CHEMICAL ENGINEERING

April 2013

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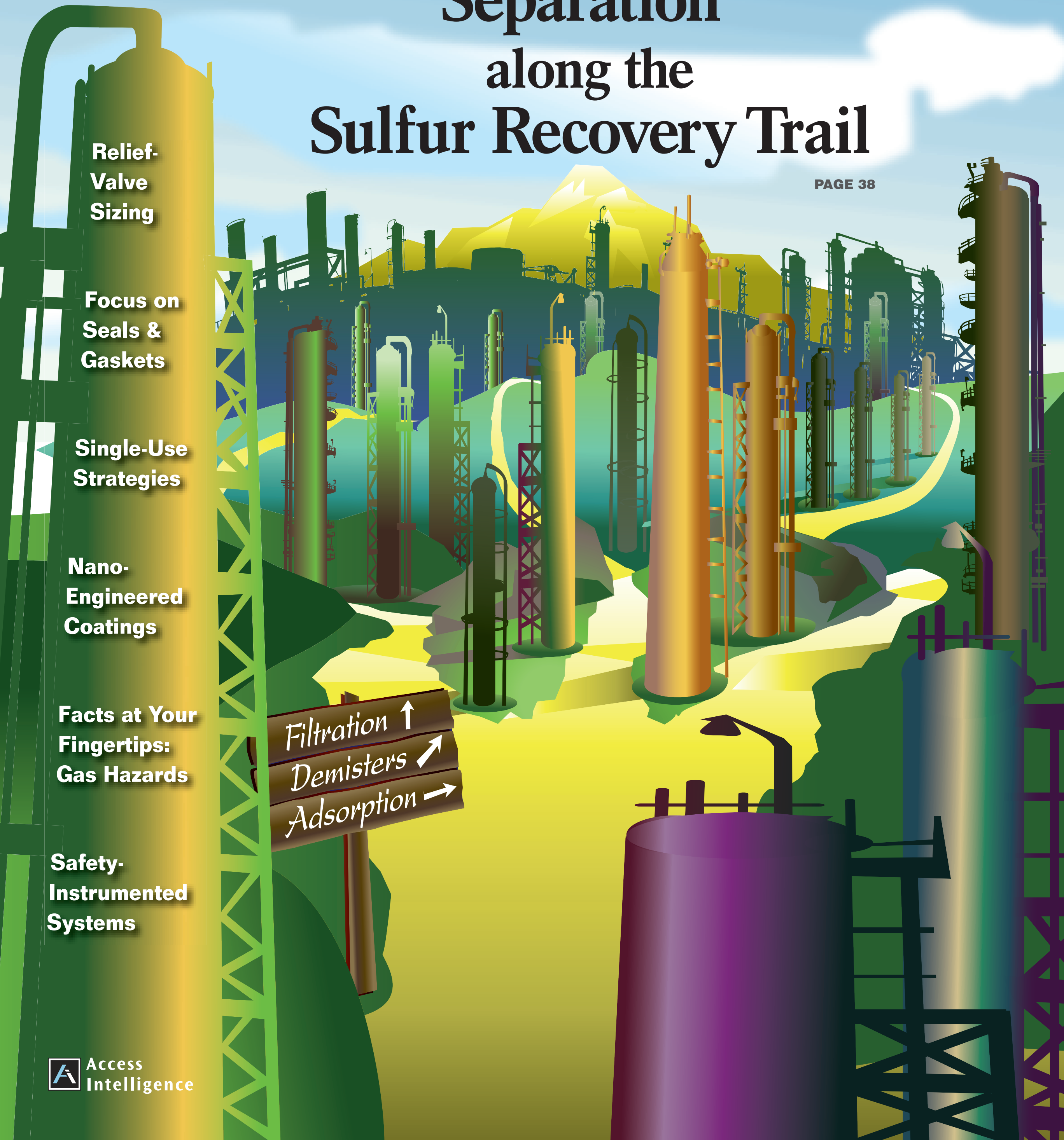

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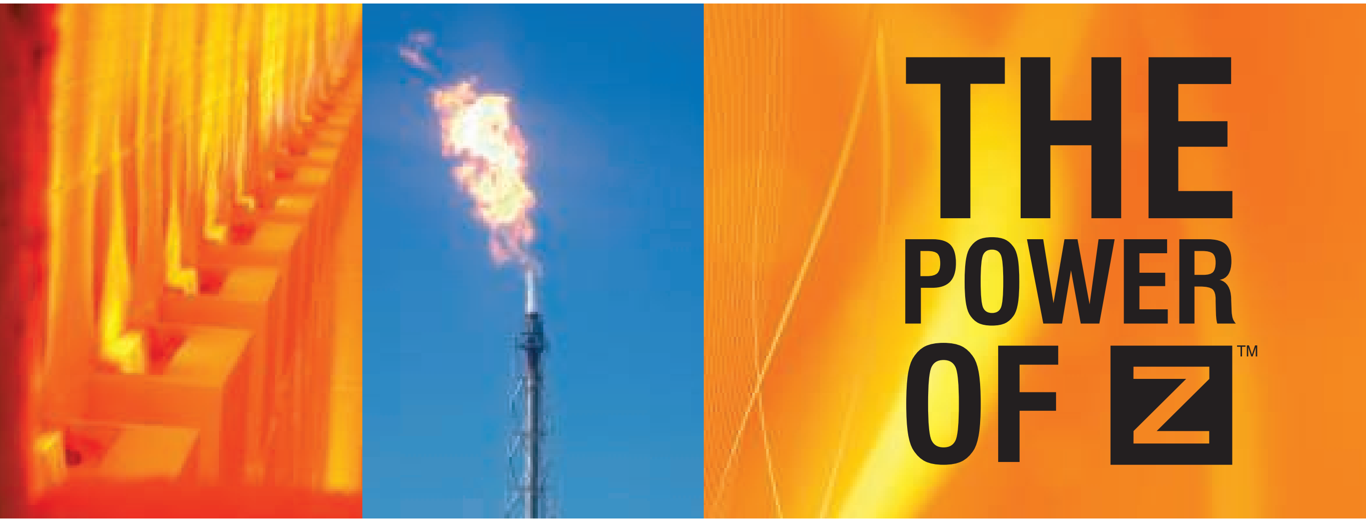

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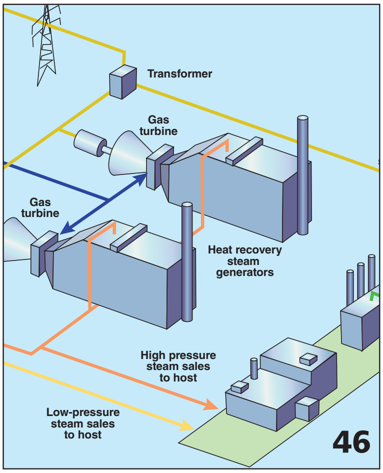

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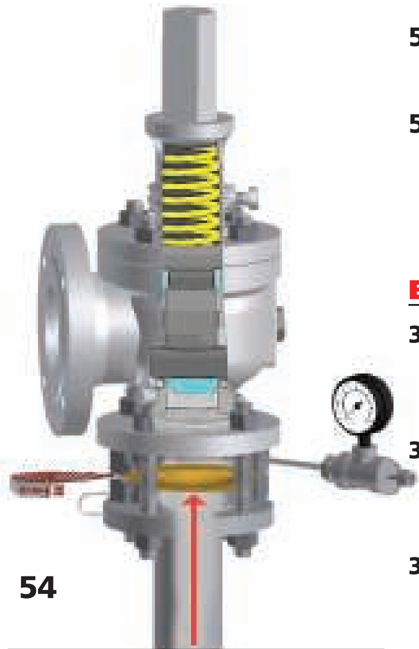

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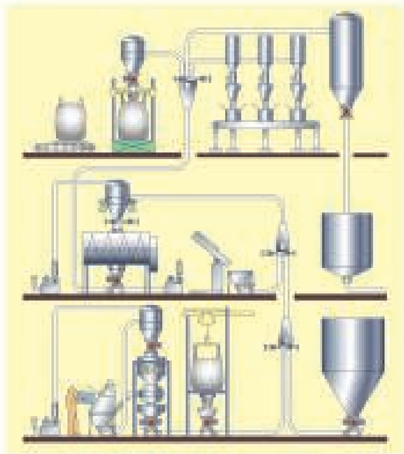

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Editor's Page

Bringing mobility to the plant

In his keynote presentation at this year's ARC Forum (February 11–14, Orlando, Fla.; www.arcweb.com), Andy Chatha, president and founder of ARC, stated that there were over ten billion WiFi-connected devices in 2012 and that over 50 billion are expected by 2020. He called this the "era of connected devices" and introduced the concept of information-driven manufacturing (IDM), where competitiveness is driven by the ability to effectively obtain and use information that is available from a wide variety of sources. Rapidly advancing technologies are driving IDM, and one of the enablers is the readily accessible, mobile device.

Mobile handheld devices, which were once forbidden on the plant floor, are moving into the realm of being sought-after solutions for manufacturing personnel. The high attendance at the ARC Forum track devoted to this topic is indicative of the growing interest in mobile devices for manufacturing.

There seems to be a convergence of several factors that are driving the movement toward mobility in the plant. First, the nature of many jobs is changing, and the need to do more with less opens opportunities where mobility of information can help. For example, many operators no longer stay in one control room all day overlooking one process unit. Instead, they need to cover several units in a plant, and so accessing information remotely is a plus. Second, the nature of the workforce itself is changing. As mobile devices have become so prevalent in our daily lives, people want to be able to get the same instant information in their workplaces that they can get outside of it. Young "millenials" entering the workforce expect mobility. Third, the availability of the technology for the mobile devices themselves, and supporting technologies like wireless instrumentation, and "smart" sensors and transmitters, is advancing rapidly.

