant environment, also protecting workers from potentially hazardous exposure. The bag dump station features a self-contained filtration system, allowing for 99.9% retention efficiency down to  $1\ \mu\text{m}$ . The filtration system eliminates the need to continually clean the equipment or have a remote dust-collection location with external ducting or auxiliary fans. The bag dump station's low-profile design makes it simple to install at floor-level, and allows the unit to fit in tight spaces. — *Hapman, Kalamazoo, Mich.*

[www.hapman.com](http://www.hapman.com)



Invensys Foxboro



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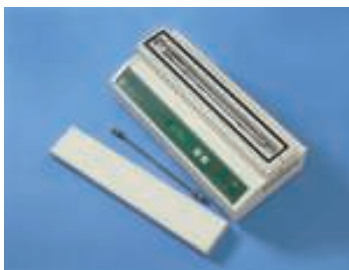


Conval

### Blowdown valves for tandem or continuous service

Clampseal blowdown service valves (photo) provide necessary control for continuous or tandem boiler or turbine blowdown and bottom blow-off service. The blowdown valves are available in sizes ranging from 3/4 to 4 in., with butt- or socket-weld and flanged ends in various materials, with customizations available. The valves feature a uniform one-piece gland that eliminates the potential for stem damage from gland cocking. With leak-proof bonnets, the cartridge-style packing chamber facilitates rapid access to the valve trim for inspection and maintenance. — *Conval, Somers, Conn.*

[www.conval.com](http://www.conval.com)



Torrey Pines Scientific

### Precise temperature regulation for chromatography columns

The new EchoTherm Model CO50 programmable chiller/heater (photo) for high-performance liquid chromatography (HPLC) columns has a readable temperature range from 4 to 100°C and setpoint capability to 0.1°C. The control software regulates temperatures to  $\pm 0.2^\circ\text{C}$ , even at ambient conditions. The CO50 has an indicator LED that illuminates when the target temperature is stable to within  $\pm 0.2^\circ\text{C}$ . The CO50 is suitable for chiral and biomedical chromatography where below-ambient temperatures help preserve bioactivity. The CO50 holds columns up to 30 cm long with 1/4- or 3/8-in. diameter in provided mounting clips. Larger-diameter columns can be used by removing the column clips. — *Torrey Pines Scientific Inc, Carlsbad, Calif.*

[www.torreypinesscientific.com](http://www.torreypinesscientific.com)



Schaffner EMC

### This software manages tags for project handover

SmartPlant Master Tag Registry (MTR) is a software module intended to simplify how companies manage equipment and instrumentation tags and register the information required for project handover. It is said to be one of the first commercially available enterprise products to target the systemic problems associated with tag management in the industry. MTR provides specific functionality to automate consistent tagging rules among supported products, and then makes

tag register generation, management and monitoring more efficient in large projects that may include thousands of individual tags. Traditional methods for handover involve the manual collection of key technical data from numerous sources. MTR automatically collects and organizes tags as they are created and used in various work processes during projects — *Intergraph Corp., Huntsville, Ala.*

[www.intergraph.com](http://www.intergraph.com)

### Extend VFD service life with these devices

This company's Sine Wave Filter products (photo) eliminate premature motor damage from overvoltages, cable ringing, motor overheating and eddy-current losses caused by variable frequency drives (VFDs). Suitable for motor frequencies up to 600 Hz, this technology converts the rectangular pulse-width modulation (PWM) output voltage of motor drives into a smooth sine wave with a low residual ripple. In addition to elongating the service life of high-speed motors, the Sine Wave filters can improve bearing life by eliminating circulating bearing currents. Typical applications include pumps, conveyors, compressors, HVAC applications and motor drives with multiple motors or long motor cables. — *Schaffner EMC Inc., Edison, N.J.*

[www.schaffnerusa.com](http://www.schaffnerusa.com)

### New coagulant blends for water-treatment applications

Two new coagulants, CDP2727 and CDP2724, have been introduced to the KlarAid product line. The custom-designed blends are based on a high-molecular-weight organic coagulant and a high-basicity aluminum coagulant. The new products have improved capabilities for reducing the turbidity and total oxygen content (TOC) in low-turbidity raw water when compared with predecessor products, says the company. CDP2727 is the more concentrated product, and CDP2724 is a diluted version that is suitable where low volumes and dosages are preferred. These products can be used alone or in conjunction with anionic flocculants. — *GE Power & Water, Schenectady, N.Y.*

[www.gepower.com](http://www.gepower.com)

## Biowaste treatment system goes global

The EDS+ BioWaste treatment system (photo) was recently launched globally for biologically hazardous waste decontamination and growth media sterilization. The systems, certified to ASME and ISO/EN standards, are now suitable for local standardization and offer dual controls in local languages and English. Following five years of successful internal and external testing, the company's newest EDS+ system has been enhanced to include a number of major technical advances, including thermal energy regeneration, dual redundancy, anti-foam control, pH neutralization, 100% positive release for treated waste and electronic records generation. The EDS+ is designed for waste levels from BSL1 to BSL4, and is typically used in the biopharmaceutical sector, laboratories and research institutes. — *Suncombe Ltd., Enfield, Middlesex, U.K.*  
[www.suncombe.com](http://www.suncombe.com)

## A new pipe system range with detectable rings and seals

The multi-way distributor (photo) that was presented at Achema 2015 is also available with the performance criteria of the company's new Detectable Design series. All of the components in this pipe system are food-grade, in accordance with both the European Directive EC 1935/2004 and the U.S. Food and Drug Administration (FDA). This applies to stainless-steel pipes and fittings, as well as to the sealing materials used, which are also metal detectable and visually detectable. Special vulcanized sealing rings are available for the easy-to-assemble pull-ring connections on this system, which are more resistant to tearing and to chemicals than conventional, bonded variants. — *Fr. Jacob Söhne GmbH & Co. KG, Porta Westfalica, Germany*  
[www.jacob-rohre.de](http://www.jacob-rohre.de)

## This gas analyzer detects traces of H<sub>2</sub> in gas mixtures

The ProCeas H<sub>2</sub> trace analyzer (photo) is said to be the world's first device capable of measuring H<sub>2</sub> via infrared (IR) radiation using optical resonance laser spectroscopy. The system enables di-

rect, continuous measurements of low H<sub>2</sub> concentrations (up to 3 parts per million) in Cl<sub>2</sub> or other gas mixtures in realtime for control of industrial processes. The system is based on the company's patented OFCEAS (optical feedback cavity enhanced absorption spectroscopy) technology and low-pressure sampling techniques. OFCEAS is said to be extremely precise for both simple and complex gas mixtures. The optimized optical resonance system produces a very long optical path — 10 km instead of 10 m achieved in conventional Fourier-transform IR and non-dispersive IR technologies — thus enabling sub parts-per-billion detection. — *AP2E, Aix-en-Provence, France*  
[www.ap2e.com](http://www.ap2e.com)

## A new field-mounted HART temperature transmitter

The 7501 (photo) is a field-mounted, Ex d explosion- and flame-proof temperature transmitter with local operator interface for easy programming, review and diagnostics of process values in the field. The optical buttons combined with the 10-mm explosion-proof window is a patent-pending technology. The optical buttons are immune to interference of ambient light sources and false triggering caused by shadows and reflections, and the device dynamically adapts to accumulation of dust and moisture, says the company. The user can also perform advanced HART programming from the front, and as a result, significantly reduce the need for handheld communicators. The transmitter delivers HART 7 functionality with HART 5 compatibility and can be configured to measure two-, three- and four-wire RTDs and 13 different thermocouple types, as well as bipolar millivolt and resistance signals, and it provides an analog output. — *PR electronics A/S, Roende, Denmark*  
[www.prelectronics.com](http://www.prelectronics.com)

## This sieve mill challenges agglutinated products

The formation of agglomerates can make additional processing of materials difficult and can negatively affect the quality of the final product. The Conika sieve mill (photo, p. 36) solves this problem, by preventing powders



Suncombe



Fr. Jacob Söhne



AP2E



PR electronics

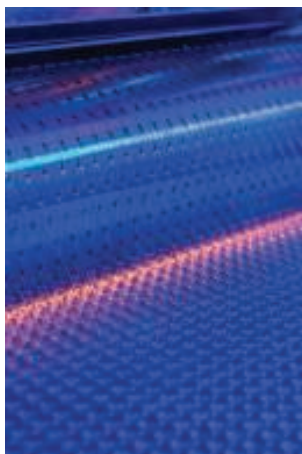


IKA Werke

and granules from forming agglomerates during storage, and thus minimizes loss for further processing. The internal rotor of the Conika shears the material through a conical sieve. The conical shape reduces the friction in the process and thus keeps the energy input into the product to a minimum. This results in less warming of the product. All wetted parts, including the sieve, are stainless steel. — *IKA Werke GmbH & Co. KG, Staufen, Germany*  
[www.ika.com](http://www.ika.com)

### Increasing pastillation productivity for viscous melts

The new ThermoCut system is the latest addition to this company's Rotoform technology, and was developed to increase productivity and result in a better-quality end product with high-viscosity melts. In the past, a common issue faced by processors handling advanced, high-viscosity products is the formation of threads, commonly known as "angel hair." These threads, created when the melt is deposited on the steel-belt cooler, can result in a less-than-perfect pastille shape, as well as an increase in dust formation and increased cleaning requirements. The ThermoCut system was developed to solve this problem. The system works by blasting a narrow jet of high-pressure, heated air at the precise location where droplets are formed between the Rotoform and the steel belt. The hot air blast cuts the threads before they have a chance to develop further. This not only leads to better quality pastilles (photo), but also results in a substantial reduction in the need to clean the machine. — *Sandvik Process Systems, Fellbach, Germany*  
[www.processsystems.sandvik.com](http://www.processsystems.sandvik.com)



Sandvik Process Systems



Alfa Laval

### Less fouling with this gasketed heat exchanger

The T35/TS35 (photo) is this company's latest gasketed heat exchanger, and comes with several innovations that minimize maintenance costs and ensure high uptime. These include the CurveFlow distribution area, which reduces fouling, and glue-free ClipGrip gaskets, which facilitate regasketing. The new, patented CurveFlow distribution area of the T35/TS35 gives it a much more uniform flow distribution

than other plate heat exchangers. This means there are no stagnant zones and significantly less fouling. The uniform flow also improves thermal efficiency. Users benefit from low maintenance costs and compact installations. — *Alfa Laval AB, Lund, Sweden*  
[www.alfalaval.com](http://www.alfalaval.com)

### A large control valve optimized for anti-surge applications

This company rounds off its proven series of Ecotrol control valves at the high end with the development of the nominal size DN 700 (28 in.). This valve has been specially optimized for use as an anti-surge valve on turbocompressors, but can also be used for all control applications involving liquid and gaseous media. The valve offers a high flow coefficient,  $K_{VS}$ , which usually well exceeds the delivery capacity of the turbocompressor. The quantities of gas trapped inside the pipelines and heat exchangers can therefore also be relaxed in a very short space of time upon safety shutdown of the compressor. Actuating times are short — usually considerably less than 2 s for the complete 0 to 100% valve opening. — *Arca Regler GmbH, Tönisvorst, Germany*  
[www.arca-valve.com](http://www.arca-valve.com)

### A device to detect dust in wet gases

Conventional dust-measuring devices are unable to differentiate between dust particles and water droplets, resulting in incorrect measurement results. The FWE200DH provides the solution: the dust-measuring device continually extracts the waste gas from the stack, dries it with a thermocyclone, and uses forward scattering to take high-precision measurements of dust concentrations, which are usually very low. The FWE200DH can be attached directly to the stack. Since no moving parts come into contact with aggressive gas, the measuring device is very low-maintenance. The FWE200DH can be used for monitoring wet-scrubbing plants, taking measurements in saturated gas following fluegas desulfurization, and measuring dust concentrations in wet exhaust air. — *Sick AG, Wildkirch, Germany*  
[www.sick.com](http://www.sick.com)  
*Mary Page Bailey and Gerald Ondrey*

# Facts At Your Fingertips

## Hot-oil heat-tracing systems

Department Editor: Scott Jenkins

Hot-oil heat-tracing systems use liquid heat-transfer fluids to reliably deliver heat to process fluids. Heat tracing may be used in place of jacketed equipment, often at lower cost and with lowered risk of cross-contact. The use of liquids reduces many of the costs associated with pressurized steam-handling systems and simplifies operation. Heat tracing is particularly beneficial in systems with highly viscous fluids or fluids at risk of freezing at ambient conditions. Properly designed heat-tracing systems can supplement overall heating systems to provide effective temperature control for applications that require a narrow temperature operating range. Proper selection and installation of heat tracing elements can ensure that process on-stream time remains optimal, and undue associated maintenance costs are avoided.

### Tubing options

The most basic hot-oil heat-tracing systems utilize metal tubing along the length of the pipe (Figure 1A). This tubing, called the tracer, is attached to the pipe by bands (preferred) or wires. A heated fluid flows through the tracer and transfers heat to the pipe and its contents. In this scenario, heat is poorly transferred through air convection. The narrow region of contact between the cylindrical piping and tubing provides very little surface area for heat conduction. Additionally, metal expansion due to heating can cause tubing to warp and pull away from the pipe, preventing best heat-transfer performance.

To overcome this problem, several methods are used to increase surface contact area. Various heat-transfer compounds are available that can fill the void between tubing and pipe. These compounds have high heat-transfer coefficients and displace air, which increases the surface area for conductive heat transfer.

In addition to heat-transfer compounds, modifications to the tubing shape can increase surface area. Specially shaped metal pieces can be fitted over the cylindrical tubing to conduct heat across a wider area on both sides of the tubing (Figure 1B). These metal pieces can be solid wedges or hollow V-shaped covers filled with heat-transfer compound. Some heat-tracing systems use specially fabricated tubes

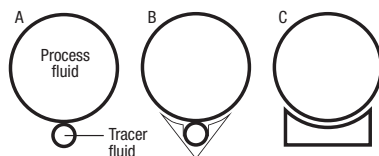


FIGURE 1. Three configurations of heat-oil tracing systems are shown

that fit the curvature of the pipe precisely without requiring separate equipment (Figure 1C). This tubing can be uniquely designed to fit piping, flanges, tanks and other process equipment. Many of these systems use short sections of tubing that are connected by hoses, allowing for simple installation around bends, flange pairs, and other structural features.

With any type of tubing, the option exists to use multiple tracers around the pipe. In some circumstances, one modified tubing tracer will transfer enough heat to replace several simple cylindrical tracers. Tubing choices are influenced by process economics and heating performance.

Materials for tubing, valves and other system components should be determined based on heat-transfer-fluid manufacturer recommendations.

### Installation

In most heat-tracing systems, tubing is held in place along the pipe by steel bands. Bands are spaced evenly along the tracer to ensure proper contact between tubing and pipe. For equipment with shapes impractical for banding, tracing systems can be designed to be clamped on. These systems require threaded bosses to be mounted on the pipe or tank prior to tubing installation.

Tracer fittings should be configured such that potential leakage will drain away from insulation. To minimize leakage, welded connections are preferred to threaded fittings. Properly sealed systems reduce fire hazard and decrease fluid replacement costs.

Weather barriers should be installed over insulation for systems exposed to the elements. Barrier seams should be oriented to prevent water from entering the insulation. Water coming into contact with the tracer or insulation will draw heat away from the process pipe and reduce the effectiveness of the heating system.

### System comparisons

Other types of heat-tracing systems include fully jacketed piping, steam tracing, and electric tracing. Fully jacketed systems provide the greatest heat-transfer surface area and are most useful for short heat-up times. Capital costs for jacketed systems are high, due to the amount of material and assembly required, and the complexity of the system can mean costly and difficult maintenance.

Steam tracing uses a constant-temperature heat source and is common for plants with readily available steam. While steam can be used in any tubing type, it is often associated with simple cylindrical tracers. Since each tracer requires its own steam trap and connections to steam sources, tubing modifications to improve heat transfer can reduce the number of tracers and associated capital costs.

Electric tracing systems have grown in popularity in recent years as safety and reliability have improved. In contrast to the other heat-tracing systems discussed here, electrical power is the only required input. Resistance heaters supply heat, and cable-modifying attachments (Figure 1B) can be used to improve heat transfer to the system. The addition of circuits and controllers for more complex systems can increase costs, often at a faster rate than for similarly complex fluid systems. Repairs to electric tracing systems can require specialized skills and tools. ■

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## Lactic Acid Production via Glucose Fermentation

By Intratec Solutions

Lactic acid is a naturally occurring chiral acid most commonly found in the L(+) form. Driven by a recent demand for more sustainable products, the lactic acid market has expanded beyond the food, pharmaceutical and cosmetics industries. Currently, a major growth application is for the production of polylactic acid (PLA) polymer. This biodegradable polymer has the potential to replace petrochemical-based plastics in applications such as packaging, paper coating, fibers, films and a wide range of molded plastic items.

### The process

Figure 1 depicts a process for lactic acid production via glucose fermentation similar to that developed by Purac (now Corbion; Amsterdam, the Netherlands; [www.corbion.com](http://www.corbion.com)). This process generates an 88 wt.% lactic acid solution in water that can be used as the raw material in polymerization processes. Rigorous control of fermentation conditions and additional purification steps are necessary to ensure both optically and chemically pure lactic acid. The quality of the PLA produced from it depends on the purity.

**Fermentation.** Glucose solution is fed to the fermenter. It undergoes a continuous anaerobic bacterial fermentation, and is converted to lactic acid. The fermentation temperature is controlled by circulating cooling water through internal coils, and the pH is controlled by the addition of CaCO<sub>3</sub> solution, which reacts with lactic acid to form calcium lactate.

**Recovery.** The fermentation broth is decanted and concentrated solids are filtered, removing mostly biomass. The aqueous solutions from the decanter and from the filter are acidified with sulfuric acid to convert calcium lactate into soluble lactic acid and CaSO<sub>4</sub>, which precipitates. The slurry outlet stream from the acidification vessel is fed to a belt filter for solids removal. The resulting aqueous filtrate is concentrated in an evaporator, then fed to ion-exchange columns for purification.

**Purification.** Concentrated lactic acid is sent to a solvent-extraction system. First, it is contacted with an organic extractant solution, forming two phases: a lactic-acid-rich organic phase and an aqueous phase containing most of the contaminants. The organic phase is then washed with water to remove contaminants. Next, it is contacted with water at 80°C to transfer the lactic acid into the aqueous phase. The resulting organic phase is washed with an NaOH solution and then with water to recover the extractant solution, while the lactic-acid-rich aqueous solution is sent to an evaporator. The concentrated lactic acid stream is fed to a distillation system where light ends are removed. Heavy ends are hydrolyzed and recycled to the process. Lactic acid is recovered as a side stream.

### Economic performance

An economic evaluation for a lactic-acid production plant was conducted, assuming a facility with a nominal capacity of 100,000 ton/yr of lactic acid solution (88 wt.%) constructed on the

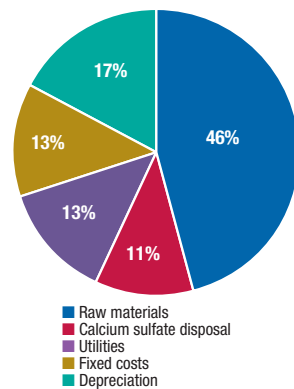


FIGURE 2. Production costs breakdown

U.S. Gulf Coast, and including storage capacity equal to 20 days of operation for feedstock (no product storage). Estimated capital expenses to construct the plant are ~\$200 million, while operating expenses are estimated at ~\$1,300/ton of lactic acid solution.

