# CHEMICAL ENGINEERING

June
2014

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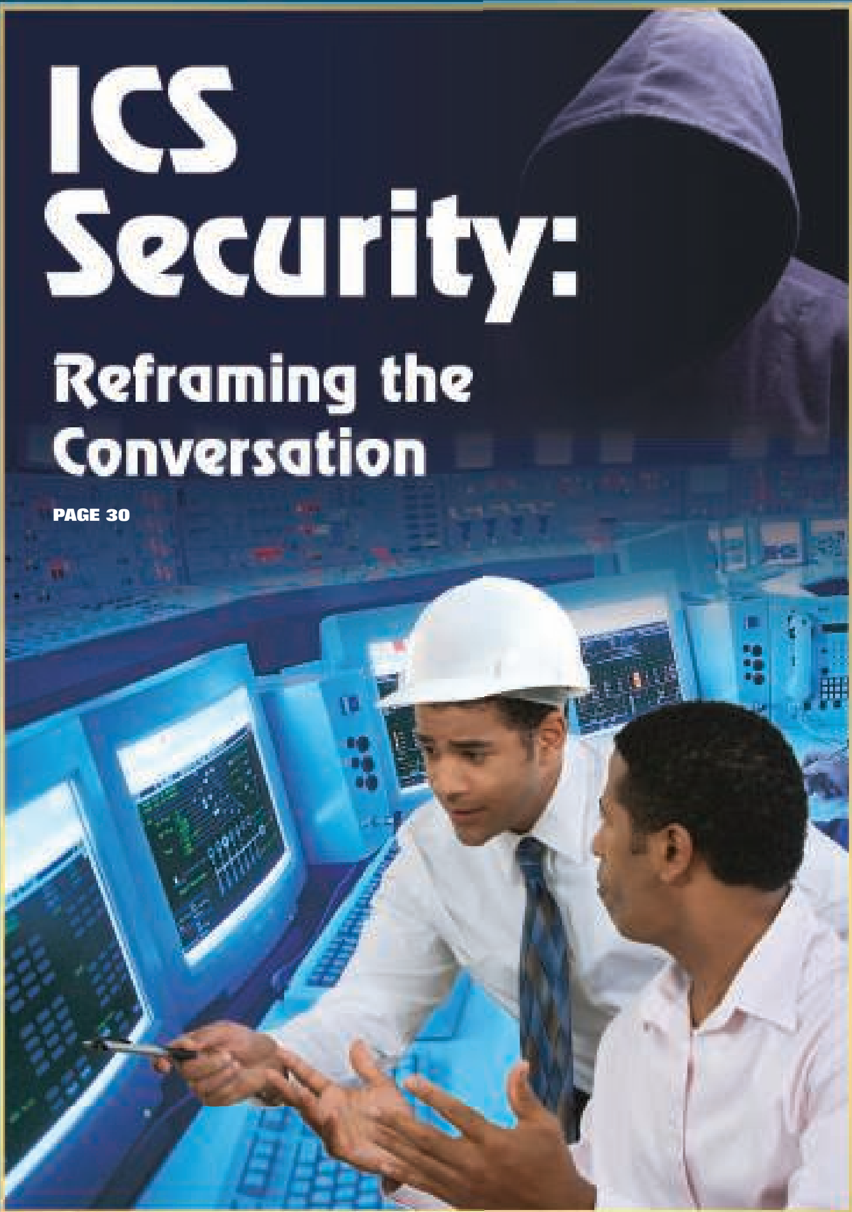


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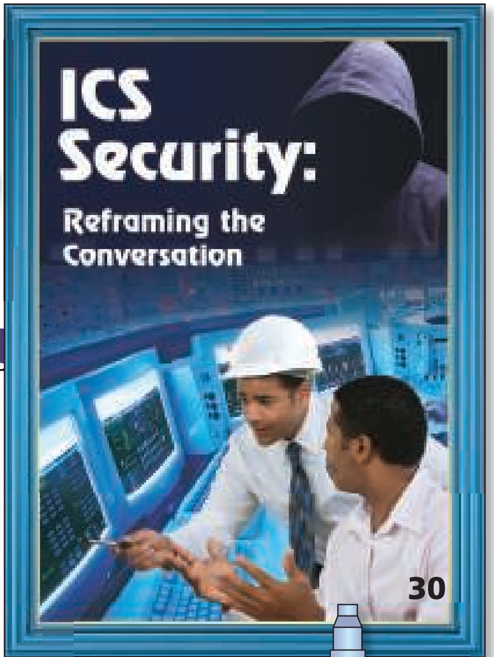
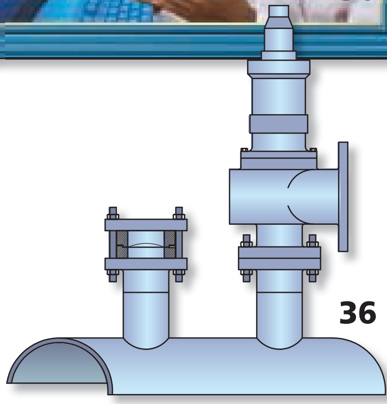



## COVER STORY



## NEWS





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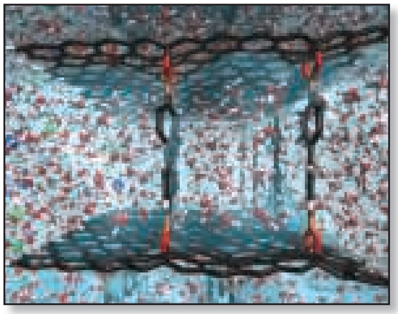

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## ENGINEERING




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Published since 1902
An Access Intelligence Publication

**PUBLISHER**

**MICHAEL GROSSMAN**
Vice President and Group Publisher
mgrossman@accessintel.com

**EDITORS**

**DOROTHY LOZOWSKI**
Editor in Chief
dlozowski@che.com

**GERALD ONDREY** (Frankfurt)
Senior Editor
gondrey@che.com

**SCOTT JENKINS**
Senior Editor
sjenkins@che.com

**MARY PAGE BAILEY**
Assistant Editor
mbailey@che.com

**CONTRIBUTING EDITORS**

**SUZANNE A. SHELLEY**
sshelley@che.com

**CHARLES BUTCHER** (U.K.)
cbutcher@che.com

**PAUL S. GRAD** (Australia)
pgrad@che.com

**TETSUO SATOH** (Japan)
tsatoh@che.com

**JOY LEPREE** (New Jersey)
jlepree@che.com

**GERALD PARKINSON**
(California) gparkinson@che.com

**ART & DESIGN**

**DAVID WHITCHER**
Art Director/
Editorial Production Manager
dwhitcher@che.com

**PRODUCTION**

**JOHN BLAYLOCK-COOKE**
Ad Production Manager
jcooke@accessintel.com

**INFORMATION SERVICES**

**CHARLES SANDS**
Director of Digital Development
csands@accessintel.com

**MARKETING**

**CRISTANE MARTIN**
Marketing Manager
TradeFair Group, Inc.
cmartin@accessintel.com

**AUDIENCE DEVELOPMENT**

**SARAH GARWOOD**
Audience Marketing Director
sgarwood@accessintel.com

**GEORGE SEVERINE**
Fulfillment Manager
gseverine@accessintel.com

**JEN FELLING**
List Sales, Statlistics (203) 778-8700
j.felling@statlistics.com

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BPA

## Editor's Page

# The wide reach of the CPI

Last year I visited Yellowstone National Park for the first time. I was appropriately awed by the beauty of the landscape and majestic wildlife. One site that stays in my memory is the Grand Prismatic Spring — a steamy, colorful hot spring that is said to be the largest in the U.S. and third largest in the world. Our expert guide explained that some of the brilliant colors were due to microbes, and that scientists from around the world come to study the microbial mats that thrive under the extreme temperature conditions of the spring.

Recently I had the opportunity to visit one of BASF Corp.'s (Florham Park, N.J.; www.basf.com) research and development hubs, in Tarrytown, N.Y., where experts were on hand to discuss a number of key technology areas. One area is "white" biotechnology, a term used to describe industrial biotechnology. Markus Pompejus, vice president of research for white biotechnology at BASF in North America and CEO for Verenium, which BASF acquired late last year, discussed how technologies are being used to adapt enzymes to target applications. Examples can range from an enzyme for laundry use that works well in cold temperatures to applications in the oil-and-gas industry. He explained that scientists seek out lifeforms, such as bacteria, in extreme ecosystems in hopes of finding enzymes that might be of interest for a specific area of application. This brought back my memory of the Grand Prismatic Spring, because enzyme research is one of the scientific studies our guide was referring to.

BASF experts further described a wide variety of technology areas that they are working on, including the following: a service to economically and ecologically optimize concrete mixtures using both cementitious and non-cementitious materials; production of both cathode materials and electrolytes for batteries; polymers and coatings for seed enhancement in agricultural use; and formulations for food, beverage and dietary supplements such as vitamins, and functional ingredients for cereals, desserts and more.

Michael Pcolinski, vice president of innovation and technology in North America described a few additional "growth fields" for BASF, including organic electronics (organic LEDs), lightweight composite materials, plant biotechnology, wind energy and water solutions (for example, using polymer beads to reduce the amount of water needed to wash clothes). BASF's logo describes itself as "The Chemical Company," and it is clear from the company's technology portfolio just how wide-reaching chemistry is.

BASF is part of the chemical process industries (CPI) and the wide breadth of subjects under its umbrella only starts to describe the CPI. Readers of *Chemical Engineering* will know that you can find the term CPI in veritably every issue. The CPI consist of: chemicals, including petrochemicals; drugs and cosmetics; fertilizers and agricultural chemicals; foods and beverages; synthetic fibers; metallurgical and metal products; paints and coatings; petroleum refining and coal products; plastics; soaps and detergents; stone, clay, glass and ceramics; pulp-and-paper products; and additional industries where chemical processing takes place.

The range of the CPI is broad enough to reach the hidden resources of Yellowstone National Park. If you haven't been to Yellowstone yet, consider putting it on your "bucket list." You won't be disappointed. ■

*Dorothy Lozowski, Editor in Chief*

# Letters

## Call for papers: Achema 2015

The organizers of Achema (Dechema; Frankfurt am Main, Germany; www.dechema.org) have issued a call for papers for Achema 2015, which will be held June 15–19, 2015 in Frankfurt am Main. The event is held every three years, and attracted over 3,700 exhibitors and over 166,000 participants in 2012.

The Achema Congress spans a wide spectrum of process engineering themes, complementing classical topics with cutting-edge lecture series, and thus reflects the multifaceted diversity of the exhibition. Abstracts on topics related to industrial practice are welcome. Some of the themes are the following:

- Energy-efficient processes
- Flexible production (resources/dynamics)
- Alternative raw materials
- Thermal and electrical power engineering
- Advanced fluids
- Nanotechnology
- Electrochemical technologies
- Bioprocesses: reactors, monitoring, modeling, downstream processing
- Novel bio-catalysts: development and application
- Single-use technologies
- Progress in laboratory and analytical techniques
- Digital plant engineering
- Modular plant concepts
- Catalysis
- Reaction engineering
- Mixing and separation technology
- Solids processing
- Safety
- Pharmaceutical production
- Plant control
- Materials and materials testing

The following three areas are focal topics for the congress:

- Industrial water management
- Innovative process analytical technology (PAT)
- Bio-based world
  - bio-refinery platforms: advanced biofuels, renewable chemical and bio-based materials
  - algae, specialty crops and biomass supply

Abstracts can be submitted at www.achema.de/congress

The deadline for submissions is August 31, 2014.

More information and regular updates about the exhibition and congress can be found at www.achema.de

**Do you have —** • Ideas to air? • Feedback about our articles?
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• Job-related problems or gripes to share?
**If so —** Send them, for our Letters column, to
Dorothy Lozowski, ***Chemical Engineering,*** Access Intelligence, 88 Pine St., 5th floor, New York, NY 10005;
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## Calendar

### NORTH AMERICA

**107th Annual AWMA Conference & Exhibition**. The Air & Waste Management Assn. (AWMA; Pittsburgh, Pa.). Phone: 412-904-6031; Web: awma.org
*Long Beach, Calif.* **June 24–27**

**Establishing and Monitoring a Clean Manufacturing Program**. Inst. of Environmental Sciences and Technology (Arlington Heights, Ill.). Phone: 847-981-0100, Ext. 6012; Web: iest.org
*Arlington Heights, Ill.* **July 16**

**2014 Chemical Sector Safety Security Summit and Expo**. SOCMA (Washington, D.C.). Phone: 202-721-4100; Web: socma.com
*Baltimore, Md.* **July 22–24**

**Cat Cracker Seminar**. American Fuel & Petrochemical Manufacturers (AFPM; Houston). Phone: 202-457-0480; Web: afpm.org
*Houston* **Aug. 19–20**

**5th Biobased Chemicals Conference: Commercialization & Partnering**. Global Technology Community (GTC; Monrovia, Calif.). Phone: 626-256-6405; Web: gtcbio.com
*San Francisco, Calif.* **Sept. 18–19**

**43rd Turbomachinery and 30th Pump Users' Symposia**. Texas A&M University Turbomachinery Laboratory (College Station, Tex.). Phone: 979-845-7417; Web: pumpturbo.tamu.edu
*Houston* **Sept. 22–25**

**WEFTEC**. Water Environment Federation (Alexandria, Va.). Phone: 571-830-1545; Web: weftec.org
*New Orleans, La.* **Sept. 30–Oct. 1**

**AFPM Q&A and Technology Forum**. American Fuel & Petrochemical Manufacturers (AFPM; Houston). Phone: 202-457-0480; Web: afpm.org
*Denver, Colo.* **Oct. 6–8**

**WJTA–IMCA Expo**. Water Jet Technology Assn. and Industrial & Municipal Cleaning Assn. (St. Louis, Mo.). Phone: 314-241-1445; Web: wjta.org
*New Orleans, La.* **Oct. 13–15**

**Next-Generation Filter-Media Conference**. American Filtration and Separations Soc. (Nashville, Tenn.).

Phone: 615-250-7792; Web: afssociety.org
*Chicago, Ill.* **Oct. 14–15**

**AFPM Environmental Conference**. American Fuel & Petrochemical Manufacturers (AFPM; Houston). Phone: 202-457-0480; Web: afpm.org
*San Antonio, Tex.* **Oct. 19–21**

**Gasification Technologies Conference 2014**. Gasification Technologies Council (Arlington, Va.). Phone: 703-276-0110; Web: gasification.org
*Washington, D.C.* **Oct. 26–29**

## EUROPE

**Carbon Footprinting**. IChemE (Rugby, U.K.), Phone: +44-1788-534431; Web: icheme.org
*Manchester, U.K.* **June 25–26**

**10th European Soc. of Biochemical Engineering Sciences and 6th International Forum on Industrial Bioprocesses**. University of Lille (Lille, France), in collaboration with the American Chemical Soc. (Washington, D.C.). Fax: +33-3-2876-7356; Web: esbes-ifibiop-lille2014.com
*Lille, France* **Sept. 7–10**

**Advances in Process Control and Automation 10**. IChemE (Rugby, U.K.), Phone: +44-20-7927-8200; Web: icheme.org
*York, U.K.* **Sept. 15–17**

**IWA World Water Congress & Exhibition 2014**. International Water Assn (IWA; London, U.K.). Phone: +31-70-315-0792; Web: iwa2014lisbon.org
*Lisbon, Portugal* **Sept. 21–26**

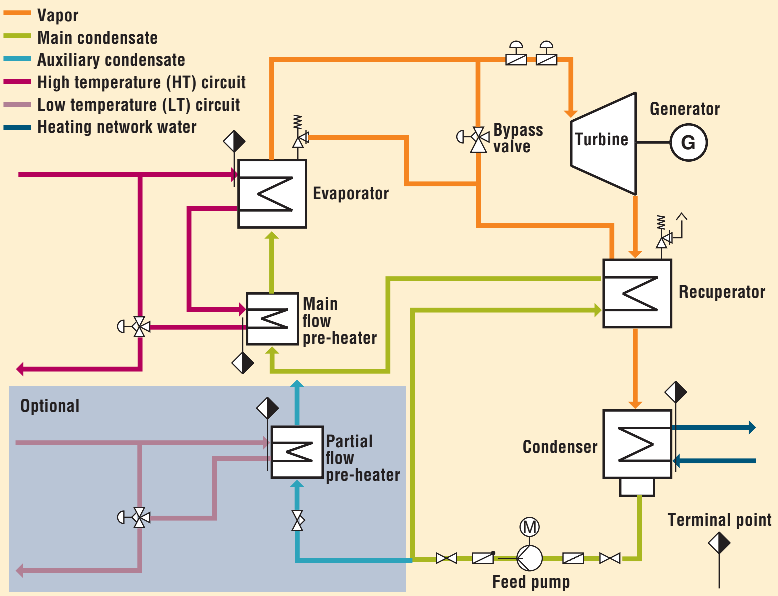
## ASIA & ELSEWHERE

**3rd Annual InaChem — Trade Fair and Conference for the Chemical, Fine & Specialty Chemical and Petrochemical Industries**. Federation of the Indonesian Chemical Industry and Indonesia Institute of Chemical Engineers (Jakarta, Indonesia). Phone: +62-21-789-2938; Web: ina-chem.com
*Jakarta, Indonesia* **Aug. 14–16**

**IndoPlas — 9th Annual International Plastics Exhibition**. Messe Düsseldorf Asia Pte. Ltd. (Singapore). Phone: +65-6332-9620; Web: indoplas.com
*Jakarta, Indonesia* **Sept. 3–6** ■

*Suzanne Shelley*

## Recover waste heat with ORC technology




Last April, Siemens Energy (Erlangen, Germany; www.siemens.com/energy) introduced the Siemens organic Rankine cycle (ORC) modules, which enable operators of industrial plants to generate power from low-temperature (around 300°C) waste heat.

Unlike a conventional Rankine cycle, which converts water to steam, the ORC vaporizes an organic fluid with a high molecular weight. The organic fluid has a boiling point lower than water, thus enabling heat to be recovered from low-temperature sources, such as biomass and biogas combustion, industrial waste heat (for example, in the glass and cement industries), geothermal heat, solar ponds and so on. The recovered heat is then converted into useful work, driving a turbine to generate electricity.

The ORC module (flowsheet) consists of a turbine (with generator), a recuperator with condenser combined in a functional unit, a feed pump, pre-heater(s), an evaporator and the interconnecting piping. The vapor produced in the evaporator is supplied to the ORC turbine, which drives the generator. After leaving the turbine, the vapor enters the recuperator to transfer the energy from the superheated exhaust vapor to the liquid side of the process. Afterwards, it is condensed. The condensate (collected in the condenser hotwell) is discharged by a feed pump back to the evaporator via the pre-heater(s). The speed of the feed pump is controlled by a frequency converter to maintain the level in the condenser hotwell.

Siemens first utilized its ORC technology in 2013, and is now offering modules commercially. Four modules are currently available with capacities from 300 kW to 2 MW, and the company plans to expand to higher capacities over the medium term.

## Reduce energy costs by combining wastewater treatment and desalination

New Energy and Industrial Technology Development Organization (NEDO; Kawasaki, Japan; www.nedo.go.jp) and Global Water Recycling and Reuse Solution Technology Research Assn. (GWSTA) have developed a new water-treatment system that integrates seawater desalination with recycle water from a sewage treatment plant. In a demonstration unit installed at the Water Plaza Kitakyushu (deveolved to Kitakyushu City on April 1), Japan, the system achieved energy savings of more than 30%, says NEDO. Because the desalination process partially uses water recovered from the sewage-treatment process, less seawater is withdrawn, thus reducing the environmental load. The organizations believe the system is an optimal water-treatment package for regions where water is scarce.

In the demonstration plant, sewage is first pretreated by a membrane bioreactor (MBR) with a flowrate of 1,500 $m^3$/d. The water is then treated in a low-pressure reverse osmosis (RO) membrane system (throughput of 1,400 $m^3$/d), to make water for industrial applications. The low-concentration brine from the RO process is then mixed with seawater, pretreated by ultrafiltration (UF; flowrate of 500 $m^3$/d) and then used as the feedwater for

(*Continues on p. 10*)

## Pilot bioreactor

A next-generation photobioreactor is scheduled to be installed in June at the Orlando, Fla. pilot facility of biofeedstock company Proterro (Ewing, N.J.; www.proterro.com). Proterro is working with advanced fabrics and engineering firm ILC Dover (Dover, Del.; www.ilcdover.com) on developing the updated photobioreactor, which is a key element of Proterro's process for converting $CO_2$ and sunlight and water to fermentable sucrose using a patented cyanobacteria strain (see *Chem. Eng.*, June 2012, p. 12). Speaking at a recent biofuels conference, Proterro CEO Kef Kasdin said that the company has met its sugar-production milestones for the first quarter of 2014, and the results from the pilot plant verify the scalability of the process.

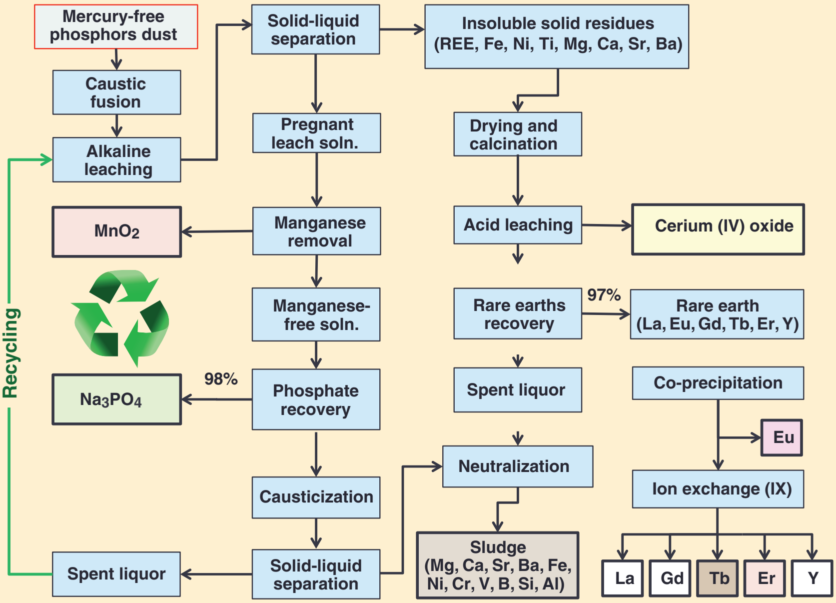
## Graphene membrane

A stable, porous membrane that is just two carbon atoms thick has been produced by a team of researchers at the Dept. of Mechanical and Pro-

(*Continues on p. 10*)

*Note*: For more information, circle the 3-digit number on p. 56, or use the website designation.

# A process that recovers rare-earth elements from 'urban mining'




Electrochem Technologies & Materials Inc. (Montreal, Canada; www.electrochem-technologies.com) has recently patented a process for recovering rare-earth elements (REEs) from mercury phosphor powders — a byproduct from recyclers of fluorescent lamps and light bulbs. This technology offers a sound and profitable alternative for the production of REEs compared to the mining of low-grade ores, which requires large tonnage operations, says company president, Francois Cardarelli. Such mining operations have both high operating and capital costs, and unavoidably produce radioactive wastes, he says.

In contrast, Electrochem's "urban mining" technology can secure the supply of affordable REEs, especially coast-to-coast in North America, where several tons of REE-rich phosphor powders are generated monthly by recyclers. Moreover, because of the limited number of REEs present and the very small-scale operation required (50 to 300 kg/wk of mercury-phosphor dust), existing and well-known REE separation technologies can be used directly downstream without impacting the operating costs significantly, says Cardarelli.

In Electrochem's process (flowsheet) Hg is first removed by vacuum retorting. The Hg-free dust is then digested in a molten alkali hydroxide at 300–800°C, followed by a caustic fusion step, in which the melt resides less than 1 h in a batch furnace or a continuous rotating kiln. In this step, essentially all of the phosphate, silicates, aluminates, vanadates, chromates and borates, along with some manganese are separated. The solidified melt is then treated with an aqueous alkaline solution, which leaches out the P, Si, Al, B, Cr and V from the insoluble solid residues. Undissolved solids, including REEs with some Fe, Ni, Ti, Mg, Ca, Sr and Ba are filtered out, washed, oven dried and eventually calcined to oxidize Ce(III) to Ce(IV). The solids are then hot-acid leached, leaving behind the insoluble cerium (IV) oxide. The REEs (Y, La, Eu, Gd, Tb, and Er) remain in the pregnant leach solution, and are subsequently recovered by selective precipitation and ion exchange.

The process has been tested at a kilogram-scale prototype with materials originating from foreign and Canadian lamp recyclers, achieving 98–99% recovery of the REEs, says Cardarelli. A preliminary cost analysis indicate that REEs can be extracted for as low as \$7/kg of mercury phosphor dust, while the "basket value" of REEs contained can be as high as \$95/kg of dust, he says.

(Continued from p. 9)

cess Engineering at ETH Zurich (Switzerland; www.ethz.ch), led by professor Hyung Gyu Park. The membrane is made of a double layer of graphene in which precisely defined pores are etched by a technique known as focused ion-beam milling. The beam of helium or gallium ions can be controlled to create the desired number and size of pores in the graphene. Potential applications include gas separation, water filtration and even waterproof clothing, since the light, flexible membrane is said to be a thousand times more breathable than Gore-Tex. (For more on graphene, see pp. 13–17).

## Curing EPDM rubber

Researchers from Lanxess AG (Cologne, Germany; www.lanxess.com) have shown that zeolites can be used as a new co-activator for resol curing, enabling both high cure speeds and high curing efficiency. The company says resol curing opens up an

(Continues on p. 12)

### NEW WATER-TREATMENT SYSTEM

(Continued from p. 9)

a medium-pressure RO membrane system (flowrate of 600 m³/d). The dilution of seawater with the low-concentration brine from the sewage treatment process makes it possible to use the medium-pressure RO for desalination. As a result, power savings of more than 30% are possible due to the reduction of electricity needed for pumping water, compared to a high-pressure RO system. The dilution of seawater also reduces the size of the UF treatment needed, thereby lowering investment costs by 30% says NEDO.

The produced water can be supplied to the surrounding industrial facilities and used, for example, as boiler feedwater at the Kyushu Electric Power Co.

## Using algae to clean wastewater . . .

Technology developed by Algal Scientific Corp. (Northville, Mich.; www.algalscientific.com) uses algae to remove soluble organics, nitrogen and phosphorous from medium- to high-strength wastewaters in a single step. The technology is an alternative to multistep treatment processes involving the use of bacteria and chemical treatment.

The company's Hypertrophic Water Treatment Process essentially creates a controlled algal bloom in compact growth troughs. The specially selected and adapted algae species lowers BOD (biological oxygen demand) and nutrients from wastewater to allow the water to meet regulatory requirements or to reduce wastewater surcharges.