A number of examples of mobile devices in operation in the chemical process industries (CPI) was given at the ARC Forum. Those examples included environments where an iPad could be used, as well as environments where intrinsically safe (IS) devices were required. Areas that could, or already are benefitting from handheld devices on the plant floor include: maintenance; inventory management; quality control; and data management, such as being able to fulfill regulatory "paperwork" requirements from out in the field rather than having to come back to an office setting to do so.

There are certainly issues that need to be addressed in this age of mobility — the most important being safety and data security. A robust approach to cybersecurity is certainly needed. This is an issue that is becoming increasingly important for manufacturing (see Chemical Plant Security: Gating More Than the Perimeter, *Chem. Eng.*, September 2012, pp. 18–22). I expect some earlier considerations about introducing handheld devices on the plant floor included concerns about lost productivity due to workers using these devices for non-work-related activities. Years ago, this was also a main concern in allowing internet accessibility in the workplace, but the resulting improvements in productivity outweighed those fears.

One suggestion that was heard during the ARC Forum was that companies need to put a set of policies in place before implementing the use of mobile devices in order to address some of the concerns, particularly about data security. With this sound advice, handheld devices are poised to help increase productivity as manufacturers embrace the new technologies available to them. ■

Dorothy Lozowski, Managing Editor

Letters

Respiratory Protection*

. . . OSHA requires employers to institute engineering and work practice controls as the primary means to reduce and maintain employee exposures to air contaminants to levels at or below the OSHA permissible exposure limits (PELs). Primary reliance on engineering controls and work practices is consistent with good industrial hygiene practice. OSHA also relies on traditional adherence to a hierarchy of controls that prefers engineering and work practice controls over dependence upon respirators. . . .

Comments

1. Page 28, middle column, 1st full paragraph — This paragraph should incorporate: "Where toxic substances are present in the workplace, OSHA requires employers to maintain exposure levels at or below the PEL by implementing engineering and work practice controls.". . .

Unfortunately, OSHA standard 29 CFR 1910.134(a)(1) fails to mention the ability of the employer to rely on work practice controls to reduce exposures. . . .

2. Page 28, right column, last paragraph — It is unclear what the author means when he states, "The aforementioned statistic reveals that too many workers are not employing appropriate protection from respiratory hazards on the job." What does "not employing appropriate protection," mean? . . .

Ira Wainless,
Senior Industrial Hygienist, OSHA (retired)

Author's response

. . . 1. In this article my intent was to give an overview of the standard and therefore I followed OSHA's wording as closely as possible. However, even if OSHA failed to mention the ability of the employer to rely on work practice controls to reduce exposure, the article did state "Where toxic substances are present in the workplace and engineering controls such as . . . are inadequate to reduce or eliminate them, it is time to turn to respirators.". . .

2. In 2011, "Respiratory Protection, General Industry (29 CFR 1910.134)" was OSHA's fourth most frequently cited standard violation. This means that too many workers are not employing appropriate protection from respiratory hazards. More specifically, workers are either not using the correct protection or are wearing it incorrectly. And by following the standards (i.e., ensuring that they are properly trained on usage, are fit tested, and are maintaining the respiratory properly) the number of violations should consequently decrease. . . .

Dennis Capizzi,
Product Line Manager Air Purifying Respirators
MSA Safety

**Editor's Note:* The above are excerpts from letters sent to the editors regarding the article, Clearing the Air About Respiratory Protection, *Chem. Eng.*, pp. 28–33, December 2012. The full letters can be found on our website (www.che.com).

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ACS Rubber Div. 183rd Technical Meeting 2013. American Chemical Society, Rubber Div. (Washington, D.C.). Phone: 330-972-7978; Web: rubber.org/183rd-technical-meeting
Akron, Ohio **April 22–24**

Advancing Green & Sustainable Chemistry. Active Communications International (Chicago, Ill.). Phone: 312-780-0700; Web: wplgroup.com
Portland, Ore. **April 24–26**

104th American Oil Chemists Soc. Annual Meeting & Expo. American Oil Chemists Soc. (Urbana, Ill.). Phone: 217-693-4821; Web: annualmeeting.aocs.org
Montreal, Que. **April 28–May 1**

AIChE 2013 Spring Meeting and 9th Global Congress on Process Safety. AIChE (New York, N.Y.). Phone: 203-702-7660; Web: aiche.org
San Antonio, Tex. **April 28–May 2**

7th International Symposium on Enabling Technologies. ETP Symposium Inc. (Sharon, Ont.). Phone: 905-589-0059; Web: etpsymposium.org
Toronto, Ont. **April 30–May 1**

Risk Management of Corrodible Systems. National Assn. of Corrosion Engineers International (Houston). Phone: 281-228-6200; Web: event.nace.org
Orlando, Fla. **June 18–20**

Valve World Americas Expo & Conference. Messe Düsseldorf North America (Chicago, Ill.). Phone: 312-781-5185; Web: mdna.com
Houston **June 25–26**

Air and Waste Management Assn. (AWMA) Annual Conference. AWMA (Pittsburgh, Pa.). Phone: 412-232-3450; Web: awma.org
Chicago, Ill. **June 25–28**

American Chemical Soc. 246th National Meeting and Exposition. American Chemical Soc. (Washington, D.C.). Phone: 202-872-4600; Web: acs.org
Indianapolis, Ind. **Sept. 8–12**

4th Annual ChemInnovations Conference & Expo. TradeFair Group, an Access Intelligence LLC Co. (Houston). Phone: 713-343-1891; Web: cpievent.com
Galveston, Tex. **Sept. 25–26**

EUROPE

In-Cosmetics. Reed Exhibitions (Richmond, U.K.). Phone: +44-207-240-2444; Web: in-cosmetics.com
Paris, France **April 16–18**

ImagineNano 2013. Phantoms Foundation (Madrid, Spain). Phone: +34-911-402145; Web: imaginenano.com
Bilboa, Spain **April 23–26**

5th International Symposium on Advances in Synthetic & Medicinal Chemistry. LD Organization (Louvain-La-Neuve, Belgium). Phone: +32-10-454777; Web: asmc2013.org
Moscow, Russia **May 5–8**

EU BC&E 2013 — 21st European Biomass Conference and Exhibition. ETA Florence Renewable Energies (Florence, Italy). Phone: +39-055-500-2174; Web: conference-biomass.com
Copenhagen, Denmark **June 3–7**

Introduction to Foundation Fieldbus. Industry Source (Mesa, Ariz.). Phone: 480-775-6020; Web: fieldbus.org
Frankfurt, Germany **June 18**

ASIA & ELSEWHERE

World Coal-to-Liquids (CTL) 2013. World CTL (Paris). Phone: +33-607-28-5247; Web: world-ctl.com
Shanghai, China **April 16–19**

Introduction and Advanced Principles of Foundation Fieldbus. Industry Source (Mesa, Ariz.). Phone: 480-775-6020; Web: fieldbus.org
Pune, India **April 23–26**

CPhi Japan 2013. UBMi BV (Amsterdam, The Netherlands). Phone: +31-204-0990544; Web: cphijapan.com
Tokyo, Japan **April 24–26**

BioTech 2013 Japan. Reed Exhibitions Japan Ltd. (Tokyo, Japan). Phone: +81-3-3349-8518; Web: bio-t.jp
Tokyo, Japan **May 8–10**

The 13th China International Environmental Protection Exhibition and Conference. Goodwill Exhibition and Promotion Ltd. (Hong Kong). Phone: +852-2893-4338; Web: goodwill-exh.com.hk/ciepec2013/
Beijing, China **May 13–16** ■

Suzanne Shelley

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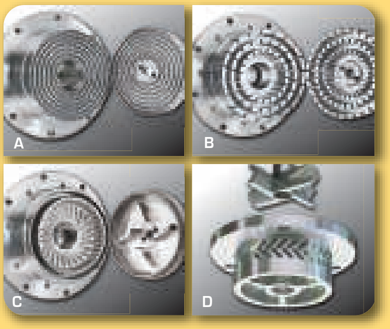

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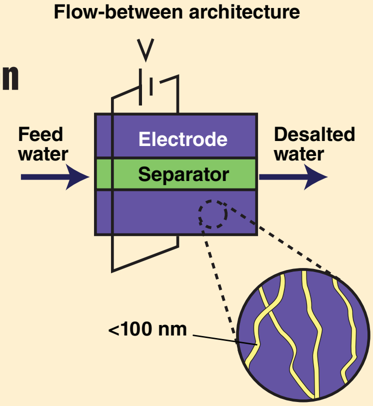
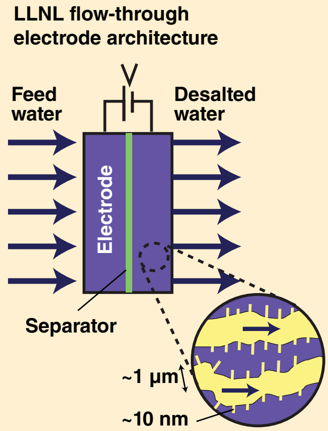
CHEMENTATOR

Edited by Gerald Ondrey April 2013

A new way to desalt water without using RO or distillation





Capacitive desalination, in which salt ions are electrically removed from saltwater, has been researched for decades as a potentially cheaper alternative to energy-intensive reverse osmosis (RO) and distillation, but so far its application has been limited to waters with very low salt concentrations. A new method, which may change that, is being developed by researchers at Lawrence Livermore National Laboratory (LLNL, Livermore, Calif.; www.llnl.gov).