### Global perspective

Major production-cost components for this process are raw material acquisition (mainly glucose and CaCO<sub>3</sub>) and CaSO<sub>4</sub> byproduct disposal (Figure 2). Recent improvement efforts focus on using less costly cellulosic material to obtain fermentable sugars, and on developing low-pH-tolerant bacteria strains, which would decrease both CaCO<sub>3</sub> use and CaSO<sub>4</sub> generation. ■

Edited by Scott Jenkins

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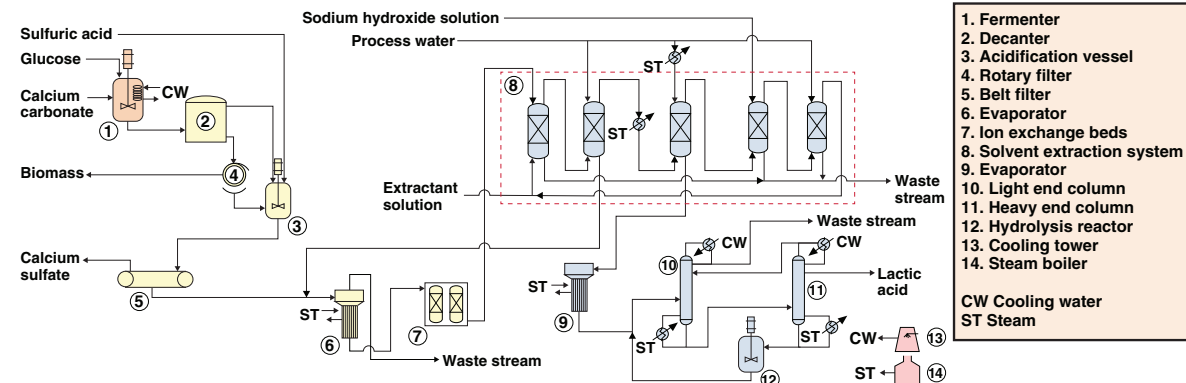


FIGURE 1. A lactic-acid production process from glucose, similar to the Purac process

# Extremely Low-Temperature Systems

Understanding the nuances of low-temperature engineering is key to safe and efficient operations

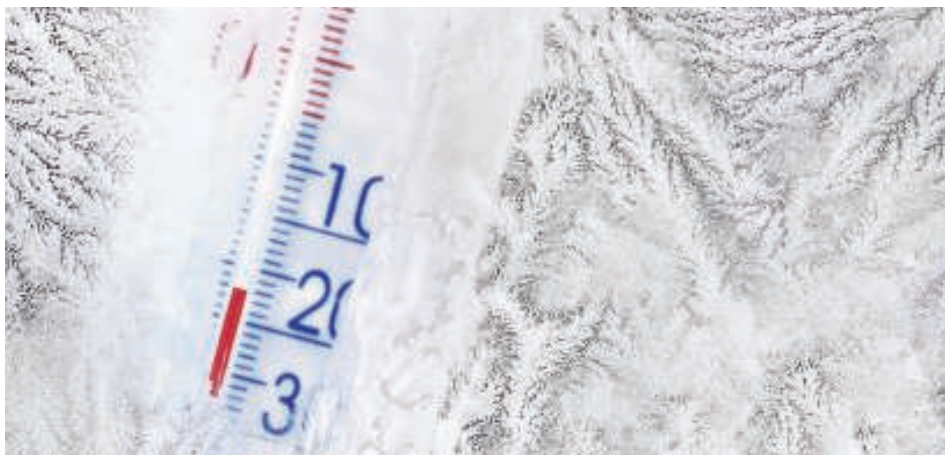
**Nancy Easterbrook, Tim Boland and Dave Farese**  
Air Products

## IN BRIEF

APPLICATIONS OF CRYOGENIC PROCESSING

SAFETY CONSIDERATIONS

CHOOSE BETWEEN CONSUMABLE AND MECHANICAL REFRIGERATION SYSTEMS



**E**xtrremely low temperatures — below  $-100^{\circ}\text{F}$  ( $-73.3^{\circ}\text{C}$ ) — have many widely diverse commercial uses, from de-flashing of molded rubber parts on the plant floor to pristine storage of biological materials in the medical and pharmaceutical industries. Understanding the special characteristics of low-temperature fluids and systems, and properly managing the associated risks, are important to the safe design and handling of low-temperature processes.

Temperatures this cold (Figure 1) rarely occur naturally, but are commonly found in modern industry and healthcare. Two technologies are typically used to generate extremely low temperatures: consumable refrigerant systems, which use products such as liquid nitrogen or dry ice, and mechanical refrigeration systems. While both mechanical refrigeration and consumable refrigerant systems share common risks of operating at extremely low temperatures, they have different costs and operational characteristics. Selecting the right technology to generate low temperatures depends on a number of specific process parameters.

Whether the refrigeration is from a mechanical or consumable source, extremely low temperatures can provide innovative

solutions to problems such as how to pulverize an intractable material or recover a difficult-to-condense volatile compound. Yet extremely low temperatures present technologists with a number of unique risks that must be safely managed.

Being aware of safety and process necessities, such as suitable materials of construction, appropriate venting, and proper personal protective equipment, will enable engineers to continue to harness the power of extreme cold to improve their operations in a safe and efficient manner.

## Common cryogenic applications

Extremely cold temperatures are routinely encountered in many products and processes, including the following:

- Magnetic resonance imaging (MRI) scanners, most of which use liquid helium to cool superconducting magnets to temperatures below  $-441.7^{\circ}\text{F}$  ( $-263.2^{\circ}\text{C}$ )
- Superconducting magnets, refrigerated with liquid helium, are the heart of the Large Hadron Collider — the machine used to discover the Higgs Boson — at the European Organization for Nuclear Research (CERN) facility in Meyrin, Switzerland
- Production, distribution and storage of in-

dustrial gases, such as oxygen, nitrogen, argon, hydrogen, and helium; for example, many plants store nitrogen as a cryogenic liquid at  $-320^{\circ}\text{F}$  ( $-196^{\circ}\text{C}$ )

- Natural gas liquefaction, which reduces the cost of transporting natural gas by liquefying it at temperatures below  $-250^{\circ}\text{F}$  ( $-157^{\circ}\text{C}$ )
- Polymerization of isobutylene, to make products such as butyl rubber, at temperatures from  $-130^{\circ}\text{F}$  to  $-148^{\circ}\text{F}$  ( $-90^{\circ}\text{C}$  to  $-100^{\circ}\text{C}$ )
- Testing aerospace components and assemblies, which must operate at very low temperatures
- The pharmaceutical and fine chemical industries, which use low temperatures in the synthesis of many intermediates, for freeze drying, and for storing biological samples and drugs
- Low-temperature metallurgy, such as quenching steel to reduce the presence of residual austenite
- Cryogenic grinding (Figure 2)

There are many additional opportunities within the industrial processing space to take greater advantage of the benefits of low temperatures and cryogenic liquids.

### Types of refrigeration systems

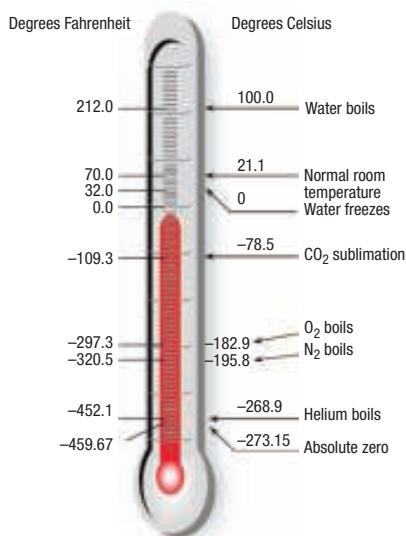
The low temperatures used in the processes outlined above are produced using either a consumable refrigerant, such as liquid nitrogen or liquid helium, or a mechanical refrigeration system.

A consumable refrigerant is produced at a central plant and delivered to the process site, where it is melted, evaporated, or sublimed to produce refrigeration. A bag of water ice in a food cooler is a simple example of a consumable refrigerant at a relatively warm temperature.

A mechanical refrigeration system uses mechanical energy to transfer the thermal energy from a refrigerant (or the machine's working fluid) at a low temperature to a higher-temperature heat sink, such as the ambient air or cooling water.

Mechanical refrigeration systems are commonly found in home air conditioning units, refrigerators, and freezers, although these systems operate at warmer temperatures than those we are concerned with here.

Both mechanical refrigeration and consumable refrigerant systems have some common risks associated with low-temperature operations that must be properly managed. These risks include the effects of exposing people to low temperatures, the effect of low temperatures on the ductility of many materials, thermal contraction, and managing the inventory of refrigerants in closed systems.



**FIGURE 1.** This temperature scale from  $212^{\circ}\text{F}$  to absolute zero shows the boiling points of nitrogen, oxygen, and helium

Because of their unique characteristics, the risks associated with low temperatures and the proper handling and use of cryogenic liquids may not be as well understood as other hazard classes.

### Safety considerations

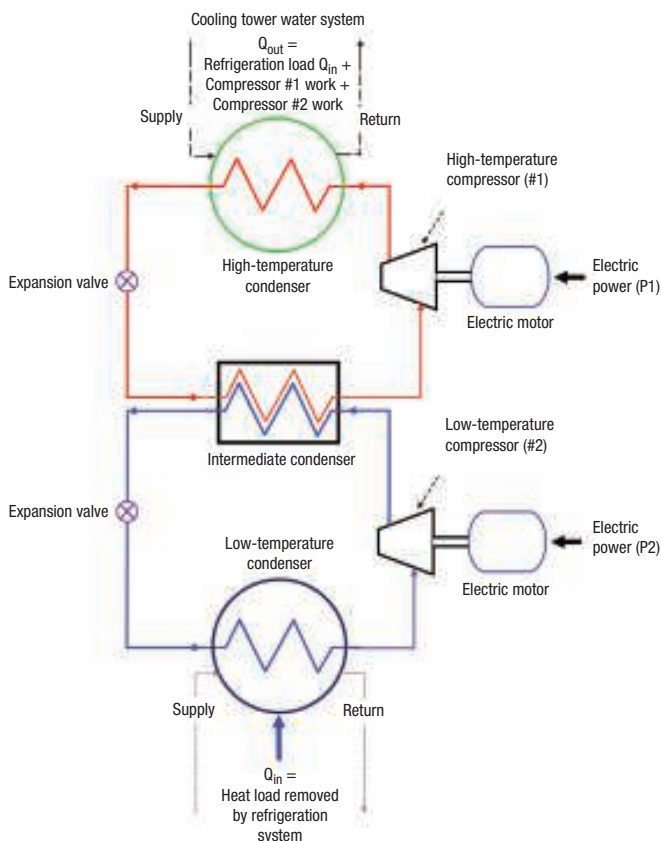
Extremely low temperatures require special attention and handling to avoid risks to plant and personnel safety.

**Freezing of tissue.** Extremely low temperature can rapidly freeze tissue, a characteristic that enables high-quality, long-term storage of cell lines and biological samples. For example, cryogenic tissue banks enable long-term storage of organs for research, while pharmaceutical companies also use cryopreservation to store cells. However, contact between a worker's bare skin and a low-temperature vapor, liquid, or solid can quickly freeze the skin tissue, resulting in a cryogenic burn.

Contact is most likely to occur when objects are being moved into or out of a low-temperature zone, such as placing samples into a liquid nitrogen storage bath; during maintenance activities; or when low-temperature fluids are being transferred. To per-

**FIGURE 2.** Cryogenic grinding allows even difficult materials like silicones to be reduced to a uniformly fine particle size





**FIGURE 3.** A typical cascade refrigeration system is illustrated here

form these tasks safely, workers should wear long sleeves, long pants, thermally insulating gloves, and face and eye protection. Eyes are the most sensitive area and can easily be harmed by cold vapor, so a full face shield over safety glasses is advisable. Pants should not have cuffs, and gloves should be loose so they can be quickly removed if necessary.

When an object is placed in a cryogenic liquid, or a cryogenic liquid is poured into a warm container, boiling and rapid vaporization will occur. These tasks should be done slowly, and tongs should be used to handle objects being dipped.

Even when low-temperature liquids are not directly handled, it is still important to identify any uninsulated pipes or vessels that contain them. If unprotected skin comes into contact with these surfaces, the skin may stick to them.

**Embrittlement.** Many materials will embrittle at cold temperatures. This is a powerful benefit of low temperatures that facilitates the size reduction of materials that would otherwise be too soft, oily, or volatile to grind. For example, rubber and other soft polymers can yield fine particles only when milled at low temperatures. Also, many spices contain volatile components that are essential to their quality. If spices are not milled at low

temperatures, qualities such as aroma and taste will diminish due to the heat generated during grinding.

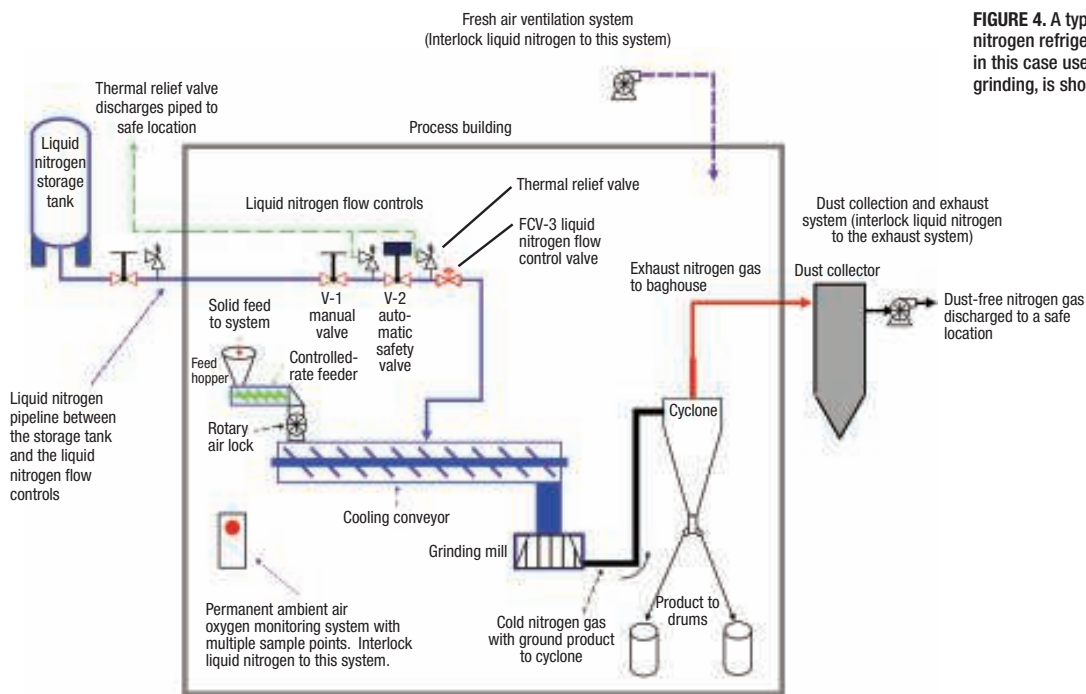
Many materials commonly used for ambient temperature systems, such as carbon steel or galvanized steel, lose their ductility as their temperature is lowered. This can result in a catastrophic failure of equipment and piping made from these materials if subjected to excessive stresses under low-temperature conditions. Piping and pressure vessel design and fabrication codes, such as ASME B31.3 (Process Piping) and B31.5 (Refrigeration Piping and Heat Transfer Components), address this hazard by specifying minimum temperatures for materials of construction, plus materials testing and design restrictions for selecting and using materials at low temperatures. Materials that remain ductile at low temperatures include austenitic stainless steel (including types 304, 316 and 321), copper, red brass, and many copper alloys and aluminum. These are the preferred low-temperature materials of construction.

In addition to the embrittlement of materials of construction, thermal contraction must also be considered when designing an extremely low-temperature system. Most materials of construction will shrink as their temperatures decrease. For example, a stainless steel or copper pipe that is 100 ft. (30 m) long will contract linearly by about 3.5 in. (90 mm) as it cools down from 70°F to -320°F (20°C to -195°C). This thermal contraction is independent of the diameter of the pipe. The stresses generated by thermal contraction are large and will severely damage an improperly designed pipeline or piece of equipment.

**Vapor expansion.** Liquid nitrogen, the most common cryogen, expands to over 700 times its liquid volume when warmed to 68°F (20°C). This expansion property is used commercially to purge, inert, and pressurize containers housing foods, drugs, and chemicals that are sensitive to air or moisture by dropping a small amount of liquid nitrogen into the container during packaging.

Low-temperature systems may need to be designed to accommodate the pressures that can be generated whenever a liquid refrigerant is trapped in a closed volume. For example, liquid nitrogen or liquid trifluoromethane can become trapped in a pipeline between two closed valves. As the cold liquid warms up to ambient temperature, the increase in vapor pressure can spring flanged joints and burst pipes.

In liquid nitrogen piping systems, this expansion is usually managed by installing a pressure relief valve, typically known as a thermal relief valve, in every piping segment that



**FIGURE 4.** A typical liquid nitrogen refrigeration system, in this case used for cryogenic grinding, is shown here

can potentially trap liquid. All thermal relief valves in a liquid nitrogen system should discharge to a safe location, ideally outdoors.

In mechanical systems, the higher unit cost and other properties of the refrigerants used, such as trifluoromethane, means that thermal relief valves are not a feasible solution. Instead, these systems typically incorporate expansion vessels to accommodate expansion without loss of refrigerant.

**Condensing or solidifying surrounding materials.** The ability of cryogenic temperatures to liquefy substances with low boiling points is useful in many process operations. For instance, it is used to help plants operate more sustainably by condensing volatiles that cannot be separated from exhaust air streams at ordinary refrigeration temperatures. Many times, the condensed material can be reused and recycled instead of being incinerated in a thermal oxidizer, and hence wasted.

If cryogenic liquids contained in vessels or piping are colder than the oxygen dewpoint of the surrounding air, an oxygen-enriched liquefied-air condensate will form on uninsulated surfaces. This can drip onto surrounding equipment and personnel, causing cryogenic burns. As the condensate warms and re-evaporates, the resulting local raised oxygen levels can also create a serious fire hazard.

In the case of hydrogen and helium, the surrounding air can even be solidified. This frozen air can block the discharge ports of pressure relief valves, preventing them from operating correctly.

## Low-temperature systems

There are two technologies for generating low temperatures in industrial processes: mechanical refrigeration systems and consumable refrigerants. Whatever the method of final delivery in the plant, however, producing extremely low temperatures requires sophisticated mechanical refrigeration technology.

Cascade refrigeration systems are very useful for producing low temperatures. A cascade refrigeration system combines two or more compressors and typically two or more different refrigerants in a series of connected refrigeration cycles operating at different temperatures (Figure 3). The low-temperature cycle removes heat from the low-temperature process and transfers it to the high temperature cycle via an intermediate condenser or heat exchanger. The high-temperature cycle removes the heat from the intermediate condenser and transfers it to the high-temperature condenser, where it is rejected to cooling water or ambient air.

“Autocascade” systems are also very effective for producing low temperatures. An autocascade system has a single compressor, a refrigerant mixture comprising two or more components with progressively decreasing atmospheric boiling points, and a series of intermediate heat exchange steps [1,2]. Most natural gas is liquefied using mixed-refrigerant autocascade-type refrigeration cycles. Closed- and open-air-cycle refrigeration systems have also been used to produce low temperatures [3,4].