Because the algae treats organics and nutrients simultaneously, denitrification and phosphorous-removal process steps are eliminated and capital and operating costs are thus lowered. Also, the algal treatment process results in byproduct biomass, which can be used as fertilizer or burned for boiler heat, says Geoff Horst, Algal Scientific CEO.

Along with developing the algae species and adapting it to the process environment, the company has developed patented protocols for growing and harvesting the algae that minimize the physical footprint of the process. Algal Scientific is now offering licenses for its wastewater treatment technology.

## . . . and to make beta-1,3- glucan

With a separate algae-based technology, Algal Scientific (see previous story) has invented a sterile fermentation process for making beta-1,3-glucan from algae that has lower costs than those associated with fermenting the molecule with yeast.

Beta-1,3-glucan is a polysaccharide that boosts immune-system function in humans and livestock animals. As a feed supplement, beta-glucan can eliminate the use of antibiotics as growth stimulants, a practice that promotes the evolution of antibiotic-resistant bacterial strains.

Traditionally, beta-1,3-glucan has been made through yeast fermentation, but the process suffers from a number of limitations. "Yeast-derived beta-glucan makes up only about 5 to 15% of cell weight, and the compound is locked up in the yeast cell wall, which lowers its bioavailability," explains Algal Scientific's Horst.

Over 50 wt.% of Algal Scientific's proprietary algae strain consists of beta-glucan after fermentation, and the compound is more bioavailable in the highly digestible algae meal the company manufactures. Yeast-based processes involve costly extraction steps in which the cell wall must be destroyed and the product isolated. In Algal Scientific's algae fermentation, no extraction is needed, so the product can be manufactured less expensively, Horst says, making it cost-effective for a wider range of applications than when it is made by yeast.

Algal Scientific started up a commercial production facility for its beta-glucan products earlier this year, and now offers three beta-glucan powders that can be mixed into animal feeds or further refined for inclusion in nutraceuticals.

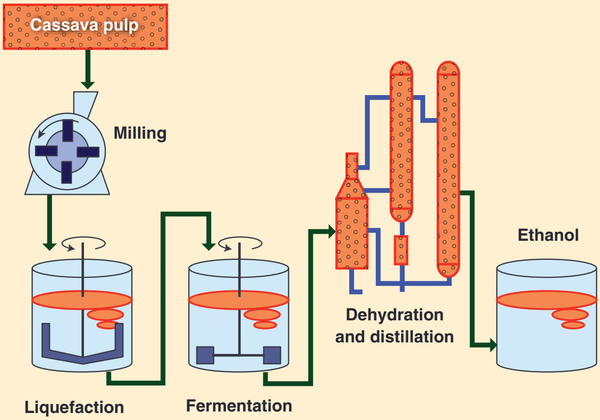
## This PEM fuel cell has $H_2$ storage built-in

Conventional hydrogen-based systems designed to store electrical energy (from solar arrays, for example) typically feed electricity to a proton-exchange-membrane (PEM) electrolyzer to generate $H_2$ from water. This $H_2$ is then compressed and stored in a cylinder, or as a metal hydride in a canister containing a powdered metallic alloy. When electricity is required, $H_2$ is supplied from the storage cylinder to a PEM fuel cell, along with $O_2$ from air, and electricity is generated again. The conversion efficiencies in the various stages of such a system lead to an overall energy efficiency of less than 50%. To improve this efficiency, a reversible PEM fuel cell with integrated metal-hydride hydrogen storage — a "proton flow battery" — has been designed by researchers from RMIT University (Melbourne, Australia; www.rmit.edu.au), led by professor John Andrews.

In the proton flow battery, many steps in the conventional process that incur net energy losses and irreversible entropy gains are completely avoided. The researchers claim their system's roundtrip energy efficiency can be comparable with that of the best battery types, such as lithium ion (about 70 to 80%), while storing more energy per unit mass and volume. The system can operate either as an electrolyzer to split water or as a fuel cell to generate electricity. In electrolyzer mode, $H^+$ ions emerging from the membrane enter the solid storage directly and then react with electrons and storage-material atoms to form a hydride, without producing any $H_2$ gas. In fuel cell mode, protons coming out of the solid storage pass through the membrane to form water molecules and generate electricity.

The proton flow battery features a composite metal-hydride, Nafion hydrogen-storage electrode. The Nafion provides a continuous polymer medium interspersed between the metal particles in the electrode. This provides pathways for conducting protons between the Nafion membrane of the cell itself and sites on the metal particles where absorption of the hydrogen atoms can take place.

## This demonstration plant makes bioethanol from tapioca residue




In April, a demonstration plant started up that makes bioethanol from cassava residue — a byproduct of tapioca production. The demonstration plant, located at the cassava starch plant of EBP Ethanol Co. in Sa Kaeo Province, Thailand, is part of a four-year, $7-million project of New Energy and Industrial Technology Development Organization (NEDO) and Thailand's National Innovation Agency. The project aims to demonstrate the effectiveness of technologies to efficiently produce bioethanol from cassava residue using newly developed heat-tolerant yeast, which can be used for the fermentation of cassava materials containing more than 30% fibers at temperatures over 40°C. The process (flowsheet) is able to treat the highly viscous pulp without additional pretreatment. The demonstration plant will operate until February 2016. NEDO aims to disseminate the new technologies throughout Thailand, as well as in ASEAN (Association of Southeast Asian Nations) countries where cassava plants are widely cultivated.

The project started in 2012 with the installation of a pilot plant at EBP Ethanol Co. NEDO selected Sapporo Breweries Ltd. and Iwata Chemical Co. as partner companies, and aims to produce 80,000 L/yr of bioethanol by processing 1,000 ton/yr of cassava pulp (wet).

Thailand is the largest cassava starch (tapioca) exporter in the world, and a large amount of cassava pulp, a residue obtained after starch extraction, is normally discarded. The project aims to contribute to increased biofuel production in Thailand by utilizing an untapped resource that does not compete with food supplies. It is expected that Thailand's annual cassava pulp generation of 2.0 million tons (as of 2012) will be utilized to produce approximately 1.8-billion L/d (656 billion L/yr) of bioethanol. Thailand's Dept. of Alternative Energy Development and Efficiency (DEDE) announced that the Thai government has set a target to supply 25% of the country's energy consumption from a renewable energy mix, which includes an increase of daily bioethanol production capacity from 2.5 billion L/d in 2013, to 9 billion L/d by 2021.

## Imitating nature leads to a better catalyst

An inexpensive and scalable composite catalyst that is said to outperform platinum for oxygen reduction in metal-air batteries and fuel cells has been developed by a research team led by professor Jaephil Cho from Ulsan National Institute for Science and Technology (Ulsan, South Korea; www.unist.ac.kr). The team includes people from Pohang Accelerator Laboratory (Pohang, South Korea), Los Alamos National Laboratory (Los Alamos, N.M.), Georgia Institute of Technology (Atlanta, Ga.) and SRM University (Kattankulathur, India).

Polarization due to the oxygen-reduction reaction contributes significantly to the energy efficiency of fuel cells and metal-air batteries. Although Pt and its alloys are the most efficient catalysts for activation of the O=O bond, their application is limited by high costs and scarce reserves.

The new catalyst — iron phthalocyanine (FePc), with an axial ligand anchored on single-walled carbon nanotubes (CNTs) — has shown a higher electrocatalytic activity for oxygen reduction than traditional Pt on carbon catalysts. The catalyst has shown exceptional durability and electrocatalytic activity in alkaline media. It has also achieved a long cycle life, attaining more than 1,000 cycles in a durability test.

The researchers were inspired by the unique feature of the active site in cytochrome c oxidase, which also contains an active site with an iron(II)-porphyrin structure. Unlike the synthetic catalysts, the iron center in biological systems contains a five-coordinated structure with an axial ligand from the backside. The team used pyridine-functionalized CNTs to anchor FePc molecules and provide the axial ligand for the iron center. At the same time, the CNTs provide an easy pathway for fast electron transfer from the current collector to the catalyst's active sites. ■

(*Continued from p. 10*)

alternative approach to EPDM (ethylene propylene diene terpolymer) rubber processing aside from the established sulfur-vulcanization and peroxide curing, which are the main crosslinking technologies for EPDM rubber. And although the company is focusing on EPDM, the activation is applicable for the resol curing of other types of rubber, including (X)IIR (isobutylene-isoprene rubber), CR (chloroprene rubber), (H)NBR (hydrogenated nitrile rubber), SBR (styrene-butadiene rubber) and NR (natural rubber). Depending on the particular resol curing system and rubber investigated, the cure rate is strongly increased, which means a reduction of scorch and vulcanization times up to 75% says the company. The final degree of crosslinking is also enhanced by a factor of two, the company says. ❏

# GRAPPLING WITH GRAPHENE: THE RACE TO COMMERCIALIZATION

**Touted as a wonder material, graphene is a key focus of scientists and engineers around the world**

In the ten years since its first isolation by a pair of Russian physicists, the single-atom-thick carbon allotrope known as graphene has been extolled as a "miracle material" for everything from television screens to tennis rackets to superabsorbent fabrics. What excites scientists so much about graphene is its myriad of impressive physical properties, some of which are illustrated in Figure 1. Its unique ability to be flexible, yet strong and light, all while conducting heat and electricity better than typically used materials, have provided scientists and engineers with countless research and development opportunities, one of the most prominent being the quest for economically feasible production processes for graphene.

While graphene's applications are seemingly endless, its effect on electronics, particularly consumer products, is gaining the most attention in the media. However, this material also holds much potential as a solution in many other industries. This article explores some of the potential uses for graphene in the chemical process industries (CPI), including biological and chemical sensors, water purification and desalination, energy storage and thermal interface materials. It also examines some innovative graphene-production processes.

The European Commission recognized graphene's revolutionary potential and launched the Graphene Flagship (www.graphene-flagship.eu) program in October 2013. The Graphene Flagship promotes industrial and academic collaboration for research and eventual commercialization of graphene-based technologies and products. Funded with €1 billion, the program is a consortium of companies and universities from 17 different countries, who, over the next 10 years, will be developing prototypes for graphene-based products and materials. Andrea C. Ferrari, professor of nanotechnology at Cambridge University (Cambridge, U.K.; www.cam.ac.uk), director of the Cambridge Graphene Center and chair of the Graphene Flagship Executive Board, stresses that a key aspect of the Graphene Flagship is its focus not only on pure graphene, but on the entire family of graphene-related materials, consisting of over 500 layered materials. The various members of the Graphene Flagship are producing graphene in solution, dispersion and polymer forms, and can manufacture it by the ton, liter or square meter, depending on format, for numerous applications.

According to Ferrari, the Graphene Flagship projects range from those that are very near commercial application to some that are a

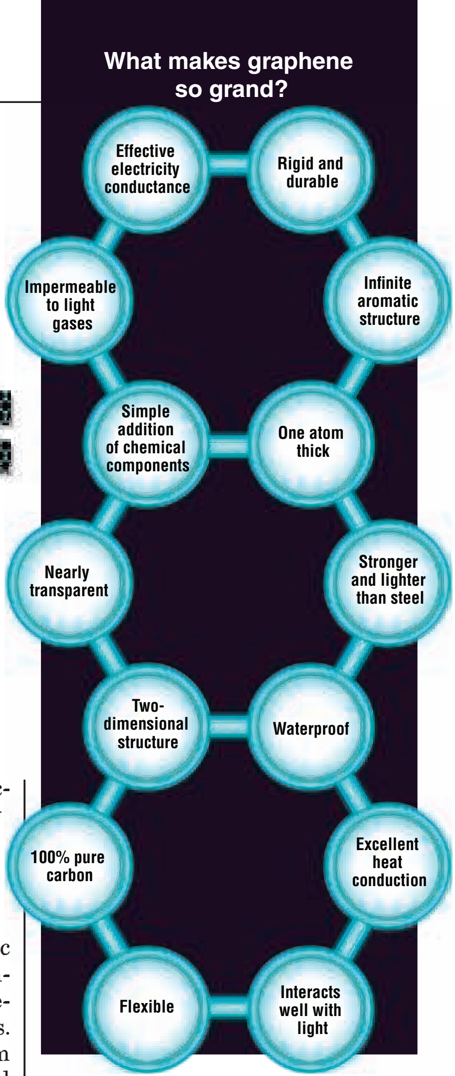



**FIGURE 1.** Graphene's unique and abundant desirable properties could provide never-before-seen solutions in a wide range of industries

decade or more away from commercial adoption. He says some of the work that is closest to commercialization involves the use of graphene composites in flexible, bendable or wearable electronics. Figure 2 shows a more detailed timeline of the Graphene Flagship's predicted commercialization progress for various applications.

## Energy storage and beyond

One of the groups associated with the Graphene Flagship is set to capitalize on graphene's extreme conductivity with a new graphene-production process. In a research partnership first formed in 2012, specialty chemicals company Thomas Swan & Co. (Consett, U.K.; www.thomas-swan.co.uk) and the Advanced Materials and Bioengi-

neering (AMBER) Center at Trinity College (Dublin, Ireland; www.tcd.ie) set out to develop a simple and scalable manufacturing process for graphene. Their findings led them to a liquid-phase, high-shear exfoliation process employing a simple industrial mixer. The process results in graphene nanoplatelets with very few defects, as explained by Andy Goodwin, commercial director of Thomas Swan's Advanced Materials Division. "The exfoliation is a physical process, and therefore we observe no oxidation or damage to the sp2 carbon platelets. The result is a low-defect graphene nanoplatelet with superior conductivity performance," Goodwin says. The simplicity of the exfoliation process makes it unique and an especially viable option in the high-cost world of graphene production. "This process uses no aggressive chemicals, no ultrasonics, no elevated pressures or temperatures. A range of solvents can be used. For obvious reasons we prefer to use water." Goodwin says. Thomas Swan holds an exclusive license to this technology.

In April 2014, Thomas Swan began producing and selling graphene in small quantities under the commercial name Elicarb from its initial pilot plant, located in Consett, U.K. The company predicts that by late 2014, the pilot plant will reach a graphene production capacity of 1 kg/d. Available in powder format or as a water/solvent dispersion, Elicarb consists of graphene platelets that are five to seven carbon layers in depth, with an average X-Y platelet dimension of around 1,000 nanometers (nm). Figure 3 shows eight transmission electron micrograph images of Elicarb graphene powder. One of the hallmarks of the material is the consistency in particle size between batches. With Elicarb first introduced at Berlin's Printed Electronics Europe tradeshow in April 2014, Thomas Swan plans to capitalize on graphene's excellent electrical and thermal conductivity and tailor its materials for use in capacitors, batteries, barriers and separators, conductive inks, and for thermal-management applications.

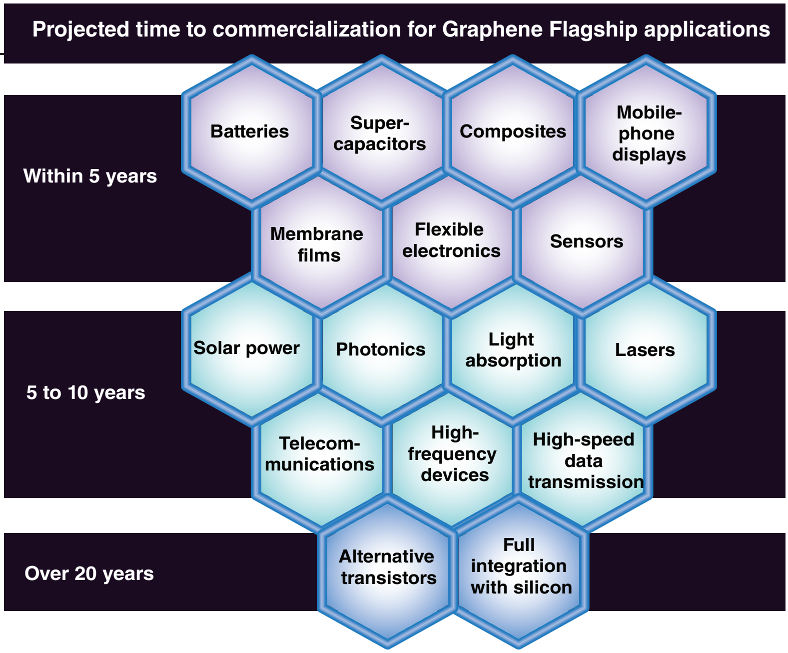



**FIGURE 2.** The Graphene Flagship involves projects that are at many different stages of development and commercial adoption

Another group focusing on graphene's energy conductivity has set their sights on solar-power applications. In a collaboration formed under the U.K.-India Education and Research Initiative (UK-IERI; www.ukieri.org), the University of Surrey (Surrey, U.K.; www.surrey.ac.uk), Tata Steel (Mumbai, India; www.tatasteel.com) and the University of Hyderabad (Hyderabad, India; www.uohyd.ac.in) are researching graphene-based carbon nanomaterials that could provide a platform for improved energy generation and storage. Their work focuses on whether graphene materials can help overcome some of the drawbacks of traditional solar photovoltaic systems: deployment costs and phase mismatch between peak energy production and peak energy usage.

The group touts graphene's conductivity, surface area and form factor as exploitable properties that can be adapted for energy storage and harvesting. The group's advanced solar cells — known as "inorganics-in-organics" — consist of large-area graphene materials that interface with bipolar steel plates (Figure 4). By integrating the graphene nanomaterials into an existing energy-generation platform, the feasibility of building solar-energy-generation systems on these plates can be directly evaluated. The research also investigates the interface between the steel substrate and the graphene materials. Additionally, the project involves methods to make the production and processing of the cells in large quantities economical.

## Sensing graphene

Another company that is beginning to produce graphene at larger and larger scales is Graphene Frontiers (Philadelphia, Pa.; www.graphenefrontiers.com), which in August 2013 received a grant from the National Science Foundation (NSF; Arlington, Va.; www.nsf.gov) that will help it take its innovative graphene-production process from batch to continuous production. Focused on creating large-area graphene tailored for use in specialized biosensors, Graphene Frontiers produces its graphene in sheets that are typically 5–12 $in^2$ in size, but hopes that the NSF grant will not only boost production capacity and lower operating costs, but also allow them to create even larger sheets.

Like other graphene-production processes, Graphene Frontiers employs a chemical-vapor deposition (CVD) technique, but its process is especially unique because it can be carried out at atmospheric pressure in a CVD furnace (Figure 5), rather than in a vacuum, allowing for roll-

Source: Thomas Swan

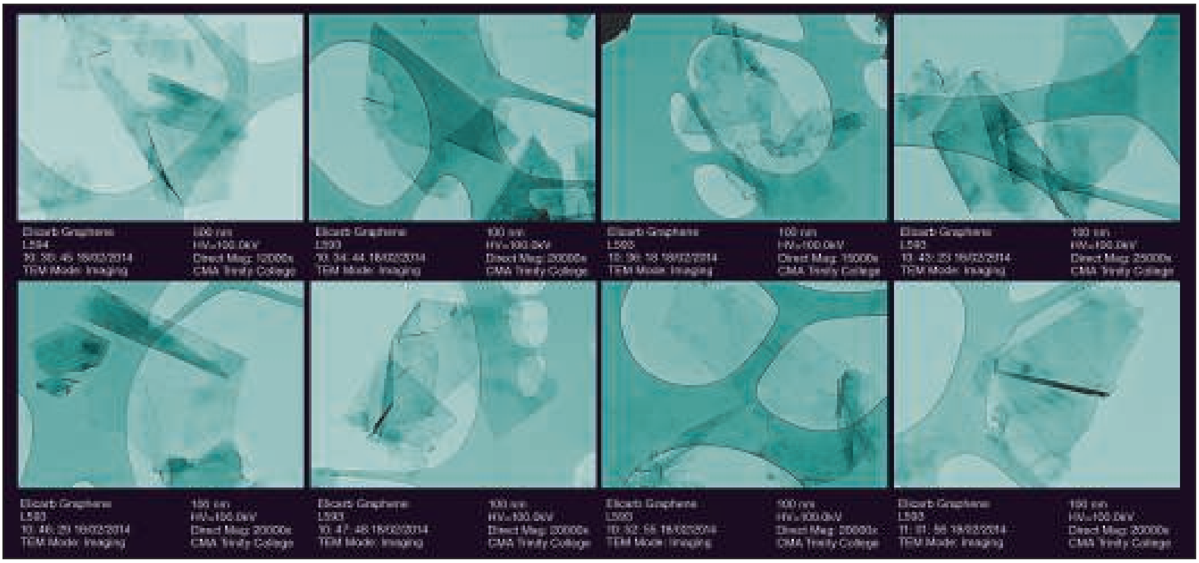


**FIGURE 3.** Eight transmission electron micrograph images of Elicarb graphene powder show typical graphene nanoplatelets

University of Surrey

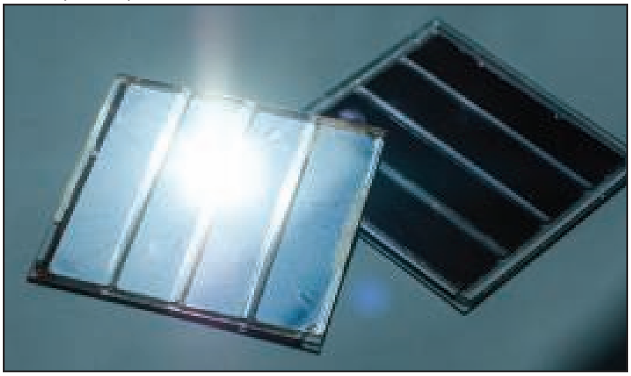


**FIGURE 4.** Large-area (39 $cm^2$) organic solar cell modules that incorporate carbon nanotechnology have been produced at the Advanced Technology Institute, University of Surrey

Graphene Frontiers

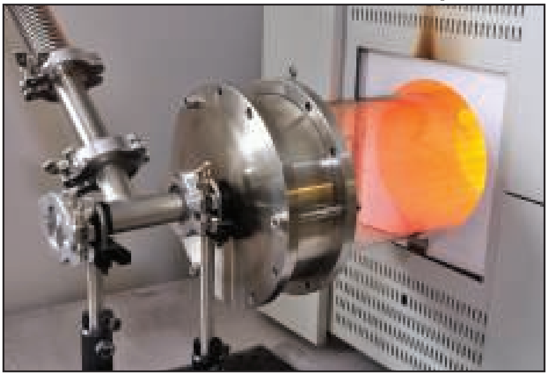


**FIGURE 5.** The CVD furnace used in Graphene Frontiers' production process is operated at atmospheric pressure

to-roll production. The elimination of vacuum requirements helps drive down costs when compared to other companies' graphene CVD processes, explains Paige Boehmcke, senior product manager for Graphene Frontiers. Boehmcke also says that continuous production at a commercial scale is expected to be achieved at Graphene Frontiers' Philadelphia facility in about 15 months, with a projected manufacturing cost of less than \$0.01/$cm^2$ of graphene. Only 10 µm wide, the biosensors produced by Graphene Frontiers (shown in a testing array in Figure 6) are field-effect transistors with graphene channels instead of traditional silicon channels. Receptor molecules can be directly attached to the graphene channel. Concentration of attached antigens can be determined from these sensors through signal analysis, as the current through the transistor is altered by the binding activity to the receptors. The two-dimensional nature of the graphene makes these sensors ultrasensitive to binding behavior, Boehmcke explains, and allows them to function well even at very low concentrations. Graphene Frontiers is beginning to form agreements with some diagnostics companies to commercialize the biosensors. Also in the early development stage at Graphene Frontiers are graphene-based chemical sensors, which could be used for air-quality monitoring, hazard detection and many other functions.

## Desalination and purification

One of the most promising uses for graphene outside of electronics is water purification and desalination. Because of its inherent structure and hydrophobicity, graphene exhibits its excellent selectivity as a porous material, even with extremely small particles. A group from Rensselaer Polytechnic Institute (RPI; Troy, N.Y.; www.rpi.edu), in collaboration with Oak Ridge National Laboratory, (ORNL; Oak Ridge, Tenn.; www.ornl.gov) has undertaken an ambitious computational modeling

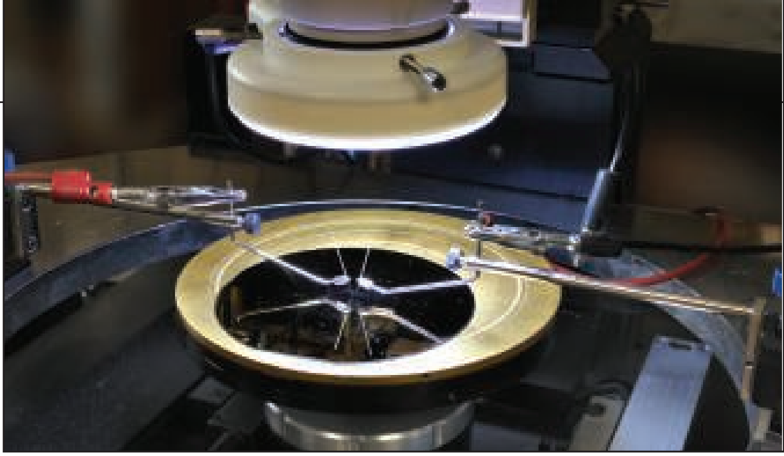


**FIGURE 6.** An array of graphene field-effect transistor-based sensors are tested using a four-point probe

Rensselaer Polytechnic Institute

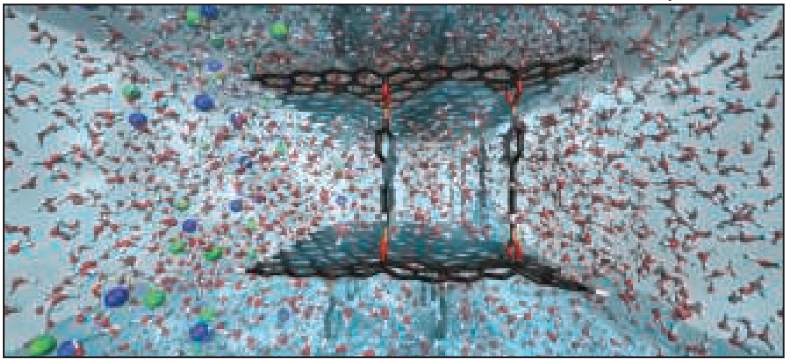


**FIGURE 7.** Salt, represented by sodium and chloride ions (shown as large blue and green spheres, respectively) is present in the water (in red) on the left of the GOF membrane (in black), but the salt is removed as the water passes through the GOF membrane

project to evaluate graphene materials' suitability for purification applications, particularly for desalination purposes. The specific materials being investigated are graphene oxide frameworks (GOFs) — a class of synthesized nanoporous materials made of graphene oxide layers that are covalently interconnected by linking pillaring units, explains Vincent Meunier, professor of physics at RPI. Meunier, along with postdoctoral researcher Adrien Nicolaï and Bobby Sumpter of ORNL, have been examining how these pillaring units — which are composed of linear boronic acid and are typically 1–2 nm long — can be configured between the graphene oxide layers to "tune" a GOF's properties for use in desalination applications. "By selecting proper linker concentration, one can tune a large set of geometrical parameters of GOF materials, such as the pore volume," Meunier says. This versatility makes GOFs especially suited for a broad range of water-filtration processes, as well as many other ultrafiltration applications in the chemical processing, pharmaceutical and biomedical industries. In their simulation-based study, it was found that, when water was passed through a GOF membrane, nearly all of the salt ions were removed at a much faster rate than typical reverse-osmosis membranes. (Figure 7).