In capacitive desalination, saltwater is typically pumped through a channel between two capacitor-electrodes (diagram). The electrodes remove the Na^+ and Cl^- ions from the saltwater by capturing them on the surface of the electrodes. LLNL has built a flow-through electrode (FTE) module that has no channel, but consists of two highly porous blocks of carbon aerogel (with negative and positive charges), divided by a thin polymer membrane. Saltwater is pumped through the entire capacitor and the ions, which can pass through the membrane, are accumulated by the appropriate electrode.

Earlier experiments (in the 1990s) used aerogels with small pores and had a channel between the electrodes. This limited ion-transport times from the channel into the electrodes, says Michael Stadermann, an LLNL staff scientist. The FTE uses a newly developed aerogel that combines large pores, of 1–3-µm dia. with small pores of 1–2-nm dia. The large pores allow easy flow at low pressure, while the small ones give the aerogel a vast surface area of up to 3,000 m^2/g for capturing ions. The structure permits elimination of the channel, thereby reducing the transport distance from millimeters to micrometers, with a corresponding decrease in desalination time, he says.

The aerogel is made by sol-gel polymerization of resorcinol with formaldehyde in water, using acetic acid as a catalyst. The water is removed by washing with acetone, which is then evaporated by heating. Finally, the material is pyrolized at 950°C and activated by a carbon dioxide etch. Pyrolysis stabilizes the pores and makes for a robust structure, says Stadermann. Laboratory tests with the FTE module achieved salt removal of 80 mmol/L in a single pass and indicate the process would require less than a hundredth of the pressure of conventional RO, he says, but the higher electricity use would even out the operating costs. However, he expects the capital cost would be lower and that the process would have an advantage over RO for processing brackish water.

Simplified CO_2-to-methanol process uses copper-oxide nanowires

A process developed by researchers at the University of Texas at Arlington (www.uta.edu) uses copper-based semiconductor nanoscale-rod arrays and solar radiation to carry out a photoelectroreduction of carbon dioxide to methanol. The process offers a pathway to utilize the greenhouse gas CO_2 as a feedstock to synthesize a widely manufactured chemical (CH_3OH) under much milder conditions that those needed for steam reforming of methanol from hydrocarbon-based feedstock. Also, the researchers note that no co-catalysts are needed for the selective conversion process. Since the method uses copper-based semiconductor materials, less abundant (Te and Ga) and more toxic (As and Cd) materials are not necessary. The two types of copper oxide were used because both are photo-active and the two have complementary solar-light absorption, the researchers say.

The semiconductor arrays are prepared using a two-step approach that consists of a thermal process of growing CuO nanorods, and a controlled electrodeposition of Cu_2O crystallites on the walls of the nanorods. The hybrid CuO-Cu_2O nanoarrays are constructed on a copper substrate. When the nanorod arrays were submerged in a water-based solution rich in CO_2 and exposed to simulated sunlight, methanol was produced without the need for excess energy input (overpotential).

Biosteel

The world's first artificial-silk fiber that is entirely made of recombinant spider-silk proteins has been produced by AMSilk GmbH (Planegg/Martinsreid, Germany; www.amsilk.com), a spin-off company of the Technical University of Munich. The fiber's tensile strength is comparable to that of natural spider silk, and has thus been tradenamed Biosteel. Fiber prototypes are smooth to the touch, pleasant to the skin and shine like silk, says the company. Potential applications include high-performance technical textiles, sporting goods, medical textiles and surgical products.

(*Continues on p. 12*)

Note: For more information, circle the 3-digit number on p. 68, or use the website designation.

First application of sulfuric acid heap leach to vanadium

Following a positive feasibility study, American Vanadium Corp. (Vancouver, B.C.; www.americanvanadium.com) will construct the world's first sulfuric acid heap-leach mining operation for vanadium at a specific geologic formation in central Nevada, known as Gibellini Hill. The project is currently in the permitting and mine-engineering stage, and once operational in 2015, will become North America's only primary producer of vanadium for making steel and titanium alloys, as well as for making vanadium electrolytes for grid-scale flow batteries for energy storage.

American Vanadium CEO Bill Radvak explains that the unique geology of the Gibellini area — heavily oxidized ore and soft rock (shale) — allows the company to produce high-purity vanadium cost effectively at high volumes.

Heap-leach mining was originally used for low-grade gold deposits. For vanadium, the ore is stacked on an impermeable liner, over which dilute sulfuric acid is sprayed. The acid leaches the vanadium out of the ore as it flows through the rock stack and into a pregnant-solution pond. The vanadium solution then moves to a solvent-extraction process and organic-stripping circuit to generate vanadium pentoxide product. American Vanadium has filed a patent on its purification process for producing high-purity vanadium onsite.

A method to produce an iron-titanium alloy for H_2-storage

A new and inexpensive way to produce TiFe — an alloy with potential as a material for reversibly storing hydrogen — has been developed by the research groups of professors Zenji Horita and Etsuo Akiba at Kyushu University (Fukuoka, Japan; www.kyushu-u.ac.jp). The TiFe is made by a technique known as high-pressure torsion (HPT), whereby a precursor is both pressed and twisted between two dies under high pressure. Treating a brittle TiFe with B2 structure as a precursor, the HPT (8 MPa pressure) yields a TiFe alloy exhibiting heterogeneous microstructures composed of nano-grains, coarse-grains, amorphous-like phases and disordered phases with a high, steady-state hardness of around 1,050 Hv. The TiFe made by this process adsorbs 1.7 wt.% H_2, which is near the theoretical maximum of 1.9 wt.%.

The TiFe alloy uses less expensive metals than other intermetallic alloys being considered for H_2 storage, and also requires no activation pretreatment at high temperatures and pressures. The group is investigating the mechanism for the formation of this TiFe alloy, enabling the adaption of a conventional rolling process for industrial-scale production.

A hybrid acid catalyst makes levulinate from cellulose

Ken-ichi Tominaga and colleagues at the National Institute of Advanced Industrial Science and Technology (AIST; Tsukuba, Japan; www.aist.go.jp) have developed a hybrid catalyst system that can be used to convert cellulose into levulinic acid. The catalyst consists of two types of acid: indium(III) trifluoromethanesulfonate ($In(OTf)_3$), a Lewis acid; and *p*-toluenesulfonic acid (PTSA), a Brønstead acid. In laboratory experiments, the combination converts cellulose in methanol (at 180°C and 0.05 MPa nitrogen pressure) directly to methyl levulinate. Yields of 60–75% were achieved after 5 h for cellulose, and 60% using powdered cedar wood as starting material. The catalysts can be recycled, and only a few mole percent of the catalyst is required — two orders of magnitude below the 600 mol% needed for the existing sulfuric acid process.

The researchers believe the new catalyst will open up new routes to making chemicals from cellulose. For example, levulinic acid can be used to synthesize 2-methyl-tetrahydroquinone, a precursor for a gasoline additive; α-methylene γ-valerolactone, an alternative monomer of methyl methacrylate (MMA); α-amino levulinic acid, a raw material for a biodegradable herbicide; as well as a promoter for photosynthesis.

The group is working to optimize the reaction using real biomass as a raw material, and looking to collaborate with a company that has biomass pretreatment technology.

(*Continued from p. 11*)

The Biosteel fibers are produced using a scalable spinning process, which is based on inventions of Thomas Scheibel, professor of Biomaterials at the University of Bayreuth, and technical developments of AMSilk engineers. The company is now optimizing the fiber and scaleup of the raw material production in its new pilot plant. The synthetic spider-silk proteins are made by a fermentation process (*Chem. Eng.,* May 2011, p. 10).

F-T catalyst

A new Fischer-Tropsch (F-T) catalyst has been developed by chemists at the Heterogeneous Catalyst and Sustainable group (Van't Hoff Institute for Molecular Sciences), University of Amsterdam (The Netherlands; www.uva.nl). The catalyst consists of spherical nanoparticles with iron-oxide cores (8-nm dia.) and cobalt-oxide shells (1-nm thickness). The core-shell catalyst is made by a process similar to that used for making audio cassette tape, known as surface nucleation. The catalyst is said to perform at least as well as pure cobalt F-T catalysts, and to produce "good diesel fractions." Because the bulk of the catalyst is inexpensive iron-oxide, the catalyst is expected to be significantly less expensive than conventional F-T catalysts, and the production process is cheap, reliable, efficient and scalable. Total S.A. (Paris, France; www.total.com) has patented the catalyst.

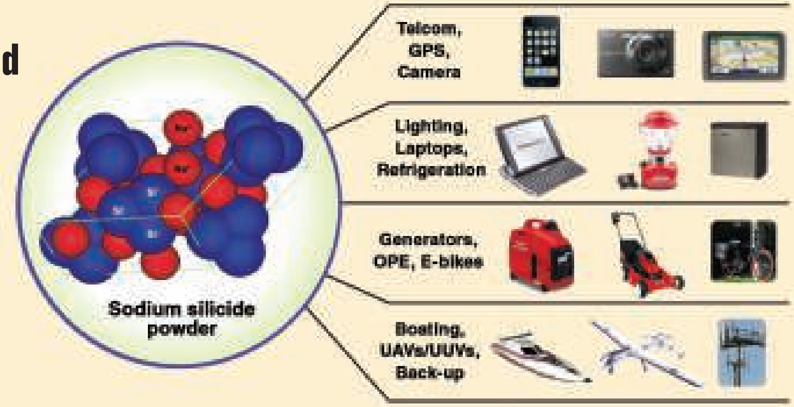
Lignin made in the U.S.

Domtar Corp. (Montreal, Canada: www.domtar.com) installed a commercial-scale lignin-separation plant at its Plymouth, N.C. mill — said to be the first U.S. facility of its type in 25 years. Production of the so-called BioChoice lignin began in February, with a targeted rate of 75 ton/d. The lignin can be used as fuels, resins and thermoplastics.