A consumable refrigerant is produced at a central plant and delivered by truck to the process site for use. Low temperatures are generated by evaporating the consumable refrigerant and releasing its vapors to the atmosphere. At the point of use, consumable refrigerant systems are typically simpler than mechanical refrigeration systems, and easier to install and operate. This simplicity means that consumable refrigerant systems can easily be designed to be very reliable.

Liquid nitrogen and liquid carbon dioxide are the most common consumable refrigerants for extremely low-temperature processes. Liquid helium can be used as a consumable refrigerant for certain high-value applications, but care must be taken due to the high unit cost of helium.

Liquid carbon dioxide, with a triple point of  $-109.3^{\circ}\text{F}$  ( $-78.5^{\circ}\text{C}$ ), can be used at the upper end of the extreme temperature range. Solid carbon dioxide, also known as dry ice, can be used to make laboratory and pilot-production-scale cold baths by mixing it with

a low-freezing-point solvent such as acetone or limonene. Such bath temperatures are limited to about  $-109^{\circ}\text{F}$  ( $-78^{\circ}\text{C}$ ).

### Liquid nitrogen refrigeration

Liquid nitrogen, with a normal boiling point of  $-320.5^{\circ}\text{F}$  ( $-195.8^{\circ}\text{C}$ ), is the most widely used consumable refrigerant for generating temperatures below  $-110^{\circ}\text{F}$  ( $-79^{\circ}\text{C}$ ). Figure 4 illustrates a typical liquid nitrogen refrigeration system. There are typically five major pieces of equipment:

**Liquid nitrogen storage tank.** This holds the inventory of liquid nitrogen for use in the process. A cryogenic liquid storage tank is a sophisticated tank-within-a-tank. The inner tank holding the liquid nitrogen is suspended from a carbon steel outer tank by a carefully engineered support system to minimize the heat transfer by conduction. The space between the inner and the outer tanks is held at high vacuum to minimize heat transfer by convection. The space between the tanks can also be filled with powder insulation, or

TABLE 1. SOME COMMON REFRIGERANTS

Chemical name	Refrigerant number	Category (1)	Molecular weight	Normal boiling point, $^{\circ}\text{F}$	Critical temperature, $^{\circ}\text{F}$	Triple point, $^{\circ}\text{F}$	Heat of vaporization, BTU/lb (2)	Flammability range, % (3)	Refrigerant type (4)
Dichlorodifluoromethane	21	MP HCFC	102.92	48.0	353.2	-210.8	102.8	NF	Working fluid
1,1-Difluoro-1-chloroethane	142b	MP HCFC	100.50	15.4	278.9	-203.4	95.9	6.3-17.9	Working fluid
Chlorodifluoromethane	22	MP HCFC	86.47	-41.2	204.8	-251.4	100.5	NF	Working fluid
Water (5)	718	$\text{H}_2\text{O}/\text{CO}_2/\text{NH}_3$	18.02	212.0	704.9	32.0	973.8	NF	Example of consumable
1,1,1,2-Tetrafluoroethane	134A	HFC	102.03	-14.9	214.0	-149.8	93.5	NF	Working fluid
Ammonia	717	$\text{H}_2\text{O}/\text{CO}_2/\text{NH}_3$	17.03	-28.1	270.1	-107.9	592.4	15-30	Working fluid
Propane	290	HC	44.10	-43.7	206.6	-305.8	183.2	2.2-9.5	Working fluid
Hexafluoroethane	116	HFC	138.01	-108.8	67.4	-149.3	49.3	NF	Working fluid
Carbon dioxide (as dry ice)	744	$\text{H}_2\text{O}/\text{CO}_2/\text{NH}_3$	44.01	-109.3	87.9	-70.0	237.0	NF	Consumable only
Carbon dioxide (liquid at 95 psia)	744	$\text{H}_2\text{O}/\text{CO}_2/\text{NH}_3$	44.01	-59.8	87.9	-70.0	145.0	NF	Both
Trifluoromethane	23	HFC	70.01	-115.9	78.7	-247.3	103.8	NF	Working fluid
Ethane	170	HC	30.07	-127.4	90.4	-297.9	210.1	3-16	Working fluid
Ethylene	1150	HC	28.05	-154.6	49.8	-272.5	207.5	2.7-34	Working fluid
Tetrafluoromethane	14	HFC	88.00	-198.5	-50.2	-298.5	57.5	NF	Working fluid
Methane	50	HC	16.04	-258.6	-116.2	-296.5	219.4	5-15	Working fluid
Argon	740	Element	39.95	-302.2	-188.4	-308.8	69.2	NF	Both
Nitrogen	728	Element	28.01	-320.5	-232.5	-345.9	85.5	NF	Both
Neon	720	Element	20.18	-410.9	-379.8	-415.5	36.8	NF	Both
Helium	704	Element	4.00	-452.1	-450.3	-455.8	8.9	NF	Both

(1) MP HCFC = an HCFC refrigerant that is subject to the Montreal Protocols; HFC = hydrofluorocarbon; HC = hydrocarbon;  $\text{H}_2\text{O}/\text{CO}_2/\text{NH}_3$  = compounds

(2) Carbon dioxide will sublime (solid-to-vapor transition) rather than vaporize (liquid-to-vapor transition) at atmospheric pressure

(3) Lower and upper flammable limits, respectively, in air; NF = non-flammable

(4) A working fluid is a refrigerant used in a mechanical refrigeration cycle

(5) Water is included as an example of a consumable refrigerant when used in a cooling tower

the inner tank wrapped with super insulation blankets, to reduce radiant heat transfer.

**An insulated pipeline.** This transfers liquid nitrogen from the storage tank to the process. Liquid nitrogen pipelines are typically insulated to conserve the refrigeration value of the liquid nitrogen and to prevent personnel contact with the very cold surfaces of the pipe. Poly(isocyanurate) cellular plastic foam is the simplest insulation system for liquid nitrogen pipelines. Vacuum jacketed (VJ) piping is more efficient than foam insulation, but is also more expensive to purchase and install. VJ piping is a coaxial piping system: the inner pipe carries the liquid nitrogen, while the outer pipe supports the inner pipe and contains a vacuum that minimizes convective heat transfer. The inner pipe is wrapped with layers of super-insulation, a material designed to minimize radiation heat transfer.

When a consumable refrigerant like nitrogen is used for a super-cold application such as cryogenic grinding or de-flashing, it is important that the nitrogen be primarily in the liquid state at the point of use. The higher the quality of the two-phase liquid-gas mixture (in other words the greater the ratio of liquid to gas), the more heat it can remove from the process material. While insulated piping is crucial to keeping the nitrogen as a liquid, other factors are also important. When a cryogenic operation starts up, for instance, the need to cool the piping and equipment from ambient to operating temperature will temporarily reduce the fraction of liquid nitrogen. In addition, flash losses are created by pressure drops along the pipe run, especially at valves, sharp bends, and the tops of vertical legs. To that end the pipe run needs to be as short as possible, with smooth bends and minimal changes in direction or elevation.

**Liquid nitrogen flow controls.** The flow of liquid nitrogen to a refrigeration process is typically controlled using a three-valve manifold. The first valve is a manual valve that is used to stop the flow of liquid nitrogen to the process. This valve should be able to be locked out. The next valve is an automatic safety valve to stop the flow of liquid nitrogen in an emergency. The final valve is a flow control valve. Valves are typically made from stainless steel, brass, or bronze; carbon steel components must not be used. Low-temperature valves are similar to those used at ambient temperatures, but feature modifications such as extended stems to keep their packing glands warm.

**Heat exchanger.** The heat exchanger is designed to evaporate the liquid nitrogen, and possibly to heat the resulting nitrogen gas, by transferring heat from the substance being refrigerated. The heat exchanger in Figure 5



is a modified screw auger for cooling plastic pellets prior to cryogenic grinding. The heat exchanger could also be a cooling tunnel for refrigerating solids that will not convey well in a screw auger. Liquids or gases can be cooled, or vapors condensed, using liquid nitrogen in heat exchangers of more familiar types: shell-and-tube, spiral plate, spiral tube, and plate heat exchangers.

**Gaseous nitrogen exhaust system.** A stream of nitrogen gas will be generated as the liquid nitrogen evaporates in the heat exchanger. This nitrogen gas must be discharged to a safe location outdoors to prevent the formation of an oxygen-deficient atmosphere. Oxygen-deficient atmospheres can be very dangerous to people if their hazards are not managed safely. Wall-mounted ambient-air oxygen sensors should be included with any consumable refrigerant system.

References 5 and 6 discuss the safe handling of liquid nitrogen in more detail.

### Selecting a low-temperature system

While mechanical refrigeration and consumable refrigerant systems face common technical challenges associated with low-temperature operation, they have very different cost and operational characteristics. Selecting the right technology to generate low temperatures depends on a number of specific parameters and process goals, as discussed below:

**FIGURE 5.** This ultrafine grinding mill uses liquid nitrogen for refrigeration

**Process temperature.** Table 1 shows some potential refrigerants, with their normal boiling points and critical temperatures. This is not a complete list, and does not include multi-component refrigerants as used in autocascade cycles. Below  $-100^{\circ}\text{F}$  ( $-73^{\circ}\text{C}$ ) the choice of refrigerants for mechanical refrigeration systems becomes restricted primarily to flammable materials, plus nitrogen, argon, and helium. Using a non-flammable consumable refrigerant, such as liquid nitrogen, can be an attractive alternative to a mechanical refrigeration system containing flammable refrigerants, especially at process temperatures below  $-100^{\circ}\text{F}$ .

Helium's properties make it an attractive refrigerant below  $-320^{\circ}\text{F}$  ( $-195^{\circ}\text{C}$ ). Helium can be used either as a consumable refrigerant or as a working fluid in a mechanical refrigeration system.

**Installed capital costs.** Consumable refrigerant systems have relatively low installed capital costs. The cost breakdown between the variable and fixed (capital) costs of a consumable refrigerant system is weighted toward the variable costs; that is, the cost of the refrigerant.

The installed capital costs of mechanical refrigeration systems are typically higher than those of consumable refrigerant systems, often including a significant cost for the refrigerant charge. Mechanical refrigeration may also depend on using existing cooling towers for heat rejection. The cost breakdown for mechanical refrigeration is weighted toward the capital costs. The variable cost of a mechanical system should be lower than that of a consumable refrigerant system. However, if the mechanical refrigeration system is under-utilized, the total cost can be significantly higher than expected.

The system that provides the highest return on invested capital should be selected. In many cases, this is a consumable refrigeration system.

**Process duration.** Consumable refrigerant systems can be attractive for processes that only require low-temperature refrigeration for a small portion of the overall cycle, even though the process as a whole may run for extended periods. An example of this type of process would be low-temperature synthesis of pharmaceuticals, where cleaning between batches consumes a significant amount of time.

Consumable refrigerant systems may also be able to provide a much faster cool-down time than a typical mechanical refrigeration system. Mechanical refrigeration and consumable refrigerant systems can even complement each other, with the consumable refrigerant system providing fast cooling cycles

and very low temperatures, while mechanical refrigeration is used for the baseload.

Low capital costs can make consumable refrigerant systems attractive for processes with uncertain market projections. Consumable refrigerants are also ideal for processes that do not reoccur frequently, such as cooling a reactor during a refinery turnaround, or using liquid nitrogen to cool down an LNG plant during startup. ■

*Edited by Charles Butcher*

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# New Measurement Practices for Cold Climates

Selecting the right pressure, temperature, flow and level meters and connection systems can help to eliminate the need for heated enclosures and sensing lines

In an environment where the ambient temperature can fall below  $-20^{\circ}\text{C}$ , outdoor measurements can suffer from poor accuracy, slow response, poor reliability and unreadable displays. To avoid these problems, users have traditionally specified heated enclosures and heat-traced sensing lines and capillaries, but these incur high capital and operating costs. A previous article [7] provided recommendations for improving steam-flow measurement by installing the transmitter

on top of the flow element to eliminate the formation of wet legs, thereby reducing the cost and risk of wet-leg plugging and freezing. It also recommended conducting liquid level measurement by using remote sensors connected by wires, rather than wet legs or oil-filled capillaries.

In recent years, additional practices have emerged that further leverage improving technology and experience, including the following

- Modern transmitters can be directly installed in the field, with no enclosures (heated or unheated)
- New seals allow pressure and level transmitters to be close-coupled, with connections heated by the hot process instead of relying on external heat tracing
- New diagnostics allow users to detect and predict cold-weather problems so they can be fixed before they cause winter shutdowns

Facilities that adopt these new practices will further reduce capital and operating costs, and improve performance, safety and reliability.



**FIGURE 1.** These pressure transmitters are connected to manifolds and mounted inside a sealed enclosure, which also contains thermostat-controlled heaters

## Heated enclosures

Figure 1 shows transmitters installed inside a heated enclosure. In addition to the transmitters, the enclosure contains one or more pressure manifolds and thermostat-controlled heaters that are used to heat the transmitter and the manifolds. Many users specify factory assembly of their enclosures, which ensures consistent assembly quality and minimizes field labor.

The assembled enclosures are then connected in the field. The sensing lines are connected by the pipefitter, the instrument wiring is connected by the instrument technician, and power for the heaters is provided by the electrician.

Reasons for using heated enclosures with older transmitters include the following:

- To avoid freezing of the transmitter, which could cause mechanical damage. Of particular concern has been the impact of a “cold restart” as older designs can suffer damage when power is restored after an extended outage during which the ambient temperatures fall below  $-30^{\circ}\text{C}$
- To ensure that local displays continue

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## IN BRIEF

HEATED ENCLOSURES

ELIMINATING LINE  
HEATING

DIAGNOSTICS

RECOMMENDATIONS



**FIGURE 2.** This image shows a cold-temperature qualification test for a pressure transmitter whose LCD is clearly readable at a temperature of  $-40^{\circ}\text{C}$



**FIGURE 3.** These modern wireless temperature transmitters have been successfully operating in Fort McMurray, in Alberta, Canada, for several years, without the need for enclosures

operating, as older liquid crystal display (LCD) designs do not update at temperatures below  $-20^{\circ}\text{C}$

- To ensure performance, as accuracy, drift, ambient temperature effects and response time tend to be compromised at temperatures below  $-40^{\circ}\text{C}$
- To meet the transmitter's agency approvals; for instance, Canadian Standards Association (CSA) may not be valid at temperatures below  $-40^{\circ}\text{C}$

Total installed cost of the heated enclosure — including procuring the enclosure and heater(s), factory assembly and connection in the field — typically exceeds the cost of the transmitter itself. After startup, the user also incurs ongoing electricity costs.

And there are other considerations: Heaters require frequent maintenance, seals can leak, and a thermostat that fails on or off can result in damage to the transmitter. Finally, any fugitive emissions from the threaded connections can become trapped and build up inside the sealed enclosure, creating a potential safety risk when it is opened by maintenance or operations personnel.

Some newer transmitters contain modern electronics that are designed and approved for extended operation in very cold tempera-

tures, without significant degradation of performance, and can even withstand multiple restarts at  $-50^{\circ}\text{C}$ . Figure 2 shows a cold-temperature qualification test for a modern, smart pressure transmitter — note that the LCD is clearly readable to  $-40^{\circ}\text{C}$ .

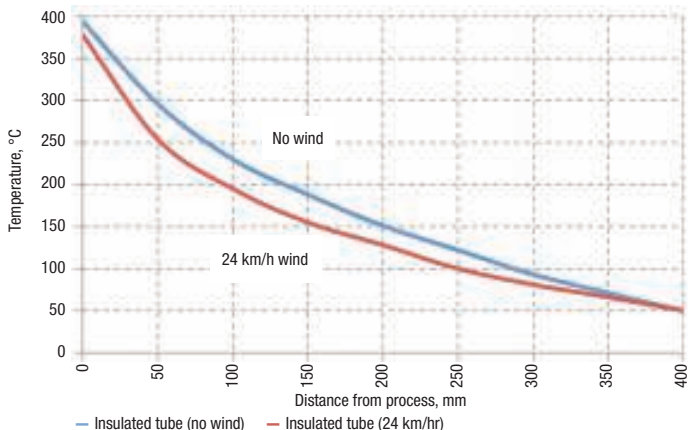
A modern transmitter that has been designed for operation and startup in very cold temperatures does not require or benefit from installation in an enclosure, heated or unheated. Eliminating the need for an enclosure and installing the transmitter directly in the environment yields savings in capital and operating costs — often exceeding the cost of the transmitter — and yields improvements in performance, safety and reliability.

Figure 3 shows one of the hundreds of smart transmitters that have been operating outdoors without enclosures in northern Alberta, Canada, over multiple winters with no failures. Although the photograph shows a wireless temperature transmitter, similar installations are also operating for wired and wireless pressure, flow, level and other transmitters.

### Eliminating line heating

In pressure or level applications, the pressure transmitter is connected to the process using sensing or “impulse” lines. These are called “wet legs” if filled with liquid, or “dry legs” if filled with vapor. Where differential pressure is measured — to obtain level in a closed vessel or pressure drop across a flow element or a filter — the pressure taps are in different locations. Sensing lines allow the transmitter to be installed between the two taps.

Even where only pressure is being measured with a single tap, the sensing line allows the user to locate the transmitter at a location that may be more convenient for maintenance. Finally, the sensing line is used with a very hot or very cold process. The sensing line is sized to ensure that the transmitter sensor is not exposed to a temperature outside of its safe operating range ( $-40$  to  $120^{\circ}\text{C}$  is typical).



**FIGURE 4.** Heat dissipation at  $0^{\circ}\text{C}$  ambient (shown here) is six times faster than at  $50^{\circ}\text{C}$ , but is three times slower than at  $-40^{\circ}\text{C}$ . This varying heat dissipation complicates the design of sensing lines in environments with widely varying ambient temperatures

**TABLE 1. COMMON FILL FLUIDS: BOILING POINTS AT ATMOSPHERIC PRESSURE AND VISCOSITY AT SELECTED TEMPERATURES**

Fill Fluid	Maximum Temp., °C	Maximum Temp., °F	Viscosity at 25°C (centistokes, cSt)	Viscosity at 0°C (cSt)	Viscosity at -25°C (cSt)
Syltherm XLT	149	300	1.6	2.1	3.5
Silicone Si200	205	400	9.5	16.1	30.7
Silicone Si704	315	600	39	183	Stops responding
Silicone Si705	370	698	175	Stops responding	Stops responding

Note: Syltherm XLT is manufactured and marketed by Dow Chemical; Boiling points and viscosity results for all silicone oils from internal Rosemount testing, not published.

In an outdoor application with a hot process, ensuring the correct length of sensing lines can be challenging. If the line is too short, not enough heat will be dissipated in summer, and the transmitter can become overheated and damaged. If the line is too long, too much heat will be dissipated in winter, and the process fluid may begin to freeze, crystallize or separate and plug in the lines before it reaches the transmitter. For example, components in some heavy crude oils start to freeze below -40°C [2]. Thawing a plugged or frozen pipe or vessel connection in winter requires the user to apply external heat, which, in the presence of hydrocarbons, presents an obvious fire risk.

The designer tasked with calculating the optimum length of sensing line will find that the rate of heat dissipation in sensing lines is not well understood or documented. A Web search reveals a range of informal guidelines, such as "100°F/ft." While a sensing line designed using this informal guideline may ensure sufficient heat dissipation in a Houston summer, for example, in an Alberta winter it will be much too long.