The next steps for this research from a simulation standpoint will be to examine GOFs' mechanical stability and the effect of structural defects on their desalination ability. Also, the production process for GOFs must be further evaluated to determine its scalability for industrial manufacturing. Overall, though, Meunier and Nicolaï are optimistic, saying that "Some technical issues might arise, but it seems that, given the quality of existing material made in the laboratory, there is solid hope for actual implementation of GOFs for filtration capabilities at large scales."

## Grounding graphene

While graphene may present many opportunities for revolutionary technological breakthroughs, many unknowns still remain with its production and use. In addition to the near-daily announcements of new, exciting uses for graphene, there are also some high-profile projects that are examining negative aspects of graphene and its related two-dimensional materials. Findings from the University of California, Riverside (Riverside, Calif.; www.ucr.edu) regarding the stability of graphene oxide in water, and the potential negative environmental impacts therein, have garnered much press attention. Also gaining traction is work published by Georgia Institute of Technology (Atlanta, Ga.; www.gatech.edu) and Rice University (Houston; www.rice.edu) that shines light on the potential for defects, structural weakness and brittleness in graphene, warning that manufacturers must properly evaluate materials for specific purposes before rushing to mass production. Brown University's (Providence, R.I.; www.brown.edu) college of medicine has been studying the cellular toxicity of graphene materials used for medical devices. Concerns have also been raised about end-of-life recycling options for graphene materials, since many products may be inherently non-biodegradable.

The Graphene Stakeholders Association (Buffalo, N.Y.; www.graphenestakeholders.org), a non-profit organization formed to promote responsible graphene developments, has embarked on a focused collaboration with the Arkansas Research Alliance (Conway, Ark.; www.aralliance.org) regarding the environmental, health and safety concerns associated with graphene. They hope that the initiative, while investigating important technical topics, will also foster informed discussions among industry professionals and the public in general about graphene and other two-dimensional materials. These projects will not likely put a damper on the flurry of research on graphene — they just serve to emphasize the importance of mindfulness in the quest to commercialize these unique graphene nanomaterials. ■

*Mary Page Bailey*

# SIZE REDUCTION RE-DO

## Improvements to grinding, milling and screening equipment help processors increase efficiency and reduce costs

Stedman

**FIGURE 1.** The H-Series impact mill is a multi-cage mill used for pulverizing, grinding, crushing and mixing abrasive and non-abrasive materials that are wet, sticky or dry

Sturtevant

**FIGURE 2.** The Micronizer jet mill can process chemicals to nanometer scale while providing a tight particle-size distribution

Fritsch

**FIGURE 3.** The premium line of planetary mills provides rotational speeds that allow ultra-fine grinding results down into the nano range

When it comes to size reduction, chemical processors seek to improve product quality, increase flexibility and uptime and, simultaneously, reduce costs. Fortunately, equipment manufacturers are working to develop grinding, milling and screening machines that can help chemical processors meet their size-reduction needs.

"Customers always want a better quality product, they always want to increase their output and they always want to do a better job, but they want to do it all at a lower cost," says Chris Nawalaniec, national sales manager at Stedman (Aurora, Ill.; www.stedman-machine.com). "Energy and maintenance costs are among critical operating expenses related to size-reduction equipment, but there are things we, as equipment manufacturers, are working on to help boost performance while reducing these costs."

### Quality, consistency & flexibility

Maximizing the first-yield pass through a milling machine is very important for processors interested in consistent size reduction and capacity, says Nawalaniec. Some of the traditional machines used for these operations are not necessarily the best equipment available for the job, he says. "The grinding elements in traditional machines are wheels and, as the wheels wear, the particle-size distribution of the product they are milling changes and the processor loses product capacity and consistency, which results in product output variation."

In an effort to combat this issue, Stedman developed updated versions of the vertical roller mill and cage mill. The vertical roller mill is an air-swept fine grinding system. According to Nawalaniec, it offers a particle-size distribution that is superior to that of traditional equipment because the consistent wear and long-life grinding zone components don't deteriorate as quickly as traditional wheel components, which allows this fine grinder to provide consistent results, increasing process efficiency and quality, for longer periods of time.

Similarly, the H-Series impact mill (Figure 1) is a multi-cage mill used for pulverizing, grinding, crushing and mixing abrasive and non-abrasive materials that are wet, sticky or dry. It offers extended wear life via better designed parts and the ability to operate in reverse, which helps maintain stability in product capacity and particle-size consistency. The cage mill can handle up to 240 ton/h.

In addition to consistent particle

Munson Machinery

**FIGURE 4. Model SCC-15 screen classifying cutter cuts hard, soft and fibrous material into controlled particle sizes with minimal fines at high throughput rates**

size, Rich Robatzek, Micronizer product manager with Sturtevant (Hanover, Mass.; www.sturtevantinc.com) says low particle size is another critical element in chemical processing size reduction operations, which has driven equipment manufacturers to develop machines that can provide this capability. "Processing chemicals to low particle size increases the surface area, thus providing better control of properties and more predictable performance of their products," he says. "The ability to achieve lower particle sizes during size-reduction processes allows processors to attain desired results while using less material."

For this reason, Sturtevant's Micronizer jet mill (Figure 2) can process chemicals to nanoscale size while providing a tight particle-size distribution, according to Robatzek. The jet mill is operated by particle-on-particle attrition to grind without the introduction of heat or wear into the grinding process and is capable of particle reduction to 250 nm. There are no dead zones to trap material, no moving parts to wear, and no grinding media or lubrication to contaminate milled products. The one-step grinding and classifying is suitable for continuous or batch processes.

In several areas of the chemical process industries (CPI), flexibility in size reduction is also key to increasing efficiency of the process and quality of the final product, says David Ekstrom, president of Bematek (Salem, Mass.; www.bematek.com).

For instance, in the food industry, a processor might want large chunks for one product and smoother consistency for another, both of which may be run on the same line on alternating days or batches. In pharmaceuticals and personal care, processors may be looking to make some products very smooth and some less smooth, he explains. "So, what they want is a machine that gives them the ability to control the amount of shear action by selecting and easily changing different components, as well as the ability to change the speed of the motor, using a variable-frequency drive," says Ekstrom.

While Bematek's in-line colloid mills have been used for wet milling tasks, including primary particle-size reduction and particle deagglomeration for solid-in-liquid dispersions for some time, the now modular-engineered design allows optimal configuration and flexibility. Quick and easy in-field modifications are simplified so the colloid mill can be configured for any application a processor may need. To process multiple products, a variety of alternative rotor/stator designs are available and can be easily installed on-site to adapt the mill for a variety of products.

Fritsch GmbH (Idar-Oberstein, Germany; www.fritsch.de) also sees smaller particle size and flexibility as essential to successful size-reduction operations. The company's premium line of planetary mills (Figure 3) provides rotational speeds that allow ultra-fine grinding results down into the nanoscale range. To achieve the best grinding results, all grinding bowls and balls are available in different sizes and materials. For grinding with inert gas and for mechanical alloying, the Fritsch premium-line gassing liquid can be used. The Fritsch Easy GTM ensures simple and easy monitoring of thermal effects, physical and chemical reactions or increases or decreases in pressure due to con-

## WASTE NOT, WANT NOT

As landfill costs continue to escalate and the desire to reduce material costs increases, many chemical processors are trying to decrease the amount of waste material sent to landfills, while finding methods to re-use the waste. Size reduction equipment is finding employment in this new, niche application, says Munson Machinery's Stephen Knauth.

"Processors are trying to reduce the amount of product they send to landfill because they don't want to pay those costs anymore," he explains. "In addition, if the waste material is properly processed, they may be able to re-introduce it into their process or sell it, helping to boost their competitiveness in the marketplace."

An example of this type of processing might be in the plastics industry where instead of throwing away or landfilling trim scrap, they are cutting it down to smaller sizes and remelting it for use as an additive in another run. Pigment manufacturers, too, often recycle waste. "Instead of tossing away an off-spec run of pigment, they recycle it by bringing it down in size again and creating a new color to sell," explains Knauth.

Titan 20 Shredders (Figure 6) from Munson are frequently used in recycling of product, says Knauth, because they are capable of cutting costs by reducing bulk waste, scrap and out-of-spec materials by 50 to 80%.

This machine shreds plastics and purging, wood products, carpets, glass, aluminum, PET bottles and various industrial and manufacturing waste products. It handles size reduction with low speed, high torque and little or no temperature rise, while reducing bulk volume up to 75 or 80%. The double-rotor shredder design creates an intensive shearing and cutting action with a self-feeding hopper design. "It was made to handle recycling applications with low maintenance, low energy usage and the ability to help processors reduce costs associated with waste," says Knauth. ❑

tinuous measurement of gas pressure and temperature directly in the grinding bowl. And, the mill is automatically controlled in a manner so that set parameters are not exceeded. When grinding in suspension, the Fritsch special emptying device can be used to provide quick and easy separation of the grinding balls and suspension. This range of accessories provides flexible and optimal grinding for a variety of applications, says the company.

### Reducing costs

One of the biggest complaints regarding size-reduction equipment is that they often require frequent maintenance due to wear and tear. "One of the most common issues faced by every processor is maintenance," says Stephen Knauth, general sales manager with Munson Machinery (Utica, N.Y.; www.munsonmachinery.com). "All processora are looking for equipment that is more durable and easier to maintain because they no longer have the budgets to maintain equipment like they used to. So, many are upgrading from an inefficient system to one that is easier and less costly to maintain."

Not only are machines being designed for easier access and simplified maintenance using front panel access, but Knauth says Munson Machinery has also been focusing on creating more wear resistance in its machines to reduce the amount of maintenance that is necessary to begin with. "We've upped the hardness on tips, we're using more coatings than we did before, using wear liners and making the equipment more user friendly in an effort to decrease the amount of time spent maintaining it, and to reduce the time spent conducting changeovers if the machine does wear out," he says.

One such example is Munson's Model SCC-15 screen classifying cutter (Figure 4), which cuts hard, soft and fibrous material into controlled particle sizes with minimal fines at high rates. The cutter features a helical rotor design with dozens of cutter heads attached to a helical array of staggered holders called "interconnected parallelograms" to continuously shear oversize materials against twin, stationary bed knives. The cutter tips are available in a variety of materials designed for wear resistance, including tungsten carbide, and can be slid onto blade holders and secured easily with one retaining socket-head screw for rapid replacement.

Triple/S Dynamics

**FIGURE 5.** **Unlike vibrating screens that shake the entire screen box, the Overstrom fine mesh screener vibrates only the screen cloth, which offers many benefits**

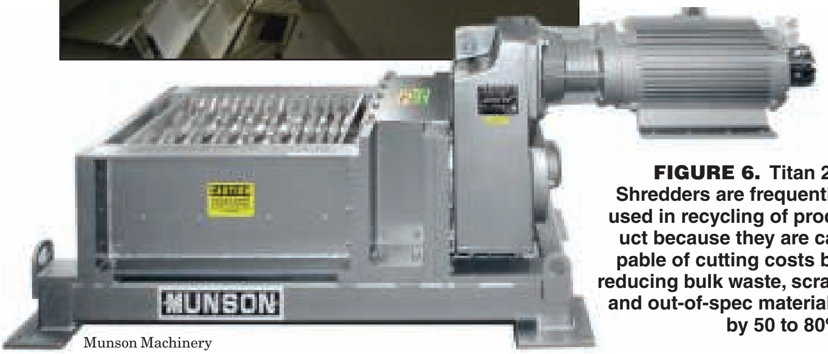



**FIGURE 6.** **Titan 20 Shredders are frequently used in recycling of product because they are capable of cutting costs by reducing bulk waste, scrap and out-of-spec materials by 50 to 80%**

Munson Machinery

The high energy consumption of size-reduction equipment is another common complaint among chemical processors, says Knauth. As a result, smarter, more-efficient equipment designs are being developed and employed. Munson's centrifugal impact mills are among such machines. The mill is capable of providing controllable particle-size distribution and is easy to clean, but a smart design also makes it more energy efficient. Metered material is gravity-fed through the centrally located inlet of the stator disc. Centrifugal forces accelerate the material and launch it into the impact zone. The action created by the stationary and rotating pins creates a "treacherous path" for material to pass through. Achieving the desired, tight particle-size distribution is obtained by controlling the rotor speed. Varying the rotor speed between a few hundred rpm and up to 5,400 rpm provides the flexibility to use the machine for coarse grinding and de-agglomeration, or as a fine grinding mill, but also increases its energy efficiency in these applications.

### Smarter screening

While screening is a different operation than size reduction, there are some similar challenges, including the need to achieve higher capacities while decreasing energy consumption and maintenance costs, according to Chris Meadows, a market development specialist, and Ferdinand Schon, product manager for screening solutions, with Triple/S Dynamics (Dallas, Tex.; www.sssdynamics.com).

To help processors increase capacity, the company developed its Overstrom fine-mesh screener (Figure 5). Unlike vibrating screens that shake the entire screen box, the Overstrom vibrates only the screen cloth, which offers many benefits including lower power consumption, low maintenance requirements and an open-hood design to allow fast access to lower decks.

The screener can screen down to 325 mesh, which was previously only possible with an air classifier, and increases the efficiency of the separation, according to Meadows. The screener can be configured as a single-deck unit or stacked up to five decks, each independent from one another, which provides more screen area in the same footprint, according to Schon.

Screen blinding, another common screening issue, is also resolved via the vibration of the screen cloth, and thus the efficiency of the screening process is also improved. Because the speed of the motor is ramped up only at certain intervals that can be set, it helps get the particles off the screen, while reducing energy use.■

*Joy LePree*

Electro-Chemical Devices

908 Devices

Mettler-Toledo

# FOCUS ON Analyzers

Automation Products



JM Science

## An all-in-one sulfide analyzer helps control odor

The new SMS-22 Sulfide Ion Measurement System (photo) can be quickly installed with no special tools, features user-selectable automated sequential sampling of sulfide ions in water and lowers plant technician and supply maintenance costs. The SMS-22 saves time and labor costs for municipal water operations by completing all the necessary steps to measure sulfide ions in a water sample. Because sulfide ions only exist at high pH values, this is a multi-step process. The SMS-22 conditions the sample, measures the sulfide, neutralizes the sample, measures the pH and drains and rinses the cell. After measurement, the highly caustic sample pH is reduced to a safe level near pH 8. The SMS-22 uses a sulfide ion selective electrode (ISE) to measure the total amount of sulfide present in the sample. — *Electro-Chemical Devices, Inc., Irvine, Calif.*
**www.ecdi.com**

## Density measurement with no moving parts

The Dynatrol CL 10-HY Density Cell and Series 2000 Density Digital Converter (photo) can be used to measure the density of ammonium hydroxide, caustic soda, ethylene glycol, hydrochloric acid, methanol, nitric acid, oleum, phosphoric acid, sodium chloride, sodium hydroxide, sulfuric acid and many more. These cells are available in a full range of corrosion-resistant materials and are both weather-tight and explosion-proof. With no moving parts, the unit it is virtually wear-free ensuring a long operating life. The Dynatrol Digital Density Converter has an onboard microcontroller, and the provided software calculates the specific gravity, percent concentration, and other parameters using the density and temperature information generated by the CL10-HY. A two-line LCD displays temperature, density, product frequency and status. — *Automation Products, Inc., Dynatrol Div., Houston*
**www.dynatrolusa.com**

## A handheld mass spectrometer for use in the field

The M908 (photo) is said to be the world's first truly handheld tool utilizing high-pressure mass spectrometry (HPMS). Specifically engineered for first responders and military teams, the M908 provides accurate, immediate detection and identification of trace-level chemical and explosive hazards at the point-of-action. The patented HPMS technology that powers the M908 uses diminutive ion traps that measure less than a millimeter in diameter and that are capable of operation at 10,000 times higher pressure than those in conventional MS systems. This enables the use of small, rugged vacuum pumps, micro-ionizers, detectors and efficient electronics. — *908 Devices, Boston, Mass.*
**www.908devices.com**

## Coulometric titration for analyzing ammonium-nitrogen

The AT-2000 Ammonia Analyzer (photo) is based on coulometric titration technology. It can measure ammonium-nitrogen ($NH_4$-N) swiftly, accurately and easily without any specific skill on the part of the end-user. This system has a wide measurement range from low to high end with accurate measurement and is not affected by turbidity or color of the sample. It has a short measurement time of typically one minute. A very small volume of sample is necessary (0.1, 1 or 10 mL) and calibration is not required prior to the sample run. The elec-



*Note*: For more information, circle the 3-digit number on p. 56, or use the website designation.


E Instruments International


Wilks Enterprise

Michell Instruments Group

trode is stain resistant, has little influence on temperature change, and has a long life compared to ion-selective electrodes. Only one type of environmentally friendly reagent (dedicated electrolyte) is used, which means lower costs for budget-minded laboratories. — *JM Science, Inc., Grand Island, N.Y.*
**www.jmscience.com**

**Transfer data easily with this analyzer**
The latest models of Professional moisture analyzers (photo, p. 22) from this company feature USB, Ethernet and WLAN connectivity for easy data transfer, and now can send measurement reports directly to a networked page printer. The built-in instrument tests are easy to perform and ensure accurate results. With a five-click guided procedure, the performance of all the instruments together can be qualified thanks to the certified reference substance SmartCal. Professional moisture analyzers are tested as with SmartCal to verify the performance of the instrument's overall functionality. After following simple instructions, normalization for ambient conditions is automatic thanks to a new plug-in humidity and temperature sensor. — *Mettler-Toledo AG, Greifensee, Switzerland*
**www.mt.com/moisture**

**This binary gas analyzer offers longterm reliability**
The XTC601 Thermal Conductivity Analyzer (photo) uses thermal conductivity technology to detect percent levels of a target gas mixed with another background gas. Typical applications would be: $H_2$ generator cooling systems; $H_2$ in heat-treatment furnaces; $H_2$ in $NH_3$, methanol or chlorine; $CO_2$ in fermentation process; $CO_2$ in biogas; $H_2$ in blast-furnace top gas; $H_2$ in hydrocarbons; He recovery; or $N_2$ in $CO_2$. The key advantages of the XTC601 binary gas analyzer is its low cost of ownership and compact dimensions (particularly in Ex d format), says the company. Thermal conductivity sensors are non-depleting which gives the analyzer minimal maintenance costs, and longterm reliable operation. The analyzer provides measurements with an accuracy of 2% full scale and a stability of ±0.5% per month of span. The sensor is highly flexible and is capable of measuring gases from 0–5% up to 0–100% (calibrated to suit application). — *Michell Instruments Group, Ely, U.K.*
**www.michell.com/uk**

**This portable IR device quickly measures sub-ppm oil-in-water**
The new InfraCal 2 Field Portable IR (infrared) Analyzer (photo) can measure down to 0.1 ppm oil-in-water accurately, onsite and in less than 15 minutes. The analyzer combines improved electronics — which significantly increase signal-to-noise ratio for greater performance — with a touchscreen intuitive display that allows for multiple calibrations, internal data logging and data transfer capabilities, as well as alarm functions to ensure results are acceptable, and security and password settings to avoid unauthorized changes. — *Wilks Enterprise, Inc., East Norwalk, Conn.*
**www.wilksir.com**

**A complete combustion-gas and emissions analyzer**
The E8500 combustion analyzer (photo) is a complete portable tool for U.S. Environmental Protection Agency (EPA) compliance-level emissions monitoring and testing. The E8500 is suitable for regulatory and maintenance use in boiler, burner, engine, turbine, furnace and other combustion applications. The E8500 also includes electrochemical sensors (for $O_2$, CO, NO, $NO_2$, $SO_2$, $H_2S$); non-dispersive IR sensors for $CO_2$, $C_xH_y$, high CO; and is low NOx and true NOx capable. The system has a realtime PC-software package with Bluetooth, and a wireless remote printer. — *E Instruments International, Langhorne, Pa.*
**www.e-inst.com**

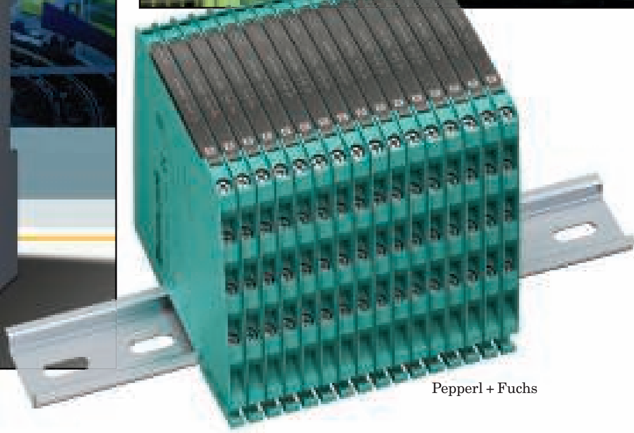
**An orthophosphate analyzer that stands alone**
The P 700 IQ orthophosphate analyzer can be used as a stand-alone analyzer or in conjunction with other sensors in an IQ SensorNet 2020 XT continuous monitoring and control system. This analyzer can run itself continuously for up to 8 months without needing a user calibration or reagent changes, says the company. Because of its two measuring ranges, the P 700 can be used throughout a wastewater treatment plant to measure orthophosphates — from pre-sedimentation to the biological tanks and to the effluent. It provides continuous data to help improve operational efficiency as well as verification of phosphate elimination. — *Xylem Analytics, Beverly, Mass.*
**www.xylemanalytics.com** ■

*Gerald Ondrey*

Prüftechnik

# JUNE New Products

Siemens AG



Pepperl + Fuchs

Bürkert Fluid Control Systems

## A new spin on flow metering

The new FLOWave (photo) flowmeter uses a new technology based on surface acoustic waves (SAW). One of the outstanding features of SAW for inline flow measurement of liquids is that it operates without any components or constrictions inside the measuring tube. The interior surface can be manufactured with the same surface finish applied to the rest of the pipeline. In terms of hygiene, cleaning and flow conditions, there are absolutely no differences compared to any other straight piece of pipe. There is no pressure drop and no effects of the fluid on sensor elements. Maintenance is minimal — if at all, says the manufacturer. In addition to volume flow, temperature and density can be measured, too. Based on this data, the mass flowrate is calculated. The new flowmeter principle works even with stagnant liquids, so results are available even with the smallest flow volumes. The high excitation frequency of 1.5 MHz avoids disturbances due to inherent vibration in the plant. Magnetic and electrical effects have no influence on measurements. This technology does not depend on the conductivity of the fluid, which significantly expands its scope of application. SAW is the only flowmeter technology that can distinguish between laminar and turbulent flows, says the company. — *Bürkert Fluid Control Systems, Ingelfingen, Germany*
**www.burkert.com**

## A new IO-Link master for the Simatic controller

This company has developed a new IO-Link master (photo) for its Simatic S7-1200 controller, based on the current IO-Link specification V1.1. The IO-Link master connects field devices to the controller via IO-Link. The field device parameters are automatically backed up in the controller. If the field device has to be replaced, all the parameters are automatically transferred to the new component, so the user does not need to back up the data. The new IO-Link master supports the rapid COM3 mode, provided as an option in the standard device, for short response times or for handling extensive data sets, as well as supporting devices with V1.0 IO-Link specifications. Thanks to the industry-wide IO-Link standard, switching devices and sensors can be connected to the control level with a low-cost point-to-point link. — *Siemens AG, Industry Sector, Industry Automation Div., Nuremberg, Germany*
**www.siemens.com/s7-1200**

## Introducing a new family of signal conditioners

The SC-System (photo) is a new family of signal conditioners, which was launched at the Hannover Trade Fair (April 7–11; Hannover, Germany). The SC-System uses high-quality three-way isolation between input, output and supply that withstands a working voltage of up to 300 V and a test voltage up to 2.5 kV. The low-power design, which ensures low self-heating, contributes to the wide temperature range of up to 70°C in which the products can be operated. Effective use of the switch cabinet space is achieved by a compact design: the housings, which are just 6-mm wide, reduce the space requirement of the signal conditioner modules to



*Note*: For more information, circle the 3-digit number on p. 56, or use the website designation.