Turbine protection

Innovnano (Porto Salvo, Portugal; www.innovnano-materials.com) has com-

(*Continues on p. 14*)



Production facility for stabilized sodium slated for startup

A new plant for manufacturing sodium silicide (Na_4Si_4; diagram), a non-flammable, air-stable powder that reacts with water to generate pure hydrogen, is under development near Niagara Falls, N.Y. by SiGNa Chemistry Inc. (New York; signachem.com). Scheduled for startup in the third quarter of 2013, the facility will produce 1,000 ton/yr of sodium silicide using a proprietary thermal process that was developed by SiGNa, says CEO Michael Lefenfeld. The process combines sodium metal and silicon powder to create a stable, free-flowing powder that can be handled easily in dry air. The nonpyrophoric powder absorbs water slowly and reacts without ignition to yield H_2 gas and the environmentally benign sodium silicate.

SiGNa's manufacturing process for sodium silicide produces no waste and is exothermic, which translates into low energy inputs for manufacturing, explains Lefenfeld.

The stabilized sodium silicide powder is used in several energy-related applications, including tertiary flooding of oil-and-gas wells to enhance recovery. In this application, the powder reacts with water from the well, producing H_2 and heat to displace heavy crude oil deposits. SiGNa has shown that sodium silicide can more than triple the amount of heavy crude oil that can be recovered, compared to current tertiary oil recovery technologies.

The material is also used as a source of pure H_2 for portable fuel-cell applications. SiGNa currently has H_2 cartridges on the market for recharging consumer electronics. It is also developing H_2 cartridges for military power, back-up power for industrial and home uses, and portable power for disaster relief efforts. SiGNa is also exploring the use of sodium silicide cartridges to extend the range of electric and fuel cell vehicles.

The company utilizes similar thermal-process technologies to stabilize other reactive alkali metals for use in industrial synthesis of chemicals via Birch and Wurtz reductions, and in other applications.

New GC based on resonating nanosensor

A newly launched gas chromatography (GC) device from Analytical Pixels Technology (APIX; Grenoble, France; www.apixtechnology.com), called GCAP, uses a nanoscale silicon-beam resonator to detect mass. A piezoelectric cross-beam initiates the vibration of the nanoscale silicon resonator, and the resonance frequency varies with the mass in contact with the silicon resonator.

Because GCAP's architecture is highly flexible, it can be used in a wide array of applications. Among them are the detection and precise quantification of volatile organic compounds (VOCs), including BTEX (benzene, toluene, ethylbenzene and xylenes), as well as natural gas and "permanent" gases, such as N_2, O_2, CO_2 and H_2, at concentrations from sub-ppm levels to 100%.

The instrument achieves sensitivities not possible with conventional GC devices, and is compact and portable enough for use in in-line process testing in an industrial or research setting. Further, the nanoscale measurement mechanism can generate near-realtime gas-analysis results. GCAP can handle a variety of carrier gases, including He, H_2, Ar, N_2 and scrubbed air.

Because the system is miniaturized compared to alternative GC technologies, the instrument requires a significantly lower power and sample volume and can be deployed in a variety of settings, says Jean-Pierre Braun, CEO of APIX. The low-maintenance product is a large step toward making GC technology ubiquitous, says Braun.

The product arose out of a joint development agreement with France's Atomic Energy Commission (CEA) and the California Institute of Technology (CalTech; Pasadena, Calif.; www.caltech.edu). GCAP is currently undergoing beta testing at customer sites, and was recently demonstrated at PittConn 2013 (Philadelphia, Pa.; March 17–21).

(*Continued from p. 12*)

mercialized a highly pure, 4 mol% yttria-stabilized zirconia (4YSZ), a thermal barrier coating material that has "exceptional"properties for withstanding extreme temperature variations. Due to its low thermal conductivity, only a very thin coating is necessary, thus the coating is suitable for low-weight applications such as turbines.

The 4YSZ has a melting point of around 2,700°C and a hardness of 14 GPa. It has been shown by CPT, an independent center for thermal spray technology, to demonstrate the same thermal diffusivity and corrosion resistance as conventional coatings, but at much lower thickness and weight, says the company. The powders are made using the company's Emulsion Detonation Synthesis (EDS) process.

Customized, sub-micron powders are available in stable aqueous or ethanol-based suspensions for suspension plasma-spraying applications, as well as spray-dried granules.

A new composite

SGL Group (Wiesbaden, Germany; www.sglgroup.com) has developed a new type of high-strength composite material for industrial heat-treatment applications. Dubbed SigraBond, the carbon-fiber-reinforced graphite material is said to have advantages over alternative carbon-fiber-reinforced carbon materials. For

(*Continues on p. 16*)

Quantum clusters serve as ultra-sensitive detectors

Detection of extremely small quantities of dangerous or hazardous substances is important in many areas, including national security, safety of public utilities and radiation prevention. Several analytical methods have been developed using structural, functional and electronic properties of nanomaterials. In particular, noble-metal quantum clusters (QCs), a family of nanomolecules exhibiting intense luminescence, are highly sensitive and selective for specific analytes. Anchoring such QCs on small particles (100 nm to a few micrometers in diameter) leads to surface-enhancement of their luminescence, and may create a new platform for ultrasensitive detection — especially when combined with the use of optical microscopy.

Now researchers from the Dept. of Chemistry, Indian Institute of Technology Madras (www.iitm.ac.in), led by professor Thalappil Pradeep, have developed a simple and reliable strategy for the selective detection of 2,4,6-trinitrotoluene (TNT) and Hg^{2+} ions at the sub-zeptomole level (zmol; less than 10^{-21} moles) in solution.

Their method involves anchoring silver clusters, which are comprised of 15 atoms and embedded in bovine serum albumin (BSA), onto silica-coated gold "mesoflowers" (MFs), denoted by $Au@SiO_2@Ag_{15}$ MFs, and using this setup for analyte detection. Gold mesoflowers are anisotropic materials with five-fold symmetric stems containing surface-enhancing nanoscale features. An MF — which is only a few micrometers in size — has a distinct shape, allowing for clear identification by optical microscopy. Thus, changes in the properties of an MF can be used for the immediate and efficient detection of analytes.

The $Au@SiO_2@Ag_{15}$ MFs have a length of about 4 µm. The BSA-protected silver cluster is a red luminescent, water-soluble quantum cluster. It exhibits a high quantum yield (10.7%) in water, and is stable over a wide pH range.

The researchers exposed varying concentrations of TNT to $Au@SiO_2@Ag_{15}$ MFs and found that even a concentration of less than 1 zmol of TNT per mesoflower quenches the luminescence of the composite mesoflowers within 1 min. The simultaneous disappearance of the luminescencne of Ag_{15} on the MF and the appearance of the luminescence of another embedded fluorescent compound allows for easy identification of the analyte.

The researchers say the same method could be extended to other QCs with brighter luminescence, opening up numerous applications in catalysis, bioimaging and other areas where novel devices can be visualized, merged and tailored. This approach can be considered a single-particle, single-molecule detection technique, which is, probably, the ultimate in ultra-trace sensitivity, they say.

MOFs that reversibly capture CO_2

An Australian team has developed a structure made of a metal organic framework (MOF) that captures and then releases CO_2 using natural sunlight. The team includes researchers from: CSIRO Division of Materials Science and Engineering, (Clayton South, Victoria; www.csiro.au) the Dept. of Chemical Engineering, Monash University (www.monash.edu.au) and the School of Chemistry, University of Sydney (www.sydney.edu.au).

"The capture and release process can be compared with soaking up water with a sponge and then wringing it out," explains Matthew Hill, who led the CSIRO group. "When ultraviolet (UV) light hits the material, its structure bends and twists and stored gas is released."

MOFs are among the most attractive potential adsorbents to replace more energy-demanding commercial adsorbents, because of MOFs' high adsorption capacity, and the possibility of incorporating light-responsive organic groups within the MOFs' pore structure. The large internal surface areas of the newly developed MOFs can adsorb large quantities of gases, including H_2, CH_4 and CO_2.

Azobenzene and its derivatives, in particular, can undergo clean and efficient reversible photoisomerization about the azo bond to *cis* and *trans* states upon irradiation (coordinated *trans*: $\lambda_{max} \approx 370$ nm, *cis*: $\lambda_{max} \approx 460$ nm). Conversion of azobenzene to the 4,4'-dicarboxylate (AzDC) delivers a ligand that can be incorporated into MOF architectures. Besides, the pillar ligand trans-1,2-bis(4-pyridyl)ethylene (4,4'-BPE) also exhibits *cis-trans* photoisomerizability when coordinated to a metal complex.

Combination of these two ligands within zinc-based MOFs generates the triply interpenetrated framework Zn(AzDC)$(4,4'\text{-BPE})_{0.5}$,which exhibits a selective and high capacity adsorption of H_2 and CO_2.

Exposure to UV light results in an instantaneous release of up to 64% of the absorbed CO_2 using broadband radiation, similar to concentrated solar sources. Furthermore, the response was found to be fully reversible. ■

(*Continued from p. 14*)

example, it is 20% stiffer and can be processed to create intricate structures. Applications for the new composite include the heat-treatment of steel components for the automotive and aerospace industries, and in the coating of silicon wafers in the photovoltaic industry, says the company.