As shown in Figure 4, laboratory testing [3] reveals that using typical insulated tubing, at 0°C with a light wind, the process temperature falls from 400°C to 50°C within 400 mm (16 in.) of the process connection. This heat dissipation is six times as fast as predicted by the guideline; at an ambient temperature of -40°C the same testing shows it is twelve times as fast. These widely varying rates of heat dissipation from season to season usually make it impossible for an engineer to design sensing lines that are both long enough to avoid over-heating the transmitter in summer, yet also short enough to avoid freezing the impulse lines in winter.

To resolve this design problem, most users in cold climates insulate and heat-trace their sensing lines. Unfortunately, as with heated enclosures, heated sensing lines add significant capital and operating costs. Worse, inconsistent heating of the sensing lines can cause condensation in dry legs or evaporation in wet legs, leading to measurement drift

and requiring additional maintenance.

To avoid the high maintenance requirements typical of heated sensing lines, many users in cold climates choose to connect the transmitter to the process using a remote seal. This provides flexibility in the choice of process connections and fill fluids, and reduces the risk of plugging due to dirty fluid. The remote seal can be mounted directly to the transmitter, or remote-mounted via an oil-filled capillary, as shown in Figure 5.

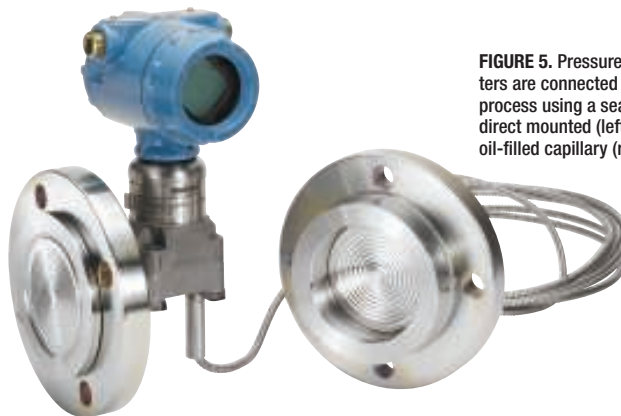
Use of the oil-filled seal and capillary does not evade the heat-dissipation challenge. The silicone oil used to fill the seal and capillary must be selected so that it does not boil at the highest anticipated operating temperature. Table 1 shows boiling points at atmospheric pressure for some common silicone oils. Exposure to the boiling temperatures will cause each oil's volume to increase dramatically, which can burst the sealed system, leading to failure.

Even extended operation near the boiling point can eventually cause thermal breakdown of the oil [4]. The user must also determine if there is any risk that, during a process upset or maintenance operation, the seal could experience momentary vacuum, since that would reduce the oil's boiling point.

Faced with these risks, many users default to higher-molecular-weight (M.W.) oils, such as Si704, which has a higher boiling point



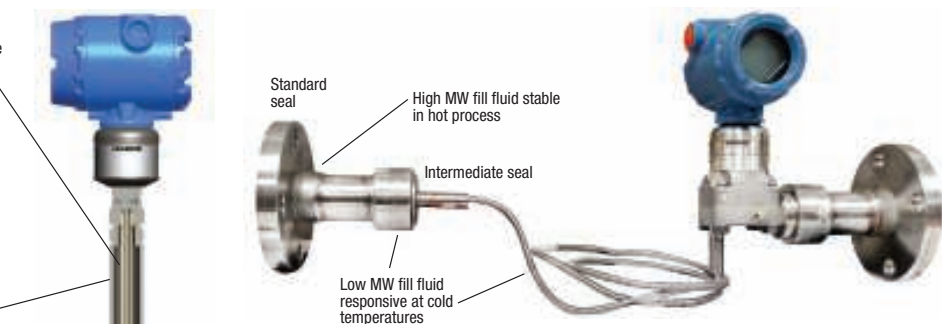
**FIGURE 6.** Heat-traced capillaries, as shown here, ensure fast response but incur high capital and operating costs



**FIGURE 5.** Pressure transmitters are connected to the process using a seal, either direct mounted (left) or via an oil-filled capillary (right)



**FIGURE 7.** A thermally optimized, single-oil seal system eliminates the need for heat tracing, without compromising measurement responsiveness, for close-coupled, gage pressure applications



**FIGURE 8.** The newer thermally optimized, two-oil seal system can help to eliminate the need for heat tracing, and works in a wide variety of applications, including differential applications, or where the transmitter must be mounted remotely from the process

and improved thermal stability. While higher-M.W. oils stay liquid at higher temperatures, the tradeoff is that they suffer from very high viscosity and thus become slower or even non-responsive at lower temperatures.

As shown in Table 1, during the winter, low-M.W. Si200 oil has higher viscosity and thus suffers from slower response than during the summer. The higher-M.W. Si704 and Si705 oils are relatively viscous and slow even in the summer months; in the winter, they can completely solidify and become non-responsive. Since nothing necessarily “fails” — the oil will just thicken or solidify when cold but should return to normal operation when warmer temperatures return — the user may not be aware that this is happening. The only symptom will be very slow or no response with corresponding poor pressure or level control, particularly during the winter season. This is of particular concern in safety installations, since the non-responding measurement will be covert and can impact multiple, redundant sensors.

To avoid these problems, most users in cold climates heat-trace their capillaries, as shown in Figure 6. Heat tracing of capillaries leads to very high installed and operating costs, as with heated enclosures and sensing lines.

Users in cold climates have historically been forced to choose between two unappealing options:

- Use unheated impulse lines or capillaries, which in winter can lead to very slow response and compromised process control and safety. The lines will require high maintenance, and in extreme cases, will freeze completely and require manual heating, which is both inconvenient and potentially dangerous
- Use thermostat-controlled heated impulse lines or capillaries, which are expensive to

install and operate, and require frequent maintenance

Newer remote seals are now available that have been specially optimized to eliminate the need for process-line heating without compromising response time. As shown in Figure 7, the seal is directly connected to the vessel or pipe, which is presumably maintained at a high temperature using a pipe- or vessel-heating blanket. The seal is insulated, and the design of the seal and internal copper tube are optimized so that sufficient heat is transferred to ensure that the temperature at the sensor is low enough to avoid damaging the transmitter, but high enough to ensure that the oil stays hot, in a liquid phase and responsive. This eliminates the risk of plugging or freezing, without any need for external heat tracing. To date, thousands of these units have operated for several winters in northern Alberta without failure.

For very hot processes, or where the user wishes to locate the transmitter at some distance from the process, a two-oil solution may be needed (Figure 8). A high-M.W. oil, such as Si705, is used near the hot process, and provides high temperature stability. Response time is fast since the oil stays hot. A relatively low-M.W. oil, such as Syltherm XLT (from Dow Corning), is used after the intermediate seal — after the oil has cooled — and runs through the capillary to the transmitter. Syltherm XLT retains its low viscosity at temperatures below  $-50^{\circ}\text{C}$ , which ensures fast response in the coldest climates without the need for heat tracing.

The thermally optimized, single-oil seal is suitable for pressure measurements that can be close-coupled. This includes level applications that use electronic remote sensors. As described in the previous article [7] electronic remote sensors are pressure sensors connected together using standard electrical cable instead of oil-filled capillaries. Electronic remote sensors are best suited to low- (including vacuum) to medium-pressure

level applications. In higher-pressure level applications, thermally optimized, two-oil seals provide a better solution. These seals would use a high-M.W. oil at the hot process connection and a low-M.W. oil in the (long) capillaries that extend to the transmitter.

## Diagnostics

Cold-weather maintenance can be minimized with the appropriate use of process and measurement diagnostics. While users in all climates seek to minimize unscheduled maintenance, this takes on added importance in cold climates. Winters in northern climates are typically characterized by temperatures below  $-30^{\circ}\text{C}$  and relatively few hours of daylight.

Working under these conditions is unsafe and inefficient. In addition to engineering the process to withstand the impact of a cold climate, users should build-in diagnostics that detect problems in the fall, so they can be fixed safely and efficiently before they cause a dangerous shutdown in the winter. For instance, the following steps should be taken to do the following:

- Detect failure of heat tracing before it causes process freezing by monitoring the surface temperatures of pipes and vessels, using clamp-on and surface-mount wireless temperature sensors (Figure 9)
- Detect failures of steam traps using wireless acoustic sensors. A bad steam trap results in year-round efficiency loss; if not corrected, it may result in process freezing in mid-winter
- Verify high-level switches and radar level transmitters using built-in remote diagnostics that eliminate the need for maintenance personnel to climb on top of tanks in winter

## Recommendations

When measurement devices are installed outdoors where the ambient temperature can fall below  $-20^{\circ}\text{C}$ , the user should be concerned about poor accuracy, slow response, poor reliability and unreadable displays. The traditional approach to obtain acceptable measurements is to use heated enclosures and heat-traced sensing lines and capillaries, but these often incur high capital and operating costs, and reliability and safety risks.

As an alternative, users should consider modern transmitters that are proven safe, accurate and reliable at low temperatures — including cold-restart applications after extended shutdown. Users should also use special, thermally optimized, seals to decouple pressure and level measurements and eliminate heated sensing lines. In applications with very high temperatures or long capillaries, two-oil seals should be consid-



**FIGURE 9.** A clamp-on wireless temperature sensor allows users to cost-effectively verify correct operation of heat tracing and avoid process freezing

ered. Users should also try, where possible, to diagnose process and measurement problems so they can be fixed before they cause winter shutdowns. Adopting these new best practices will yield lower capital and operating costs, and improve safety, reliability and performance. ■

*Edited by Suzanne Shelley*

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# Engineering for Plant Safety

Early process-hazards analyses can lead to potential cost savings in project and plant operations

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Consultant

## IN BRIEF

CPI PROJECT LIFECYCLE

PROCESS HAZARD IDENTIFICATION

WHEN TO USE A GIVEN METHOD

SAFE-DESIGN OPTIONS

ADDRESSING HAZARDS EARLY

FINAL REMARKS



The chemical process industries (CPI) handle a wide variety of materials, many of which are hazardous by nature (for example, flammable, toxic or reactive), or are processed at hazardous conditions (such as high pressures or temperatures). The risks associated with CPI facilities not only extend to the plant personnel and assets, but can potentially affect the surrounding population and environment — sometimes with consequences having regional or international scale, as in the case of toxic vapor or liquid releases.

It is for this reason that process safety is recognized as a key element throughout the entire life of the plant, and several industry and professional associations and government authorities have issued norms, standards and regulations with regards to this subject.

Process safety, as defined by the Center for Chemical Process Safety (CCPS), is “a discipline that focuses on the prevention and mitigation of fires, explosions and accidental chemical releases at process facilities. Excludes classic worker health and safety issues involving working surfaces, ladders, protective equipment and so on.” [1] Process safety involves the entire plant lifecycle: from visualization and concept, through basic and detailed engineering design, construction, commissioning, startup, operations, re-

vamps and decommissioning.

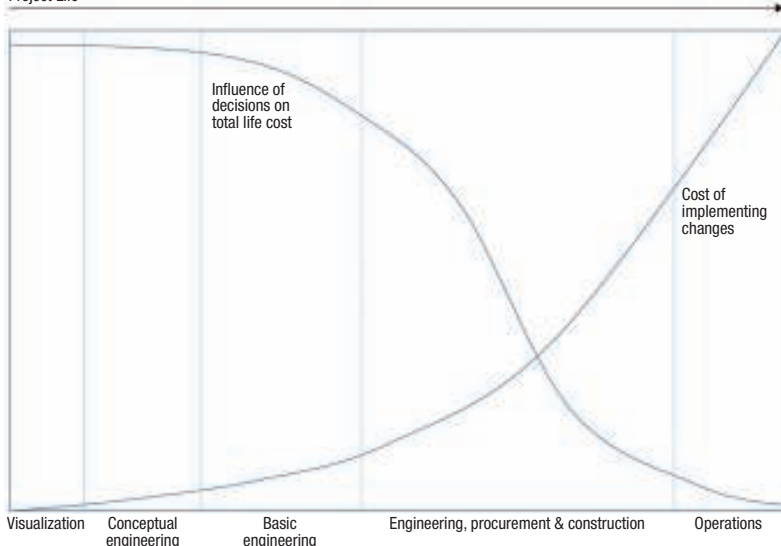
In each of the plant life phases, different choices are made by engineers that have a direct impact on the overall risks in the facility; however, the highest opportunities for cost-effective risk reduction are present in the earlier phases of the project. In contrast, the cost of implementing changes in the later stages of the project increases dramatically. Hence, it is important for the design team to identify risks, and implement effective design solutions as early as possible.

This article covers some of the typical decisions that the project design team has to make over the course of a project, with examples of how the incorporation of process safety throughout the entire design process can significantly reduce the risk introduced by a new CPI facility, while also avoiding potential cost-overruns, or unacceptable risk scenarios at later stages.

### CPI project lifecycle

A project for a new chemical process facility usually involves different phases, which are outlined here:

**A screening or visualization phase.** In this phase, the business need for the plant is assessed. Typical choices at this stage involve defining plant throughput, processing technology, main blocks and plant location



**FIGURE 1.** The relative influence of decisions on total life cost, and cost of implementing changes throughout the project lifecycle

earlier in the project lifecycle have the greatest impact on the total plant life cost; in contrast, the cost of implementing changes in the later stages of the project increases dramatically, as can be seen on Figure 1.

The same holds true for overall plant risk, as the impact of decisions on overall facility risk is greatest in the earliest stages of the project.

### Risks and hazards

A risk can be defined by a hazard, its likelihood (or probability) of occurrence, and the magnitude of its consequence (or impact).

A hazard, as defined by the Center for Chemical Process Safety (CCPS), is “an inherent chemical or physical characteristic that has the potential for causing damage to people, property or the environment” [2].

Process hazards can be classified in terms of the following:

1. Their dependence on design choices:

- Intrinsic — not dependent on design decisions (that is, always associated with the operation or process). For instance, hazards associated with the chemistry of the materials being handled (flam-

(high-level), with the goal of developing a high-level project profile, and a preliminary business case based on “ball-park” estimates, benchmarks and typical performance ranges, in order to identify project prospects.

**A conceptual engineering phase.** In this phase, the design team further develops the concept of the plant, leading to a more-defined project description, an improved capital-cost estimate, and a more-developed business model. At this stage, the process scheme is defined, along with the characteristics of the major pieces of equipment and their location on the layout (which would ideally be set over a selected terrain). The needs for raw materials, intermediate and final product inventories, as well as utility requirements are also established.

**A basic engineering, or front end engineering design (FEED) phase.** This sets the basis for the future engineering, procurement and construction (EPC) phase, by generating a scope of work that further develops the process engineering, and includes the early mechanical, electrical, instrumentation and civil/structural documents and drawings. This phase also serves to generate a budget for the construction.

**An EPC phase.** The EPC phase also includes the detailed engineering for the development of the “for construction” engineering deliverables, the procurement of equipment and

bulk materials, the execution of the construction work, the pre-commissioning, commissioning and startup of the facilities.

Table 1 shows typical engineering deliverables, along with their degree of completion, for each phase of project development.

After the plant construction is finished, the facility enters the operations phase. At the end of its life, the plant is decommissioned.

It is a generally accepted fact in project management that decisions made

**TABLE 1. TYPICAL ENGINEERING DELIVERABLES AND STATUS PER PROJECT PHASE**

Deliverable	V	CE	BE	DE
Project scope, design basis and criteria	S	P	C	C
Soil studies, topography, site preparation		S	C	C
Construction bid packages			P	C
Process block diagrams	S/P	P/C	C	C
Plot plan		S	P/C	C
Process and utility flow diagrams (PFDs / UFDs)		S/P	P/C	C
P&IDs		S	P/C	C
Material & energy, utility balances		S	P/C	C
Equipment list		S/P	P/C	C
Single line diagrams		S/P	P/C	C
Data sheets, specifications, requisitions		S	P/C	C
Mechanical equipment design drawings and documents		S	P/C	C
Piping design drawings and documents			S/P	C
Electrical design drawings and documents			S/P	C
Automation and control drawings and documents			S/P	C
Civil / structural / architectural design drawings and documents			S/P	C
Cost estimate	C5	C4	C3	C2/C1

Key: V = visualization; CE = conceptual engineering; BE = basic engineering; DE = detailed engineering; S = started; P = preliminary; C = completed; C5, C4, ..., C1 = Class 5, Class 4, ..., Class 1 cost estimate (AACE)

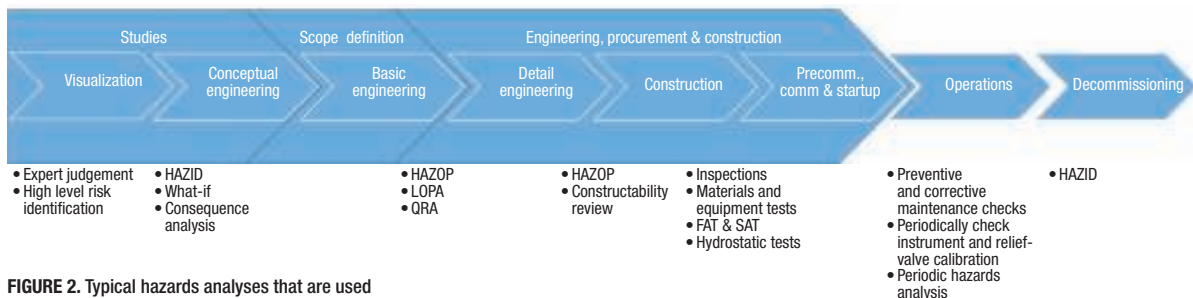


FIGURE 2. Typical hazards analyses that are used throughout a CPI project lifecycle

mability, toxicity, reactivity and so on); these properties cannot be separated from the chemicals

- Extrinsic — dependent on design decisions. As an example: hazards associated with heating flammable materials with direct burners can be avoided by using indirect heating

2. Their source:

- Process chemistry — associated with the chemical nature of the materials (for example, flammability, toxicity, reactivity and so on)
- Process variables — associated

with the operating conditions (pressure, temperature), and material inventories. As general rules:

- o higher pressures increase the impact of potential releases, whereas vacuum pressures increase the probability of air entering the system
- o higher temperatures increase the energy of the system (and hazards, especially when near the flashpoint or self-ignition temperature), whereas very low temperatures could pose the risks of freezing, formation of hydrates, or

material embrittlement

- o higher material inventories increase the impact of potential releases, whereas lower material inventories reduce response times in abnormal operating conditions
- Equipment failures — associated with damages to plant equipment
- Utility failures — associated with failures in utilities supplied to the facility, such as electricity, cooling water, compressed air, steam, fuel or others
- Human activity — associated with activities by humans over the facil-