Samson Controls

a minimum. The housing height is so low that the modules fit between the narrow-seated cable channels. — *Pepperl + Fuchs GmbH, Mannheim, Germany*
**www.pepperl-fuchs.com**

**Machine alignment with cardan shaft in place**

Premature wear of cardan shaft joints is one of the most common causes of machine downtimes. But cardan shaft misalignment is no longer a fatality. With patented brackets and new measurement methods (photo, p. 24), this company says it has reinvented cardan shaft alignment. No longer is cardan shaft removal necessary, along with the associated costs and risks (crane hire, safety hazards, machine damage and intensive manpower). Using the specially designed bracket with a rotating arm, cardan shaft alignment now takes a matter of minutes. The alignment condition is accurately measured only after as little as a 60 deg shaft rotation and the angular cardan misalignment can be corrected by moving the machine according to the displayed feet-correction values. The cardan shaft application is available with the company's Rotalign Ultra iS alignment platform as of firmware 3.03. — *Prüftechnik AG, Ismaning, Germany*
**www.pruftechnik.com**

**Precise, fast response when controlling large valves**

Larger valves (200 mm and above, with flowrates in excess of 200 $m^3/h$) tend to be fitted with pneumatic actuators. However, the air-output capacity required to achieve fast positioning in larger actuators is simply too high for normal positioners and accuracy can be compromised. An added complication in these situations is that the positioner controls a volume booster, which results in a much higher air capacity than the positioner and this can lead to an overshoot on responding to small signal changes. To overcome these problems, this company developed a control-valve positioner package (photo) that ensures precise positioning with fast response times, over the entire operating range, for example strokes of 60 mm and greater. The package consists of a positioner and a special hookup of

# New Products

Levelese

Morse Manufacturing

Charles Ross & Son

several volume boosters with different air-output capacities that perform this task. Closing and opening times of less than 2 s can be achieved over the entire positioning range. — *Samson Controls, Redhill, Surrey, U.K.*
**www.samsoncontrols.co.uk**

### A stainless-steel drum lifter that provides ample support

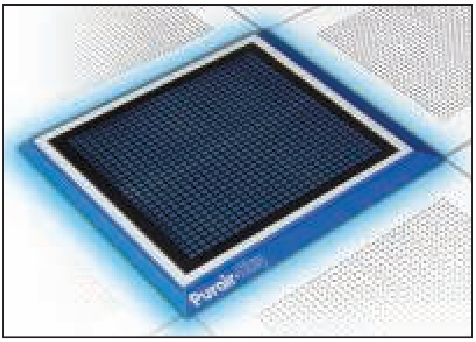
The 86-SS below-the-hook drum lifter (photo) is made from 304 stainless steel and provides lifting services for a wide range of industries, including construction, marine, oil-and-gas, food, pharmaceuticals and more. The stainless-steel construction makes the 86-SS model ideal for applications where cleanliness and washdown are required. It is also corrosion-resistant for use in harsh environments. The lifter's design provides support from below by two bars, and support around the drums' girth by a positive-grip cinch chain with a ratchet mechanism. — *Morse Manufacturing Co., East Syracuse, N.Y.*
**www.morsedrum.com**

### Smart transmitters for a variety of level-sensing applications

This company's line of LevelBest smart transmitters (photo) for application-specific level sensors feature onboard diagnostics and completely configurable alarms, providing an accurate inventory of stored products, reduced-cost support for startup and commissioning and ongoing reductions in scheduled maintenance. LevelBest transmitters are outfitted with better than 12-bit resolution and automatic zero, with a 4–20-mA output powered by 10–26 V d.c. and turn-on current of less than 3.8 mA. Upscale and downscale alarms are configurable for both process and sensor. Standard- and corrosive-service models are available, along with a wide selection of mounting accessories. Also offered are versions of the units for open-channel flow and water monitoring. These units feature configurable weir and flume level-to-level linearization, as well as user-defined curve-fitting, remote configuration, wired or wireless options and a wide range of temperature monitoring, from –40 to 65°C. — *Levelese, Inc., Denver, Colo.*
**www.levelese.com**

### These blenders deliver uniformity for both wet and dry materials

The new line of Model 42C cylindrical ribbon blenders (photo) are designed to mix dry powders, wet granulations and paste-like materials with densities up to 100 lb/$ft^3$. Available in many capacities, from 0.5 to 1,000 $ft^3$, these blenders can be built for atmospheric, full-vacuum or internal-pressure operation. A full range of standard, heavy-duty and sanitary models are offered. Uniform mixing and heating are accomplished by a variable-speed, double-ribbon agitator that is driven by a gear motor. Precisely pitched inner and outer ribbons produce a well-balanced axial and radial flow pattern within the batch. Tightly controlled clearances between the horizontal agitator and the cylindrical vessel further ensure thorough blending and promote nearly complete discharge. Stainless steel 316 is the standard material of construction. Options for stainless steel 304, carbon steel and other alloys are also available. — *Charles Ross & Son Co., Hauppauge, N.Y.*
**www.mixers.com**



Air Science USA

### Ceiling-mounted units outfitted with dynamic filtration chambers

Purair SKY ceiling-mounted filtration units (photo) provide personnel protection in areas where hazardous substances are handled. Outfitted with a dynamic filtration chamber and a sliding filter clamp that allows for quick filter changes, these units are designed for a wide range of laboratory and industrial applications. Units come with an epoxy-coated steel support frame with LED lighting and wall-mounted controls. An electrostatic pre-filter helps ensure good trappage of particulate materials. Two different model sizes are available: the SKY-24 (which requires a rough


Flottweg SE

opening of 22.75 × 22.75 in.) and the SKY-48 (which requires a rough opening of 45.5 × 22.75 in.). — *Air Science USA, Fort Myers, Fla.*
**www.airscience.com**

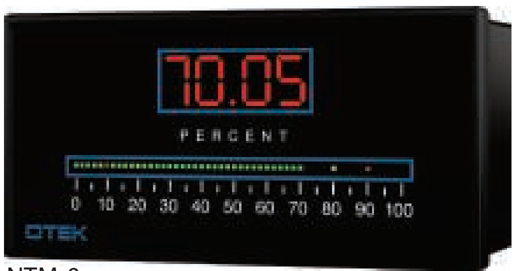
### A new decanter series for sludge dewatering

With the C-XI decanter series (photo), sewage treatment plants can achieve higher dry-substance values, saving money on disposal and further processing, says the company. The new decanters are said to offer optimum conveying of solids, greater purity of the liquid phase, an optimized scroll slope and improved geometry of the scroll body. High speeds and the associated forces require ongoing performance in the gearbox. The manufacturer's Simp Drive gearbox reacts automatically to different load conditions, and transmits a reliable force at high load, while featuring reinforced belt drive, belt guard and gears. — *Flottweg SE, Vilsbiburg, Germany*
**www.flottweg.com**

### Modular piping system now has adapter flanges, too

This company has simplified transitions to its modular pipework system by introducing adapter flanges, which are available in square or round form. The new adapters are welded onto the machine outlet or inlet ports. Matching counter flanges from the company's modular range can then be used to connect the adapters to the pipeline simply and quickly, using bolts. Counter flanges are available in unprimed mild steel or stainless steel. They are used in conjunction with silicone or NBR gaskets to ensure reliable connections to the pipe system. Previously, the customer had to provide a counter flange or adapt it to the correct hole pattern. The new counter flange completes the wide range of the company's transitions and connection adapters, which also includes welding ends and transition pieces, for example. The series manufactured range covers diameters from DN 80 to DN 300 mm. Larger diameters are available on request. — *Fr. Jacob Söhne GmbH & Co. KG, Porta Westfalica, Germany*
**www.pipe-systems.eu** ■

*Gerald Ondrey and Mary Page Bailey*

CHEMICAL ENGINEERING
FACTS AT YOUR FINGERTIPS
Department Editor: Scott Jenkins

# Construction-Cost Indices

Cost estimation is a critical dimension of project planning in asset-heavy industries such as the chemical process industries (CPI). Construction cost indices are useful tools in cost estimation and are used to compare plant construction costs from one time period to another. Understanding them can improve the accuracy of the cost estimates, as well as the effectiveness with which they are applied. This column discusses the use of cost indices and historical trends.

## Indices for the CPI

Cost indices are dimensionless numbers that compare prices of a class of goods or services to the corresponding prices at a base period. They are widely used in the construction industry and can be customized to various industry segments.

Predesign estimates are usually made for equipment and assets that will be built in the future, but must be assembled from prices of the past.

The mathematical relationship between costs and indices is the following:

*(Cost at Time 1)* ÷ *(Cost at Time 2)* =
*(Index at Time 2)* ÷ *(Index at Time 1)*

A number of cost indices are relevant to the CPI, including the *Chemical Engineering* Plant Cost Index (CEPCI) and the Marshall & Swift index (www.marshallswift.com) for the chemical industry and the Nelson-Farrar index (www.ogj.com), which is designed for petroleum refineries.

## CEPCI

The CEPCI is a composite index, and is built from four sub-indices: 1) Equipment; 2) Construction Labor; 3) Buildings; and 4) Engineering & Supervision. The Equipment subindex is further broken down into seven component indices, as follows:

- Heat exchangers and tanks
- Process machinery
- Pipe, valves and fittings
- Process instruments
- Pumps and compressors
- Electrical equipment
- Structural supports and miscellaneous

The component indices are compiled with weighting factors and combined to make up the equipment index. The three other sub-indices are compiled independently and also weighted and normalized.

The CEPCI includes the costs to design, purchase and install chemical plant equipment, and is weighted as follows:

- 61% equipment, machinery, and supports
- 22% construction labor
- 7% buildings
- 10% engineering and supervision

Combining the four sub-indices gives rise to the composite CEPCI index. Annual values for the overall CEPCI and for each of the sub-indices are calculated from the arithmetic mean of the monthly values for that year, for each subindex.

The overall CEPCI for 1959 was assigned a value of 100 as a benchmark. Figure 1 illustrates the historical escalation of the CEPCI and its sub-components for the past 37 years. The CEPCI value, along with those for the sub-indices, has risen unevenly, but steadily for the past three decades.

**CEPCI Historical Trends**

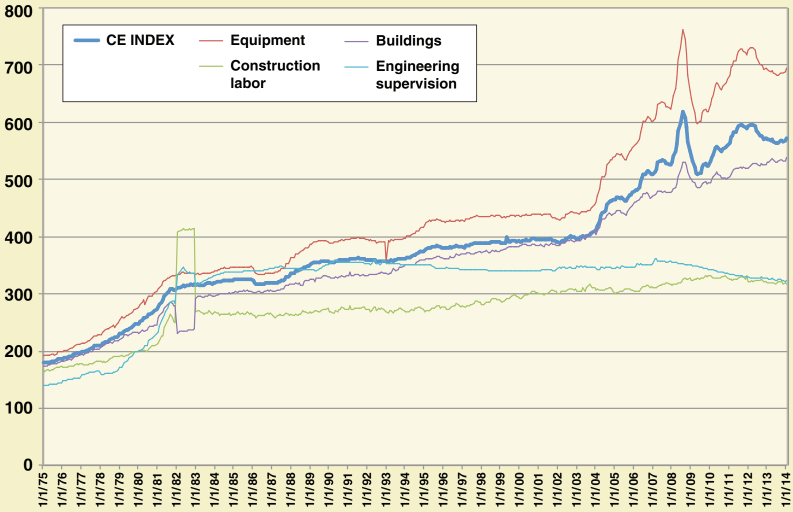



**FIGURE 1. The CEPCI, along with the four sub-indices that contribute to it, has risen unevenly over the past three decades**

## Location factors

Cost indices are traditionally tied to a particular geographic region. As industries and markets globalize, the ability to translate costs has become increasingly important. The cost engineering professional organization AACE International (formerly Assoc. for Advancement of Cost Engineering; Morgantown, W.Va.; www.aacei.org) defines a location factor as an estimating factor used to convert the cost of an identical plant from one location to another. Location factors recognize differences in labor costs, engineered equipment and material freight, duty, taxes, engineering, design and project administration costs. The cost of land is not included in location factors.

Accurately and effectively applying location factors can be challenging and doing so requires an understanding of how each factor was derived and exactly what it represents. In most cases, location factors are not industry-specific, but rather are designed for a broader mix of facility types, so they may not reflect the unique characteristics of buildings in a particular sector. Location factors also typically do not consider cost effects that are associated with site-specific conditions, such as local climate, earthquake risk and other geological differences. Countries may not have the capability to manufacture certain specialized equipment and need to import it, so the degree of importing required needs to be part of the calculation.

When used, location factors should be reserved for preliminary project evaluations — they are not intended to be used when preparing appropriation-quality estimates. Resources for more information on location factors include AACE International and the International Cost Engineering Council (Sydney, Australia; www.icoste.org).

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**Editor's note:** The editor would like to acknowledge AACE International for allowing access to its virtual library, and EC Harris LLP (London, U.K.; www.echarris.com; an Arcadis company), for insight related to this column.

# Bio-based adipic acid

By Intratec Solutions

Adipic acid is a dicarboxylic acid that is widely applied to the production of nylon, polyurethanes, plasticizers and other polymers. Its main use is as one of the raw materials for the manufacture of the polyamide nylon 6,6. In 2012, 80% of the total adipic-acid demand was directed to this application.

The traditional production method for adipic acid is based on the oxidation of cyclohexane, a petroleum-based feedstock. However, adipic acid is among the growing group of industrial compounds for which more-sustainable production methods have been developed. A bio-based route to adipic acid has emerged as an alternative to petroleum-derived adipic acid. Bio-adipic acid can be produced from renewable feedstocks, such as sugar and plant-based oils.

## The process

Adipic acid is produced by a microorganism-based fermentation process that uses a sucrose solution as the feedstock (Figure 1). The process described below was compiled based on information available in the published literature.

***Sugar inversion.*** Sucrose is hydrolyzed to fructose and glucose, which are the compounds actually consumed in fermentation. The reaction occurs in an acidic medium, obtained by adding HCl. After inversion, the pH is adjusted with $Na_2CO_3$, and the resulting stream is cooled before being sent to the fermentation stage.

***Fermentation.*** The sugars are converted to adipic acid through an aerobic fermentation. The microorganism culture is prepared for inoculation in a two-stage seed train. An ammonia ($NH_3$) solution from the recovery area is added to the fermenters to maintain a pH of about 7. The adipic acid produced in the fermentation step reacts with ammonium hydroxide, forming diammonium adipate.

***Cell separation.*** The fermentation broth is sent to agitation tanks and centrifuges in order to completely remove cell biomass. Ultrafiltration is used to remove remaining contaminants, such as cell debris and precipitated proteins. The aqueous solution, now free of microorganisms, is then sent to the product-recovery area.

***Product recovery.*** The solution is sent to the concentration column, where diammonium adipate is split into free ammonia and adipic acid. The overhead stream, which contains mostly ammonia and water, is sent to the ammonia-recovery column and, afterwards, is recycled to the fermentation step. The bottoms product stream contains dissolved adipic acid that is crystallized at low temperatures. The slurry from the crystallizer is centrifuged, and part of the mother liquor is recycled to the concentration column. The solid adipic acid product is dried and sent to storage.

## Economic performance

An economic evaluation of the bio-based adipic acid production process was conducted. The following assumptions were taken into consideration:

- A 83,000-ton/yr capacity plant erected on the U.S. Gulf Coast
- Storage of product is equal to 30 days of operation, and there is no storage for feedstock
- Outside battery limits (OSBL) units considered: ammonia refrigeration system, cooling towers and steam boilers

The estimated capital investment (including total fixed investment, working capital and other capital expenses) for the construction of such a plant amounts to about $340 million, and the operating cost is about $2,150 per ton of product, as depicted in Figure 2.

Due to the availability of low-cost feedstock, the bio-based process can be a good alternative when compared to petroleum-based process for adipic acid production. Research activity to achieve higher product yield and selectivity may eventually lead to the bio-based process becoming more cost-effective than the conventional petroleum-based process.

Aside from the possible economic advantages, the production of bio-based adipic acid can have an impact on environmental issues. The bio-based process is more environmentally friendly and leads to an overall reduction in oxides of nitrogen (NOx) and other greenhouse-gas emissions compared to what is normally produced during the petroleum-based adipic acid process. ■

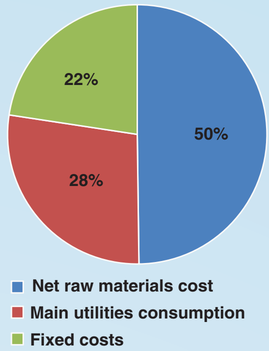


**FIGURE 2.** Raw materials costs account for the half of the operating expenses for a bio-based adipic acid unit

***Editor's Note:*** The content for this column is supplied by Intratec Solutions LLC (Houston; www.intratec.us) and edited by *Chemical Engineering*. The analyses and models presented herein are prepared on the basis of publicly available and non-confidential information. The information and analysis are the opinions of Intratec and do not represent the point of view of any third parties. More information about the methodology for preparing this type of analysis can be found, along with terms of use, at www.intratec.us/che.

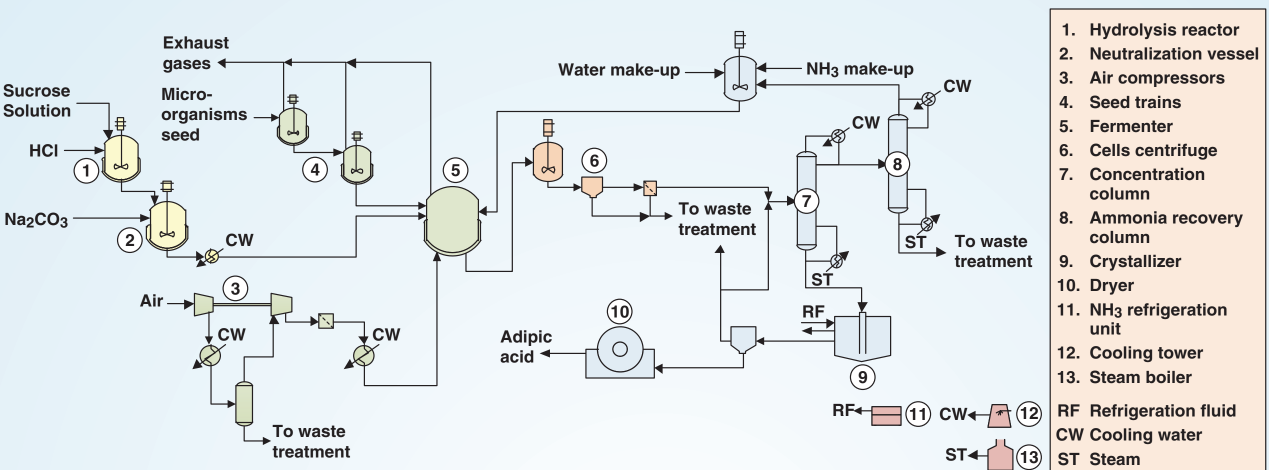


**FIGURE 1.** Bio-based adipic acid production is based on fermentation, ultrafiltration and crystallization processes

# INDUSTRIAL CONTROL SYSTEMS SECURITY: The Owner-Operator's Challenge

**Addressing the cybersecurity of industrial control systems requires a collaborative response, beginning with a realistic assessment**

**Eric C. Cosman**
The Dow Chemical Company

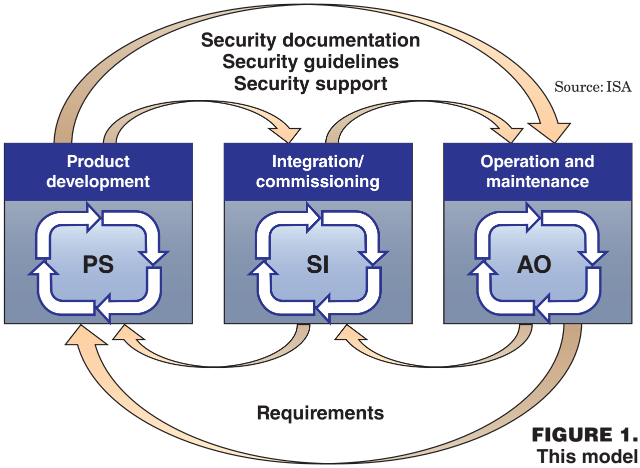


**FIGURE 1. This model shows an industrial control system lifecycle**

Ensuring the security of industrial control systems (ICS) can be a daunting and complex task, requiring expertise and experience in several areas from a variety of contributors. The rapid pace of improvements to both technology and practice adds to the challenge. Finally, the amount and complexity of information, guidance and advice that is available can easily become overwhelming.

Rather than focusing on the details about security products and technology, an approach from an engineering perspective is necessary — concentrating first on why security is important, and then moving on to the roles and responsibilities involved in an effective response. This article provides practical guidance on how to address this challenge.

While the ultimate accountability for the security of these systems lies with the owner-operator, successfully addressing the subject requires a collaborative approach that includes contributions from several stakeholders. Understanding, agreeing on and coordinating the respective responsibilities of these contributors is actually more important than any specific technology or method.

While accountable, the owner-operator need not feel alone. Specific responsibilities can be assigned or delegated to others who have specific experience or expertise. The most important message is that this is a challenge that cannot be ignored. Inaction is simply not an acceptable response.

## IT AND OT

Both information systems and ICS security have received significant attention over the past several years. Much has been written on the subject, including articles on the subject that have appeared in the pages of this magazine [*1–3*]. There is considerable discussion and debate within the security community about the degree to which ICS security is similar to or distinct from more traditional information systems security. Recently this has led to the common use of the terms information technology (IT) and operations technology (OT) to describe these two areas of attention. Regardless of the specific terms used, there is general agreement that the technology is evolving rapidly; perhaps too rapidly for all but the most dedicated specialists to track.

### Engineering and operations

Throughout all of this debate it is not clear that there has been sufficient involvement from the engineering and operations communities. Given the rapid pace of change and the sometimes confusing or arcane nature of the subject, what is the typical process or operations engineer to do about security? They are often the most direct representatives of the owner-operator, who are expected to address the accountability previously mentioned.

## CHANGING THE CONVERSATION

The current state of affairs requires a change in tone and context of the conversation about cybersecurity. Typically much of the security-related dialog is concentrated on protecting and acting on "the system" or "the network." The reality is that this approach is often too abstract when applied to industrial systems. Why should a design or operations engineer care about these abstract terms, when they face more pressing and tangible challenges and problems on a daily basis? It is not primarily about protecting the sys-

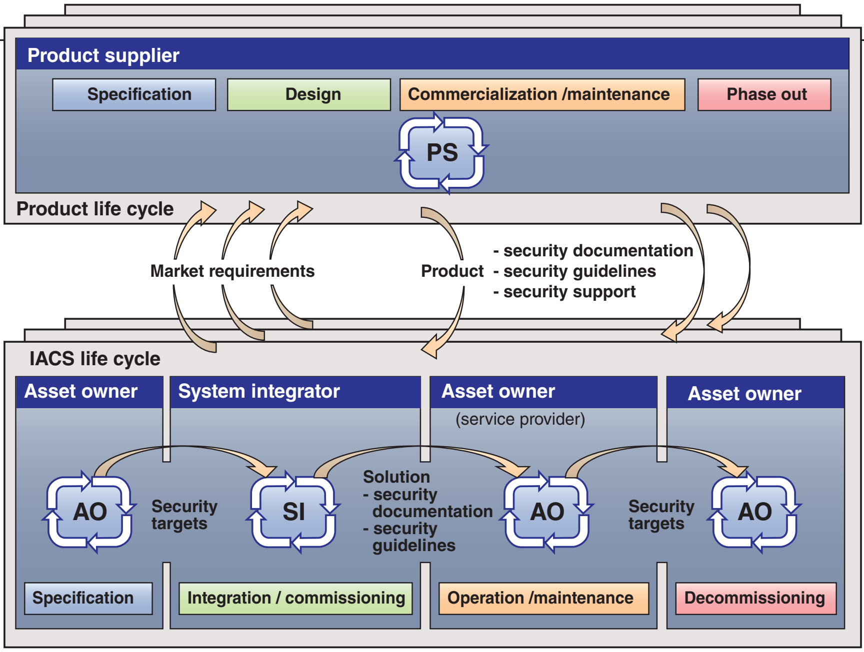


**FIGURE 2.** A different view of the ICS lifecycle is depicted here

tem, the network or the data. It is about the potential implications of not protecting them.

An informed and balanced assessment of these implications has to be the first step in this refocused conversation. The people who are the most experienced in the design and operation of the process or equipment have the best understanding of what those implications may be. It is important that they consider a breach in security to be another possible source of failure or compromise, just like equipment failure, uncontrolled reactions or other factors. With this concept, what has been framed as a security opportunity now becomes an engineering opportunity.

## THE CHALLENGE

A realistic assessment of implications in no way changes the technical nature of the challenge. Securing the systems, networks and data provides a means to an end, and that end is the safety, availability and reliability of the underlying process or equipment. Compromising the security of these system components can result in negative impacts in the physical world. Even in situations where ICS elements (such as a safety instrumented system) are physically isolated and not connected to a network, it is still possible for poor practices to lead to security compromise, which can in turn affect plant or process operations.

### Risk is real

The reality of this type of scenario has been demonstrated in both simulations and real incidents. Although companies are often reluctant to publicly discuss such incidents, those with experience in this area know that they have occurred. Discussions of this subject inevitably lead to a debate about exactly what constitutes a "cyber incident." Some have taken the position that only deliberately malicious acts should be considered, while others have argued that inadvertent acts must also be included. In fact, this distinction is largely artificial and not particularly important. What is important to the owner-operator company is a realistic assessment of risk to its operation.

## BASIC ELEMENTS

Before such a risk assessment can be conducted, it is necessary to have an understanding of certain basic elements of ICS security, taking into account the implications for both design and operation of industrial operations. This assessment does not require a great deal of complicated or technical analysis, but it is helpful to have an appreciation of a few important concepts.

### Lifecycles

Perhaps the most fundamental of these concepts is that of the lifecycle of the industrial control system. Several views of this lifecycle have been proposed and developed. One of these is being developed for use in international standards on this subject. This model is shown in Figure 1.*

As seen in Figure 1, this model describes three interconnected processes that correspond to the phases of the solution lifecycle, as well as to the stakeholder groups that have the primary responsibility for each.

- The product supplier (PS) has primary responsibility for the product development lifecycle, which must include security in the design of the individual products
- The integration or commissioning lifecycle combines a variety of products to create the overall control solution. This phase is primarily the responsibility of the system integrator (SI)
- The operation and maintenance lifecycle is the principal focus of the asset owner (AO) or owner-operator (for the purpose of this discussion, the terms owner-operator and asset owner are considered to be synonymous)

As shown in the model, these lifecycles are connected through the sharing of system and support documentation, as well as requirements.

A different view (also under development for use in standards) is shown in Figure 2.* In this case, the focus is on how the development of products is related to the ownership and operation of the resulting systems.

The phases of ownership and operation range from initial specification, through to eventual decommissioning and removal or replacement. Through all phases of this cycle, it is essential to have a realistic as-

* Figures 1 and 2 are unpublished submissions to the ISA99 Committee on Industrial Automation and Control Systems Security by R. Schierholz and P. Kobes

sessment of the risk associated with a cyber-related incident, where risk is generally considered to be a function of threat, vulnerability and consequence. Each of these factors has to be considered separately.

The owner-operator is either accountable or directly responsible for activities in each of these phases. Response to security challenges typically takes the form of a cybersecurity management system (CSMS).

### Industry standards

Industry standards represent accepted conventions and practices with respect to a particular subject. In the case of ICS security, they are the ISA-62443 [*4*] series of standards under development by the ISA99 committee of the International Society for Automation (ISA) and the International Electrotechnical Commission (IEC). An overview of this series is shown in Figure 3.