Largest CSP launched

Last month, Masdar (Abu Dhabi, UAE; www.masdar.ae) and partners inagurated Shams 1, which is said to be the largest concentrated solar power (CSP) plant operating in the world. The 100-MW CSP incorporates 258,000 mirrors mounted on 768 tracking parabolic-trough collectors, which concentrate heat from sunlight onto oil-filled tubes. The heat is used to make steam to drive a turbine for generating electricity. The $600-million facility was designed and developed by Shams Power Co., a joint venture of Masdar, Total and Abengoa Solar. ❑

P2i

NANO-ENGINEERING INFUSES GROWING COATINGS MARKET

Controlling the structure of coatings at the nanoscale level is increasingly common in a coatings market projected for growth

The continuous quest to maximize process efficiency and boost product performance, in which all sectors of the chemical process industries (CPI) are engaged, is driving demand for technologically advanced coatings. And engineering at the nanoscale level is an increasingly common avenue to reach these goals in commercial coatings.

Progress in nano-steps

As the traditional coatings market continues to grow, engineering on the nanoscale will play an increasingly prominent role in infusing the newest coatings with specialized properties and enhanced performance (Figure 1). Nanostructured materials are being applied in all industry sectors to improve properties such as corrosion resistance, specific chemical and mechanical characteristics, flame retardance, ultraviolet radiation stability, gloss retention and others. According to a recent market research report from Future Markets Research Inc. (Rockville, Md.; www.marketresearch.com), nanocoatings are likely to replace traditional coatings "in the medium to long-term in end-use segments such as packaging, anti-microbial coatings, architectural coatings, industrial manufacturing, marine, wood, auto refinish, transportation and protective coatings" (see sidebar, p. 22).

"There's definitely a drive to impart more and more functionality to existing products, especially onto powders," explains Tony Taylor, CEO of AeonClad Coatings (Austin, Tex.; www.aeonclad.com), "and nano-engineered coatings are a good way to do that."

Currently, though, Charles Bangert, partner at the management consulting company Orr & Boss (Knoxville, Tenn; www.orrandboss.com), says that "Nanotechnology-based coatings have not yet changed the overall coatings market on a macro level, although they are being used for many niche applications." Along with Orr & Boss colleague Scott Detiveaux, Bangert recently authored a report on the global coatings market (see sidebar, p. 18).

Though the presence of nanotechnology in the coatings sector is significant and growing, the pace of advance is likely to be halting and uneven, rather than sweeping and revolutionary. Progress in nanocoatings will likely continue to be an evolutionary process where incremental advances will be the general rule, say Victor Castaño and Amlan Gupta, senior scientific consultant and president, respectively, of CG^2 NanoCoatings Inc. (Ottawa, Ont.; www.cg2nanocoatings.com). "Nano-engineered coatings have huge potential, but the science and engineering problems to which they are being applied are very difficult," says Castaño, adding, "nanotechnology is not magic — you've still got to work hard on the science and technology development."

FIGURE 1. **Coatings with specially designed structure at the nanoscale level, such as this one from P2i, generate properties like liquid repellancy**

"The industry has generally struggled in moving nanotechnology from the laboratory to the production floor," says Jack Angel, CEO of DuraSeal Coatings Co. (Kansas City, Mo.; durasealcoatings.com).

Nanohybrids for automotive

One way nanotechnology has entered the market has been through the creation of "hybrid" coatings that incorporate nanotechnology features into a more conventional coating. One example of this approach has been developed by BASF SE (Ludwigshafen, Germany; www.basf.com) for the automotive market, which is a large end-use segment of coatings.

BASF has created a topcoat for automobile paint that combines two types of materials in a nanostructured hybrid. Between 90 and 95% of the coating is organic material, making the finish flexible, elastic and weather-resistant, the company says, while the remaining 5 to 10% consists of nanoscale clusters of inorganic silicate that are particularly hard and scratch-resistant. The organic and inorganic components are covalently bound.

"The special nanostructure of the coating does not form until the paint, with the addition of a hardener together with a catalyst, is baked on the surface of the car," says BASF.

The coating performs better than conventional topcoats in springing back after contact by a potential source of scratching, such as brush bristles. While existing topcoats have an elastic recovery of around 70%, the BASF hybrid nanocoating achieves 90%, the company says, adding that the coating can be applied using existing paint application apparatus, and has been used by carmaker Daimler for several automobile models at its Bremen plant.

Nano agglomerations

While chemical makers like BASF are integrating nanotechnology into their coatings businesses, small solutions-based companies are applying their expertise in nano-engineered coatings to address complex customer problems. CG^2 NanoCoatings Inc. is an example. As a way of imparting new functionality onto materials, CG^2's proprietary coating platform is based on functionalizing nanoparticles with specific chemical groups for use in base coatings.

The company has made an important advance with its method to prevent nanoscale particles from agglomerating, a pervasive challenge that creates non-homogeneous environments at the nanoscale and negates the potentially useful behaviors of the nanoparticles. "We make a trade with nature," says CG^2's Castaño, "by minimizing the energy of the nanoparticles with specific chemical modifications, we offset the thermodynamic instability of the tiny particles, which would ordinarily cause them to agglomerate." In some cases, researchers at CG^2 chemically modify commercially available nanoparticles for a specific application. Rather than selling a specific products, the company applies its expertise and methodology to various industry challenges.

Corrosion and abrasion

Two cross-industry issues where nanocoatings are poised to have an impact are resistance to corrosion and abrasion. DuraSeal is one company that is marketing a nanocoating in this area. The company has developed a corrosion-resistant nanoscale coating that can be applied to metal surfaces, such as pipes, pump assemblies, well casing, valves, fittings and others as an alternative to fusion-bonded epoxy coatings. The coating is designed for use in hostile environments, including H_2S, HCl, H_2SO_4, brines, CO_2 and bicarbonates across a wide temperature range. To date, DuraSeal has coated more than five million feet of pipe, primarily tubulars and rods, and has seen performance levels improve by 200–500% or more, the company says.

DuraSeal's highly hydrophobic coating depends on reactive nanoscale silicon particles forming a cross-linked barrier at the surface. The coating forms covalent bonds with the metal substrate and forms a glass-like surface that withstands corrosive substances and has abrasion-resistant hardness. Further, the surface's relative smoothness can reduce frictional losses, the company says.

"Once you get down to the nanoscale level, conventional coatings are not able to completely fill in all the crevices of an irregular metal surface," explains DuraSeal's Jack Angel. "Nanoscale particles in these coatings can fit into all those spaces on the surface, forming a hydrophobic, cross-linked barrier film."

The method of application is just as important as the engineering of the coating itself, Angel notes. "We have seen more uptake of our technology since we have focused the same attention and effort on improving application techniques and production processes — including pre-cleaning of metal surfaces to minimize dust particles, and adjusting coating thickness and rotational speed of the applicator — as we do on the coating itself." Dust particles, can weaken the bond between coating and substrate.

In DuraSeal coating, a thermal process initiates the interaction between the coating and the free ions of iron within the surface metal. Combined with the penetration of the nanoparticles into the surface irregularities of the metal, the resulting bond of coating to substrate is strong.

Plasma for nanocoatings

Several companies are looking at coating-application methods as a way to help engineer surface properties at the nanoscale. One significant area of development is in modifying plasma-enhanced chemical vapor deposition (PECVD) processes to allow improved control over nanostructure. PECVD deposits thin films onto solid substrates from molecules in the gas phase by generating plasmas with electrical discharges. Among the challenges of traditional PECVD is that the continuous plasma-generating discharge can erode newly deposited coating material, as well as underlying substrate.

A modified version of PECVD developed by Aeonclad Coatings (Austin, Tex.; www.aeonclad.com) is aimed at creating a gentler environment for building a nanocoating. The Aeonclad method avoids surface ablation by using pulsed discharges, rather than continuous. This approach allows fine control over the surface composition and thickness (Figure 2).

"By carefully controlling the pulse length, the pulse power, the pressure of the vacuum chamber and the type of vapor, we can put down a surface gently, without ablating the surface," says Tony Taylor, president of Aeonclad.

The pulsed radio-frequency plasma technique also allows the company to engineer surfaces by incorporating different chemical functional groups

GLOBAL COATINGS REPORT

The Global Market Analysis Report for the Paint and Coatings Industry (3rd ed.) was sponsored by the International Paint and Printing Ink Council (IPPIC) and was assembled by analysts at Orr & Boss. The following represent a selection of the points made in the report.

- The global paint and coatings industry took in $106.1 billion in revenue in 2011 on a volume of 33.1 billion liters of product, the report estimates.
- The decorative-coatings market segment dominates, with 56% of the volume sold and 44% of the value. Other coatings market segments include general industrial (10% by value), auto OEM coatings (8% of value); powder coatings 8%; wood coatings (6%) and others.
- The report forecasts growth for the global coatings market to $144 billion in projected revenue in 2016.
- The global coatings industry has become increasingly consolidated over the past two decades, and the trend toward consolidation will continue, the report says
- Coatings demand tends to follow overall economic activity — a strong correlation exists between gross domestic product (GDP) per capita and coatings demand.
- Migration away from solvent-based coatings to water-based coatings has largely happened already for the decorative paint segment, but the transition is still occurring in the industrial coatings market segment. Often, the move there is toward high-solids, solvent-based technologies and powder coatings.
- End-use segments for coatings are diverse and fragmented compared with other industries. ❑

at varying densities to tune surface properties and control behavior at interfaces. For example, "the pulsed technique can selectively break the most labile chemical bond, while leaving others in tact," explains Taylor, "so we can access many different surface chemistries in a flexible way." The pulsed-plasma technique is compatible with a wide range of chemical groups and substrates — "We've coated discrete objects, as well as powders," notes Taylor, and we've made surfaces with a host of properties, including chemical inertness, hydrophilic, lubricious and others."

Since May 2012, the company has operated a pilot plant in Austin, Tex. that has the capability to coat up to 2,000 lb of material at a time using the pulsed-PECVD approach.