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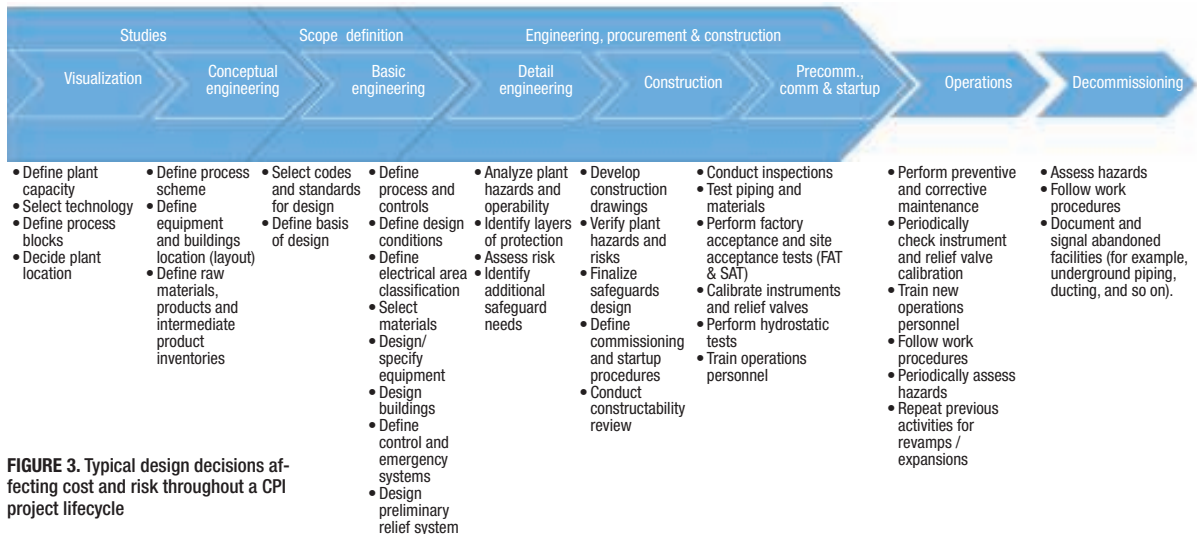
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**FIGURE 3.** Typical design decisions affecting cost and risk throughout a CPI project lifecycle

ity (for example, operator error, tampering with facilities, security threats and so on)

- Environmental — associated with environmental conditions (for example, earthquakes, hurricanes, freezing, sandstorms and so on)

The likelihood of a risk can be expressed in terms of an expected fre-

quency or probability of occurrence. This likelihood can be either relative (low, medium, high), or quantitative (for instance, 1 in 10,000 years). Quantitative values of the likelihood of different categories of risk, or equipment failures, as well as risk tolerability criteria, can be obtained from literature sources, such as Offshore and

Onshore Reliability Data (OREDA), American Institute of Chemical Engineers (AIChE), Center for Chemical Process Safety (CCPS), American Petroleum Institute (API), U.K. Health and Safety Executive (HSE), Netherlands Committee for the Prevention of Disasters by Dangerous Materials (CPR), or local government agencies,

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**TABLE 2. EXAMPLES OF CHANGES IN DESIGN AS RESULT OF PROCESS HAZARDS ANALYSES IN DIFFERENT PROJECT PHASES**

Impact on	Project description	Conceptual engineering	Basic engineering	Detailed engineering
Process definition	Industrial solvents manufacturing facility	1. Preliminary process design and equipment characteristics were defined based on process simulations and best engineering practices.	2. Tower diameter, reboiler, condenser, and pump capacities changed, spare equipment, alternate lines and valves added following <i>What-if</i> analysis.	3. Line routings changed after <i>constructability review</i> , adding pressure drop, which altered pumps and control valves.
Plant layout / area	High-pressure gas plant	1. Preliminary plot plan was arranged based on available terrain and recommended equipment spacing. 2. After <i>consequence analysis</i> , plant area was increased by 50% and equipment and buildings were relocated to prevent impact areas from reaching occupied buildings and public spaces.	3. <i>Relief systems design</i> required further modifications to plot plan, and an additional 10% of space for flare exclusion area. 4. After <i>QRA</i> , proper safeguards were selected in order to reduce risk contours to tolerable levels in occupied buildings and public spaces, hence reducing space requirement by 25% versus that required by <i>consequence analysis</i> .	5. Location of some lines and equipment was slightly changed as result of <i>constructability review</i> , to allow early operations in parallel with construction.
Automation and controls	Crude-oil central processing facilities	1. Only summary description of major control system items developed in conceptual engineering.	2. Control system designed according to P&IDs. 3. Approximately 30% more instruments and control loops added as result of <i>HAZOP</i> . 4. The overall system was increased from SIL-1 to SIL-2 after <i>LOPA</i> , as result of one section of the plant handling light ends.	5. Some additional modifications were required after reception of vendor information.

and they can be especially valuable when performing quantitative, or semi-quantitative studies.

The consequence of a risk can be expressed in terms of its impact on several recipients, such as assets, personnel, society and environment.

The combination of likelihood and consequence defines the risk. The risk is then analyzed versus tolerability criteria, either qualitatively (for example, in a risk matrix), or quantitatively (for example, in risk contours). Company management and the design team may then select measures to eliminate or reduce individual risks, if they are not in the tolerable range.

### Process hazards identification

An experienced engineering design team, with proper design basis documentation, and working under approved industry standards and best engineering practices, is the first

factor in ensuring that plant hazards can be avoided or reduced as early as possible in the design.

Aside from the experience of the team, it is generally accepted that different methodical approaches can be applied in a timely manner to the engineering design process, in order to detect possible hazards that were not addressed by the design team. These structured reviews are called process hazards analyses (PHAs), and are often conducted or moderated by a specialist, with participation of the design team, owner's employees or experienced operators.

Several methodologies exist for conducting a PHA, each suitable for specific purposes, processes, and for certain phases of project development and plant lifecycle (Figure 2). Below is a brief description of some of the most used PHAs in the CPI.

**Consequence analysis.** This is a

method to quantitatively assess the consequences of hazardous material releases. Release rates are calculated for the worst case and alternative scenarios, end toxic points are defined, and release duration is determined.

**Hazard identification analysis (HAZID).** HAZID is a preliminary study that is performed in early project stages when hazard material, process information, flow diagram and plant location are known. It's generally used later on to perform other hazard studies and to design the preliminary piping and instrumentation diagrams (P&IDs).

**What-if.** This is a brainstorming method that uses questions starting with "What if...", such as "What if the pump stops running?" or "What if the operator opens or closes a certain valve?" It has to be held by experienced staff to be able to foresee possible failures and identify design alternatives to avoid them.

**Hazard and operability study (HAZOP).** This technique has been a standard since the 1960s in the chemical, petroleum and gas Industries. It is based on the assumption that there will be no hazard if the plant is operated within the design parameters, and analyzes deviations of the design variables that might lead to undesirable consequences for people, equipment, environment, plant operations or company image. If a deviation is plausible, its consequences and probability of occurrence are then studied by the HAZOP team. Usually an external company is hired to interact with the operator company and the engineering company to perform this study. There are at least two methods using matrices to evaluate the risk (*R*): one evaluates consequence level (*C*) times frequency (*F*) of occurrence; and the other incorporates exposition (*E*) as a time value and probability (*P*) ranging from practically impossible to almost sure to happen, in this method, the risk is found by Equation (1):

$$R = E \times P \times C \quad (1)$$

**Layer-of-protection analysis (LOPA).** This method analyzes the probability of failure of independent

protection layers (IPLs) in the event of a scenario previously studied in a quantitative hazard evaluation like HAZOP. It is used when a plant uses instrumentation independent from operation, safety instrumented systems (SIS) to assure a certain safety integrity level (SIL). The study uses a fault tree to study the probability of failure on demand (PDF) and assigns a required SIL to a specific instrumentation node. For example in petroleum refineries, most companies will maintain a SIL equal to or less than 2 (average probability of failure on demand  $\geq 10^{-3}$  to  $< 10^{-2}$ ), and a nuclear plant will tolerate a SIL 4 (average probability of failure on demand  $\geq 10^{-5}$  to  $< 10^{-4}$ ).

**Fault-tree analyses.** Fault-tree analysis is a deductive technique that uses Boolean logic symbols (that is, AND or OR gates) to break down the causes of a top event into basic equipment failures or human errors. The immediate causes of the top event are called "fault causes." The resulting fault-tree model displays the logical relationship between the basic events and the selected top event.

**Quantitative risk assessment (QRA).** QRA is the systematic development of numerical estimates of the expected frequency and consequence of potential accidents based on engineering evaluation and mathematical techniques. The numerical estimates can vary from simple values of probability or frequency of an event occurring based on relevant historical data of the industry or other available data, to very detailed frequency modeling techniques [4]. The events studied are the release of a hazardous or toxic material, explosions or boiling liquid expanded vapor explosion (BLEVE). The results of this study are usually shown on top of the plot plan.

**Failure mode and effects analysis (FMEA).** This method evaluates the ways in which equipment fails and the system's response to the failure. The focus of the FMEA is on single equipment failures and system failures.

### When to use a given method

Some studies have more impact in some phases than in others. For

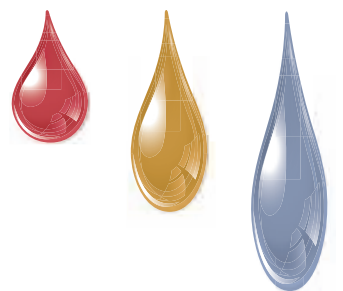
example, if a consequence analysis is not performed in a conceptual or pre-FEED phase, important plot plan considerations can be missed, such as the need to own more land to avoid effects over public spaces; or the fact that the location might have a different height with respect to sea level than surrounding public places impacted by a flare plume.

Some other studies, like HAZOP, cannot be developed without a control philosophy or P&IDs, and are performed at the end of the FEED or detailed engineering (for best results, at the end of both) to define and validate pressure safety valves (PSVs) location and other process controls and instrument safety requirements. QRA or LOPA (or both) are done after HAZOP to validate siting and define safety instrumented systems SIL levels, and finally meet the level required by the plant.

Figure 2 shows the typical CPI project phases, with a general indication of when it is recommended to conduct each study; however, this may vary depending on the specific industry, corporate practices, project scope and execution strategy. AIChE's CCPS [2] has an Applicable PHA technique table that indicates which study to perform in each project phase, which also includes research and development (R&D), pilot plant operations, and other phases not covered in the present article.

Table 2 includes some real-life examples of how the results of some of these studies can impact the development of the plant design at different project phases.

Out of the previously mentioned studies, a properly timed HAZOP, at the end of the basic engineering phase, is key to identifying safety and operability issues that have been overlooked by the engineering design team, especially when involving an experienced facilitator and plant operators in the study, given that they have a fresh, outsiders' view of the project, and they can provide input on daily operating experience. Also, the deviations identified in the HAZOP can serve to detect the need for additional safeguards that were



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**TABLE 3. ADDITIONAL COSTS OF CHANGES ASSOCIATED WITH HAZOP RECOMMENDATIONS DURING EPC PHASE**

Sample project number	Project description	Estimated cost of changes associated with HAZOP recommendations in EPC phase (as % of approved budget)	PHAs and proper safe-design practices implemented in previous design phases?
1	Gas dehydration unit	3%	Yes
2	Gas compression unit	3%	Yes
3	Crude oil atmospheric unit	1%	Yes
4	Fuel storage tank farm	2%	Yes
5	Petrochemical plant relief and flare systems	2%	Yes
6	Crude oil dehydration station	1%	Yes
7	Crude oil evaluation facilities	1%	Yes
8	Heavy crude oil dehydration unit	3%	Yes
9	Propane/air injection plant	1%	Yes
10	Oil pipeline + two gas compression units	1%	Yes
11	New flare system in existing refinery	1%	Yes
12	Refinery gas concentration unit revamp	7%	No
13	Extra-heavy oil deasfaltering unit	5%	No
14	Demineralized water plant	13%	No
15	Hydrogen compression unit	35%	No

not considered by the design team. When the recommendations are implemented correctly, and no other changes to the process or plant are done between the preparation of the basic engineering design book and the EPC phase, then a HAZOP significantly reduces the probability of significant cost impacts in the latter as a result of changes due to additional PHAs.

Even though “what-if,” HAZID and consequence analyses have impact on the capital cost of the project, the cost of implementing their modifications to the design are typically included on the EPC bidding process, as they are realized at the beginning of the project lifecycle. Fault-tree analysis and LOPA are used to define the redundancy level of controls and instrumentation. The changes derived from these studies generally represent a minor portion of the total capital expenditure. That leaves HAZOP and QRA as the most important studies

to identify design improvements to prevent process hazards in the latter project phases.

### Safe-design options

At the early project phases, it is not possible to identify all possible risk-reduction measures that could be included in the design. However, a safety-oriented design team might be able to pinpoint sources of project risk due to lack of data, and opportunities for risk reduction that could be evaluated in later stages, as the design progresses and further details are known.

Some large organizations have collected the pool of their experiences within risk checklists and proprietary design standards, thus paving the way for future work. Where organizations have not established their own standards and engineering practices, the design team should look for accepted codes and standards that are the result of best engineering practices in a particular field or industry.

The design options include, in descending order of reliability: inherently safer design, engineering controls (passive and active) and administrative controls (procedural).

*Inherently safer design* involves avoiding or reducing the likelihood of a hazard in a permanent or inseparable fashion. For example, when designing a centrifugal pump discharge system, an inherently safer design would be to specify the design pressure at the centrifugal pump shut-off pressure, thereby largely reducing the risk that an increase in the pump discharge pressure (for example, due to a blocked outlet) could cause a rupture in the pipes with consequent loss of containment.

*Engineering controls* are features incorporated into the design that reduce the impact of a hazard without requiring human intervention. These can be classified as either passive (not requiring sensing and or active response to a process variable) or active (responding to variations in process conditions). In the previous centrifugal pump example, a passive solution would be to contain possible leaks within dikes, and with adequate drainage. Examples of active solutions could be: a) providing a high-pressure switch associated with an interlock that shuts the pump down; and b) providing a pressure safety valve (PSV) designed for blocked outlet.

*Administrative controls* require human intervention. These are the least reliable, because they depend on proper operator training and response. In the previous example, an administrative control would be to require operators to verify that the valves in the pump discharge lines are open.

Throughout the engineering phases leading to the EPC phase, different safe-design choices can be made, as further information is made available. Figure 3 shows some of the typical design choices made by the engineering team throughout a chemical process plant lifecycle, which have direct impact on lifecycle cost and risk.

In the visualization phase, safety can be included in the analysis as a

factor to decide key items, such as production technology and plant location. These key items are typically selected based on other technical criteria, such as overall efficiency, production cost, or vicinity to either raw materials, or markets (or export facilities). For instance, when selecting a technology, health, safety and environmental concerns could be included as a criteria on the evaluation matrix, by adding positive points to technologies that reduce risks to their environment by using less-toxic materials, operating at lower pressures or temperatures, or yielding non-toxic byproducts. When selecting a high-level plant location, management could opt to locate the plant away from large population centers, in order to minimize risks to communities. In this case, planning authorities also have an important role in defining allowable land-uses.

In the conceptual engineering phase, safety can be included in the analysis, for example, in the following ways:

1. Defining a simple, yet functional process scheme, as relatively simple processes have less equipment and consequently lower failure probability (this can conflict with other design goals); also, the types of equipment selected can have an important effect on process safety (for example, selecting indirect over direct heating).
2. Including safety concerns in the early layout definition. For instance, a design by blocks — keeping the main process, storage, and utility areas separate from each other — can reduce overall risk. Other good practices include: maintaining an adequate separation between pieces of equipment; separating product inventories taking into account their flammability, toxicity or reactivity, and considering dikes around tanks containing dangerous materials; placing flares and vents in locations separate from human traffic, taking into account wind direction (for example, so that flames or plumes are directed farther from personnel or population); and allowing sufficient plot space for an adequate exclusion area.
3. Keeping flammable and toxic ma-

terial inventories to the minimum required to maintain adequate surge/storage capacity and flexibility in shipping.

In the basic engineering or FEED phase, many design choices are made over the specific mechanical, piping, electrical, automation and civil design that impact on the overall facility risk. The first decision involves selecting the codes and standards that will be used for design, and defining the design basis and criteria for each engineering discipline. Then, throughout the design, some other decisions may include: selecting between automated and manual operation, setting equipment and piping design conditions, defining the electrical area classification, designing or specifying equipment, structures and buildings, defining control and emergency systems (including appropriate redundancy, where applicable), and designing appropriate relief systems, among others. Then, there are equipment and system-specific hazards and available safeguards that need to be considered. Ref. 2 contains a comprehensive list of hazards and safeguards for various types of unit operations.

When hazards have been properly identified and addressed in the earlier design phases, this reduces the probability of significant costly changes being made during the EPC phase as a result of unsafe process conditions.

### Addressing hazards early

When hazards are identified, and proper design choices are taken early in the engineering design to address them, significant benefits can be obtained.

Table 3 compares the additional cost of changes arising from recommendations made during a HAZOP at the EPC phase. The costs are expressed as a percentage of the budget that was approved during the bidding stage, of projects of different scope and plant type, executed by different companies in different countries, including the U.S. and Latin America, with approved budgets between \$5 million

and \$200 million.

The projects are divided into two categories: a) projects where the design contractor applied best engineering standards and employed PHAs at optimum points during the conceptual engineering and FEED phases; and b) projects where adequate PHAs and safe-design practices were not applied in the previous design phases.

As can be seen in Table 3, there is a significant difference between the cost of the changes arising from HAZOP recommendations when proper safe-design practices and PHAs were applied during the FEED phase, and when they were not.

For the first category, changes were typically in the range of 1 to 3%. In the upper end of this category, changes were higher when the owner requested some minor modifications to the FEED design without properly assessing the risks associated with said changes.

As an example, the heavy crude oil dehydration unit (Project 8) was designed according to best engineering practices, and adequate analyses (HAZOP, LOPA) were conducted during the engineering phase. However, the owner decided to implement changes in the design in order to compress the schedule, by removing several long-lead items that included emergency shutdown system (ESD) valves and components, without updating the PHAs. With the unit in operation, the owner asked the contractor to include the ESD items that were in the original design.

For the second category, changes exceeded 5%, and in one case reached as high as 35% of the approved budget. Below is a description of what went wrong in each of these projects:

The refinery gas concentration unit revamp (Project 12) FEED considered hand operations in key pieces of equipment. As a result of a HAZOP during the EPC, the operations had to be automated, which changed the equipment specifications and design. The number of loops added after the HAZOP exceeded the capacity of the controller, and another

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one had to be installed.

The extra-heavy oil deasphalting unit (Project 13) was designed during the basic engineering phase as a mostly hand-operated facility, with minimum supervisory controls. As a result of a HAZOP during the EPC, the risk was not tolerable to the owner, and the whole unit had to be automated.

The demineralized water plant (Project 14) was delivered by the vendor as a package unit, and no PHAs were conducted by the vendor. When received, the plant had many safety and operability issues and a number of important modifications had to be made, including: additional lines, block and control valves, relief valves and associated lines, among others. Aside from the costs associated with the changes, the project was delayed by six months.

The hydrogen compression unit (Project 15) basic engineering design did not address all of the safety considerations associated with hydrogen handling. Some of the modifications recommended by the HAZOP/LOPA studies during the EPC phase included changing the compressor specification, and increasing the SIL of the SIS from SIL-1 to SIL-3.

### Final remarks

Hazards are present in the CPI; some are avoidable, while others cannot be separated from the plant, as they are tied to the very nature of the chemicals or the unit operations, or both. However, a proper design team, one that is trained to identify hazards, and address them using the best engineering practices in safe-design from early on in the project lifecycle, along with properly timed and executed PHAs, can be very valuable in avoiding costly changes during the EPC phase, or even worse: potential damages to persons and the environment. ■

*Edited by Gerald Ondrey*

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## A Pragmatic Approach to Powder Processing

Modern powder-testing methods can offer valuable insights into bulk material behavior and lead to efficient powder-processing solutions

**Jamie Clayton**  
Freeman Technology

Process simulation is a powerful tool for engineers in the chemical process industries (CPI) and one that plays an important role in commercializing new manufacturing routes and supporting process optimization. While it might be costly to construct a robust process model, once such a model is in place, it will deliver value throughout the lifetime of the process. For powder processors, however, comprehensive simulation is not yet an industrially practical option, largely because the mathematical modelling of powder behavior cannot yet be reliably achieved. Establishing a successful manufacturing solution by applying experimentation and process simulation in combination is therefore not viable, so alternative methods must be considered.