Source: ISA

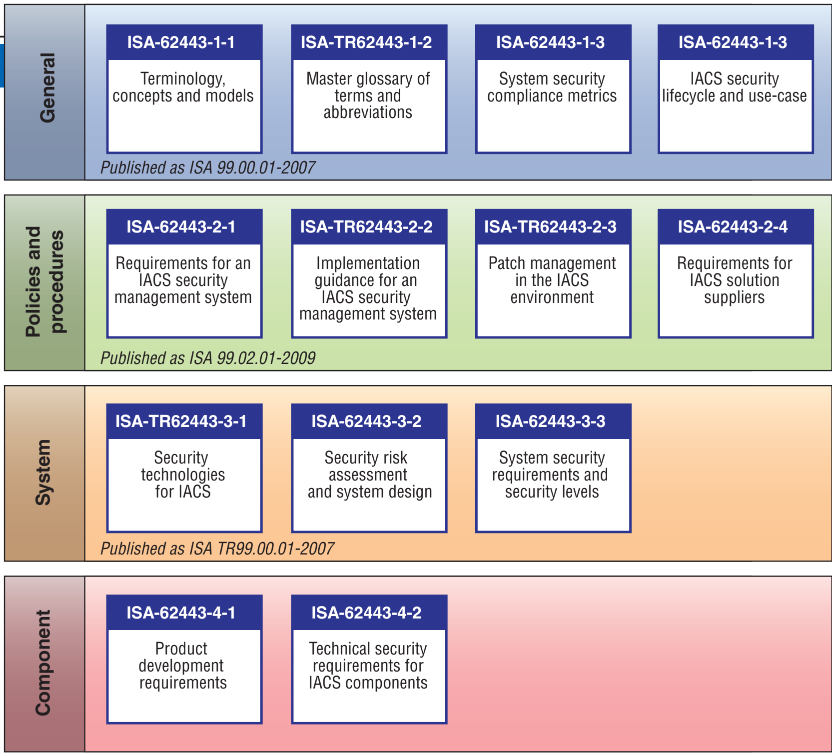


**FIGURE 3. The ISA-62443 Standards are prominent references for industrial automation and control systems (IACS) security**

The ISA-62443 standards are purposely structured in such a way that the primary focus for the owner-operator community will be on the top two levels in the above-mentioned model. For example, the ISA-62443-2-1 standard provides specific direction on how to structure a cybersecurity management system that complements similar practices for general-purpose IT systems.

The standards in the two lower levels of Figure 3 are directed primarily to those who develop and assemble industrial control systems.

### Product certification

The owner-operator requires a reliable means of determining whether specific control systems and components adhere to standards such as ISA-62443. This is accomplished through formal programs, such as ISASecure** [*5*] that certify these products as being compliant to a set of formal specifications taken from the standards.

Although products that are certified as compliant provide the basic building blocks for security, a complete response by the owner-operator also requires more practical guidance and assistance.

### NIST Framework

In the U.S., an important recent development in cybersecurity has been the creation of the NIST (National Institute of Standards and Technology) Cybersecurity Framework [*6*]. Developed in response to Presidential Order 13636, this framework provides the owner-operator with a specific approach to the development of a comprehensive cybersecurity program. Figure 4 shows a view of the information flows required to address cybersecurity, as described by the framework.

The NIST Framework consists of the following elements:

- The Framework Core
- The Framework Profile
- The Framework Implementation Tiers

The Framework Core (Figure 5) describes the set of activities that an organization should perform as part of a cybersecurity management system. Starting with five functions — identify, protect, detect, respond and recover — the Framework Core is divided into categories (such as asset management, risk management, awareness and training) and subcategories (such as investigate anomalies and perform forensics). References to sector, national or international standard requirements, or clauses are then listed within these subcategories.

Although the scope of application for the framework includes all aspects of cybersecurity in the critical infrastructure, it also has applicability to the narrower scope of industrial control systems security. The ISA-62443 series of standards are prominent references in this area.

## ADDITIONAL RESOURCES

Additional information, including sector, industry and international standards, as well as technology, product- and sector-specific guidance is available from many sources. There are also many training programs offered. Several resources are available to assist and guide the owner-operator in planning and executing an effective cybersecurity program. The irony is that the problem is not a lack of in-

** ISASecure is a trademark of the Automation Standards Compliance Institute

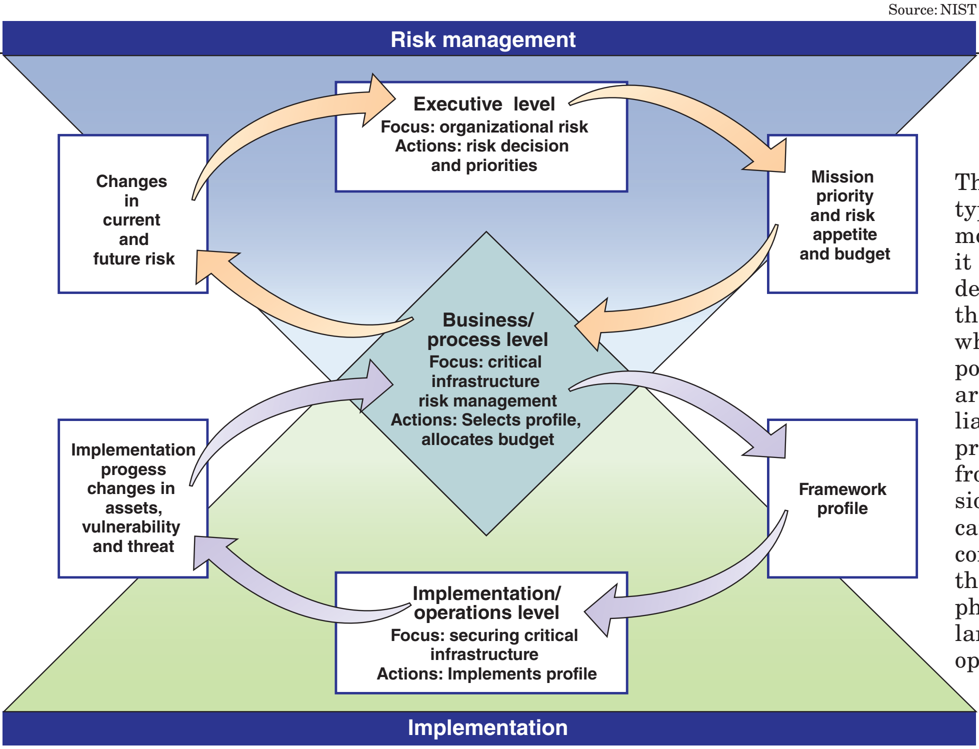


**FIGURE 4.** The NIST Cybersecurity Framework describes information and decision flows

formation, but an over-abundance of it. The result is often confusion and uncertainty about where and how to begin.

If a company is a member of an industry trade association, there may be guidance available from that source. For example, members of the American Chemistry Council (ACC; Washington, D.C.; www.americanchemistry.com) have access to sector-specific guidance, as well as vehicles such as the Responsible Care Security Code [7]. Professional associations, such as the Institute of Electrical and Electronics Engineers (IEEE; Washington, D.C.; www.ieee.org) or the International Society for Automation (ISA; Research Triangle Park, N.C.; www.isa.org) [8] also have information available in the form of standards, practices and training.

Once the references, guidance and training resources have been identified, the owner-operators must assemble the components of their cybersecurity program. As mentioned earlier, the first step is to accept this accountability and acknowledge the opportunity. If management support is required, it is useful to explain the potential implications of a security incident in terms of impact on the process or business.

### Threat information

Information about possible threats often comes in the form of anecdotal reports of attacks or incidents that have occurred elsewhere. While some may have very specific relevance to industrial control systems (such as Stuxnet and Shamoon), others are much more generic. Sources for this information include security companies and perhaps government sources such as ICS-CERT (The Industrial Control Systems Cyber Emergency Response Team) [9].

### Vulnerabilities

Sources for information on vulnerabilities include suppliers of the systems and component software, as well as reporting sources, such as ICS-CERT. In many cases, vulnerabilities are not disclosed until a patch or some other mitigation is available. However, this is not a universal practice. It is for this reason that it is important to maintain regular communications with systems and technology providers.

### Consequences

The consequence component should receive considerable attention from the owner-operators, as they are in the best position to understand it. The best source of this type of information is most often internal, since it will be those who have designed and operated the plants or processes who best understand the possible consequences in areas such as safety, reliability, availability and product quality. Recall from the above discussion that a cyber incident can just as easily lead to consequences similar to those of a more-expected physical failure, particularly in highly automated operations.

When considering these consequences it is important to be realistic. Far too often when this topic is discussed in the context of industrial control systems, there is a tendency to lapse into hyperbole and to talk about explosions, release of noxious materials and other catastrophic events. While these events may in fact be possible, they are probably not likely. Industrial processes have long been designed with these possibilities in mind, and mitigating measures have already been taken in both design and operation.

When talking about the importance of security, dwelling on such scenarios in conversations with experienced operations people often has the result that they simply tune out the discussion. Engaging the engineering community requires that the conversation take place in a context that they can relate to.

## CREATING THE PROGRAM

The best choice of a starting point depends somewhat on the individual situation. In some companies there may already be corporate programs in place that address a portion of the subject. Assistance is also available from a variety of external sources. In either case, it is important to remember that effective ICS security requires a multi-disciplinary approach, drawing on expertise in security, engineering and operations.

### Basic steps
Regardless of the details of a specific process, the basic steps for creating a program can be expressed as follows:
1. Identify contributors (Plan)
2. Establish the scope of interest (Plan)
3. Acquire or develop an inventory of systems within scope (Do)
4. Conduct an assessment of the current state of security (Check)
5. Establish and execute a plan to improve performance (Act)

### Contributors
Many of us have received advice to avoid allowing the business IT function to be responsible for industrial cybersecurity. However, rather than ignoring this source of expertise or building barriers to its involvement, a better approach is to reach out to IT professionals to form a partnership. Much of their technology and security-related expertise does have value in this situation, as long as it is carefully applied, with a full understanding of the operational characteristics and constraints.

Effective methods of establishing this partnership could easily be the subject of an entirely separate discussion, but any such relationship has to begin with a description of the shared vision or intent, and an open and frank exchange of wants and offers. What does each party hope to gain through the relationship, and what do they bring to it?

### Establish scope
The first task is to assemble an accurate inventory, describing the systems and components that are considered to be in scope for the program. This inventory can be assembled using a variety of criteria, as described in the ISA-62443 Standards. These criteria include the following:

***Functionality included.*** The scope can be described in terms of the range of functionality within an organization's information and automation systems. This functionality is typically described in terms of one or more models.

***Systems and interfaces.*** It is also possible to describe the scope in terms of connectivity to associated systems. A comprehensive program must include systems that can affect or influence the safe, secure and reliable operation of industrial processes.

***Included activities***. A system should be considered to be within scope if the activity it performs is necessary for any of the following:
- predictable process operation
- process or personnel safety
- process reliability or availability
- process efficiency
- process operability
- product quality
- environmental protection
- compliance with relevant regulations
- product sales or custody transfer affecting or influencing industrial processes

***Asset-based.*** The scope should include assets that meet any of the following criteria, or whose security is essential to the protection of another asset that does any of the following:
- is necessary to maintain the economic value of a manufacturing or operating process
- performs a function necessary to the operation of a manufacturing or operating process
- represents intellectual property of a manufacturing or operating process
- is necessary to operate and maintain security for a manufacturing or operating process
- is necessary to protect personnel, contractors and visitors involved in a manufacturing or operating process
- is necessary to protect the environment
- is necessary to protect the public from events caused by a manufacturing or operating process
- is a legal requirement, especially for security purposes, of a manufacturing or operating process
- is needed for disaster recovery
- is needed for logging security events

***Consequence-based.*** Scope definition should include an assessment of what could go wrong and where, to disrupt operations, what is the likelihood that a cyber-attack could initiate such a disruption, and what are the consequences that could

Source: NIST

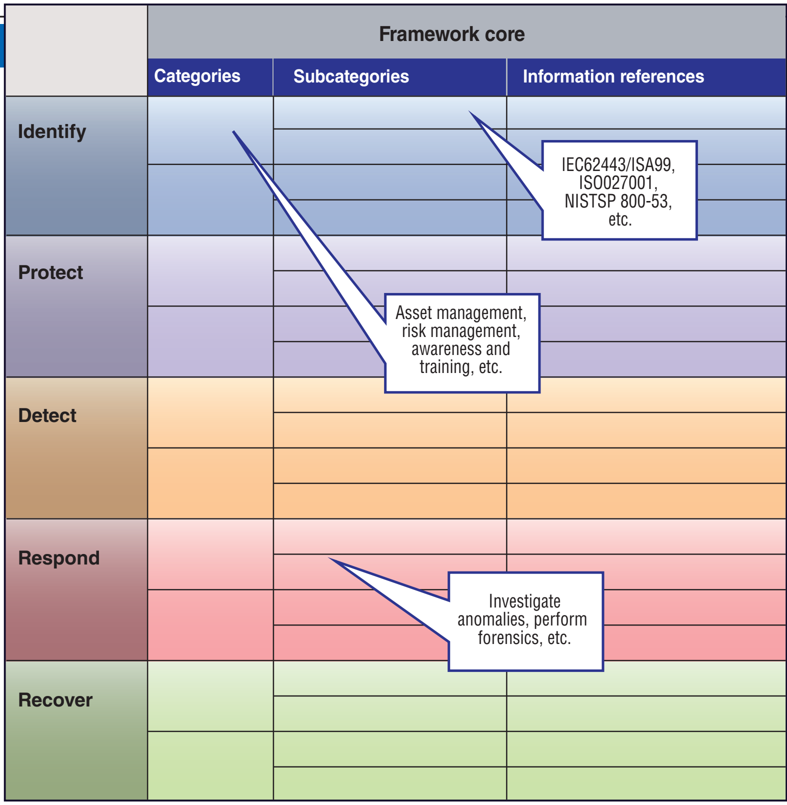


**FIGURE 5. The Framework Core describes activities that an organization should perform as part of a cybersecurity management system**

result. The output from this determination shall include sufficient information to help the ordinary user (not necessarily security-conscious ones) to identify and determine the relevant security properties.

### Inventory

Having considered the above criteria to define the focus or scope of the cybersecurity response, the next task is to develop an inventory of the systems, components and technologies that are included in scope. This is the "Do" step. Sources of such information include design and maintenance documents for the control systems and connecting networks.

In cases where the configuration has evolved over time, it may be necessary to confirm and supplement the records by physically surveying the installation. It may be necessary for this process to be repeated several times, adding more detail each time. Although this can be a tedious process, it is essential to have an accurate description of the system to be protected.

An accurate and current inventory can only be prepared with involvement of the owner-operators, since they are most familiar with the installation. In this case, the owner-operator is both accountable and responsible.

It is important to take note of the potential for using some sort of automated or programmable scanning as a means of identifying systems and devices. Tools such as this should be applied very carefully, as there is a very real potential for such scans to disrupt normal operations.

### Assessment

With a thorough understanding and appreciation of the systems to be protected, attention now moves to the assessment phase. This is the "Check" step. It is at this point that the state of system components are compared to expectations drawn from sources, such as standards and recommended practices.

The important choice here is the exact standards or measures against which to measure performance. To some extent, this choice depends on factors such as sector or industry affiliation. For example, facilities that are part of the U.S. chemical sector may be expected to demonstrate compliance with the Chemical Facility Anti-Terrorism Standards (CFATS) [*10*] or the cyber-related elements of the Responsible Care Security Code.

Similar expectations exist for other sectors. Assistance in selecting appropriate assessment standards is available from several sources. Several of these have been developed and are readily available from the U.S. Department of Homeland Security (DHS; www.dhs.gov).

One of the more recently available tools is called a Cyber Resilience Review (CRR) [*11*]. It can be used for a "non-technical assessment to evaluate an organization's operational resilience and cybersecurity practices." Intended to be applied to all aspects of a company's security program, this tool has some relevance for ICS security. The expectation is that it would be applied at a corporate level.

### Improvement plan

Regardless of the method or approach used to complete the assessment, the results will have little value if they are not used to make improvements. A common approach is to develop a "multi-generation" plan that characterizes each of the required improvement steps or tasks as short-, medium- or long-term. Regardless of the specific method used, it is essential to address the findings of the assessment in order to make regular improvements in the state of control systems security.

## FINAL THOUGHTS

Addressing the cybersecurity of industrial control systems can be a daunting task, but it is not one that can be ignored. Although the complex and arcane nature of the subject can tempt design and operations engineers to dismiss it as an "IT problem", the reality is that accountability for the protected systems most often lies with engineering and operations.

A collaborative response, beginning with a realistic assessment of potential implications and consequences, is needed to develop an effective ICS cybersecurity management system. ■

*Edited by Dorothy Lozowski*

### Author

**Eric C. Cosman** is an Operations IT consultant with The Dow Chemical Company (Michigan Operations, Midland, MI, 48667; Email: eccosman@dow.com). He specializes in the application of information technology to all areas of manufacturing and engineering, including the definition of policies and practices, system and technical architecture, and technology management and integration planning processes for manufacturing and engineering systems. With over thirty years of experience in areas including process engineering, process systems software development, telecommunications, IT operations, architecture definition and consulting, he has also held leadership roles in various industry groups, standards committees, focus groups and advisory panels. Cosman is an industry leader in the area of industrial systems cybersecurity, with a specific emphasis on the implications for manufacturing operations. He is a founding member and the current co-chairman of the ISA99 committee on industrial automation and control systems security and the vice president of standards and practices at the International Society for Automation (ISA).

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**Feature Report**

# Combining the use of Rupture Discs with Relief Valves

## Using the two devices together offers significant benefits in chemical processes. Here is how to take advantage of them

**Roger Bours**
Fike Corp.

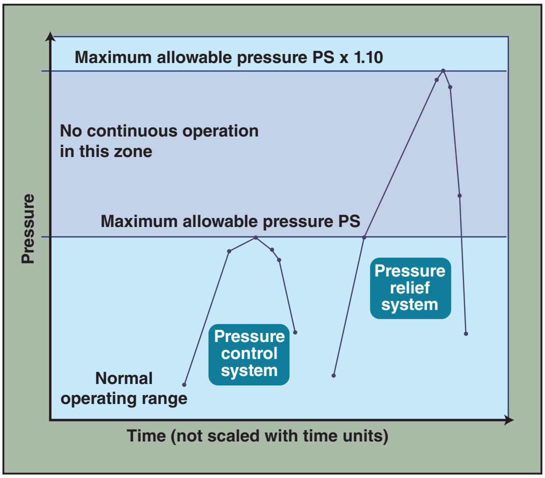


**FIGURE 1.** Pressure-control systems may not ensure the required level of pressure safety, so dedicated pressure-relief systems are also needed in many situations

Pressure-relief solutions are commonly used in the chemical process industries (CPI) to ensure safe working environments for personnel and to protect equipment and assets. Relief valves (RV) and rupture discs (RD) are the most commonly used pressure-safety devices and are generally specified and selected according to application-specific requirements. For pressure-system designers looking for viable pressure-relief solutions, the two device classes offer specific pressure safety features and require consideration of different factors.

Using rupture discs in combination with relief valves offers a wide range of benefits to end-users. Possible benefits include improved environmental performance, cost savings, better emissions control, higher safety and reliability levels and improved performance of plant safety systems. To realize the benefits, process system designers need to evaluate the effects of each of the devices and select the arrangement that works best, based on the individual plant requirements.

Various industry standards and legislation exist to help set up safe and effective solutions. In most applications, using rupture discs together with relief valves offers higher overall value and a host of worthwhile benefits.

### Pressure control versus relief

Since the beginning of the industrial revolution, industrial processes have operated at pressures that differ from atmospheric pressures (both overpressure and vacuum). With the advent of pressurized processes the requirement to adhere to mandatory safety measures has also arisen. A body of national and international legislation has been developed and is in place for promoting pressure safety and reducing risk to personnel, the environment and to investments.

The first line of defense for pressure safety are typically pressure-control systems. These systems monitor the pressure changes in the process equipment and interact in a timely fashion with the process-control system to adjust the pressure to acceptable levels. Control and monitoring devices, which are not specifically a part of a safety system, are usually excluded from safety design standards, since they are typically active in advance of a safety system.

The efficiency of pressure-control systems depends on the input received from instrumentation devices, and requires extensive and validated reliability analysis, based on probability of failure on demand (PFD) or safety integrity level (SIL) assessment. Because pressure-control systems may not ensure the required level of reliability under all service conditions, the use of pressure-relief systems as a last line of defense is often required. In situations where the pressure-control systems do not achieve the required pressure-safety levels alone, dedicated pressure-relief devices must be installed to protect the process when the critical pressure threshold is reached. Figure 1 illustrates the correlation between pressure control-and-monitoring systems, and pressure-relief systems.

When designing an effective pressure-relief system, it is essential to consider the complete system holistically to maintain the full pressure-relieving capacity of the pressure-relieving devices and avoid having the operation of the devices interfere with each other. Operating problems within pressure-relief systems, when they are observed, frequently result from one or more of the following: incorrect device selection; improper pressure handling;

**FIGURE 2.** Installation of rupture discs upstream of relief valves allows for in-situ calibration testing of relief valves

**FIGURE 3.** Rupture discs can prevent leakage through the relief valve and prevent corrosion of relief-valve internals when positioned on the process side of a valve

**TABLE 1. RUPTURE DISCS VERSUS RELIEF VALVES**

| Properties | Rupture disc | Relief valve |
|---|---|---|
| Complexity of device | Low | High |
| Investment cost | Low | High |
| After activation | Replace | Reset |
| Protection against overpressure | Yes | Yes |
| Protection against vacuum pressure | Yes | No |
| Mounting-position restrictions | No | Vertical only |
| Installation cost | Low | High |
| Maintenance cost | Low | High |
| Requires regular recalibration | No | Yes |
| Affected by backpressure | Yes | Yes |
| Operational testing possible | No | Yes |
| Leak-tight | Yes | No |
| Selection of materials of construction | Large | Limited |
| Size range | Large | Limited |
| Change of set pressure | No | Yes |
| Suitable for gas-liquid two-phase systems | Yes | No |
| Reaction time | Low | High |
| Unrestricted opening | Yes | No |

incorrect device installation; or improper (or lack of) maintenance.

## Reclosing versus non-reclosing

Pressure-relief devices are categorized as reclosing and non-reclosing types. Both offer unique characteristics for design engineers seeking to protect against pressures that exceed allowable levels. Most industry sectors traditionally work with reclosing relief valves or non-reclosing rupture discs to achieve pressure-relief action.

Reclosing devices — sometimes referred to as safety relief valves (SRV), pressure-relief valves (PRV) or relief valves (RV) — are designed to open at a selected set pressure. Opening allows the overpressure to evacuate and the pressure to return to an acceptable level, whereupon the valve recloses. Pressure-relief valves come as spring-operated or as pilot-operated units.

To protect installations against unacceptable vacuum pressures, the use of vacuum-relief valves (VRV) may be considered. These devices will similarly open and allow for atmospheric pressure to be re-established when the set-to-open vacuum pressure is reached.

Rupture disc devices are often preferred as a means to achieving instant and unrestricted pressure relief (both overpressure and vacuum pressure). They consist of a calibrated (metallic or graphite) membrane that ruptures when the set pressure is achieved. After activation, the membrane remains open, resulting in a complete discharge of the pressure in the installation.

The main properties of rupture discs and relief valves are summarized in Table 1.

Depending on the equipment that needs to be protected and the required performance, reclosing and non-reclosing devices can be complementary, offering unique advantages and limitations. The appropriate selection of devices must be determined by the design engineer and end-user, depending on the needs of a specific application.

## Complementary RDs & RVs

Using rupture discs in combination with relief valves can utilize the properties of both, often arriving at an optimal solution (Figures 2 and 3). Combinations can employ the RD and RV either in parallel or in series, offering a combination of features that achieves operating and safety objectives. The goal of design engineers and safety specialists is to determine which combination provides the desired features, while keeping the consequences of exceeding pressure limits in balance.

| TABLE 2. ASME VERSUS ISO REQUIREMENTS FOR COMBINATION RD/RV SYSTEMS | | | |
|---|---|---|---|
| **Requirement** | **ASME Sect. VIII, Div. 1 (API)** | **EN ISO 4126-3** | **Comments** |
| Definition of an RD/PRV combination | None | Rupture disc is within five pipe diameters of the inlet of the PRV | If the RD is not within five pipe diameters, then a combination capacity factor is not applicable |
| The three-percent rule | Pressure drop between the vessel and PRV inlet, including the effect of the rupture disc, shall not exceed 3% of the valve set pressure at valve nameplate flowing conditions | Pressure drop between the vessel and PRV inlet, including the effect of the rupture disc, shall not exceed 3% of the set pressure of the valve at maximum flowing conditions | The difference between flowing at nameplate capacity or another maximum could be significant. That is, what if the PRV is set well below the MAWP (maximum allowable working pressure) but sized to prevent exceeding 110% of MAWP. It may be impossible to meet the ISO requirements in this situation |
| Certified combination capacity factor (CCCF) | One-size method applicable to all sizes equal to and larger than the tested combination | One-size method for a single size or three-size method to be applied to a family | Pursuit of the ISO three-size combination capacity factors is cumbersome due to the cost and logistics. With a default of 0.9 the pay-back on three-size testing is minimal |
| Protrusion of petals into valve | No specific requirement | Petals shall not protrude into the PRV inlet unless the influence of the petals on the capacity and performance of the PRV has been assessed and proven to meet the requirements of Clause 7 (Combination Performance) | Both codes use language prohibiting the RD to impair the performance of the PRV. The ISO document seems to take a firm stand on the petal protrusion issue but points to Clause 7 which allows a default CCF (Fd) of 0.9 |
| Documentation of the combination | Nameplate marking for the combination provided by the user, PRV, RD, or vessel manufacturer | Supplier of the combination shall provide the nameplate, certification, assembly and installation instructions taking into account the results of a hazards analysis | In both codes, there are gaps in these requirements. In practice these requirements are rarely followed |

The two major standards governing combination of rupture discs and relief valves are the ASME Boiler and Pressure Vessel (BPV) Code (Section VIII Division 1, § UG-125 (c)(1)) and European Pressure Equipment Directive 93/23/EC (EN764-7 § 6.1.4, as defined in EN/ISO 4126-3). The requirements for the two are outlined in Table 2. The elements of API Recommended Practice (RP) 520 (Sizing, Selection and Installation of Pressure-Relieving Devices) are taken directly from the ASME BPV code, Section VIII, Division 1.

## RD and RV in parallel

When using relief valves and rupture discs in parallel (Figure 4A), the main objective is to allow the relief valve to initially handle overpressure situations — bleeding the pressure until an acceptable, reduced pressure is achieved — while allowing the process to continue. When the overpressure cannot be effectively reduced by the relief valve (due to malfunction, blockage or generation of excessive pressure), the pressure may continue to rise until a higher set pressure of the rupture disc is reached. Upon activation, the rupture disc provides an additional backup relief path for the overpressure, resulting in a safer process.

When using RDs and RVs in parallel, a suitable margin of set pressure needs to be designed into the system to avoid premature failure of the rupture disc. This requires that the set-to-open pressure of the relief valve be below the burst-pressure range of the rupture disc, with an appropriate margin to separate them.

Regulatory and legislative requirements for pressure limitation and size determination, range from what is found in the ASME BPV Code — which says "Sizing of the secondary relief devices (the rupture disc) [should] be such that the pressure does not exceed 116% of the equipment design pressure" — up to the language in the European Pressure Equipment Directive, which says the "Maximum achieved overpressure [is] not to exceed 110% of the equipment design pressure."