Aeonclad

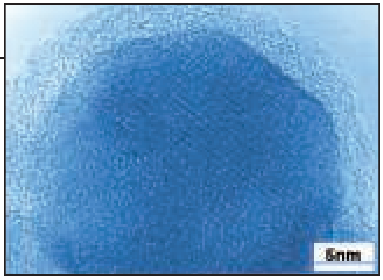


FIGURE 2. Pulsed-plasma techniques, like the one Aeonclad Coatings used to make this epoxy nanocomposite, allow better control of surface chemistry and avoid surface ablation

Liquid repellancy

Pulsed-plasma vapor deposition also figures heavily into the technology of P2i Ltd. (Abingdon, U.K.; www.p2i.com). The company has developed a process to apply an engineered nanoscale fluorocarbon-polymer coating in a way that minimizes surface energy of the coating and minimizes the interaction it has with liquids. The result is a surface that can repel both water-based liquids and oils, as well as alcohols and others.

At the molecular level, the coating application process induces a structure whereby fluorocarbon chains point upward, "like a forest of trees," with CF_3 groups at the top, says Delwyn Evans, P2i senior principal chemist, so the surface energy is significantly reduced (about 8 dyn/cm) compared to that of PTFE (polytetrafluoroethylene; about 18–21 dyn/cm), whose polymer chains have CF_2 groups lying flat.

The coating is applied to finished products inside a vacuum chamber using a two-stage plasma polymerization process. In the first stage, a room-temperature plasma is applied to a product under vacuum, a process that creates free-radical sites on the surface of the substrate material, while also freeing the surface of water and other materials that could disrupt bonding. In a second stage, a vapor of the fluorocarbon monomer is introduced and the polymer is grown on the surface. The reactive radical sites form covalent bonds with the polymer nanocoating, making the coating durable.

The pulsed plasma, with specific "dark" times, allows us to create the surface reactive sites and form the coating more gently than with a continuous discharge, explains Evans. Growing the polymer in a vacuum

allows a greater degree of vapor penetration, Evans adds, so that a coating only tens of nanometers thick has high performance and durability.

The key to P2i's commercial success, the company says, is the ability to provide a high-volume manufacturing solution. P2i's customers integrate its plasma-coating-application process into their own manufacturing lines. For small-volume manufacturing, the company also does toll processing. In addition to the electronics market (hearing aids and mobile phones), P2i's coating is also being applied to filter media, to prevent hydrocarbon liquids from blocking flow through the media.

Ultratech International Inc. (Jacksonville, Fla.; www.spillcontainment.com) is also among the host of companies working in the liquid repellant space. The company has developed a superhydrophobic and oleophobic nanocoating that repels liquids because of a molecular architecture that traps a layer of air at the surface, preventing liquids from contacting the surface.

Previous efforts in this area have given rise to coatings that generally lacked the durability needed for many applications, explains Ultratech CEO Mark Shaw. The innovation that the company has developed involves a two-layer coating that greatly increases the abrasion resistance of the coating. Both the top coat and bottom coat can be applied with air sprayers directly to a wide range of surfaces, including steel, aluminum, plastic, fabric, wood and others.

Aculon Inc. (San Diego, Calif.; www.aculon.com) is another company that markets liquid repellency products. The company's approach is to employ a layer of self-assembled monolayer of phosphonates (SAMPs) to impart surface properties. The phosphonate head groups form covalent bonds with the substrate, while the tails are constructed with a number of chemical groups that face outward and give the surface desired properties, Aculon director of business development Mario Gattuso says. The SAMP coating can be used to functionalize both surfaces and particles used in highly specialized applications, the company says. ■

Scott Jenkins

NANOCOATING MARKETS

Nanocoatings are opening new market opportunities in the global coatings arena. A wide range of possible applications for nano-engineered coatings are being explored, but it is sometimes difficult to predict what will be the most significant applications for nanocoatings even five years from now. Coatings that impart characteristics, such as easy cleaning, antifouling, dirt resistance, self-healing, electrical conductivity, abrasion resistance, antibacterial coatings and others, are likely to generate a great deal of interest in the near future. A Research and Markets (www.researchandmarkets.com) report from 2009, entitled "Nanostructured Coatings: Applications and Markets," projected that the global market for nanoscale coatings and thin films would grow from approximately \$2.4 billion in 2009 to over \$13 billion by 2016. The fastest growing markets to 2016 will be in interior and exterior household protection, textiles and medical markets, driven by the increased demands for protective and repellent coatings, the report said. ❏

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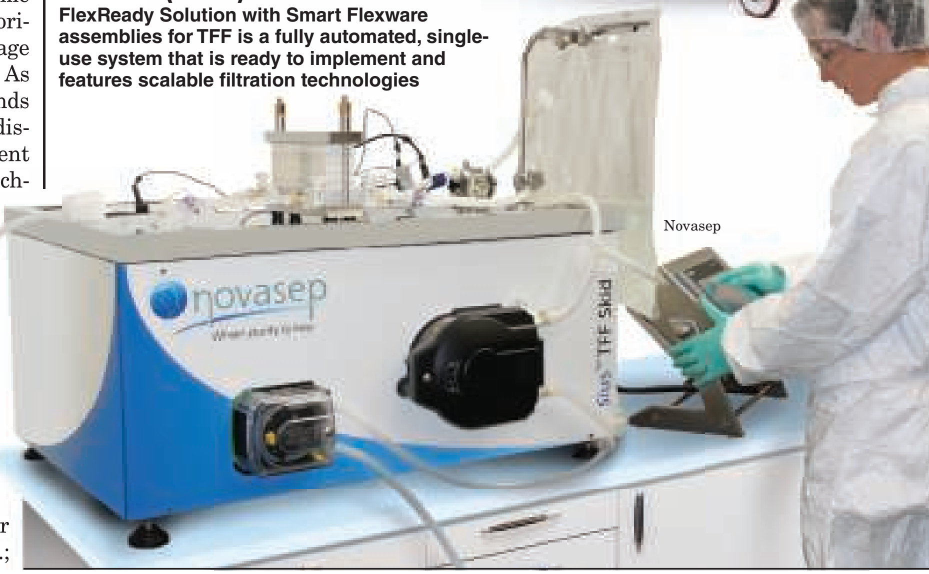
SINGLE-USE STRATEGIES

Disposable equipment systems satisfy strategic drivers for the biotech industry

EMD Millipore

FIGURE 1 (above). The Mobius FlexReady Solution with Smart Flexware assemblies for TFF is a fully automated, single-use system that is ready to implement and features scalable filtration technologies



Novasep

FIGURE 2. Sius is a single-use, skid-mounted system for TFF for pre-clinical to clinical cGMP biomanufacturing applications

Single-use equipment has come a long way from its modest origins as a convenient storage and transportation method. As a matter of fact, recent market trends and business drivers have made disposable equipment, with its inherent benefits, so attractive to the biotechnology and biopharmaceutical industries that they've begun requesting and implementing entire systems for use throughout the process line.

"The initial use of disposables for storage and transportation purposes eventually led to the use of bags in bioreactors and other in-process technologies, which has more recently led to advances in downstream use," says Michael LaBreck, global product manager with Novasep (Marcus Hook, Pa.; www.novasep.com). "As the confidence has begun to build, we see biotech companies doing actual processing in disposables and moving single-use equipment closer and closer to the purified product."

Single-use perks

One of the first benefits that attracted the biotech industry to single-use equipment was the reduction of cross-contamination risk in high-purity applications where even a few molecules remaining in the equipment after a process run could not be tolerated. In a traditional stainless-steel world, such a critical application would have required a strict cleaning regimen followed by validation. However, the introduction of disposables allowed operators to simply throw away the contaminated components and start over fresh. "The cost of doing this was much, much less than dealing with the possibility of cross contamination and having to throw away an entire day's work," says Wallace Wittkoff, director of global hygienic segment marketing with Pump Solutions Group (PSG; www.psgdover.com), a division of Dover Corp. (Oakbrook Terrace, Ill.).

While the reduced risk of cross contamination itself was enough of a benefit to attract attention, it was the ability to reduce the labor associated with cleaning and validation that made single-use technology especially attractive. LaBreck says one of Novasep's customers conducted a Six Sigma project that compared downstream processes using traditional clean-and-reuse methods to the same processes employing single-use technology. What they found, he says, was that the use of disposables eliminated non-value-added steps like flushing, cleaning and storage, which reduced labor and actual process time by 50%.

"Reduced labor time not only provides a cost savings, but it also increases productivity for the facility,"

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Sartorius Stedim Biotech

FIGURE 3. FlexAct is a new system that enables custom-configurable single-use solutions for entire biomanufacturing steps

LaBreck explains. "If a facility runs a process in the traditional way over eight hours, entire shifts are devoted to running and cleaning that process, but by employing single use to eliminate some of the non-value-added steps, the same process can run in just four hours. The facility can get more throughput because each unit operation will take less time, and getting more product through the facility in a shorter time cycle is a huge benefit to end users."

This speedier method also comes in handy during R&D. "The R&D process is long and expensive, and there's a lot of different runs and variables involved and batches to fine tune," says Wittkoff. "And if traditional methods are employed, the process must be cleaned after each run and then re-documented and revalidated, which is time consuming. By throwing the equipment away after each run, operators get rapid acceleration in the R&D process. This means they can get the product through R&D faster and recoup their investment sooner."