Traditionally, companies that handle and manufacture powders have relied heavily on processing experience, supported by basic forms of powder testing. However, this approach is more difficult to sustain within the lean-manufacturing environments that now prevail across all industrial sectors. Developing a knowledge base to extend powder processing expertise is seen as increasingly important, whether in the development of continuous processes for pharmaceutical products or in advancing the application of additive manufacturing (AM).

This article provides a review of the challenges associated with powder processing and characterization, and demonstrates how modern powder-testing methods provide increased insight to support efficient process development and optimization. An example case, describing work carried out by a leading global powder processor, illustrates the potential



**FIGURE 1.** Powders are bulk assemblies consisting of solids, liquids and gases. Interactions between all three of these phases determine the bulk powder behavior

benefits of adopting a pragmatic approach to powder handling that is based on measuring and applying powder properties that are directly relevant to a specific process.

### Powder behavior

Powders are ubiquitous throughout the CPI. As raw materials and intermediates, they are used in the production of goods, ranging from processed foods and pharmaceuticals to paints, coatings and metal components for automotive and aerospace applications. Learning how to control powder behavior to meet advancing manufacturing requirements is critical, especially as markets become more competitive, and as the use of new production techniques, such as additive manufacturing, grows.

Powders are bulk assemblies (Figure 1) comprising solids, in the form of particles, gases (normally in the form of air), and liquid, (usually present in the form of moisture on the surface of the particle or within its structure). A network of complex interactions between these constituent elements dictates the bulk properties of powders, such as flowability, compressibility, permeability and the ability to aerate, which give powders their industrial versatility.

The behavior of a powder is a function of the physical properties of the particles, as well as other external variables. For example, physical properties include particle size, mor-

phology and surface texture. Variables associated with the bulk assembly include the extent of air and the level of moisture content in the bulk. The complex, numerous and varied interactions complicate efforts toward process modeling and also make the measurement and control of a bulk powder challenging.

### Modeling and powder testing

Considering modeling first, there have been many attempts to mathematically model powder behavior, and this area remains one of continuing study, particularly as computing power increases. The starting point for the mathematical modeling of powders tends to involve uniformly sized, spherical particles — typically microscopic glass beads — and interesting, important progress has been made. However, powder modeling is still a predominantly academic activity and not yet a practical tool for industrial processors. Progressing from the modeling of “ideal” particles, to a situation that more accurately represents the types of materials in routine industrial use, increases the complexity of the modeling significantly. The reality is that all of the particle properties and environmental conditions that influence powder behavior cannot yet be reliably replicated and there is still a lack of robust correlations between the physical properties of solid particles and bulk powder behavior.

These acknowledged limitations

place the focus on powder characterization and invite the question of how to best use it in process-related investigations. As discussed, many variables have an impact on powder behavior, making robust characterization considerably more challenging than for a liquid or gas. For example, if a powder sample absorbs even a small quantity of moisture from its environment, then a repeat measurement of its flowability may be compromised. There are many aspects of powder behavior to investigate, and gathering reliable data requires close control of both the analytical methodology and the powder sample.

When considering the behavior of powder in a given process, it is necessary to identify robust, reproducible powder-testing methods capable of delivering data that directly correlate with in-process performance. The various unit operations in a given process subject powders to very different stress and flow regimes. These vary from unconstrained, highly aerated flow in a fluidized bed or pneumatic conveyor, to high-pressure compaction in a tablet press. Optimizing such processes relies on capturing relevant information that defines performance in each specific unit operation.

### The multivariate approach

There are many methods available for measuring the properties of powders, and in particular the flowability of powders, which is a defining characteristic for many industrial applications. Simple flowability methods include flow-through-orifice, angle-of-repose and tapped-density techniques, such as Carr's Compressibility Index and the Hausner Ratio. While these may provide some value in classifying powders, their ability to directly simulate the stresses and flow regimes experienced by a powder during processing is limited. This, and other limitations, restricts their use as tools for reliably predicting process performance.

More sophisticated testing techniques include shear-cell analysis and dynamic testing. Shear testing (Figure 2) involves measuring the forces required to shear one consolidated powder plane relative to another. Tests are conducted using

consolidated powders and help to quantify how easily a powder transitions from a static to dynamic state following exposure to moderate- to high-stress conditions (consolidation). A good example of the application of shear testing is in the design of hoppers, where they bring considerable value. However, shear-cell analysis is much less relevant when predicting how powders will behave in low-stress dynamic environments, for example, when loosely packed or aerated.

Dynamic testing generates properties, such as basic flowability energy (BFE) and specific energy (SE), that directly quantify how a powder flows under different conditions. These properties are generated by measuring the rotational and axial forces acting on a blade, or impeller, as it is rotated through a powder sample along a fixed helical path. BFE is measured using a downward traverse of the blade, which exerts a "bull-dozing" action that compresses the powder against the confining base of the test vessel. The resulting data are indicative of flow behavior in a low-stress, confined environment. In contrast, SE is measured during an upward traverse of the blade, and quantifies the flow properties of a powder in low-stress, but unconfined, conditions. Uniquely, dynamic testing offers the ability to evaluate powders in consolidated, moderate stress, aerated and even fluidized states to investigate how a powder responds to air — a critical aspect of behavior in many applications.

The above discussion underlines the value of adopting a multifaceted approach to powder testing, because no single powder property is relevant to every process. Powder



**FIGURE 2.** Shear-cell testing is a valuable technique for hopper design and, more generally, for assessing the performance of powders under conditions of moderate to high stress

testers based on just one technique are unlikely to provide the information required to define critical operational parameters, whereas a device that provides multiple methods is able to quantify a wider range of process-relevant properties (Figure 3). For every powder, a unique combination of these properties will determine a powder's in-process performance.

### Property-behavior correlations

A multifaceted testing strategy should provide information on a range of properties in order to capture all characteristics of a given powder. However, this approach invites the question of how to identify the most valuable variables for predicting performance in any given powder application. This is essential for developing a streamlined testing regime that will efficiently elucidate processing behavior and enable successful control. To identify these key variables, powder processors must draw on operational observations and results, and correlate them with powder-characterization data.



Operational experience often resides within a company in a subjective form and, as a result, it can be difficult to apply. Correlating such experience with reproducible powder properties converts it into more generally applicable knowledge (Figure 4).

### Solid blends example

The following example offers an illustration of this approach. A powder is selected for use in additive manufacturing (AM), on the basis of trial and error, due to its ability to process well in a specific machine and to produce high-integrity products with the desired finish. To reduce waste, and associated costs, the decision is made to recycle residual powder from the process, mixing it with fresh feed before processing. Experimental data show that certain fresh-recycled blends work well while others do not.

There are two approaches to establishing some operational guidelines for successful powder re-use. One is to conduct a series of experiments, using mixtures with different fresh-to-recycled ratios, check the performance of each on the AM machine and define an operating ratio of fresh-to-recycled materials that falls somewhere within the pass/fail boundaries. The alternative is to correlate the results from trials using mixtures that have different fresh-to-recycled ratios with a database of powder properties gathered for each mixture. The output from this study might reveal that performance of a mixture within the AM machine correlates directly with, for example, BFE, permeability and shear strength. This makes it possible to define a clear and detailed specification for a powder that is suitable for processing in this machine.

Both approaches may initially be successful, but the benefit of the second is that there is now a robust specification to identify powders that are compatible with the process so, if the feed is changed again, if a new powder is used, or if waste powder is recycled not once but twice, the impact can be assessed with confidence simply by testing the powder. In contrast, the former approach provides no generally applicable guidance with which to assess a new powder, and therefore cannot

support extrapolation beyond the tested experimental window.

In this way, rigorous, relevant and robust powder testing can engender a systematic approach to powder processing that moves beyond the empirical, experience-based practice that is traditionally employed. Ultimately, such a strategy pays dividends in the development of new formulations, the advancement of process design, the day-to-day operation of the plant and in-process troubleshooting. The following case study demonstrates the potential for this type of approach.

### Pragmatic approach in action

AZO GmbH + Co. KG (Osterburken, Germany; [www.azo.com](http://www.azo.com)) is a specialist supplier of bulk-material handling equipment for a range of industries. The company serves a wide variety of customers across the food, pharmaceutical, bulk chemical and polymer sectors. Clients rely on AZO to specify turnkey solutions for powders and granules that include: silos and hoppers for storage; filling stations for either intermediate bulk containers (IBCs) or sacks; dosing and weighing systems; and pneumatic-conveying plants. The company operates a full-scale test facility that can conduct trials and optimize handling solutions, but these trials are relatively costly to run. With over 500 new materials assessed each year, there is a considerable economic incentive to streamline process design and optimization for each new powder.

For a number of years, AZO relied on two powder-measurement systems to support process development and optimization: a shear cell and a basic powder tester that measured angle-of-repose and Carr's Index. However, these methods were not ideal in terms of efficiency or applicability. In particular, neither provided detailed information on fluidization behavior, which is highly relevant to certain unit operations. Furthermore, shear-cell analysis, although valuable for silo and hopper design, was both time-consuming and highly operator-dependent (relying heavily on the skills of a single experienced technician).

In 2010, AZO made the decision to invest in new powder-testing in-

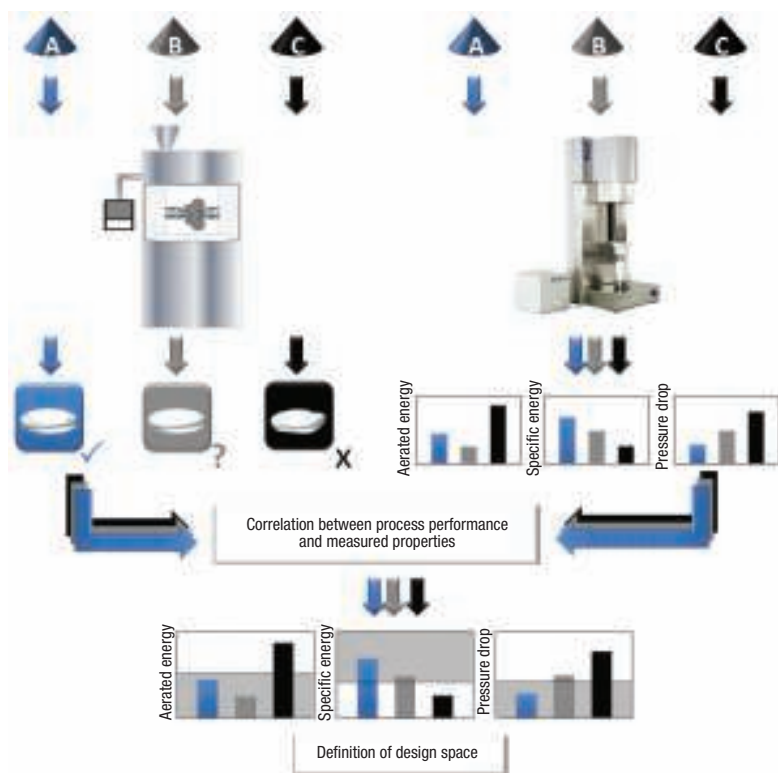


**FIGURE 3.** Powder testers that offer multifaceted powder characterization, including dynamic testing, have a proven track record of supplying valuable information for process-related studies

struments to overcome these limitations and secure more relevant information on powder characteristics. This decision was strongly influenced by the instruments' ability to provide both automated shear testing and dynamic powder testing. Today, AZO's approach to process design and optimization is far more efficient than before, in a number of key areas.

**Storage.** With automated shear analysis in place, together with integrated software that applies Jenike's well-established hopper-design algorithms, the AZO team now has tools that enable them to design optimal storage solutions far more easily and effectively than they could when using the old shear cell. Measurement times for shear properties have been reduced by a factor of four and more people can confidently perform the analysis. Wall-friction testing, which quantifies the strength of the interactions between a construction material and a powder, is now carried out routinely to establish the best material of construction or coating for any given powder.

Jenike's protocols are designed to generate hopper dimensions that support mass flow, but when powders are extremely cohesive, this cannot always be achieved. In these instances, the Jenike methods may fail. For example, they may generate values for outlet size that are wider than the diameter



**FIGURE 4.** Correlating measurable powder properties with processing performance enables the definition of the “design space” — the identification of the powder characteristics and their values that ensure acceptable process behavior

of the silo. For these powders, efficient hopper discharge depends on the use of mechanical aids, such as vibrating devices or air-injection systems. The new testing regime rapidly and robustly identifies powders that fall into this classification, thereby helping to define an optimal storage solution.

A further issue in powder storage is the potential impact of consolidation or “caking” over time. The compressing load on a powder stored under its own weight can have a significant impact on its flowability. Using dynamic powder testing, researchers at AZO have directly investigated this aspect of behavior and used the results to guide operational practice. For example, testing helped determine the frequency with which a silo must be emptied.

**Powder discharge.** During discharge from a storage vessel, there is potential for a powder to draw in air and become fluidized, to the point of uncontrollable flooding from the hopper. Rotary valves are an option for controlling powders that readily flood

in this way, while doping screws are an alternative for more challenging materials. Deciding on which system to adopt is critical.

The AZO team has assessed whether the ability of the powder to fluidize in this way. Aeration data have certainly proven to correlate with discharge characteristics, however, investigations have shown that other powder properties also influence this behavior, further supporting the need for a multifaceted test regime.

**Pneumatic conveying.** In pneumatic conveying, powders may be transported in a fluidized state — a state that can be studied directly using dynamic powder characterization. Through appropriate testing, it is possible to determine whether a material can be fluidized or not, and to measure the air velocity needed to reach fluidization. At AZO, such testing now supports

the optimization of operating parameters for fluidization.

When designing pneumatic-conveying systems, two parameters are especially important: throughput and pressure drop. Tests suggest that a number of dynamic powder properties and the bulk property of permeability are all important when determining these parameters. AZO researchers are establishing closer correlation between pneumatic-conveying performance and a number of powder properties, enabling them to strengthen design capabilities in this area.

### A dynamic industry

Developing, operating and optimizing powder processes introduces significant challenges, particularly in the absence of tools, such as accurate process simulation, that may now be taken for granted in other fields. However, powder-characterization techniques and technologies have advanced considerably in the last decade or so, supporting a move away from the inefficient “trial-and-error” approach to powder processing.

Experience suggests that by combining test methods — most productively dynamic, bulk and shear-property measurement — processors can characterize powders in ways that successfully elucidate, rationalize and control in-process behavior. By employing a pragmatic approach, based on the use of multiple powder properties and operational experience, powder processors can access new levels of efficiency and meet industrial requirements for more competitive, leaner processing. ■

*Edited by Scott Jenkins*

### Author



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# Solids processing



Access  
Intelligence

special advertising section

## Solids: from ingredients to products, across the CPI

*Solid materials feature in almost every process, whether as ingredients, intermediates or finished products, so knowing how best to handle them is important*

### Inside:

AERZEN  
Berndorf Band  
Brookfield Engineering Laboratories  
Flexicon  
Ross  
Jenike & Johanson  
Sandvik Process Systems

Powders, granules, pellets and pastilles are found almost everywhere across the chemical process industries (CPI). Frequently, raw materials are delivered in the form of bulk solids – diverse examples include metal ores, sodium hydroxide prills,



PHOTO: JENIKE & JOHANSEN

wheat flour, and citric acid powder. The product itself may be a powder, such as a laundry detergent or an active pharmaceutical ingredient, or a granular solid such as polymer chips. Even processes that are almost entirely liquids-based, such as petroleum refining, may use solid materials in the form of catalysts and water treatment chemicals, while yielding solid coke and sulfur as by-products.

All these bulk solids, with their widely differing mechanical characteristics, require specialized equipment to store and handle them. The design of bins and hoppers, for instance, is based on many decades of academic research, as well as centuries of practical experience. While hardly a glamorous topic, designing hoppers and feeders to maintain consistent flow is enormously important. Clogged discharge chutes and rat-holing in bins can cause embarrassment and, frequently, financial losses. Accurate measurement and interpretation of powder properties is vital here.

### Quality control typically depends on representative sampling

Similarly, there is a wide variety of equipment for conveying, metering, packaging and mixing granular materials. Belt conveyors, screw conveyors, and pneumatic conveying systems are just a few of the technologies used to move bulk solids around.

This special advertising section on solids processing includes input from companies offering a wide variety of equipment and services, from pastillation to powder testing. ■

## Making better pastilles from highly viscous products

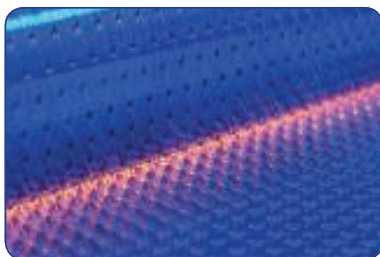
*Sandvik's ThermoCut system eliminates thread formation from melts*

Sandvik has introduced an innovative new hot-air-based cutting device that integrates into its Rotoform pastillation system to deliver perfectly formed pastilles from high-viscosity melts.

The ThermoCut system has been designed to overcome a problem known as “angel hair”. This refers to the formation of threads during the pastillation of hot-melt adhesives and resins, which can have viscosities as high as 40,000 mPas.

These threads or tails, created when the melt is deposited on the steel belt cooler by the Rotoform, can result in a less than perfect pastille shape, with more dust pollution and increased cleaning requirements. ThermoCut overcomes this to deliver a significantly better-quality product.

The system works by blasting a nar-



**ThermoCut system for high-viscosity melts – delivers perfectly formed pastilles**

row jet of high-pressure, heated air at the point where the droplets form between the Rotoform and the steel belt. This powerful blade of hot air cuts the threads before they have chance to develop further.

As well as delivering a more stable process with better-quality pastilles, ThermoCut also eliminates dust pollution and reduces cleaning and maintenance requirements.

ThermoCut has been designed to work with Sandvik's flagship Rotoform, a pastillation system widely used across the chemical industry. Rotoform consists of a heated cylindrical stator supplied with molten product, and a perforated rotating shell that turns concentrically around the stator, depositing drops of the product across the width of the steel belt. The heat from the pastilles is transferred to cooling water sprayed against the underside of the belt, resulting in quick, controlled solidification and a consistent product.

[www.processsystems.sandvik.com](http://www.processsystems.sandvik.com)

## The right compressor for each application

*AERZEN manufactures gas compressors and blowers for pneumatic conveying, among many other applications*

**A**ERZEN has supplied blowers and compressors to the process industries for over 60 years. Among the products it offers are positive-displacement blowers and screw compressors suitable for pneumatic conveying, as well as rotary-piston compressors, turbo blowers, and gas meters.

With a history dating back 150 years, AERZEN prides itself on the high quality and energy efficiency of its products – factors that in some cases allow the equipment to remain in service for more than 30 years. The company has ISO 9001 quality assurance. Each compressor or blower system is individually tailored to the customer, and there is a range of customizable service offerings too.

AERZEN GQ series oil-free rotary positive-displacement blowers adapt themselves automatically to changing operating conditions. Standard units are suitable for pressure differentials up to 1.2 bar, with intake flowrates in the range 15,000–100,000 m<sup>3</sup>/h. Special versions are also available as single-stage units for pressure differentials up to 1.5 bar, or up to 3.0 bar as two-stage units.