## RD upstream of RV

Rupture-disc devices may also be installed either upstream or downstream of a relief valve (Figure 4B). Each geometry offers particular benefits for the user. Using rupture discs upstream of relief valves is a common practice to achieve one or more of the following, each of which is explained further below:

- Prevent plugging of the RV
- Prevent corrosion on RV internals
- Prevent leakage through the RV
- Allow for in-situ testing of the RV

***Prevent plugging or gumming of the relief valve***. Through the

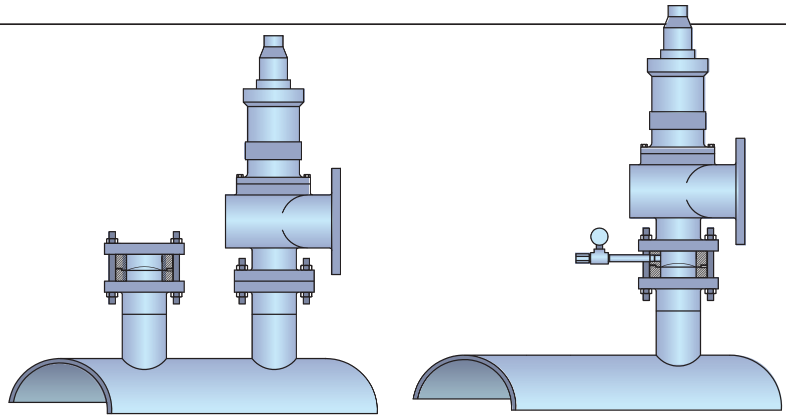

**FIGURE 4A and 4B.** Rupture discs and relief valves can be used in parallel (left) and in series (right)

selected use of suitably designed rupture discs upstream, product buildup or polymerization can be limited. Most relief valves are not suitable for use with media that create a buildup layer, because it interferes with the ability of the relief valve to open. The use of an upstream rupture disc reduces the need for regular inspection, maintenance or cleaning of the relief valve, leading to increased productivity and more reliable safety.

***Prevent corrosion of the relief-valve internals.*** When the process media require that specific corrosion-resistant materials are used, relief-valve options can be limited and those valves that are available could carry higher costs, longer delivery times and more difficulty obtaining spare parts. By installing a high-alloy rupture disc upstream of the relief valve, the valve is physically isolated from the process. Exposure of the valve to the process media is restricted to the overpressure event only. Until this emergency event occurs, the relief valve remains in pristine condition, unaffected by the process.

An in-series arrangement allows for the use of valves and related spare parts that are made from "standard" materials, resulting in substantial cost savings at the initial investment, a wider range of potential valve and parts suppliers, and shorter equipment lead times.

***Prevent leakage through the relief valve.*** To achieve leak tightness, most spring-operated relief valves rely on special metal-to-metal sealing surfaces and on the applied spring-load force. Such systems inevitably result in some leakage, which increases as the operating pressure approaches the valve set pressure. Relief-valve leakage rates are addressed in industry standards, and acceptable leakage rates are defined (for example, in API Standard 576, "Inspection of Pressure Relieving Devices"). Where such leakage rates are unacceptable, the user may choose soft-seated or pilot-operated relief valves. Both options require higher investment and may still have restrictions, such as the availability of suitable O-ring material with sustained performance characteristics when exposed to the process media, as well as pilot-valve leakage and corrosion or plugging, and so on.

Rupture discs offer reduced leakage rates, and designs are available with virtually leak-free construction. The installation of rupture discs upstream of the relief valve eliminates emissions in a simple and cost-effective manner.

***Allow for in-situ testing of relief valve.*** The acceptable use of relief valves to protect installations is linked to the need for periodic calibration of these safety devices. Depending on the local regulatory requirements, such calibration may be required annually. Since process shutdown and removal of the relief valve from the process equipment is required for such calibration testing — often to be done at a special test institute or qualified service center — important economic reasons exist to try to extend the calibration intervals. Longer calibration intervals may be allowed by the supervising authorities if the user provides evidence of unaffected set pressure over time.

This can be achieved by regular testing of the relief valve in-situ (that is, without removing the relief valve from the installation) and demonstrating that the valve's performance is unchanged.

By installing a rupture disc upstream of the relief valve, a limited volume is created, allowing for the controlled introduction of pressure between the rupture disc and the valve inlet from the outside. This pressure (possibly combined with special "pulling force" test and measuring equipment applied to the valve spindle to overcome the spring force and keep the relief valve in closed condition) can be measured and registered as evidence of acceptable valve performance. The relative cost related to adding the rupture disc device is generally far less than what results from the loss of production time when removing and re-assembling the RV.

## Considerations for selection

The following guidelines should be considered when selecting a rupture disc upstream of a relief valve:

- The rupture disc cannot interfere with the relief-valve operation. For example, the rupture disc cannot fragment upon bursting, because pieces may obstruct the valve orifice or prevent the valve from fully reclosing. Sufficient distance is required for the rupture disc to open without blocking the relief-valve nozzle (after opening, a single-petal rupture disc may extend beyond the height of the holder reaching into the inlet section of the relief valve)
- To assure proper functioning of the relief valve, the rupture disc device should be "close-coupled" with the relief valve, assuring that the pressure drop during flow at the inlet of the relief valve does not exceed 3% of the valve set pressure, as required. The 3% value is a requirement, and is described in API RP 520. In most cases, this restricts the distance between the rupture disc and relief-valve inlet to a maximum distance of five pipe diameters. This situation is often achieved by installing the rupture

disc device directly upstream of the relief valve. Longer distances between the rupture disc and relief valve — by introducing pipe sections or spacers — may result in the creation of reflective pressure waves when the rupture disc opens. This phenomenon can result in undesired fragmentation or re-closing of the rupture disc, and should be avoided

- Since, like the relief valve, the rupture disc is a device that reacts to differential pressure between the upstream and downstream side, measures should be taken to avoid any unnoticed pressure increases in the closed cavity between the rupture disc and relief-valve inlet. Most industry standards and related regulations require that the pressure in the cavity be monitored or vented to the atmosphere. This is commonly achieved through the use of a so-called tell-tale assembly, consisting of pressure gage or indicator, try cock and free vent

## Sizing and set pressure

When installing rupture disc devices upstream (at the inlet) of relief valves, the size of the rupture disc should be, at a minimum, the same nominal size of the inlet of the relief valve. Additionally, the rated relief capacity of the relief valve, as stated by the relief valve manufacturer, should be reduced by 10%, or alternatively, reduced to the certified combination capacity value (when the specific valve-disc combination has been capacity-tested and certified by a recognized third party).

The set pressure of the rupture disc device should be set in accordance with the applicable standards and guidelines, as follows:

- ASME VIII Division 1 UG-127 footnote 52 states that the selected pressure should "….result in opening of the valve coincident with the bursting of the rupture disk." For combination capacity testing, ASME UG-132(a)(4)(a) says: "The marked burst pressure shall be between 90 and 100% of the marked set pressure of the valve."
- API RP520 paragraph 2.3.2.2.2 states: "…the specified burst pressure and set pressure should be the same nominal value."
- EN ISO4126-3 paragraph 7.2 states: "The maximum limit of bursting pressure…shall not exceed 110% of the…set pressure or a gauge pressure of 0.1 bar, whichever is greater…" and "The minimum limit…should not be less than 90% of the…set pressure."

While the statements differ slightly, the basic guidance is the same: make sure the rupture disc's specified burst pressure and relief-valve set pressure are at the same nominal value (ignoring tolerances). Doing so is relatively easy and meets the intent of each standard.

There may be special cases where it is desirable to have these pressures significantly different. In such cases, the user should carefully evaluate the function of both the rupture-disc and the relief-valve to ensure that there are no adverse effects on the performance of either.

## Combination capacity factor

The process of sizing the relief valve is exactly the same whether it is used in combination with a rupture disc or as a standalone relief valve, except for the addition of the combination capacity factor (CCF; known as Fd in EN/ISO 4126-3). This factor represents the ratio of the capacity of the combination to the capacity of the valve by itself.

*CCF = Capacity of the combination / Capacity standalone relief valve*

The default CCF for most codes is 0.90 (in other words, the combination is assumed to have a capacity equal to 90% of the RV rated capacity, if nothing more is known about the actual capacity). EN ISO 4126-3 adds an additional condition on the use of the default CCF, and requires that the petal(s) of the rupture disc be fully contained within the holder after rupture, in order to use the default CCF. Otherwise, a tested or certified value must be used.

CCF values greater than 0.90 may be used in certain cases where specific testing has been conducted on a particular combination of RD and RV product. This is often referred to as a "certified" combination capacity factor (CCCF). For CCFs on specific RV-RD combinations, see the supplemental table at the end of the online version of this article, which be found at www.che.com.

Methods for establishing CCCFs vary based on the applicable code, and are summarized as follows:

***ASME.***

- Testing must be done by an authorized testing laboratory and results registered with the National Board of Boiler and Pressure Vessel Inspectors
- Testing only one size is required to establish a CCCF for a range of other sizes
- Testing with the smallest size and minimum corresponding pressure covers all higher pressures in that size and all larger sizes

***EN ISO 4126-3.***

- No certifying body or laboratory requirements
- One-size method and three-size method are accepted
- One-size method is applicable to all combinations of the same size and design of rupture disc and relief valve equal to or above the tested pressure
- The three-size method is applicable to all combinations of the same design of rupture disc and relief valve in all sizes equal to or greater than the smallest tested size; and pressures equal to or greater than the appropriate minimum pressure for the size

Both ASME and EN/ISO include requirements for establishing nameplate marking to reflect the capacity (or the CCF) of the combination, model and manufacturer of both the rupture disc and the relief valve. Although these are requirements of both ASME and EN/ISO, in reality, this nameplate is rarely supplied, because the components are generally purchased independently, with neither manufacturer aware of the other.

## RD downstream of RV

The following are primary reasons for applying rupture discs downstream of pressure relief valves:

- Prevent corrosion of relief valve
- Prevent fouling or sticking of the relief valve
- Prevent variable superimposed backpressure from affecting the relief-valve operation
- Detect opening or leakage of the relief valve

***Prevent fouling and plugging of the relief valve.*** In situations where relief systems are vented into a common header, the risk exists that blowdown material may enter the vent side of the installed relief valves. Where such vented media can result in either corrosion or polymerization, the external side of the relief-valve mechanism may be affected, resulting in failure to operate when required. By installing a rupture disc with suitable properties at the downstream side of the relief valve, vented media are isolated from the relief valve, therefore avoiding the effects of corrosion and polymerization, and increasing the reliability of the safety system and reducing the need for inspection and maintenance. To ensure that the downstream rupture disc will not impede the proper performance of the relief valve, the burst pressure of the rupture disc should be as low as possible, whereas the provided minimum net flow area of the rupture disc needs to be at least as large as the relief area of the relief-valve outlet.

***Prevent corrosion of the relief-valve internals.*** To avoid corrosion and the resulting need for inspection, maintenance and repair of the relief valve, the use of a downstream rupture disc can be considered. By installing a rupture disc downstream of the relief valve, the rupture disc will act as a chemical seal between the valve outlet and the common header, which could contain potentially corrosive agents from process media released through other emergency relief valves. In this way, corrosion effects of the process media on the valve internals can be eliminated.

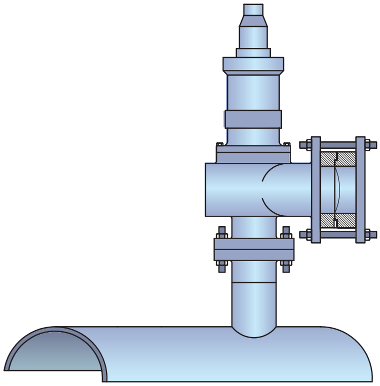

**FIGURE 5. In some cases, it can be advantageous to positon a rupture disc at the relief-valve outlet**

***Prevent backpressure from affecting relief-valve performance.*** Where backpressure can be present, its effects on the performance of the relief valve should be considered. Users can do so by selecting relief-valve attributes like balanced metallic bellows, or by using pilot-operated relief valves. These options will like increase cost and will require additional spare parts and maintenance. As an alternative, the use of downstream rupture-disc devices installed at the relief-valve outlet prevents the relief valve from being exposed to backpressure (Figure 5).

***Detect the leakage or activation of relief valve.*** By detecting the rupture of the downstream rupture-disc device, plant operators can be informed about the upset condition, leading to blowoff. When the interspace between the relief valve outlet and rupture disc is monitored, the leakage of the relief valve can be detected and emissions avoided.

## Benefits

While the use of rupture discs at the downstream side of relief valves is relatively uncommon, that arrangement can offer an array of benefits to the plant owner. The acceptable use of this setup has to comply with the following sizing and set-pressure requirements:

- The minimum net flow area of the rupture-disc device installed at the relief-valve outlet needs to be equal to or larger than the relief-valve-outlet relief area
- The burst pressure of the rupture disc needs to be as low as practical to reduce any effect on the relief-valve performance
- Where applicable, the selected rupture disc needs to be capable of withstanding the backpressures expected from the effluent handling system
- The opening of the rupture disc shall not interfere with the relief-valve opening or performance
- The system design shall consider the adverse effects of any leakage through the relief valve, or through the rupture disc, to ensure performance and reliability
- The relief valve may not fail to open at the expected opening pressure regardless of any backpressure that may accumulate between the relief-valve outlet and the rupture disc. The space between the relief-valve outlet and the rupture disc should be vented and drained (or suitable means should be provided to ensure that an accumulation of pressure does not affect the proper operation of the relief valve). Venting, pressure monitoring and selection of low rupture-disc burst pressures are commonly used to meet these requirements
- The bonnet of a balanced bellows-type relief valve shall be vented to prevent accumulation of pressure in the bonnet that can affect relief-valve set pressure ■

*Edited by Scott Jenkins*

## Author

**Roger Bours** is the pressure-relief sales manager for Fike Europe (Toekomstlaan 52, B2200, Herentals, Belgium; Email: roger.bours@fike.com; Phone: +32 14-210-031). Bours has held the position for nearly 30 years, and has expertise in pressure-relief solutions for a wide range of applications and industries. He specializes in engineered solutions with extended knowledge in industry needs and requirements. Bours is the author of multiple technical papers, white papers and articles, and regularly conducts workshops on pressure relief applications, requirements and issues. He is active in international standardization committees, including: ISO TC185 "Pressure Relief Devices" (Belgian representative since 1986); CEN0 TC 69 WG10 SG2 "Bursting Disc Devices" (Belgian representative since 1990); CEN TC 69 WG10 SG3 "Bursting Disc Devices in Combination with Safety Relief Valves" (Convenor since 1996); CEN TC 305 WG3 "Explosion Venting Devices & Systems" (Belgian representative since 1998).

# Fine Tune the Design of Sour-water Strippers Using Rate-based Simulation

**A look at the benefits of using simulations in the design of SWS units, and some practical tips on SWS operation**

**Soumitro Nagpal**
Fluor Daniel India Pvt. Ltd.

Sour-water strippers (SWSs) are common in petroleum refineries, as well as gas-processing and gasification plants, and are used to remove ammonia, hydrogen sulfide and carbon dioxide from contaminated process waters. The design of these units is typically done using the equilibrium or theoretical stage method. The actual number of trays is determined from the required theoretical stages, using tray efficiencies. The tray efficiencies are not based on actual tray performance, but on experience-based rules of thumb. These rules of thumb are usually conservative to compensate for reduced tray efficiency — due to tray malfunction, foaming or plugging — and to add some design margin for future capacity expansion. Reliable rate-based process simulators are now available for the design of SWS units, and can be used for improved estimation of tray efficiency and for estimating the actual number of trays for optimal design of these units. For existing columns, this approach can be useful for troubleshooting and evaluating available spare capacity.

Equilibrium-stage models, which form the basis for rate-based calculations, are first evaluated by looking at the impact of sour-water feed $H_2S$ and $NH_3$ content, stripper column equilibrium stages on stripped water $NH_3$ and $H_2S$ content, and stripping energy consumption. Two commercial process simulators are used and the results are compared with SWS operating and design data from several sources. Subsequently, a commercially available rate-based model was used to simulate stripper performance and estimate tray efficiencies. The rate-based model calculated significantly higher $NH_3$ tray efficiency (~60%) compared to the common industry practice of using $NH_3$ tray efficiencies of of 20–25%.

Stripper and associated equipment design and operational guidelines are also discussed.

## Conventional methodology

The prevailing design methodology for SWSs is the equilibrium-stage approach. The method determines the number of equilibrium or theoretical stages required to attain a required ammonia level of treated water for a given feed sour-water condition and specific steam consumption. Phenol and hydrogen cyanide removal are also evaluated. The following two commercial process simulators are used:

- HYSYS v7.0 with PR-SOUR property package (HYSYS)
- AspenPlus v7.0 with ELECNRTL property package (ELECNRTL)

A typical SWS unit configuration is shown in Figure 1. The stripper column comprises a steam-heated bottoms reboiler and an overheads condenser. A pump-around type overhead condensing arrangement is another common SWS configuration, and is sometimes preferred, because it can reduce plugging and corrosion problems. This approach, however, entails higher capital expenditure than the overheads-condensing configuration [*1*].

The sour-water feed conditions selected for the evaluation are shown in Table 1.

| TABLE 1. SOUR-WATER FEED AND STRIPPER CONDITIONS | |
|---|---|
| Flow, $Nm^3/h$ | 100 |
| Temperature, °C | 45 |
| Composition, ppmw | |
| $NH_3$ | 8,000 |
| $H_2S$ | 12,000 |
| Phenol | 100 |
| HCN | 100 |
| $H_2O$ | balance |
| Column feed temperature, °C | 110 |
| Column top tray pressure, bara | 2.4 |
| Column bottom tray pressure, bara | 2.7 |
| Reflux drum pressure, bara | 2.1 |
| Reflux drum temperature, °C | 85 |

## Equilibrium model evaluation

Gantz [*2*] presents data from an API-sponsored survey of a large number of SWS operating plants. Data from "questionnaire No. 34" are used. The data represented a packed column with 15 feet of 3-in. Raschig rings, and 1,400 ppmw (parts per million by weight) $NH_3$ in feed. The packed column was found

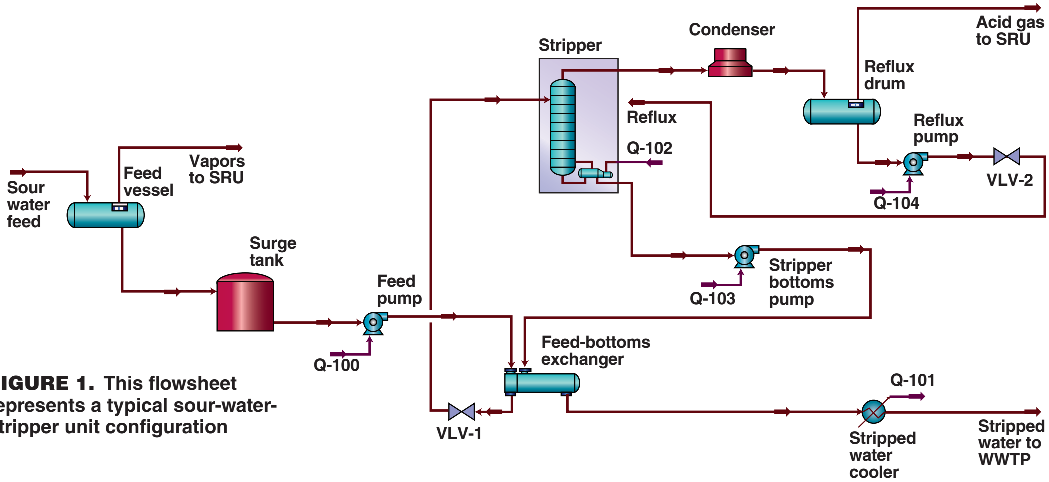


**FIGURE 1.** This flowsheet represents a typical sour-water-stripper unit configuration

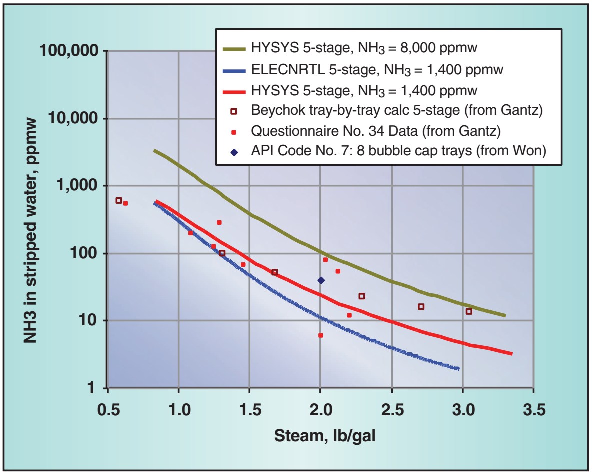


**FIGURE 2.** This graph compares the HYSYS and ELECNRTL models with API and Beychok data

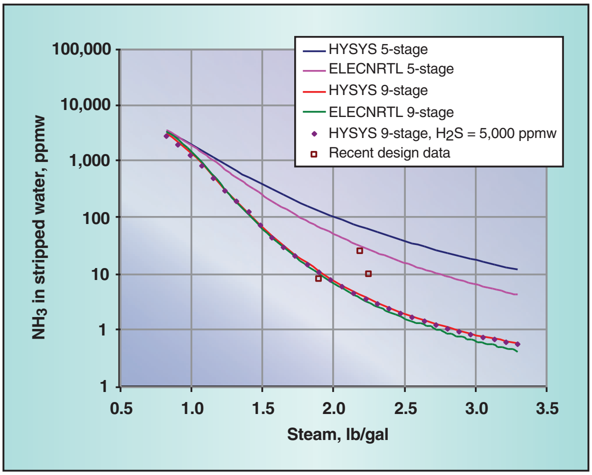


**FIGURE 3.** This graph shows a comparison of HYSYS and ELECNRTL models with design data, and the impact of feed $H_2S$ variation (feed conditions as per Table 1)

to be equivalent to ~4.6 equilibrium stages estimated with the Beychok (see Ref.4, p. 303, for a brief description) tray-by-tray calculation method. Won [3] presents data for a column (API Code No.7) with eight bubble-cap trays. The column had 960 ppmw $NH_3$ in feed, and required ~2 lb/gal of steam to produce stripped water with 40 ppmw $NH_3$. These data were used for the equilibrium model evaluation. The Beychok tray-by-tray calculation for 5 theoretical stages is also compared with the simulators in Figure 2.

## $NH_3$ stripping

The variations of stripped-water $NH_3$ concentration with specific steam consumption (SSC, lb/gal) is shown in Figure 2. SSC is defined as the ratio of reboiler steam flowrate (lb steam/h) and feed sour-water flowrate (gal /h). Also shown are data from questionnaire No. 34 data and the Beychok 5-stage tray-by-tray calculation from Gantz [2]. Both HYSYS and ELECNRTL model results are in fair agreement with the data and Beychok correlation. At high SSC (above 2.0 lb/gal), the Beychok correlation deviates, and $NH_3$ concentrations calculated with both HYSYS and ELECNRTL are lower.

Figure 2 also shows the impact of feed $NH_3$ concentration. Exit $NH_3$ concentrations are quite sensitive to feed $NH_3$ loading, and higher steam consumption can be expected as feed $NH_3$ concentration increases.

Figure 3 compares HYSYS and ELECNRTL results for the base-case feed for 5 and 9 theoretical stripping stages. At SSC below 1 lb/gal, the exit $NH_3$ approach-feed concentrations for all cases are shown. The 5-stage ELECNRTL model gives significantly lower exit $NH_3$ than the HYSYS model at a given SSC. However, for the 9-stage models the results are much closer. Thus, the HYSYS model would give a more conservative design, particularly when considering a low number of stages. Recent design data using 8–10 theoretical stripping stages are shown for comparison. Note that for nine theoretical stages, both simulators calculate an SSC of ~1.9 lb/gal for attaining 10 ppmw $NH_3$ in stripped water.

Incremental reduction in SSC with increasing stages diminishes above nine theoretical stages. Figure 4 shows the relative reduction in SSC attainable as the number of theoretical stages is increased above five.

**TABLE 2. HENRY'S CONSTANT AND ACID DISSOCIATION CONSTANTS OF SOUR-WATER COMPONENTS (25°C)**

| Component | H′ (mol/L-atm) | $pK_a$ | |
|---|---|---|---|
| $CO_2$ | 0.034 | 6.3 | Weak acids |
| $H_2S$ | 0.1 | 7.0 | |
| HCN | 10–12 | 9.4 | Weak bases |
| $NH_3$ | 50–60 | 9.2 | |
| Phenols | 200-3,000 | 9.9 | |

H′ is the Henry's law constant.
$pK_a$ = –log[$K_a$], where $K_a$ is the acid dissociation constant.
For example: $H_2S = H^+ + HS^-$ ; $K_a(H_2S) = [H^+][HS^-]/[H_2S]$

### $H_2S$, $CO_2$, HCN & phenol stripping

Relative solubility or ease of stripping of various sour-water impurities can be evaluated using the Henry's law parameter and dissociation tendency. Table 2 gives Henry's law constants (or physical solubility) and acid dissociation constants ($pK_a$) of various species commonly present in SWS feeds. These are listed in order of increasing physical solubility in water. Species at the top of the table can be expected to strip out more easily than those at the bottom. Thus, $H_2S$ is more easily stripped than $NH_3$ for all cases discussed above, and the stripped-water $H_2S$ concentration was about an order of magnitude lower than the stripped-water $NH_3$ concentration. Increasing feed $H_2S$ levels has a very small impact on the SSC compared to the exit $NH_3$ trend discussed above.

On the other hand, strong acids will complex with basic ammonia and will form heat-stable salts and will be more difficult to strip.

Carbon dioxide is only a marginally stronger "acid gas" than $H_2S$, as evident from its lower $pK_a$, but has a slightly lower physical solubility compared to $H_2S$. Stripping of $CO_2$ can thus be expected to follow the same pattern as that for $H_2S$. HYSYS and ELECNRTL simulation results confirm this, and only a small decrease in SSC is calculated for $CO_2$ stripping as compared to $H_2S$ stripping.

Petroleum refinery hydrotreaters and catalytic crackers produce sour-waters containing hydrogen cyanide as well as water-soluble metal-complexes known as complex cyanides or thiocyanates. Cyanide, if present as free hydrogen cyanide (HCN) can be expected to strip out in the SWS column, because it is a weak base and has lower water solubility than $NH_3$. HYSYS and ELECNRTL models showed >99% removal of free HCN from sour water into the sour gas produced from the stripper. Complex cyanides and thiocyanates are, however, difficult to strip, and can be assumed to remain in stripped water.

Phenols have high water solubility, as evident from their high Henry's law constant, and is difficult to strip out from sour-water. HYSYS and ELECNRTL models both showed <1% removal of phenol ($C_6H_5OH$) from sour water in our simulations. Phenols removal on the order of 0–60% has been reported by operating SWS units.

### Stripper pressure

Stripper pressure is usually governed by process considerations. The simulators indicated small (~5%) reductions in SSC for a stripper- (top) pressure reduction from 2.4 bara to 1.8 bara. The stripper pressure is limited on the low side by flow considerations to the downstream sulfur recovery unit.