The same flexibility provided on the R&D side also applies, on a larger scale, to full-scale processes that require agility. One of the biggest trends in the biopharmaceutical industry right now is the trend toward personalized medicine, says John Stover, director of product management with New Age Industries (South Hampton, Pa.; www.newageindustries.com). "In order for these facilities to keep up, they need the ability to make smaller batches of multiple drugs with quick conversions," he says. "Disposables give them the ability to plug a single-use system into the production line for making 'Drug X' today and a week later plug in a fresh new system for making 'Drug Y.' It is so much faster and more flexible than trying to force different drugs through the same type of equipment while stopping to clean and validate in between."

This agility serves another purpose, as well. Many countries have mandated that pharmaceuticals consumed in a country must be manufactured in that country, says Paul Priebe, director of fluid management technologies with Sartorius Stedim Biotech

FIGURE 4. The incorporation of single-use pumps in biologic-handling applications is gaining wider acceptance among biotech and pharmaceutical manufacturers because they can help optimize end-product speed to market. Disposable pumps feature a replaceable wetted pump chamber. These single-use pumps reduce downtime between two batches, save time and money and are quick and easy to change. Quattroflow positive displacement pumps are suitable for single-use systems because they incorporate a four-piston-diaphragm technology with no mechanical seals that is driven by an eccentric shaft and motor

Pump Solutions Group

North America (Bohemia, N.Y.; www.sartorius.com). For a lot of western countries, this means transferring technology to sites around the world. “A flexible facility, or a plug-and-play, single-use facility that can be dropped anywhere in the world and still function as it was designed and operated by the ‘mothership’ really is an enabling technology that is being driven by our customers,” explains Priebe.

For example, in the vaccine world, western vaccine producers have very little footprint in the emerging markets around the world, but many want to participate in that space. “However, building up a stainless-steel facility for hundreds of millions of dollars in a five-year project is no way to try your luck in that market,” he says. “But, with a relatively small capital investment of tens of millions of dollars and a six-month project using platform-single processes that are ready to go anywhere they are dropped into place, it becomes a more feasible investment.”

Systems versus components

Faster implementation, more flexible facilities, avoidance of cross-contamination risk and elimination of costly and labor-intensive cleaning and validation steps are the main drivers behind the success of single-use equipment. And, because these are primarily business or strategic drivers, biotech companies started requesting more and more components and even complete systems from equipment providers.

“What we’re seeing is a strategic initiative among major biopharmaceutical manufacturers to establish plug-and-play platform processes using single-use technology,” says Priebe. “They want to be more flexible and more agile and able to expand into emerging markets, and we can help them do that. It’s not child’s play. It’s definitely on the cutting edge of where single-use technology is going, but customers are pulling and suppliers are responding and it’s a reality that’s not far away.”

Currently, single-use technology is available for all applications throughout the process, says Roman Rodriguez, global group product manager with EMD Millipore (www.emdmillipore.com), a division of Merck KgaA (Darmstadt, Germany). “But the real effort is to provide full coverage or interconnected materials so there are no gaps anymore, providing the ability to process from raw materials down to final product using single-use technology,” he says.

“There are a lot of different products available to cover all different applications. We have single-use for liquid handling, storage, transportation, taking samples, connectors, mixing systems, bioreactors and so on, but everyone is striving to create a flexible facility using all these components together. We are not there. There are still some technical gaps. In the meantime, we are providing single-use systems for downstream applications that offer flexibility to the end user."

For example, Millipore's Mobius FlexReady Solution with Smart Flexware assemblies (Figure 1) is a fully automated, single-use system for tangential-flow filtration (TFF). Modularity provides the ability to switch from TFF to chromatography with a minimum number of component changes, making it suitable for pilot plants and contract-manufacturing organizations (CMOs) that have rapidly changing demands. The system is suited for a range of concentration and diafiltration processes. In addition to the feed pump and retentate-valve-control loops, the system also includes an automated flow-control valve on the filtrate line to enable open ultrafiltration and microfiltration operations, increasing overall flexibility for clinical development and manufacturing facilities.

A variety of recycle vessel configurations are available to provide maximum process capability. In addition to different vessel volumes, users can select a plastic carrier or a double-jacketed stainless-steel carrier, depending on whether or not temperature control is required. All recycle vessels have been designed so that the containers can be easily and accurately installed by a single operator.

Similarly, Novasep has launched the Sius single-use TFF skid (Figure 2), which the company says is the first 100% single-use TFF solution for pre-clinical to clinical cGMP biomanufacturing. The skid is comprised of an automated bench-top unit with a fully disposable flowpath and is compact. It is capable of laboratory-to pilot-scale TFF operations. The skid's flowpath can be combined with the company's TangenX Sius TFF cassettes and filter-plate inserts with the option of a feed bag to provide a complete single-use TFF solution from a single supplier.

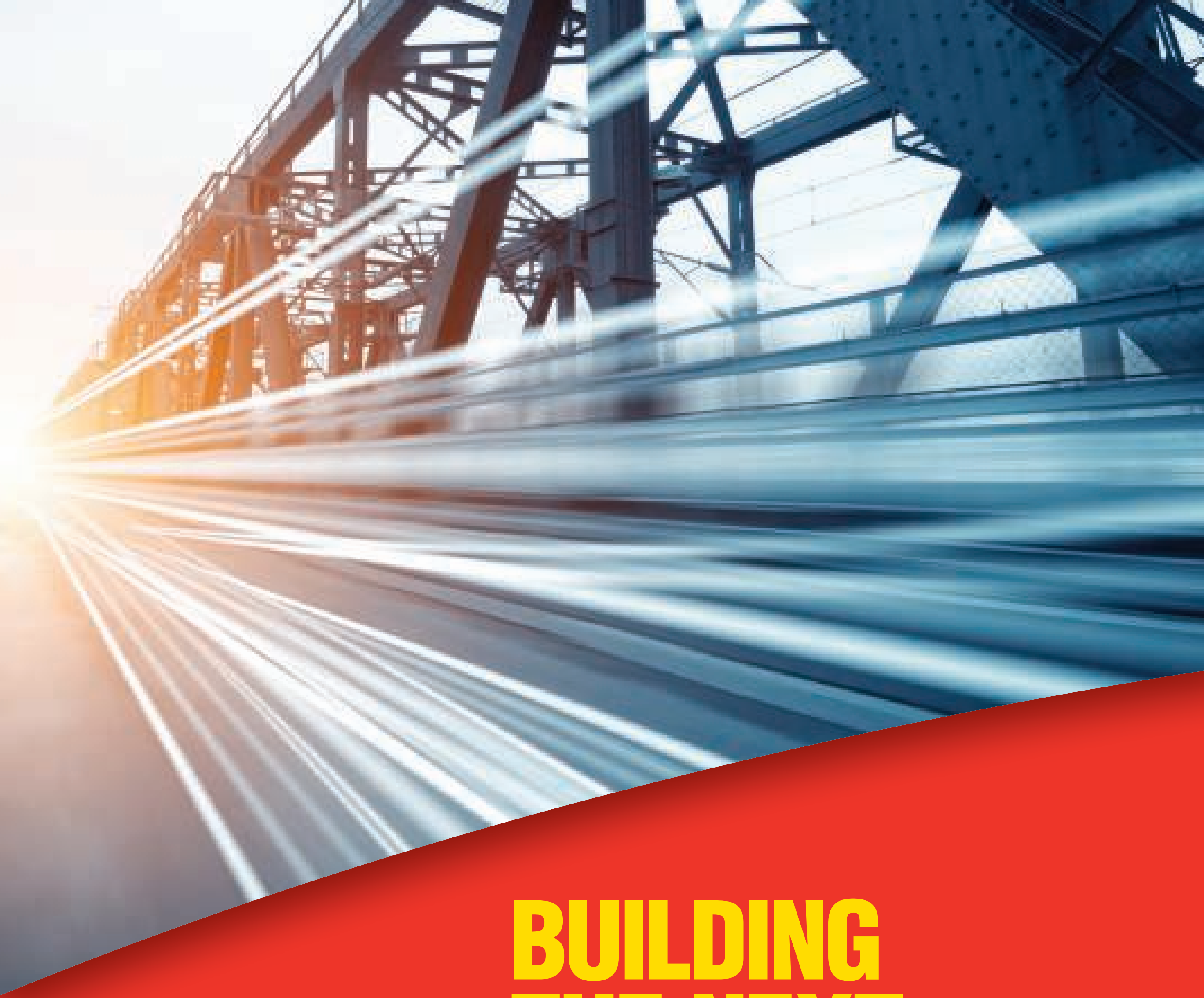
SaniSure

FIGURE 5. On a smaller scale, laboratory, pilot and prototype applications are also employing systems, which may include just a bottle, connections and filters, such as this single use integrated molded cap and tube system providing for aseptic transfer and storage

And Sartorius Stedim Biotech is now offering FlexAct (Figure 3), an innovative system for pre-configured single-use solutions for entire biomanufacturing steps in both upstream and downstream processing. The FlexAct system, a workstation, consists of the central operating module that offers a variety of configuration options. Priebe says the level of integration and standardization makes it easy for users of stainless-steel equipment or individual disposable components to convert to single-use operations. FlexAct disposable solutions are available for buffer preparation, cell harvest, virus inactivation, media preparation, virus removal and manifold filling.