The VR series of oil-free screw compressors provide pressure differentials of 3.5–25 bar per stage, with maximum final pressures of 53 bar in multi-stage versions. Outside of pneumatic conveying, these units have proven themselves in process gas service. By adding water injection, operators can use VR screw compressors for gases with fluctuating molecular weights, impu-



**Screw compressors in AERZEN's VR range offer oil-free delivery over a wide range of discharge pressures**

rities, or a tendency to polymerize.

AERZEN also offers oil-free vane compressors for pressure differentials up to 1,500 mbar and intake flowrates of 100–50,000 m<sup>3</sup>/h, plus the VMY series of oil-injected screw compressors. The latter's high reliability and low operating costs, plus a built-in control system offering turndown to 20% of full capacity, makes them an ideal choice for a wide range of applications in refrigeration and process gas handling.

[www.aerzen.com](http://www.aerzen.com)

## Powder density: what is the correct value?

*For powders that undergo large changes in bulk density as they consolidate, Brookfield Engineering Laboratories explains how accurate measurements can save headaches*



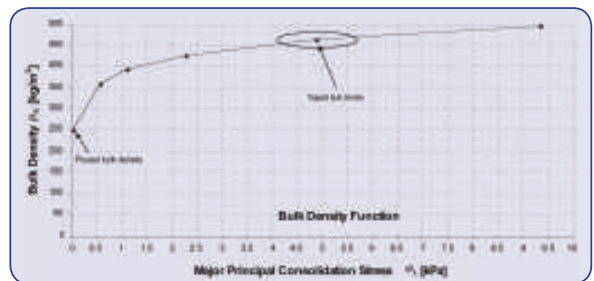
**P**ackaging specialists who work with powders know that density is a variable property, notes **Brookfield Engineering Laboratories**. “Loose-fill” density is the condition that exists when a powder is spread out on a flat surface. Consolidated powder, on the other hand, characterizes particles that are pushed together like a snowball into a compacted state. When powder is poured into bags, containers, or bins in a loose-fill condition, it will eventually consolidate over time due to the self-weight of the powder

pressing down on the particles beneath. The result is a more-dense material that may not be acceptable to the end user.

Manufacturers need to know how much a powder consolidates for several reasons. This phenomenon directly affects the flowability of the powder during processing. The final product when packaged may reduce in volume and leave significant unused space in the container. Powder can ultimately cake into a brick-like object, rendering it useless until it is broken up again.

The traditional tap test is a quick way to get an approximate idea for how much powder density can change. Use a cylinder with known volume, fill it with powder, tap it up and down 100 times, then measure the change in the fill level of the powder.

A more precise measurement is available through the use of a



**Shear cell instrument (photo, left) and powder density curve**

shear cell (photo, left), an instrument that accurately measures the change in volume as the sample transitions from a loose fill condition to a consolidated state. The shear cell applies a precise axial load to the powder. A compression plate pushes down on the sample, while a position sensor measures the change in volume.

Shear cells provide detailed graphs for density behavior (chart, above). Powders that consolidate by 50% or more can pose major challenges. With this information, manufacturers are better prepared to cope with processing issues and packaging requirements.

[www.brookfieldengineering.com/products/pft/powder-flow-tester.asp](http://www.brookfieldengineering.com/products/pft/powder-flow-tester.asp)

## Handle virtually any bulk solid material

*Flexicon stand-alone equipment and automated plant-wide systems convey, discharge, condition, fill, dump and weigh batch bulk materials dust-free*

**F**lexicon engineers and manufactures a broad range of equipment that handles virtually any bulk material, from large pellets to sub-micron powders, including free-flowing and non-free-flowing products that pack, cake, plug, smear, fluidize, or separate.

Individual bulk handling equipment includes: flexible screw conveyors, tubular cable conveyors, pneumatic conveying systems, bulk bag dischargers, bulk bag conditioners, bulk bag fillers, bag dump stations, drum/box/container dumpers, and weigh batching/blending systems. Each of these product groups encompasses a broad range of models that can be custom engineered for specialized applications, and integrated with new or existing upstream and downstream processes and storage vessels.

All equipment is available to food, dairy, pharmaceutical and industrial standards.

For large-scale bulk handling projects, Flexicon's separate Project Engineering Division provides dedicated Project Managers and engineering teams on four continents to handle projects from concept to completion. Working with each customer's preferred engineering firm or directly with their in-house team, Flexicon adheres strictly to the customer's unique standards, documentation requirements and timelines through a single point of contact, eliminating the risk of coordinating multiple suppliers.

Flexicon's worldwide testing facilities simulate full-size customer equipment and systems, verify performance prior to fabrication, demonstrate newly constructed equipment for visiting customers,



**Flexicon offers stand-alone bulk handling equipment as well as plant-wide systems integrated with new or existing processes**

and study the performance of new designs.

In 2015 the company doubled the size of its manufacturing facility and world headquarters in Bethlehem, PA, and also operates manufacturing facilities in Kent, United Kingdom; QLD, Australia; and Port Elizabeth, South Africa.

[www.flexicon.com](http://www.flexicon.com)

## Steel belts made from NICRO 85 stainless steel

*Berndorf Band is the first manufacturer to use NICRO 85 super-duplex for its endless steel belts, with the new material already proven in refinery service*

**B**erndorf Band GmbH, one of the world's leading vendors of steel belts for use in industrial processes, is the first producer to use NICRO 85 stainless steel grade for its endless steel belts.

NICRO 85 is a super-duplex material that offers high static and dynamic strength, with increased resistance to chloride-induced stress corrosion cracking, pitting and crevice corrosion. Such corrosion can occur especially in aggressive environments, for instance in sulfur cooling lines of refineries in hot and humid locations close to the sea. All applications which show an increased corrosion risk are potential applications for NICRO 85 steel belts.

The new material has already proven its quality in sulfur applications at a major refinery in Spain (photo).

NICRO 85 belts are available in standard dimensions of 1,200 mm by 1.0 mm and 1,500 mm by 1.0 mm; other widths are available on request. The new material fulfils all the other quality criteria for which steel belts from Berndorf Band are renowned: flatness, straightness, tracking and vee rope adhesion.

Berndorf Band, with headquarters in Berndorf, Austria, and a total of 250 employees, is a leading manufacturer of endless steel belts used in continuous industrial processes like pressing, drying, cooling, freezing, baking and transporting. The company is a member of the Berndorf AG Group and has an export rate of over 90%. The company is represented in all regions of the world for sales and service of steel belts. Subsidiaries exist in Austria, the U.S., China, Japan, South Korea and Colombia. Berndorf Band is highly involved



**A Berndorf Band steel belt made from corrosion-resistant NICRO 85 in use for sulfur recovery at a refinery in Spain**

in research and development, in cooperation with universities and other research institutes.

[www.berndorf-band.at](http://www.berndorf-band.at)  
[www.berndorfband-group.com](http://www.berndorfband-group.com)

## Ultra-high speed powder dispersion made simple

*Ross SLIM Technology employs high shear for rapid and complete mixing of powders into liquids, avoiding agglomerates and dust formation*

The **Ross** Solids/Liquid Injection Manifold (SLIM) is a technology for dispersing challenging powders like fumed silica, gums, thickeners and pigments using a specially modified high shear rotor/stator generator.

In both batch and inline designs, the SLIM is easy to retrofit into almost any process. In an inline set-up, the SLIM mixer pumps liquid from the recirculation tank while simultaneously drawing powders from a hopper. As the liquid stream enters the rotor/stator assembly, it immediately encounters the powder injection at the high shear zone. The mixture is then expelled through the stator at high velocity and recirculated back into the tank. In just a few short turnovers, solids are completely dissolved or reduced to the desired particle size.

This method for high-speed powder injection is ideal for dispersing small concentrations of hard-to-wet solids like CMC or xanthan gum (>5%). It is equally effective for solid loadings as high as 70%, as in the case of titanium dioxide or magnesium hydroxide slurries. By introducing solids sub-surface where they are instantly subjected to vigorous agitation, issues like floating powders, excessive dusting and formation of stubborn agglomerates ("fish eyes") are eliminated. Because the SLIM generates its own vacuum for powder induction and does not rely on external ductors or pumps, it is free of clogging and simple to operate.

Several models are available including automated skid packages where the SLIM mixer is piped to a jacketed tank and supplied with



Ross Inline SLIM powder induction mixer with built-in control panel mounted on a portable cart with work bench

flowmeters, load cells, solenoid valves, level sensors and thermocouples all integrated into a PLC Recipe Control Panel. Each ingredient addition and process step can be pre-programmed so that mixer speed, mixing time, temperature, composition and batch weight are accurately replicated in every run.

Established in 1842, Ross is one of the world's oldest and largest manufacturers of process equipment, specializing in mixing, blending, drying and dispersion. [www.highshearmixers.com](http://www.highshearmixers.com)

## Five decades of expertise in materials handling

*Jenike & Johanson offers a wide range of consulting and project services to optimize solids handling in both new plants and existing installations*

**Jenike & Johanson, Inc.** is the world's leading technology company for bulk material handling, processing, and storage. Using sound theory backed by more than 55 years of project experience, the company's experts deliver engineered solutions to achieve reliable powder and bulk solids flow.

Bulk materials and their flow properties are at the core of all Jenike & Johanson's work. Every project (over 7,500 to date) is unique, and clients have maximum flexibility in selecting services to meet their bulk material handling needs. Decisions made during the feasibility and engineering stages of a project are critically important to success. If bulk solids systems are not engineered from the outset to handle the unique characteristics of the materials, process start-up can be significantly delayed and design capacity may never be reached.

75% of all chemicals are handled in bulk solid form during manufacturing. When feeding powders to reactors or conveying wet cake from a centrifuge to a dryer, poor material flow can result in throughput limi-



Testing powder properties is a key step in equipment design

tations, non-uniform product, dust emissions, or spillage. Reliable solids handling improves unit operations involving blending, extrusion, pneumatic or mechanical conveying, and heating/cooling. Reliable powder flow and a first-in, first-out vessel discharge pattern (mass flow) is desired to

minimize cross-contamination of batches and ensure process efficiency.

The key to preventing or solving handling problems lies in knowing the causes. Jenike & Johanson offers an extensive range of services to support engineering projects in the chemical industry:

- on-site audit to review process or flow problem;
- raw material, intermediate, and final grade flow testing;
- design of storage vessels, chutes, and feeders;
- testing pneumatic conveying;
- silo structural engineering (both new and retrofit); and
- pilot-scale process modeling.

The key to preventing or solving handling problems lies in knowing the causes. Jenike & Johanson offers an extensive range of services to support engineering projects in the chemical industry. The company's website provides technical papers, case studies, and more information on solutions for chemical handling needs. [www.jenike.com](http://www.jenike.com)

## Reactor systems: Improving mass-transfer-limited reactions

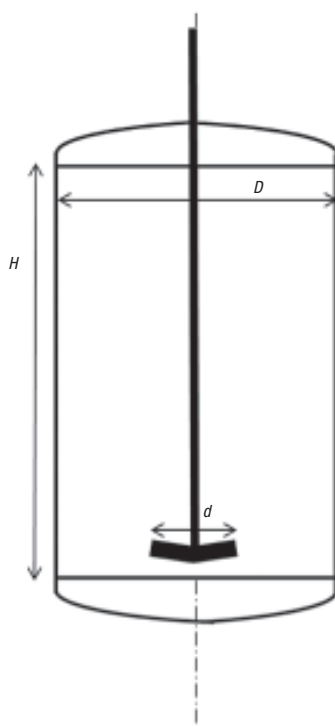
The step-by-step procedure described here can help operators to optimize reactor operation and output

Vinit D. Makwana, G. Sivalingam  
and Suketu M. Vakil  
Reliance Industries Limited

Engineers throughout the chemical process industries (CPI) commonly encounter situations where different materials have to be contacted inside a reactor to drive the chemical transformations needed to produce the required products. Reactors perform several important functions, such as bringing the reacting species into intimate contact, providing an appropriate environment to drive the desired reactions in terms of temperature, pressure and concentrations, appropriate reaction time and providing the means for proper removal of product streams or by-products or unreacted products.

The reactants are often present in different phases, adding to the complexity of the system. Typical reactions, in order of increasing difficulty, may occur in the gas, liquid or solid phase, or in a combination of any two or three of these phases. Further nuances are added to the problem in cases where the solid reactant could either be catalytic or non-catalytic, or two immiscible liquid phases may be present.

The stirred-tank reactor is the most commonly used type of equipment for batch or semi-batch reactions. Many well-developed tools and methods for modeling of chemical kinetics and reactions are available and today they are routinely used in practice for the design of reactors. However, in the case of multiphase reactions, a variety of factors — including reactor fluid dynamics, mixing, heat and mass transfer — need to be considered critically to really optimize the reactor design [1,2]. During the lifetime of an installed reactor, its original operating objectives



**FIGURE 1.** In a typical stirred-tank reactor, an agitator is provided for mixing and providing contact between the reactants, with a jacket or coil for heating or cooling. Various nozzles allow for reactants to be introduced into, and products to be withdrawn, from the reactor. These components provide all the necessary conditions to effect the desired chemical transformation. The dimensions used in the equations discussed are shown

may be extended due to capacity expansions, process changes, utility changes and retrofitting. This article provides an algorithm for evaluating the performance of commercial stirred-tank reactors for non-catalytic, multiphase reactions, with the aim of optimizing their operation.

### Basis

Consider a batch tank reactor with a jacket for heating and cooling and an agitator for mixing, as shown in Figure 1. The reaction is conducted

NOMENCLATURE	
$Re$	Reynolds number, unitless
$N_p$	Power Number, unitless
$D$	Reactor dia., m
$d$	Impeller dia., m
$H$	Reactor height, m
$p$	Pressure, bar
$P$	Applied power, kW
$\rho$	Density, kg/m <sup>3</sup>
$\mu$	Viscosity, kg/m-s
$t$	Time, min
$N$	Agitator speed, rps
$T$	Temperature, °C
$V$	Reactor fluid volume, m <sup>3</sup>

for a certain amount of time  $t$  (min) at a temperature  $T$  (°C) and pressure  $p$  (bar). The reactor diameter, reactor height, agitator diameter, agitator type and agitator motor power are known. The first step when evaluating the reactor's performance is to assess the rate-determining step of the chemical processes being conducted inside the reactor.

**Step 1. Identify the rate-determining step.** To understand the rate-controlling regime existing inside the reactor, the mass transfer and kinetic effects of the chemical reaction system must be segregated. This is typically done in a laboratory reactor by carrying out the reaction at different agitation speeds. The same reaction should be performed in the laboratory under identical operating conditions as used in the plant.

In general, the effect of increased agitation on mass transfer is much greater than on reaction kinetics. Thus, the reaction is repeated at different stirring speeds and the extent of reaction is measured at the end of each trial. The results are plotted as a graph of revolutions per minute (rpm; on the x-axis) and either time

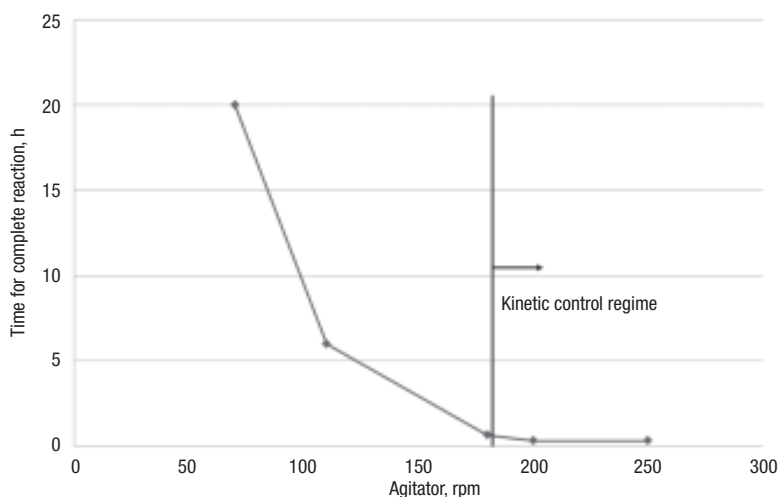
to achieve complete conversion, or time to attain equilibrium, on the y-axis (Figure 2). The graph typically shows exponential behavior, with a sharp increase in conversion — or a decrease in the time required to achieve complete reaction — produced initially with increasing mixing. This increase eventually levels off to an equilibrium value.

The region of the graph with varying slope indicates that the reaction is operating under mass-transfer control regime. By contrast, the region where the slope of the graph is close to zero represents the kinetic control regime. This laboratory-scale experiment should be repeated under conditions favoring kinetic control regime to determine the true kinetics of the reaction. This first step provides important information regarding the rate-determining process inside the reactor and appropriate design strategies that can be followed.

### Step 2. Replicate the industrial process at laboratory scale.

Step 1 provides information about intrinsic characteristics of the reaction, but it does not give a clear picture of the hydrodynamics and heat and mass transfer dynamics inside of an existing commercial reactor. This requires an exact replication of the plant reactor at the laboratory scale — not only in terms of operating parameters but also replicated in terms of mixing behavior. Geometric, kinematic and dynamic similitude between commercial and laboratory reactor is necessary for complete replication of the commercial-scale reactor at laboratory scale. For instance, the laboratory reactor should be constructed such that dimensions and ratios (for instance,  $H/D$  and  $d/D$ ) should match the commercial reactor for maintaining geometric similitude. The laboratory reactor should be operated at exactly the same operating conditions as the plant reactor, in terms of density, viscosity, solids loading and gas dispersion, in order to achieve kinematic similitude.

The final aspect for replication — that is, dynamic similitude — relates to the extent of mixing and fluid velocities that exist within the reactor. The typical parameters that can be adjusted to achieve dynamic similitude are impeller tip speed and power dissipated per unit volume



**FIGURE 2.** In this graph of the kinetics of a ZN catalyst hydrolysis reaction versus agitator rpm, one sees the classical exponential profile. The time required for the completion of the reaction decreases initially as mixing increases and then levels off beyond 170 rpm. This point indicates the transition from the mass-transfer-controlled regime to kinetic-control regime. The low plateau in the kinetic-control region indicates the intrinsically fast kinetics of the reaction

( $P/V$ ). The  $P/V$  value essentially describes the momentum transferred to the fluid and relates to the effective motion created by the agitator. This parameter provides a reliable estimation of the conditions existing inside the reactor. As turbulence increases and the situation moves toward well-mixed conditions, the tip speed similitude closely approximates the  $P/V$  similitude. Thus, the final laboratory reactor should have the same geometry, operating conditions and  $P/V$  as the plant reactor in order to achieve true scaledown.

The reactor geometry and geometric ratios are straightforward to calculate and implement, as are the operating conditions. The agitator diameter, vessel diameter, agitator motor power, agitator speed are known for plant operation. The relation between speed, diameter and power is shown in Equations (1) and (2):

$$P = N_p \rho d D^3 N^5 \quad (1)$$

and

$$Re = d^2 N \rho / \mu \quad (2)$$

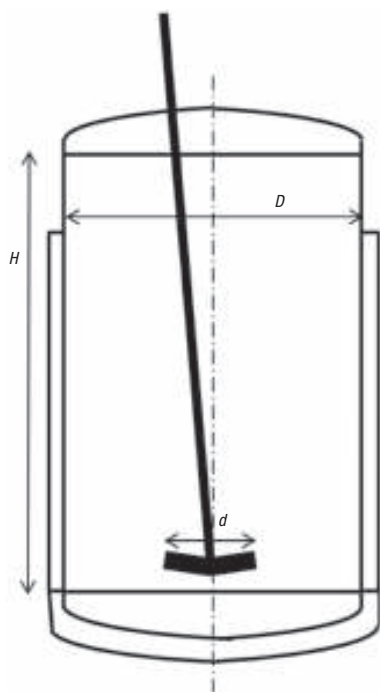
The plant agitator geometry is known and the Power Number,  $N_p$ , can be easily found from published  $N_p$ -versus- $Re$  graphs. These data are available in published literature for

a variety of impeller types and over the whole range of Reynolds number values [3].