### Rectification stages

Three to four rectification stages are usually provided, and can allow reductions in stripping steam. Absence of a rectification section with reflux returned on the feed stage can lead to 20–25% higher steam consumption. The advantages of not having a separate rectification section include cost savings resulting from a common nozzle-inlet device for reflux and feed, and improved phenol removal.

### Feed-bottoms exchanger

The stripper-feed-bottoms exchanger is usually provided to reduce steam consumption. A higher stripper feed temperature reduces reboiler energy input, but increases the condenser and feed-bottoms exchanger duties. This leads to a capital-versus-operating cost trade-off that needs to be analyzed for the optimal feed temperature. A recent analysis for identifying the optimal feed temperature was carried out for pump-around-type overhead systems [*5*]. This study found that lower feed temperatures than commonly employed can result in a more economical design.

### Rate-based design method

Conventional design practice determines the required number of stripping trays by using a tray efficiency of 20–25%. Thus, a design with nine equilibrium stages would translate into a stripper with 36–45 actual trays. Several reliable rate-based process simulators are now available for design of SWS units. In these models, the stripper column is simulated with actual trays, and tray performance is calculated using mass-transfer, interfacial area and heat-transfer correlations. Tray efficiencies are thus an output from the models. These simulators can be used for improved estimation of the actual number of trays required to attain the required level of stripped water quality. The rate-based modeling approach was evaluated using AspenPlus v7.0 with ELECNRTL property package, and RateSep rate-based simulation (referred to as RateSep).

Tray performance and efficiency are dependent on tray design (tray type, tray active area, weir height, and so on). Since these parameters are selected based on column hydraulic calculations and gas-liquid traffic, rate-based models integrate what has been two separate calculations: estimation of contacting stages and number of trays, as well as tray design and sizing.

The SWS column with feed and operating conditions as defined in Table 1 was simulated. A total of 40 trays was used, with 4 trays in the rectification section, and 36 trays in the stripping section with feed on the 5th tray from the top. The Scheffe and Weiland mass-transfer correlation was used. Reboiler duty

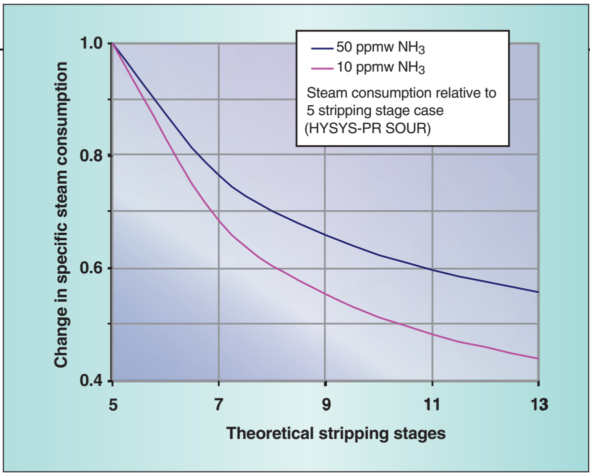


**FIGURE. 4. Shown here is the reduction in specific steam requirement with theoretical stages**

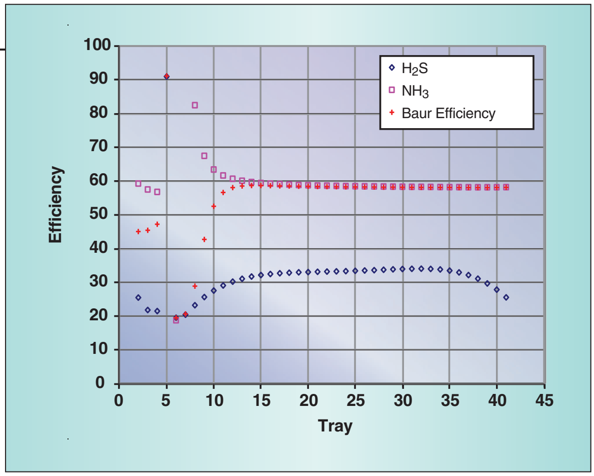


**FIGURE 5. $NH_3$ and $H_2S$ Murphree efficiencies calculated with Aspen RateSep for the base-case feed. Tray numbers are from the column top. Tray 5 is the feed tray. The overall tray Baur [*6*] efficiency is also shown**

was set as a design specification to achieve 10 ppmw $NH_3$ in stripped water. Other column specifications were as follows:

Column diameter: 2.6 m
Tray active area: 70%
Weir height & passes: 5 cm/2-pass
Tray type: Glitsh ballast, V-1 valves

RateSep calculated the specific steam consumption (SSC) to be only 1.25 lb/gal, which is significantly lower than the SSC of 1.9 lb/gal calculated for the nine theoretical-stage simulation. Note that 9 theoretical stages correspond to 36 actual trays, for a tray efficiency of 25%. The lower SSC for the 36 stripping stage RateSep column results from the relatively high $NH_3$ tray efficiency calculated. The Murphree tray efficiencies (EMV) for $NH_3$ and $H_2S$ calculated by the RateSep model are shown in Figure 5. The efficiency range was:
EMV-$H_2S$ ~ 20–35%
EMV-$NH_3$ ~ 58–60%

These rate-based results are supported by plant operating data reported by Gantz [*2*] (See Table 4 in the reference) and Won [*3*] (see Figure 6 in the reference), and suggests that a $NH_3$ tray efficiency of 50–70% is common in sour-water strippers. Also notable is that the eight bubble-cap tray API column data from Won, shown in Figure 2, indicates a tray efficiency of ~60%.

## Murphree versus Baur efficiency

Note that stripped-water $H_2S$ concentrations calculated with RateSep was <1 ppmw in spite of a lower tray efficiency for $H_2S$ than $NH_3$. Table 1 feed conditions modified to contain 4,000 ppmw $CO_2$ and 8,000 ppmw $H_2S$, were run to check the RateSep estimation of $CO_2$ Murphree efficiency. An EMV-$CO_2$ value of 8–16% was calculated, with $CO_2$ in stripped water <0.1 ppmw. These results are consistent with Won's [*3*] findings that "the more volatile the component, the smaller the stage efficiency for that component."

For the SWS column, the Murphree efficiency for $NH_3$ is representative of tray performance. However, the use of Murphree efficiency can be misleading for multi-component systems since higher $H_2S$ and $CO_2$ removal is attained than that for $NH_3$ in spite of lower $H_2S$ and $CO_2$ Murphree efficiencies.

An alternative is to use an overall tray efficiency like the Baur [*6*] efficiency. Figure 5 shows that the Baur efficiency is representative and is close to the $NH_3$ Murphree efficiency in the stripping section of the column.

## Operational issues

***pH adjustment.*** Heat-stable salts (HSS) of $NH_3$ with strong acids cannot be easily stripped, and increasing stripping steam does not reduce the amount of $NH_3$ present as HSS. Operating data that does not account for the presence of $NH_3$ HSS can be misleading, and may suggest an excessive steam requirement (or low tray efficiency) for attaining low stripped $NH_3$ levels.

It is typical to adjust pH in SWS only for removal of HSS of $NH_3$ and $H_2S$. When HSS of $NH_3$ are present, caustic (NaOH) is added to "un-fix" the ammonium salts. The pH above which ammonia is present in solution in its un-ionized form is 10 or higher (pH considered at ambient temperature). Injection of caustic into the stripper column should occur below the point at which most $H_2S$ has already been stripped — usually no higher than four trays from the bottom of the column. The quantity of caustic to be added is estimated from the expected quantities of strong acids (HCl, $H_2SO_4$, $HNO_3$) and weak acids stronger than $H_2S$ and HCN (that is, carboxylic acids) present in the feed. Alternatively, this is estimated by titration of the stripped water that contains the ammonium HSS. The recommended pH measurement-control point for caustic addition is downstream of the stripped-water trim cooler. Use of lime as the alkalinity source is not recommended.

A strong acid is sometimes injected into the sour-water feed to enhance the $H_2S$ removal. However, this is not recommended for sour-waters containing significant levels of feed $NH_3$, as it would result in the formation of the undesirable $NH_3$ heat stable salts.

***Foaming.*** Sour water can contain oil, grease and entrained particulate matter. These result in high foaming and plugging potential in sour-water strippers. This can be addressed by providing a feed tank with adequate holding time allowing separation of the oil and particulate matter. Particle and carbon filters are sometimes provided for troublesome feeds. A low system factor of 0.6–0.65 is used in the stripper-column-diameter calculation because of foaming potential. Excessive foaming with liquid carryover can lead to reduced separation efficiency.

## Concluding remarks

Several commercial simulators now offer rate-based models for sour-water stripper calculations. These models can allow optimal design, revamp and trouble shooting of these strippers. However, they require more time for set-up and experienced users are needed to produce reliable design calculations. This article validates equilibrium models and evaluates commercially available rate-based models to estimate tray efficiencies attainable in the sour-water stripper columns. The rate-based model calculates significantly higher $NH_3$ tray efficiency compared to the common industry guideline of using tray efficiency of 20–25%. The conventional industry design method includes a significant design margin, which may not always be justified and should be reviewed based on the specific feed composition and inclusion of feed surge drum and filters in the design allowing for removal of impurities and effective use of the trays in the stripper column. ■

*Edited by Gerald Ondrey*

**Disclaimer**
The conclusions presented in this article are solely those of the author, and cannot be ascribed to Fluor Corporation and/or any of its subsidiaries. The reference to the commercial simulators does not constitute an endorsement of these products or compare them to other simulators available in the market.

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## Author

**Soumitro Nagpal** is a senior process engineer with Fluor Daniel India (Building 8B, 3rd Floor, DLF Cyber City, Gurgaon, Haryana 122002, India; Email: soumitro.nagpal@fluor.com). He has 24 years work experience in the oil and gas industry in process development, design, engineering and management. He has wide-ranging experience in process modeling and simulation, and is a Fluor team leader for executing dynamic simulation projects for the oil-and-gas and power industries. Soumitro is a chemical engineering graduate of BITS, Pilani (B.E.) and the University of Utah (Ph.D.).

Engineering Practice

# Compressors for Specialized Applications

**Follow this guidance to improve the selection, operation and safety of compressors that face challenging conditions**

**Amin Almasi**
Consultant

Siemens

**FIGURE 1. This high-pressure centrifugal compressor has a single shaft and vertically split casing. This compressor model has been qualified for many industrial process situations and is suitable for use with a wide range of process gases, in applications including hydrocracking, synthesis gas handling, gas storage and injection. Its benefits include high discharge pressure and an excellent degree of efficiency**

Compressors for specialized chemical process industries (CPI) applications — particularly high-pressure applications (involving large- and medium-sized machines) and those calling for small compressor units — present particular challenges to system designers and operators. This article discusses a variety of engineering recommendations that should be considered to address these challenges. This discussion focuses on five specific areas:

- High-pressure compressors
- High-pressure dry-gas seals
- High-pressure internal seals
- Dynamic simulation of high-pressure turbocompressors
- Issues associated with the surge zone

Each section discusses high-pressure centrifugal compressors, in the medium to large size range (1–40 MW) for high-pressure service (100–900 barg). The final two sections also discuss engineering challenges that arise with small compressors (10–100 kW) for specialized CPI applications.

## Taking a closer look

***High-pressure compressors.*** A vertically split compressor casing with a heavy-bolted head is a classic design for high-pressure applications, but this heavy-bolted design is not practical for very high pressures because there may not be enough space on the compressor head for the heavy bolts that are required. As a general rule of thumb, for some specific high-pressure services that handle low-molecular-weight gases, heavy-bolted designs are not recommended for pressure above 150 barg.

Today, the improved — and now commonly used — configuration for modern, high-pressure applications is the use of a shear-ring cover-head that eliminates bolts at the compressor head. The end covers are retained by shear-ring segments. The compressor casing is generally forged from a suitable grade of steel.

Different arrangements and driver types are available for high-pressure turbocompressors. Electric-motor drivers are used in small- and medium-sized compression trains. Traditionally, these were popular in the 1–15 MW range, but for the last decade or so, modern electric motors have been used in the 15–40 MW range for different applications.

One successful design for electric-driven trains is a two-end, electric-motor arrangement that uses a dedicated gear unit at each end to drive two compressor casings. Using this configuration, an optimum speed can be achieved for each compressor casing. Compressor bundles can easily be removed from each side.

***Small compressors.*** The small CPI compressor market is a special, evolving and challenging market.

Small compressors are required in many CPI units for different services. Examples include flare-gas recovery units, process-gas recovery systems, small booster compressors, small recycle machines, some small-flow extraction services, tiny refrigeration units, small gas-blowing systems, tiny process-vacuum machines, small nitrogen or air compressors, small fuel-gas units and many more.

The market for small and tiny compressors is different from that of medium and large compressors. Two important characteristics of the small and tiny compressor package market are as follows:

- For many situations that require tiny compressors, these units are purchased in higher quantities (for instance, a facility may purchase several small, identical gas-recovery compressor packages for use in different units of the same plant or different plants and facilities). The specific aspects and capabilities of tiny compressors tend to be very vendor-dependent. The specific vendor, competing designs and specific optimization options all play a critical role, beyond just selection of the compressor type and model
- Tiny compressors are highly dependent on vendor design (manufacturing details) and proper size (or model) availability. For instance, a given compressor vendor may have an appropriate size and model available that can be a perfect match for applications that require a tiny compressor. In this situation, other compressor types, sizes or models (and other vendors) may not be able to compete with that specific compressor package

Small compressor packages are highly specialized machines that are highly reliant on the application, size, manufacturing details, available models and vendors.

***High-pressure compressors.*** Old-fashioned, high-pressure reciprocating compressors and oil-sealed, high-pressure centrifugal compressors with heavy bolted heads have been used in CPI plants for decades. They are no longer suitable for modern CPI plants. Vertically split, centrifugal compressors with shear-ring heads and dry-gas seals are advanced options for high-pressure CPI service. Modern, high-pressure turbocompressors are designed with special focus on critical areas and functions, such as rotor-dynamics, thermodynamic performance, potential excitations, the risk of rotating stall, auxiliary systems, fabrication processes, internal seals, dry-gas seals and anti-surge systems.

High-pressure compressors generally use closed-type, three-dimensional impellers. Both efficiency and pressure coefficients are higher for three-dimensional impellers compared to old-fashioned two-dimensional ones. Impellers are usually manufactured from low-alloy steels. In-depth investigations of material characteristics should be carried out to examine the material integrity with regard to potential failure modes (such as sulfide stress cracking; SSC). One major problem is that even after the transition of the gas phase to a dense phase (fluid passing the critical point when it becomes supercritical fluid), the water could condense if the gas is sufficiently cooled. SSC can occur in minutes if a susceptible material under stress is exposed to a sour wet condition inside the compressor in the event of any wet upset.

For high-pressure compressors, the thrust force generated by an impeller assembly may be much greater than what the thrust bearing can handle. Therefore, thrust forces should be balanced by using a back-to-back impeller arrangement (double-entry impeller series) and balance piston(s)) — usually with two balance pistons. The remaining axial force is the difference between the forces, which are relatively large in magnitude compared to the load on the thrust bearing. In various operating conditions, the load on thrust bearing can vary considerably — it might even change the direction. In many cases, an advanced, double-acting, tilting-pad thrust bearing should be used.

Vibrations in high-pressure turbocompressors are usually controlled by employing very stiff rotor designs. Sufficient stability of the rotor should be ensured under any potential anticipated operating or malfunction conditions. Maximizing rotor stiffness is achieved by limiting the number of impellers per casing. In general, the best recommendations involve the shortest possible bearing span and the maximum shaft diameter.

Modern high-pressure compressors (which tend to be high-speed machines) typically only use tilting-pad bearings or magnetic bearings. These bearings help to inhibit bearing cross-coupling excitation forces and other instabilities.

In high-pressure applications, the fluid density can become high, which presents a great challenge for the design and dynamics of the machine. Above 200 barg, an average gas density of around 150–300 kg/m$^3$ has been reported. This high fluid density lowers the rotating assembly's natural frequency, which increases the risk of resonance (a source of instability). For example, in a case study for a high-pressure centrifugal compressor handling process gas, the natural frequency at the operational pressure was decreased to 70% of the natural frequency at a low-pressure operation. To prevent such instability, the operating speed range should often be limited. The ratio of outlet width to passage width of an impeller is an important factor in this regard.

An accurate calculation of gas properties is very important for the realistic modeling of a high-pressure compressor. A "shaker test" is usually required for a high-pressure compressor to verify rotor-dynamics behavior and stable operation. The main aim of this test is to determine the damping of a rotor system at various operating conditions, over the entire range of compressor speeds and characteristics.

Generally, a suitable, broad-range device for generating vibration (a shaker) should be employed. One modern procedure for performing this test involves fitting the end cover of the barrel carrying the rotor with a temporary extension that is designed to host an active magnetic bearing at the rotor-shaft extension. This magnetic bearing is then operated in a manner that induces vibration on the rotating shaft. The overall damping of the compressor is determined by evaluating the rotor's vibration response to the nonsynchronous excitations produced by the exciter.

During the test, around 7–20 operating points are typically measured, and the modal frequency and damping are evaluated. Generally, the compressor should reveal satisfactory stability (supercritical damping) behavior and a very low vibration level under all operating conditions and excitation modes. A limited number of compressor manufacturers can offer the "shaker test" for verifying a high-speed compressor's dynamic performance. Requirements should be established before a compressor's order.

Specific attention should be given to the rated head. Specifically, a suitable margin should be established, to cover uncertainties related to gas properties, fouling and potential performance deterioration in high-pressure service. The material selection, stress analysis, fatigue study and quality control of compressor shafts are also important during the selection and design of compressors for high-pressure use.

Sufficiently low stresses (as low as 20% of the minimum yield strength) are recommended for regions of the shaft that might be wetted by the process gas. Impeller cracking caused by welded fabrication is a widespread problem. The electro-discharge machining process (as an alternative to welding) is recommended for the fabrication of high-pressure impellers. Modern finite element (FE) studies using fine meshing techniques are required, with a special focus on localized areas that are likely to experience high stress.

***High-pressure dry-gas seals.*** Shaft end seals in modern, high-pressure compressors are generally dry-gas seals. Oil seals were common in older designs, but are no longer used because of high power losses, low reliability, potential oil contamination, and many other operational problems. However, high pressure presents challenges to any dry-gas seal applications. There are a few dry-gas seal sub-suppliers for high-pressure services. Special attention is required because of the possibility of damage upon a rapid depressurization and a potentially explosive decompression.

The best sealing system for a CPI high-pressure compressor is usually the tandem dry-gas seal with an intermediate labyrinth. At high pressures, tremendous forces are caused by the gas load acting on the seal in both the radial and axial directions. To ensure the maximum stability of seals at such high loads, cross-sections of metal sleeves in seal cartridges should be large enough. For instance, if only one sleeve is used for a high-pressure seal, then relatively small cross-sections would be too weak (with improperly distributed load, and high localized stresses) to handle a high axial load. In a high-pressure design, sleeves should be split into several sections (two or three) to distribute the load and ensure the maximum stability of the seal.

The seal gap should be designed to be as small as possible. However, free movement of the seal mating faces should be ensured under all operating conditions. The functional gap should be no more than a certain level (usually micrometer levels). This very small gap value makes it a tough challenge to design and manufacture the seal precisely because the variation of gap under the influence of temperature and pressure has to be minimized. In other words, thermal expansions and contractions, as well as component deflections and displacements under pressures could be more than several micrometers. These thermal movements and pressure displacements can affect the seal's very small gap, unless all these effects are evaluated and properly considered.

Startup of a high-pressure compressor can be the most severe running condition for the seal since the operating gap could reach the lowest value. If a seal design is completed based on a relatively large gap margin at low speed and maximum pressure, this would certainly lead to excessive leakage rates. To avoid this, high-pressure seal design values should be optimized to avoid circumferential rubbing marks caused by insufficient gap sizes or a high leakage rate. In other words, as much as possible, an optimum design must be achieved for the seal, to account for all required operating conditions.

Critical areas for a high-pressure seal design are:

- Detailed features on the seal face (especially with respect to specific components, such as the primary ring and the mating ring, the manufacturing taper, groove design, tuning step and so on). In particular, detailed design of the support ring and the sleeve (usually if fabricated from tungsten carbide) can directly affect the seal gap
- A robust structural design for major seal components that are subject to the gas at high pressure

Note that the seal design can become problematic when extreme operating conditions are present and a large number of design parameters have to be optimized to accommodate different operating modes. In addition, there are rarely isolated effects (or parameters). Many parameters could affect one another. For instance, changing one parameter or design aspect in an effort to optimize the system will have an impact on other parameters. Moreover, effects are often contradictory. For example, although increasing the initial taper in a dry-gas seal can essentially improve the lift-off characteristics, at the same time, the radial gas-film stiffness (that is, the stiffness at radial direction assigned to the gas film in a seal) and seal performance at the maximum speed could deteriorate.

***High-pressure internal seals.*** The high density of fluid in a high-pressure compressor increases the tendency to whirl. This high density could magnify excitation forces generated in balance-piston seals and other internal seals, which can result in instability. The stiffness and damping properties of various seals within a high-pressure compressor are strictly related to the seal's shape, gap, design, manufacturing and quality-control efforts. As a result, special attention is required during the selection, design, inspection and operation of internal seals.

Generally, various methods are used to eliminate the magnitude and effect of cross-coupling forces. Features that inhibit the tangential swirl can help to reduce the cross-coupling forces. However, such special features may only be needed above pressures of 150 barg. These solutions are usually based on empirical data, comparison with successful designs and testing at full pressure.

Traditional labyrinth seals provide much lower levels of stiffness, damping and forces compared to modern internal seal designs (such as honeycomb seals or hole-pattern seals, which are discussed in this article). These traditional labyrinth seals usually do not influence rotor dynamics and damping. The forces generated by this type of seal tend to be much less sensitive to seal-clearance changes, seal gaps and seal details.

In the case of modern internal seals (such as honeycomb seals or the hole-pattern seals, which usually present nonlinear behavior), the stiffness and damping are higher than old ones. Overall, these modern internal seals are better than traditional labyrinth seals. However, seal stiffness, vibration modes (such as bending modes) and natural frequencies can be significantly affected by seal operation. As a result, while these modern seal options offer better operation, they could result in rotor instability if the system is not modeled and designed properly.

For traditional labyrinth seals, damping coefficients can be identified with reasonable accuracy by carrying out a linear curve fit to measure forces in the radial and tangential directions. However, this old-fashioned description of forces (in terms of relatively constant stiffness and damping coefficients) cannot be applied in the case of a modern seal, such as a honeycomb seal or a hole-pattern seal. An accurate nonlinear approach must be selected for proper modeling of modern seals.

Honeycomb seals and hole-pattern seals have been successfully used in different high-pressure CPI compressor applications. In these applications, good damping capability and a tremendous positive stiffness for seals are most important. In simple terms, modern honeycomb seals and hole-pattern seals are designed — and their engineering and details are set — in a way that offers suitably high damping and stiffness at different operating scenarios. All these can result in a better stability of any compressor rotor. In order to ensure high stiffness of seals, it is necessary to keep the seal clearance within defined range at all times. During any transient operation (such as a startup or a shutdown), the seal clearance could become particularly reduced as a result of transient impacts such as thermal effects. In steady-state operation, the thermal effect could be less pronounced, whereas the deformation caused by the pressure difference (for instance, over balance piston) can affect the seal geometry. The higher the pressure difference across the seal, the more the seal gap tends to be affected.

Modern hole-pattern seals and honeycomb seals provide positive stiffness, increasing by a considerable amount (even a 100% rise or more) from the unloaded operation (usually the lowest vibration mode) to the loaded. This is a desirable behavior. With this high positive stiffness, the seal acts like an additional bearing, and can offer better damping and dynamic behavior. The first natural frequency often increases from the ones at an unloaded operation to around 1.5 times (compared to the unloaded operation) at the fully-loaded (100% load) condition. In this way, the flexibility ratio of a loaded machine is reduced (rotor assembly becomes stiffer when machine-loaded), meaning that this configuration could be inherently stable.

The margin of the stability threshold (the distance between the operating point and points of potential instability) should be sufficiently high. The compressor seal should be optimized with respect to leakage, performance, reliability and stability, particularly without impact to the rotor dynamics. In other words, the above-mentioned parameters (leakage and so on) should be optimized, but in a way that does not adversely affect rotordynamics.

Hole-pattern seals present a superior damping ratio (usually twice the damping ratio of a labyrinth seal). In particular, a modern hole-pattern seal exhibits good damping performance in the middle of a compressor characteristic curve (say, around the normal operating region). The damping ratio increases with increased discharge pressure.

If the seal gap in the operation could not be kept within the defined range — and particularly, if the gap's variations and its changing rates are not within allowable limits — then the seal (such as a honeycomb or hole-pattern seal) can show some variations in the rotor natural frequency with increasing discharge pressure. Efforts to increase damping are encouraged, but variation in seal stiffness (and the rotor natural frequency) over time is not desired. This is not the recommended design, because the turbocompressor stability could be affected over time by fouling, degradation or similar effects. Seal clearance should be kept as low as possible, relatively constant and within the defined range at all operating conditions.

## Dynamic simulation

The use of dynamic simulation for high-pressure turbocompressor systems has been established as a powerful tool for analyzing the behavior of a compressor system in different situations and under different operating conditions. The use of dynamic simulation and an anti-surge system design are extremely important for high-pressure turbocompressor applications. For example, specific dynamic simulations should be carried out to do the following:

- Support the design (in terms of anti-surge system selection and sizing, advanced process control system and more)
- Support safety and reliability analyses (related to, for instance, surge events, unit blowdown and so on)
- Verify operational parameters and operator training

An important aspect of the complexity of a high-pressure turbocompressor system is the overlay of dynamic behavior of different turbo-compressors and the dynamic interactions that result from the entire system of piping and components that are connected to the turbo-compressors. Operators should carry out dynamic simulations that cover the most likely combinations of operational situations for turbo-compressors within different operational modes of a CPI unit.

At least three dynamic simulations are required for a typical CPI turbo-compressor system:

1. The first dynamic simulation — at the front-end engineering design (FEED) or basic design stage. This simulation is mainly to support the basic system arrangement and to avoid making major changes later. The resulting insight is critical to support appropriate decisions at an early stage of a project.
2. The detailed-design dynamic simulation — during the detailed design stage. This simulation is mainly for the verification of design (or sizing) of various components or sub-systems.
3. The operational dynamic simulation — during commissioning or startup. This is mainly for the verification of real operational parameters such as control details, alarm points, controller settings and similar items. It is important to identify correct operating procedures, and best operator reactions (for instance, operator actions in the event of an upset or alarm). As-built and final data should be employed at this stage.