The plant operating conditions are used to calculate the power dissipated in the fluid using Equations (1) and (2) and  $N_p$ -versus- $Re$  charts. The calculated power is divided by the volume of the reaction mass to calculate the  $P/V$  parameter. The same is maintained for the laboratory reactor. Using the volume of the laboratory reactor and the  $P/V$  value from the commercial reactor, the power required for the laboratory reactor can be calculated. The laboratory agitator is chosen and can be the same or different from the plant agitator. The geometric similitude factors are maintained and appropriate  $N_p$  values are used for the laboratory agitator. Using Equation (1), the required speed for the laboratory agitator is calculated. The physical characteristics of the laboratory agitator and motor are checked to confirm if the calculated speed is achievable. A useful cross-check is to calculate  $Re$  at the obtained speed and confirm if the chosen  $N_p$  value is valid for that  $Re$ . If both conditions are met, then the scaledown of the plant reactor is said to be complete. If not, then the laboratory impeller is changed accordingly.

**Step 3. Validate in a laboratory simulator.** The test reaction is per-





**FIGURE 3.** From the case study discussed, this schematic of the commercial hydrolysis reactor shows the stirred-tank reactor with jacket and agitator. The agitator is a single, backward-curved turbine impeller with four blades. The shaft is mounted at 10 deg to the vertical. The dimensions used in the equations correspond to this drawing

formed in the scaled-down laboratory reactor at the appropriate mixing to exactly predict the performance of the plant reactor. This can be used to troubleshoot existing processes or explore the feasibility of a given reactor to be retrofitted for a new or modified process.

The following case study gives an example of the above algorithm.

### Case study

This case study deals with a stirred-tank reactor used to deactivate catalyst fines before discharge into a waste water treatment plant (WWTP). Polyethylene is manufactured via a gas-phase process using Ziegler-Natta (ZN) catalysts. The catalyst, being extremely air sensitive, is usually made at the site in the catalyst section of the plant. The particle size distribution (PSD) of the catalyst is an important performance parameter for the main polyethylene reactor. The catalyst is elutriated after synthesis to ensure that the appropriate PSD specification is met before use.

Any catalyst fines that are removed

during the elutriation process cannot be discharged directly to the WWTP, due to their high reactivity with water. The residual catalyst and polymer fines suspended in hydrocarbon are deactivated by hydrolysis in a batch, stirred-tank reactor before discharge to the WWTP.

The deactivation reaction between water and Ziegler-Natta (ZN) catalyst slurry in hydrocarbon is performed in a typical liquid-liquid-solid stirred-tank reactor. The aqueous reactor effluent stream is drained after a reaction time of 4 h, and shows significant unreacted catalyst. Renewed contact of the active catalyst with the flushing water in a downstream filter leads to further reaction, an evolution of gas bubbles and a temperature rise due to exothermicity. The catalyst deactivation is completed in the filter and benign solids are separated from the aqueous phase, which is then further discharged to the WWTP.

When this occurs, the recovery of hydrocarbon solvent by decantation is also poor due to poor layer separation of aqueous and organic layers. This is a clear indication of incomplete reaction and the need to re-evaluate the performance of the reactor.

**Step 1. Identification of the rate-determining step.** The reaction described above was performed in a laboratory reactor at various agitation speeds. The extent of the reaction, in this case, is easily determined by the color change of the reaction mixture, as is the end of the reaction, because the active ZN catalyst is dark in color while the deactivated catalyst has a dull, pale yellow color. The time for completion of reaction was measured at various speeds and plotted, as shown in Figure 2. The figure shows a transition from the regime controlled by mass transfer, to the region controlled by kinetics beyond 170 rpm. Also the intrinsic kinetics of the hydrolysis reaction is fast, with the reaction going to completion within 20 min.

**Step 2. Replication of the industrial process at laboratory scale.**

The plant reactor for this case study is shown in Figure 3. The impeller is a backward-curved turbine with four blades. The agitator is mounted at an angle of 10 deg to the vertical,

to introduce an element of axial mixing. The impeller blade is positioned at the bottom tangent line of the reactor. The agitator is operated by a 1-hp motor, at a speed of 28 rpm.

Based on Equation (2):

$Re = 81,650$ , which is in the turbulent region

Using Equation (1):

$$P/V = 2.45W/m^3$$

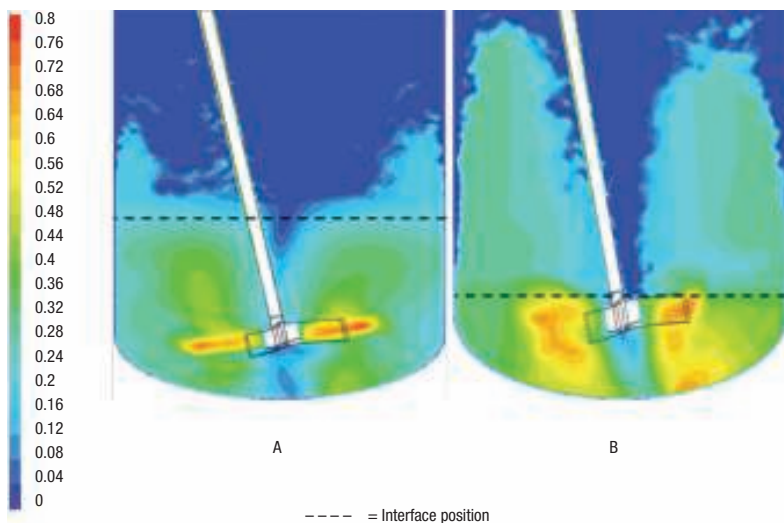
This is the number that needs to be replicated in a laboratory-scale reactor, apart from the geometry. The laboratory-scale reactor was fixed at 1 L with a similar turbine impeller agitator. The laboratory reactor was equipped with a 0.05-hp agitator motor. Again using Equation (1), the required speed for achieving the same  $P/V$  is  $N = 100$  rpm.

This speed is easily achievable with the laboratory motor. It should also be noted from Figure 2 that the plant reactor is being operated under the mass-transfer-controlled regime. The  $N_p$  assumed for calculation is valid for the  $Re$  range at this speed.

**Step 3. Validation in laboratory simulator.**

As we can see from Figure 2, the reactor is operating under mass-transfer-controlled regime at 100-rpm agitation speed. Since the reaction system consists of water (liquid), hydrocarbon (liquid), and catalyst fines (solid), the mass-transfer-control regime clearly shows the reason for incomplete reaction observed in the plant. The glass laboratory reactor allows an added benefit of visually observing the mixing patterns and extent of reaction over time. The conclusion from calculations was validated by observations during the reaction where the interface remained stagnant. Only some turbulence was seen around the impeller edges, which was quickly dampened so that no mixing occurred between layers.

**Troubleshooting.** The important conclusions from the above exercise were that, while the intrinsic kinetics of the hydrolysis reaction are very fast, the actual reaction remains incomplete in the plant reactor. Within the laboratory-scale reactor, using plant-simulated conditions, the polymer and catalyst fines,



**FIGURE 4.** CFD studies clearly show the aqueous layer velocity profiles in m/s. The light blue region denotes the aqueous layer while the dark blue region denotes the hydrocarbon layer. When the interface is moved lower to coincide with the plane of agitation in the modified operation, the agitation is more effective with higher penetration of the aqueous layer into the hydrocarbon layer, indicating better mixing

being of intermediate density, were concentrated at the interface. The power dissipated into the fluid by the agitator was dampened immediately at a very short distance away from the impeller, while the interface was 0.4 m away from the impeller in the plant reactor. As a result, the two layers barely mixed and reaction remained incomplete.

### *Geometric, kinematic and dynamic similitude between the commercial and the laboratory reactor is necessary for complete replication of the commercial-scale reactor at laboratory scale.*

This is also seen from the computational fluid dynamics (CFD) profile of water-velocity distribution that is shown in Figure 4A. As shown in this figure, the water velocity is quickly dampened close to the impeller, and the layer inter-mixing is almost negligible. The simple solution to this problem would be to increase agitator speed/motor power to increase the mixing intensity and thus complete the reaction. However, this approach would involve hardware modifications, capital expenditures and plant shutdown. This unique situation of fast kinetics was harnessed to develop a novel solution for this mass-transfer-control problem.

The agitator level lay close to the bottom of the aqueous layer, while the catalyst fines were in the organic layer. The agitator speed and power were kept constant. Instead of expanding the turbulent zone by increasing mixing, the reaction zone — that is, the interface — was brought closer to the impeller by adjusting the quantity of water added

to the reactor. The water added was in large stoichiometric excess in the original recipe and decreasing this amount did not lead to any limiting reactant issues. Adding less water brought the interface lower and nearer to the impeller. The turbulence around the impeller edges led to mixing of the two phases, and the fast reaction kinetics ensured that the reaction proceeded to completion.

The increased mixing efficiency was also validated by CFD modeling. Figure 4B shows the water velocity profile for modified operation, where the interface is lowered to be aligned with the plane of agitation, resulting

in increased turbulence. The current of the aqueous layer (light blue) penetrates the hydrocarbon layer (dark blue) much farther and reaches almost the top of the reactor with modified operation. Earlier, due to incomplete reaction inside the reactor, when the aqueous layer was drained and further washed with water in the effluent pit, the reaction proceeded to completion resulting in a rise in temperature in the effluent pit (due to exothermicity). With this modified operation, when the aqueous layer is drained to the effluent pit and further contacted with water, it remains at ambient temperature. No gas evolution was seen and the material was lighter in color, indicating completion of reaction. This simple operational modification resulted in inherently safer operation, better hydrocarbon recovery, and reduced chemical oxygen demand (COD) of the effluent. The COD of the effluent has decreased to a third of its original value, from 185,000 to 60,000 ppm, as a result of this modified operation.

### **Summary**

Commercial reactors undergo modifications over their lifetime, when their original objective is changed due to expansions, process changes, utility changes and retrofitting. Engineers must evaluate their performance with respect to mass transfer, kinetics and mixing regime. The step-by-step procedure for evaluating reactor performance discussed here, along with the case study presented about troubleshooting a commercial hydrolyzer reactor, can help engineers to better optimize reactor operation. Specifically, the use of this method identified poor mixing between the hydrocarbon and aqueous layer as the cause of poor reactor performance.

The laboratory reactor was instrumental in identifying the lack of layer inter-mixing as a critical bottleneck. This investigation also suggested an approach for manipulating the interfacial level as a means to improving mass transfer and, consequently, driving the reaction. When the solution was implemented in a plant, it resulted in inherently safer operation, increased hydrocarbon recovery from the reactor (due to better layer separation due to complete reaction), and

decreased environmental impact (by reducing effluent COD). ■

Edited by Suzanne Shelley

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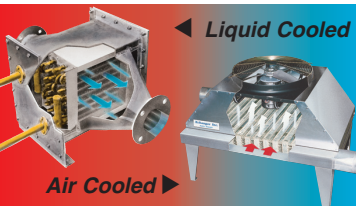
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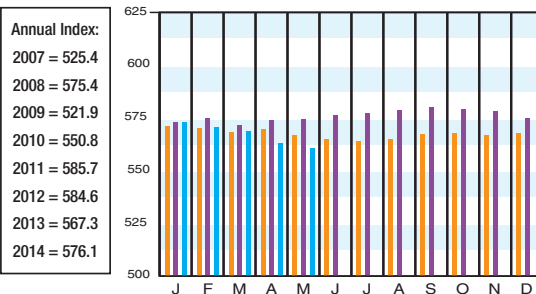
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(1957-59 = 100)	May '15 Prelim.	Apr. '15 Final	May '14 Final
CE Index	560.7	562.9	574.3
Equipment	675.6	678.8	697.0
Heat exchangers & tanks	603.5	609.7	635.1
Process machinery	658.8	663.8	665.0
Pipe, valves & fittings	843.6	845.6	876.2
Process instruments	402.7	402.0	410.8
Pumps & compressors	958.0	958.4	938.6
Electrical equipment	513.0	511.9	515.3
Structural supports & misc	740.1	741.7	767.4
Construction labor	323.2	323.8	320.5
Buildings	542.9	545.0	543.2
Engineering & supervision	320.6	319.0	321.0

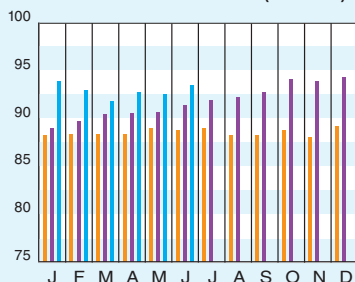


Starting with the April 2007 Final numbers, several of the data series for labor and compressors have been converted to accommodate series IDs that were discontinued by the U.S. Bureau of Labor Statistics

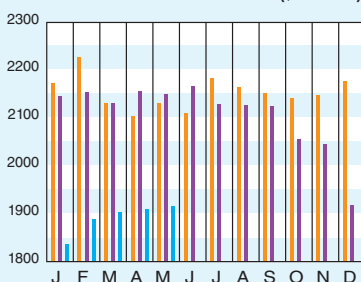
## CURRENT BUSINESS INDICATORS

	LATEST	PREVIOUS	YEAR AGO
CPI output index (2000 = 100)	Jun. '15 = 93.4	May '15 = 93.1	Apr. '15 = 93.0
CPI value of output, \$ billions	May '15 = 1,915.2	Apr. '15 = 1,911.6	Mar. '15 = 1,902.6
CPI operating rate, %	Jun. '15 = 77.9	May '15 = 77.7	Apr. '15 = 77.7
Producer prices, industrial chemicals (1982 = 100)	Jun. '15 = 241.4	May '15 = 237.3	Apr. '15 = 238.5
Industrial Production in Manufacturing (2002=100)*	Jun. '15 = 101.7	May '15 = 101.7	Apr. '15 = 101.6
Hourly earnings index, chemical & allied products (1992 = 100)	Jun. '15 = 157.5	May '15 = 159.1	Apr. '15 = 158.1
Productivity index, chemicals & allied products (1992 = 100)	Jun. '15 = 108.1	May '15 = 108.4	Apr. '15 = 108.4

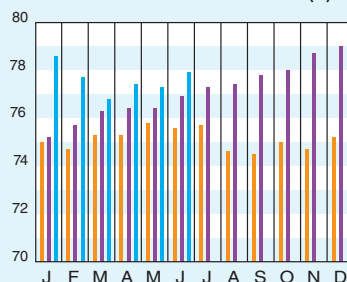
### CPI OUTPUT INDEX (2000 = 100)



### CPI OUTPUT VALUE (\$ BILLIONS)



### CPI OPERATING RATE (%)



\*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board. Current business indicators provided by Global Insight, Inc., Lexington, Mass.

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## CURRENT TRENDS

The preliminary value for the May 2015 CE Plant Cost Index (CEPCI; top; the most recent available) continued the trend of small monthly declines since the beginning of the year. The May CEPCI is 2.4% lower than the corresponding value from a year ago at the same time. This is a larger gap than the 1.8% for the April year-ago value. The Engineering & Supervision subindex rose slightly in May, but the other subindices saw small declines. Meanwhile, the latest Current Business Indicators (middle) numbers were generally slightly higher than the previous month's values. The CPI output index for June 2015 rose from the previous month and was higher than the corresponding value from 2014. The CPI value of output rose from the previous month, but was lower than a year ago.





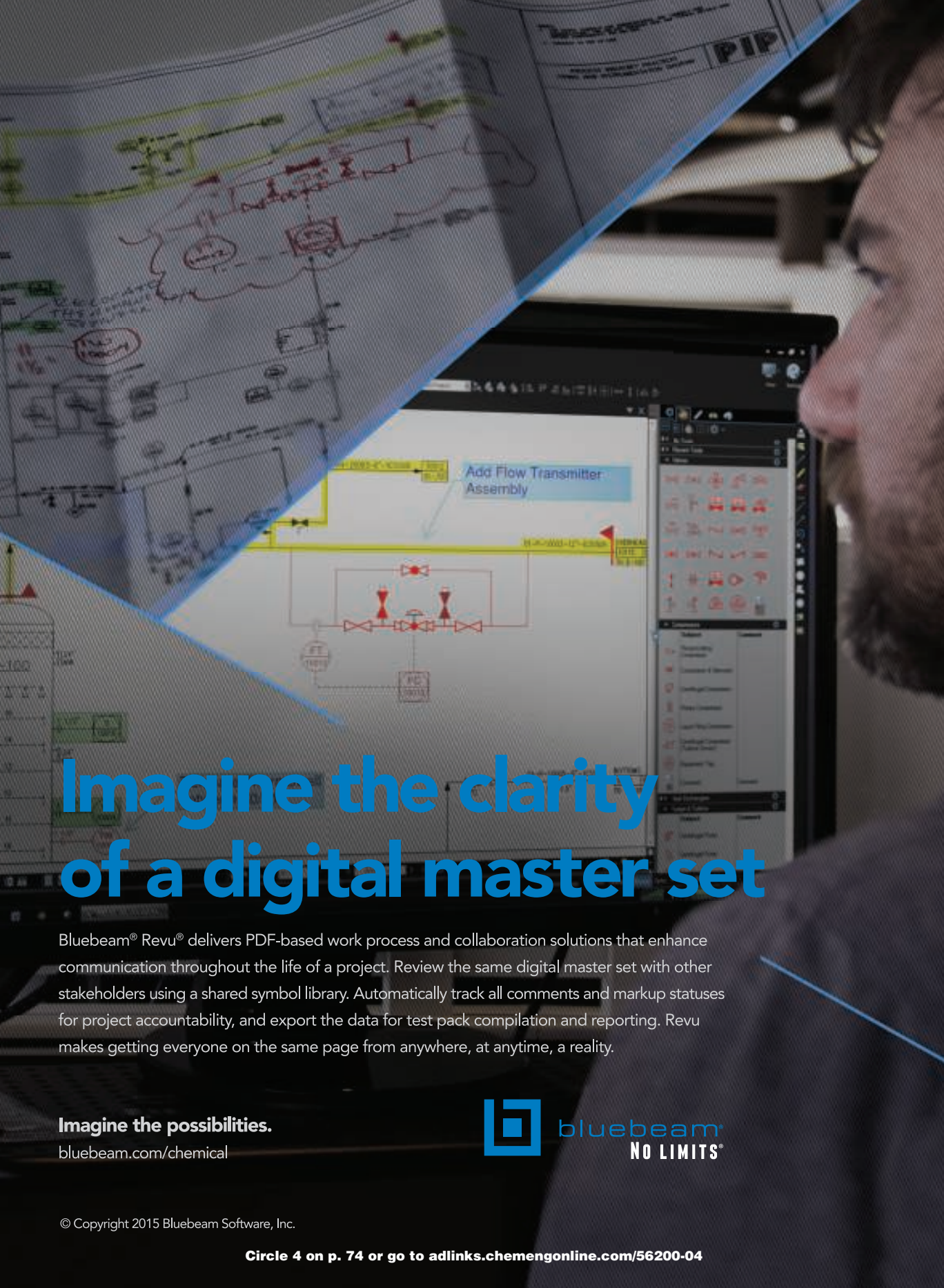
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