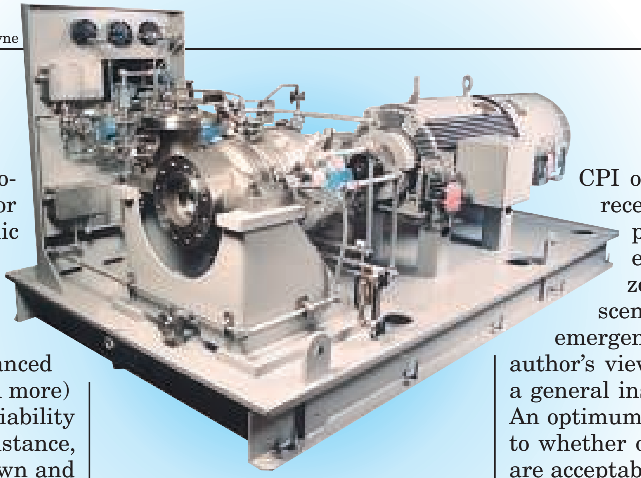



**FIGURE 2.** This horizontal-train, electric-motor-driven, high-pressure compressor shows the vertically split casing. The single-casing compressor, integral gear unit and an electric motor driver with accessories and auxiliaries were installed on a heavy-duty skid

One of the most important decisions for high-pressure turbocompressors is the selection of hot-gas-bypass valve. Performing selected dynamic scenarios in an early phase of the project can aid in the decision of whether to include (or exclude) a hot-gas-bypass valve and can help to inform related modifications such as the finalization of the check-valve position or the need for additional vent valves. The ability to make these decisions early in the process can have a significant economic impact on a CPI project budget.

## Consider the surge zone

Some high-pressure turbocompressors could experience a short excursion into the surge zone during an emergency shutdown.

For instance, when a hot-gas-bypass valve has not been selected, the willingness to allow for a short excursion into the surge zone is adopted as a suitable response. In other words, a short excursion into the surge zone is becoming acceptable by many vendors and some CPI operating companies.

Historically, a wide ranges of explanations and reasons have been presented by different vendors to justify short excursions into the surge zone. These include "controlling a surge event," or saying "a few surge cycles can be easily tolerated," or something similar. Today, CPI operators can expect to receive a centrifugal compressor with a short excursion into the surge zone for some operating scenarios (particularly an emergency shutdown). In the author's view, it is difficult to give a general instruction on this issue. An optimum design (that is related to whether or not short excursions are acceptable or not) depends very much on the application. In the author's view, regardless of whether this design is an optimum design or not, a correct operating strategy should be selected for these so-called marginally designed, high-pressure turbocompressors.

In this way, turbocompressor emergency shutdowns should be limited to necessary situations — such as a pressure trip caused by low lubrication-oil pressure — since cutoff of lubrication oil can be the most critical issue for turbocompressors. The second fatal case could be a full-power surge. Other emergency situations (such as high vibration, high temperature, a CPI plant trip and others) — if allowed to cause an immediate trip — could potentially shut down the high-pressure turbocompressor at an operating point very close to the surge line, which can result in a high-energy surge that is often more destructive and dangerous than the initial reason for the emergency trip.

Using this approach, in the event of an operational issue (as noted, except a lubrication-oil-related trip), a suitable alarm will be issued and the operating system should be designed to move the turbocompressor to an operating point far from the surge point (and probably initiate the anti-surge valve operation), before a well-coordinated shutdown in a short time. The time delay for this well-organized shutdown (probably 1–4 s) is much less risky than a high-head, high-energy surge in a high-pressure turbo-compressor.

## Options for small compressors

***Sliding (rotary) vane compressors.*** This type of compressor has a solid rotor mounted inside a liquid-jacketed cylinder, similar to that of the jacketed section of a reciprocating cylinder. The liquid jacket around the cylinder is used for cooling. The rotor is filled with blades, which are free to move in and out of longitudinal slots in a rotor. Blade configurations range from 8 to 12 blades, depending on the manufacturer and required pressure differentials. Blades are forced out against the cylinder wall by a centrifugal force, creating individual cells of gas that are compressed as the rotor turns. As the gas approaches the discharge port, the area is reduced and the gas discharged.

Sliding (rotary) vane compressors are designed for use in harsh environments. When it comes to dirty process gases, sour gases, wet gases, flare-recovery gases, vapor-recovery services and other dirty or low-pressure-ratio CPI applications, the sliding-vane compressor is widely used. Unlike other types of compressors that are used for small compression units (such as oil-flooded screw compressors, where the lubrication oil is mixed with the gas stream and is then reused), the lubrication of a sliding-vane compressor usually relies on once-through lubrication, thus not contaminating or breaking down the oil viscosity.

The sliding (rotary) vane system design does not have critical tolerances, due to its design and working principles. This is in contrast to a reciprocating compressor or an oil-flooded screw compressor, which require tight tolerances for proper operation. Since sliding (rotary) vane compressors do not operate at close tolerances, it could be considered a rugged and reliable machine for many applications.

***Reciprocating compressors.*** There are some disadvantages for tiny reciprocating compressors. The first disadvantage of reciprocating compressors is low reliability (and low availability). Another critical issue is pulsation. Some CPI operators of low-flow (small) services still prefer small reciprocating compressors to other compressor types (such as oil-flooded screw compressors or sliding-vane compressors). These CPI operators have reported acceptable reliability and acceptable maintenance expenses associated with these units. The reality is that the reciprocating-compressor technology is a mature technology. There are more manufacturers that supply these machines compared to other options (such as oil-flooded screw compressors), and today, competition among vendors has improved reliability and reduced spare-part and maintenance costs for specific industries. These vendors have also accumulated a wealth of knowledge and experience in design, fabrication, operation and troubleshooting of reciprocating compressors.

For many applications, reciprocating compressor technology may be inherently less reliable and more expensive than competing alternatives, and requires a bigger and heavier machine. However, reciprocating compressors are often a good option for some specific small or tiny compressor units.

***Oil-flooded screw compressors.*** To provide lubrication, sealing and cooling and to avoid any metal-to-metal contact, the lubrication oil is injected into the compression chamber of oil-flooded screw compressors. As screw rotors mesh, the compression chamber volume decreases, compressing the gas/lubricant mixture toward the discharge port, where the stream exits as the chamber passes over the port. Oil-flooded screw compressors are often a good option in medium- and low-volume applications and those with low suction pressure. The continuous flow of cooling lubricant permits much higher single-stage compression ratios and suction-pressure variations. In some cases, this can help to eliminate the need for an aftercooler for many small machines. An oil-flooded screw compressor could be a good option for many CPI tiny or small compressors.

There is no general rule for the selection of small/tiny (10–100 kW) compressor type. One of the above-mentioned three compressor types can usually be used for a small-compressor unit application. The oil-flooded screw compressor is technically the best option for many CPI applications, however, the reciprocating compressor or sliding-vane compressor could also be used.

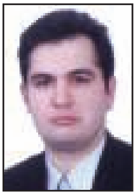
Generally, a rotary-vane compressor offers the minimum cost. For some applications, this option may cost 10–25% less than a comparable oil-flooded screw machine. Meanwhile, sliding-vane compressors are often selected if the oil-gas combination can be anticipated to cause problems in the oil-flooded screw compressor. This could be the case for some process-gas streams with heavy gases and other elements.

## Final notes on small compressors

The small compressor market (10–100 kW) is an evolving and changing market. When it comes to the need for small or tiny compressors, one of the following compressor types should be used: the oil-flooded screw compressor, the reciprocating compressor and the sliding (rotary) vane compressor. The oil-flooded screw compressor could technically be the best option for many applications. The small compressor package should usually be flexible both for the suction pressure and for the capacity with an acceptable efficiency at a part-load operation. The desired suction-pressure range often is from a low pressure (even 0 barg or lower) to a higher suction pressure (more than two-times the rated suction pressure). ∎

*Edited by Suzanne Shelley*

## Author



**Amin Almasi** is a rotating equipment consultant in Australia (Email: amin.almasi@ymail.com). He previously worked at Worley Parsons Services Pty Ltd. (Brisbane, Australia), Technicas Reunidas (Madrid, Spain) and Fluor Corp. (various offices). He holds a chartered professional engineer license from Engineers Australia (MIEAust CPEng – Mechanical), a chartered engineer certificate from IMechE (CEng MIMechE), RPEQ (registered professional engineer in Queensland) and he also holds M.S. and B.S. degrees in mechanical engineering. He specializes in rotating machines including centrifugal, screw and reciprocating compressors, gas and steam turbines, pumps, condition monitoring and reliability. Almasi is an active member of Engineers Australia, IMechE, ASME, Vibration Institute, SPE, IEEE, and IDGTE. He has authored more than 60 papers and articles dealing with rotating machines.

# Counterintuitive revamp results

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

At the 2011 Spring AIChE meeting (Chicago, Ill.), Sean Hennigan of BP gave a presentation regarding a column troubleshoot that yielded a counterintuitive result. A column bottleneck was ultimately traced to the presence of a vacuum cleaner in a tray downcomer. I wondered out loud, after that presentation, whether all tray downcomers should be equipped with vacuum cleaners to reduce liquid back-ups.

At the 2014 Spring AIChE meeting (New Orleans, La.), Hennigan gave another interesting presentation entitled, "A High Capacity Re-Tray that Wasn't — a Practical Lesson in Counter-Intuitive Thinking." His story and contentions follow. Generally, in distillation and absorption columns, the replacement of sieve trays with fixed-valve trays yields 5–10% capacity increases. Compared to sieve trays, fixed-valve trays have lower froth heights and lower pressure drops, especially when those trays have larger deck open-areas. Along those lines, Hennigan described an experience where a simple atmospheric-pressure distillation column was revamped by replacing sieve trays with such fixed-valve trays. Upon re-start, the new column's pressure drop was unstable, and higher, not lower. More importantly, the column's capacity was lower, not higher.

During the subsequent troubleshooting efforts, appreciable data from before and after the revamp were compared. The increased pressure drop and the decreased capacity were both confirmed. Eventually, the reasons were identified. The inherently lower pressure drop of the fixed-valve trays led to a lower bottom pressure in the tower. By the ideal gas law, the lower pressure increased the vapor volumetric flowrate, which proved to be unmanageable for the new trays. Thereafter, several solution possibilities were considered, including leaving the new trays in place and increasing the column's operating pressure. Indeed, that proved to be the correct solution.

When the column's top operating pressure was increased to higher than atmospheric pressure, as generally happens, the relative volatility between the key components decreased, and the separation became more difficult. To the contrary, however, the increased bottom pressure led to an increased bottom vapor density and a decreased vapor volumetric flowrate — and an increased column capacity. In retrospect, immediately following the tray changeout, the lower pressure drop of the higher-capacity trays had led to a counterintuitive result: a lower column capacity. The capacity of those new trays was truly higher, but the lower pressure drop had yielded a lower bottom pressure, a lower bottom vapor density and a higher vapor volumetric flowrate. The pressure effect had dominated.

Hennigan stressed the importance of at least two process simulations for any column revamp involving trays or packings. Near the end of his presentation, he said that he was wary of another similar revamp of an atmospheric-pressure column. I wondered, out loud, whether all revamps of atmospheric-pressure columns should be replaced by simple increases in column operating pressures. Hennigan just smiled. ■

*Mike Resetarits*

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

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Fax: +49-69-5700-2484
Email: ptrautes@che.com
**Austria, Czech Republic, Benelux, Eastern Europe, Germany, Scandinavia, Switzerland, United Kingdom**

***Dipali Dhar***
*Chemical Engineering*
88 Pine Street, 5th floor, New York, NY 10005
Tel: 718-263-1162
*E-mail:* ddhar@accessintel.com
**India**

***Katshuhiro Ishii***
*Chemical Engineering*
Ace Media Service Inc., 12-6, 4-chome
Nishiiko, Adachi-ku, Tokyo 121, Japan
Tel: 81-3-5691-3335; Fax: 81-3-5691-3336
E-mail: amskatsu@dream.com
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Silvera Pubblicita
Viale Monza, 24 Milano 20127, Italy
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*E-mail:* ferruccio@silvera.it/www.silvera.it
**Andorra, France, Gibraltar, Greece, Israel, Italy, Portugal, Spain**

***Rudy Teng***
**Sales Representative**
*Chemical Engineering;*
8F-1 #181 Wulin Road
Hsinchu 30055 Taiwan
Tel: +86 13818181202, (China),
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Fax: +86 21 54183567
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## JUNE WHO'S WHO


Kane


Sandmeyer


Kelley


Hambrecht


Johnson

**Pittsburgh Corning Corp.** (Pittsburgh, Pa.), a maker of specialized insulation and glass block products, names *James Kane* chairman and CEO, succeeding *Phillip Martineau*.

*R. Paul Sandmeyer III* joins **Sandmeyer Steel Co.** (Philadelphia, Pa.) as materials procurement manager, replacing *Gerald Geiger, Sr.*, who is retiring.

Specialty chemicals maker **Evonik Industries AG** (Essen, Germany) names *Michael Pack* head of its Performance Polymers Business Unit. He succeeds *Gregor Hetzke*, who assumes leadership of the company's Advanced Intermediates Business Unit.

Engineering, construction and services company **KBR** (Houston) names *Stuart Bradie* president and CEO. He succeeds *William "Bill" Utt*, who is retiring.

*Paul Kelley* joins **Allylix Inc.** (San Diego, Calif.), a renewable chemicals company, as vice president of operations.

**BASF SE** (Ludwigshafen, Germany) names *Jürgen Hambrecht* chairman.

*Juan Johnson* becomes general manager of **Seal Pots USA** (Broken Arrow, Okla.), a unit of sealing company EagleBurgmann GmbH & Co KG.

Specialty chemicals maker **Archroma** (Reinach, the Netherlands), appoints *Miguel De Bellis* president of emulsion products and Americas, in Sao Paulo, Brazil. ■

*Suzanne Shelley*

# BUSINESS NEWS

## PLANT WATCH

**Kemira to build sodium chlorate plant for pulp mill in Brazil**

May 9, 2014 — Kemira Oyj (Helsinki, Finland; www.kemira.com) will supply sodium chlorate to Klabin's new pulp mill in Paraná, Brazil. Kemira will build, own and operate the sodium chlorate plant, which is scheduled to begin production during the first half of 2016.

**BASF and Yara plan to build ammonia plant in Freeport, Tex.**

May 8, 2014 — BASF SE (Ludwigshafen, Germany; www.basf.com) and Yara (Osöo, Norway; www.yara.com) plan to jointly build an ammonia plant on the U.S. Gulf Coast. The proposed plant would be located at the existing BASF site in Freeport, Tex., and would have a capacity of 750,000 metric tons per year (m.t./yr), based on a hydrogen-synthesis production process.

**AkzoNobel inaugurates membrane-electrolysis plant in Germany**

May 6, 2014 — AkzoNobel N.V. (Amsterdam, the Netherlands; www.akzonobel.com) has inaugurated a new chlorine membrane-electrolysis plant in the Rhein-Main area of Germany. The plant is the result of a major conversion and expansion project begun in 2011 involving a €140 million investment. Capacity has been expanded by 50%, resulting in a chlorine production capability of 250,000 m.t./yr.

**BASF and Sinopec to build a new NPG-production plant in Nanjing**

May 6, 2014 — BASF SE and Sinopec Corp. (Beijing, China; www.sinopec.com) will build a new production plant for neopentylglycol at their Verbund site, BASF-YPC Co., a 50-50 joint venture (JV) in Nanjing, China. The plant, with a planned total capacity of about 40,000 m.t./yr, is scheduled to go onstream at the end of 2015.

**LyondellBasell granted key permits for ethylene expansion project**

May 2, 2014 — LyondellBasell (Rotterdam, the Netherlands; www.lyondellbasell.com) has received a key permit required in the company's multi-plant ethylene expansion program, which, when fully operational, is expected to increase annual ethylene capacity by an estimated 840,000 m.t./yr, for a total estimated capacity of around 5.35 million m.t./yr in North America. The ethylene expansion program began in 2013 and represents a total investment of approximately $1.3 billion in the company's Channelview, La Porte and Corpus Christi, Tex. plants. Startup at the Channelview plant is planned for early 2015. Construction at Corpus Christi is expected to begin in the second half of 2014, with startup scheduled for late 2015.

**Arkema inaugurates new electrolysis unit in Grenoble, France**

April 30, 2014 — Arkema (Colombes, France; www.arkema.com) has inaugurated a new electrolysis unit at its Jarrie plant near Grenoble, France. This €100-million investment has enabled the facility to adapt to the latest regulations on industrial risks and to convert its mercury electrolysis process to membrane technology.

**Celanese will add polyphenylene sulfide compounding line in China**

April 24, 2014 — Celanese Corp. (Irving, Tex.; www.celanese.com) has announced that the company will expand its compounding capabilities at its Nanjing, China, integrated chemical complex to include polyphenylene sulfide (PPS). The PPS compounding expansion is expected to be operational by year-end 2014.

**Milliken to double production capacity for polypropylene clarifying agent**

April 24, 2014 — Milliken & Co. (Spartanburg, S.C.; www.milliken.com) has unveiled plans for a major capacity expansion of its Millad NX 8000 clarifying agent for polypropylene at its Allen facility in Blacksburg, S.C. The company will double its production and add an additional redundant manufacturing line. The expansion is scheduled for completion in 2015.

**Shin-Etsu to construct rare-earth magnet plant in Vietnam**

April 22, 2014 — Shin-Etsu Chemical Co. (Tokyo, Japan; www.shinetsu.co.jp) will establish a new rare-earth magnet manufacturing plant in Vietnam. The manufacturing capacity will be 2,000 m.t./yr., with an investment of about $117 million. The new plant will be built in two phases in Hai Phong Province, with construction work commencing in October 2014. The first-phase portion of the plant construction work is scheduled to be completed in September 2015 and have a production capacity of 1,000 m.t./yr. The second phase is to be completed in September 2016 with another 1,000 m.t./yr of capacity.

## MERGERS AND ACQUISITIONS

**Eni sells refining and marketing assets in Romania, Czech Republic and Slovakia**

May 7, 2014 — Eni S.p.A. (Rome; www.eni.it) has signed an agreement with MOL Group (Budapest, Hungary; www.mol.hu/en) for the sale of its 32.445% stake in Ceská Rafinérská a.s., a petroleum refining company in the Czech Republic. Eni also signed further agreements for the sale of its subsidiaries Eni Ceská Republika, Eni Slovensko and Eni Romania to MOL Group. These refining and marketing subsidiaries operate in Czech Republic, Slovakia and Romania respectively.

**Songwon acquires SeQuent's specialty chemicals business**

April 24, 2014 — Songwon Industrial Co. (Ulsan, Korea; www.songwon.com) and SeQuent Scientific Ltd. (Bangalore, India; www.sequent.in) have signed an agreement whereby Songwon will acquire SeQuent's specialty chemicals business. The acquisition includes SeQuent's entire polymer-stabilizer business and one production site in Panoli, Gujarat, India, along with the local research and development team. The transaction is expected to close in the third quarter of 2014.

**Chemtura to sell agrochemicals business for $1 billion**

April 17, 2014 — Chemtura Corp. (Philadelphia, Pa.; www.chemtura.com) has entered into a definitive agreement to sell its agrochemicals business, Chemtura AgroSolutions, to Platform Specialty Products Corp., for approximately $1 billion. The acquisition will be funded with $950 million in cash and two million shares of Platform's common stock.

**Sumitomo acquires stake in CEPSA subsidiary in China**

April 14, 2014 — Sumitomo Corp. (Tokyo, Japan; www.sumitomocorp.co.jp) and CEPSA (Madrid, Spain; www.cepsa.com) have entered into an agreement for Sumitomo to acquire 25% of CEPSA's subsidiary CEPSA Química China, resulting in a JV that is owned 75% by CEPSA and 25% by Sumitomo. CEPSA Química China is currently undergoing the final phases of construction for a production plant in Shanghai. Once complete, the facility will have the capacity to produce 250,000 m.t./yr of phenol, 150,000 m.t./yr of acetone and 360,000 m.t./yr of cumene. ■

*Mary Page Bailey*

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

**June 2014; VOL. 121; NO. 6**

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2012 — 2013 — 2014

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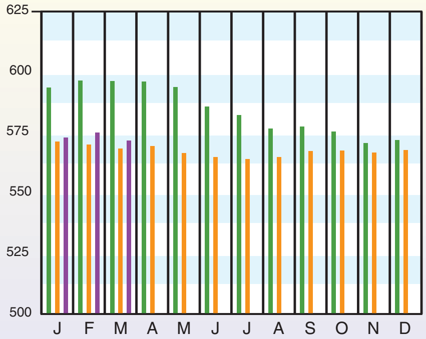
## CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

| (1957–59 = 100) | Mar. '14 Prelim. | Feb. '14 Final | Mar. '13 Final |
|---|---|---|---|
| ***CE* Index** | 571.6 | 574.9 | 568.3 |
| Equipment | 692.9 | 697.6 | 688.2 |
| Heat exchangers & tanks | 627.9 | 637.2 | 624.2 |
| Process machinery | 662.9 | 663.9 | 651.1 |
| Pipes, valves & fittings | 876.1 | 881.9 | 879.8 |
| Process instruments | 410.6 | 412.9 | 414.3 |
| Pumps & compressors | 932.9 | 931.7 | 920.4 |
| Electrical equipment | 514.6 | 515.5 | 514.4 |
| Structural supports & misc | 760.5 | 759.6 | 741.1 |
| Construction labor | 319.7 | 321.5 | 319.2 |
| Buildings | 542.4 | 541.4 | 534.4 |
| Engineering & supervision | 323.0 | 322.8 | 326.4 |

**Annual Index:**

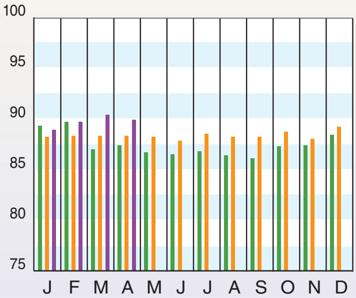
2006 = 499.6
2007 = 525.4
2008 = 575.4
2009 = 521.9
2010 = 550.8
2011 = 585.7
2012 = 584.6
2013 = 567.3



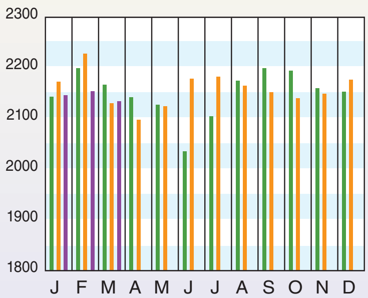

## CURRENT BUSINESS INDICATORS*

| | LATEST | | | PREVIOUS | | | | | | YEAR AGO | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| CPI output index (2007 = 100) | Apr. '14 | = | 89.9 | Mar. '14 | = | 90.4 | Feb. '14 | = | 89.7 | Apr. '13 | = | 88.3 |
| CPI value of output, $ billions | Mar. '14 | = | 2,134.0 | Feb. '14 | = | 2,154.1 | Jan. '14 | = | 2,146.8 | Mar. '13 | = | 2,131.0 |
| CPI operating rate, % | Apr. '14 | = | 75.8 | Mar. '14 | = | 76.3 | Feb. '14 | = | 75.7 | Apr. '13 | = | 75.3 |
| Producer prices, industrial chemicals (1982 = 100) | Apr. '14 | = | 295.7 | Mar. '14 | = | 293.7 | Feb. '14 | = | 299.6 | Apr. '13 | = | 303.4 |
| Industrial Production in Manufacturing (2007 = 100) | Apr. '14 | = | 98.6 | Mar. '14 | = | 99.0 | Feb. '14 | = | 98.3 | Apr. '13 | = | 95.8 |
| Hourly earnings index, chemical & allied products (1992 = 100) | Apr. '14 | = | 157.0 | Mar. '14 | = | 156.6 | Feb. '14 | = | 157.1 | Apr. '13 | = | 155.3 |
| Productivity index, chemicals & allied products (1992 = 100) | Apr. '14 | = | 108.3 | Mar. '14 | = | 107.3 | Feb. '14 | = | 108.0 | Apr. '13 | = | 104.8 |

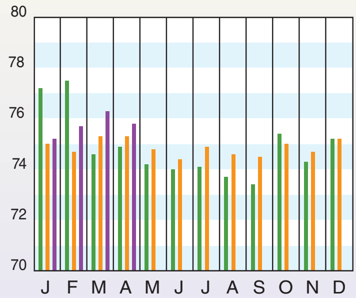
### CPI OUTPUT INDEX (2007 = 100)




### CPI OUTPUT VALUE ($ BILLIONS)




### CPI OPERATING RATE (%)




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

## HIGHLIGHTS FROM RECENT ACC ECONOMIC DATA

The American Chemistry Council's (ACC; Washington, D.C.; www.americanchemistry.com) monthly Chemical Activity Barometer (CAB; a leading economic indicator) for April reached its highest level since March 2008. Even accounting for seasonal adjustments, the unusually harsh winter skewed data from earlier in the year, and the CAB released at the end of April reflects upward revisions for January, February, and March of 0.1, 0.2 and 0.1 points, respectively. On a three-month moving average (3MMA) basis, the April CAB reading showed a healthy 0.5% gain over March. Together with the upward revisions, this suggests further growth momentum in the broader economy in the months ahead.

Meanwhile, in its Weekly Chemistry and Economic reports, the ACC discussed other economic data, including the the JPMorgan Global Manufacturing PMI (purchasing manager's index), which slipped 0.5 points to 51.9, in late March. The decrease signals that the upturn in the global manufacturing sector continued to ease at the start of the second quarter, the ACC said. A reading above 50 for the index indicates expanding business activity, while readings below 50 signal contraction.

"The growth rate of the global manufacturing sector eased to a six-month low at the start of the second quarter," the ACC report said, adding, "The slower rate of improvement reflected weaker gains in production and new orders." Production declines were observed in Japan, China, South Korea, India, Taiwan, Brazil and Russia, the report said, while the U.K. and the U.S. saw higher output and new orders accelerating."

Another ACC report discussed import/export information from the U.S. Census Bureau. "The [U.S.] chemical industry posted a surplus in all major segments except pharmaceuticals and agricultural chemicals in the first quarter of 2014," the ACC noted. ❑

## CURRENT TRENDS

The preliminary value for the March *CE* Plant Cost Index (CEPCI; top) fell 0.6% from the final February value. The decline was driven by lower Equipment and Construction Labor subindices. The Engineering Supervision and Buildings subindices increased slightly in March, but not enough to offset the decreases in the other subsections. The overall PCI value for March 2014 stands at 0.6% higher than the value from March of last year. Meanwhile, updated values for the Current Business Indicators (CBI) from IHS Global Insight (middle) saw both the CPI output index and CPI value of output indicator for April fall from the previous month. Their current values are close to those from a year ago at this time. ❑