In addition, Sartorius Stedim has formed a partnership with G-Con Manufacturing (College Station, Tex.; www.gconbio.com), a manufacturer of portable, configurable cleanroom Pods that allows the pre-configured unit operations to be placed inside portable cleanrooms, so it is fully self-contained and can travel on the back of a truck. "You just plug it in and are ready to go the next day," says Priebe. "We call it FlexMoSys because it's a flexible and mobile system that provides rapid response and easily reconfigurable operations that really address the needs of this emerging market." ■

Joy LePree



Trelleborg Group

FOCUS ON

Seals & Gaskets

Freudenberg Process Seals

Avoid pipe corrosion with this spacer

Flexspacer (photo) has been developed to effectively create an air gap between topside pipelines and thermal insulation, thereby avoiding the corrosion that can occur when using traditional insulation systems such as mineral wool. By stopping direct contact between the insulation and the pipe, Flexspacer prevents any damage to the corrosion protection on the pipe, thus helping to guarantee thermal performance. Flexspacer is a new rubber-based solution that provides a 1–2-cm air gap between the pipe and insulation. It can be easily installed without using hot works or special tools, and can be connected and split to the desired length using just a pair of scissors, says the company. It has been qualified for use up to 150°C continuous service temperature, for more than 30 years. — *Trelleborg Group, Stavanger, Norway*
www.trelleborg.com/offshore

This packing system removes particles from stuffing boxes

W.A. Chesterton

The SuperSet (photo) combines sealing capabilities of this company's mechanical packing products with the patented design of the EnviroSeal SpiralTrac Version P Environmental Controller for packed stuffing boxes. This total sealing set is designed and proven to increase packing and equipment service life while dramatically reducing flush rates, says the company. The savings are real — increased reliability with lower maintenance and operating costs. SpiralTrac enhances the utilization of the flow and centrifugal effects around the shaft, and performs like a centrifugal separator. The built-in lantern ring and helical grooving system impart motion to the flush, and the resulting centrifugal force throws particles in the fluid outside of the bushing. The patented SpiralTrac grooving system then hydrodynamically pumps the solids to the bottom of the stuffing box and back out into the pump casing. — *W.A. Chesterton Co., Wobum, Mass.*
www.chesterton.com

Very soft gaskets prevent water ingress into enclosures

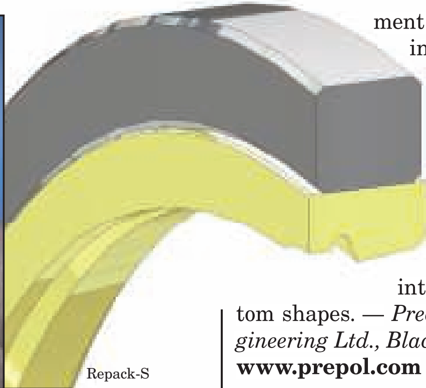
This company recently introduced molded liquid silicone rubber (LSR) gaskets with 10 and 20 durometer Shore A. The gaskets provide improved water sealing and protection for enclosures and portable devices. Low durometer LSR gaskets are said to have improved sealing properties over silicone foam or closed-cell silicone sponge gaskets. Enclosures and portable devices are frequently required to pass water ingress requirements, such as IP65, IP66 and IP67, and silicone rubber provides consistent deflection properties in cold and hot climates while resisting the damaging effects of ultraviolet and sunlight. The 10 and 20 durometer LSR compounds are Wacker Silicones LR3003/10TR and LR3003/20TR respectively. This company injection molds 12×12-in. LSR sheets in 10 and 20 durometer, with standard thicknesses from 0.032 to 0.125 in. — *Stockwell Elastomerics, Inc. Philadelphia, Pa.*
www.stockwell.com

This elastomer meets the demands of deionized water

The elastomer compound 70 EPDM 291 (photo) offers very good properties when in contact with both water and deionized water. The material exhibits no significant increase in mass or volume when submerged in water at 200°C over long periods of time, nor after prolonged immersion in deionized water at 100°C. O-rings and O-ring-type seals in special shapes or seals for valves and fittings made from 70 EPDM 291 remain reliably resistant in water up to 180°C, deionized water up to 160°C and in air up to 150°C. The material meets the specifications of the European standard DIN EN 681 — 1 WB and the U.S. standard

Note: For more information, circle the 3-digit number on p. 68, or use the website designation.

Precision Polymer Engineering



Repack-S

NSF 61, as well as German, British and French standards. The material also meets U.S. Food and Drug Amin. standards for both food and pharmaceutical products, and has USP Class VI approval for its high purity. — *Freudenberg Process Seals GmbH & Co. KG, Weinheim, Germany*
www.freudenberg-process-seals.com

Cold is okay for this chemically resistant elastomer
The Perplast ICE G75LT offers a combination of "excellent" chemical resistance and low-temperature performance. This perfluoroelastomer material has been specifically developed to perform under extreme conditions, in temperatures as low as –40°C or lower. The elastomer has been formulated to provide increased resistance to a broad range of chemicals by carefully controlling the molecular architecture. Also, this material has low permeability and as a result, it is less prone to swelling, leading to extended in-service performance in valves, pumps and mechanical seals. Said to be ideal for use in exploration and completion applications, and equipment operating or stored in sub-zero conditions, Perlast ICE G75LT is suitable for both dynamic and static applications and can be fully molded into O-rings (any size up to 2.5-m internal dia.) and custom shapes. — *Precision Polymer Engineering Ltd., Blackburn, England*
www.prepol.com

This composite improves hydraulics sealing performance
High-frequency, hydraulics cylinder operation is particularly demanding on seals. Traditional wedge-profiled PTFE composite seals are prone to early failure under the effect of repetitive short strokes and often dry-running conditions. This French seals maker has developed a better alternative — the Danastep HF seal (photo). It uses a modified PTFE seal profile by adding a back heel for additional stability, as well as a rectangular elastomer energizer, making it impossible for the PTFE seal to tilt and break free.

In addition, the PTFE grade is capable of sustaining dry running conditions. The seal fits within standard ISO/DIN grooves. — *Repack-S, Cuisery, France*
www.repack-s.com

Garlock Sealing Technologies

This high-temperature sealant is also available in sheets

This company's Therma-Pur ultra-high-temperature sealing material (photo) is now available in sheet form and in cut gaskets. Since its production in 2011, the material has been field proven to seal connections in biomass gasification, marine and land-based exhaust systems, turbochargers, mineral and fertilizer processing, incineration and cogeneration. The material is available in 40 × 40 in. sheets with an available fiber core thickness of 1/16 and 1/8 in. It is also available in precut gaskets as well as in Kammprofile and corrugated metal gaskets. The proprietary material provides sealing at temperatures of up to 1,000°C, including extreme thermal cycling conditions. Because of its low organic-fiber content and non-oxidizing formulation, Therma-Pur exhibits only minimal weight loss at these extreme temperatures. — *Garlock Sealing Technologies LLC, Palmyra, N.Y.*
www.garlock.com

Save on maintenance and operating costs with this elastomer

Kalrez perfluoroelastomer parts, such as O-rings, gaskets, valve seats or diaphragms, can withstand aggressive chemicals and maintain tight seals over a very wide temperature range. In real-life applications, the seals have been shown to save from 10 to 200% in total system operating costs in chemical, pharmaceutical and petrochemical processes, says the company. Data on file at the company showed that seal lifetimes of O-rings in an aggressive chemical process were about six months for non-perfluoroelastomer O-rings and about three years for Kalrez, resulting in savings of 98% in cost of O-ring seals, installation, downtime and loss or productivity, says the manufacturer. — *DuPont Performance Polymers, Wilmington, Del.*
www.dupont.com ■

Gerald Ondrey

CHEMICAL ENGINEERING
FACTS AT YOUR FINGERTIPS
Department Editor: Scott Jenkins

Gas Hazard Definitions and Data

The detection of gases in plant environments has a critical and wide-ranging role in the chemical process industries (CPI). Among the major applications of gas detection are limiting personnel exposure to hazardous chemicals, preventing explosive atmospheres, protecting the environment and identifying leaks in process equipment. Table 1 summarizes the main reasons for gas monitoring.

All CPI workers should be conversant with gas-detection terms to promote safety, health and environmental quality. The following is a collection of terms and data involving hazardous gases (Table 2).

PEL (permissible exposure limit). Set by OSHA to limit workers' exposure to an airborne substance, PELs are based on an eight-hour time-weighted average. PELs are enforceable legal limits.

TLV (threshold limit value). Established by the American Conference of Governmental Industrial Hygienists, TLVs are based on known toxicity of chemicals in humans or animals, and are recommendations, rather than legal limits.

IDLH (immediately dangerous to life and health). Defined in the U.S. by the National Institute for Industrial Safety and Health (Part of the Centers for Disease Control and Prevention) as a level of exposure that is likely to cause death or immediate or delayed permanent adverse health effects

LC_{50} (median lethal concentration). A measure of the toxicity of a surrounding medium that will kill half of a sample population of test animals in a specified period through exposure via inhalation.

Oxygen deficiency

Normal ambient air contains 20.8 vol.% oxygen. When oxygen concentration dips below 19.5 vol.% of the total atmosphere, the area is considered oxygen deficient. Oxygen deficiency may result from O_2 being displaced by other gases, such as carbon dioxide, and can also be caused by rust, corrosion, fermentation or other forms of oxidation that consume oxygen. Table 3 outlines the physiological effects of oxygen deficiency by concentration.

If oxygen concentrations in the air rise above 20.8%, the atmosphere is said to be oxygen-enriched. Higher oxygen levels can increase the likelihood and severity of a flash fire or explosion, because the oxygen-enriched atmosphere tends to be less stable than air.

Combustible atmospheres

Vapor and gas. Although these two terms are sometimes used interchangeably, they are not identical. Vapor refers to a substance that, though present in the gaseous phase, generally exists as a liquid or solid at ambient temperatures. Gas refers to a substance that generally exists as a gas at room temperature.

Vapor pressure and boiling point. Vapor pressure can be defined as the pressure exerted by a vapor in thermodynamic equilibrium with its condensed or solid form. Vapor pressure is directly related to temperature, and along with boiling point, determines how much of a chemical is likely to become airborne. Substances with low vapor pressures generally present less of a hazard because there are fewer molecules of the substance to ignite, but they may require higher-sensitivity instrumentation to detect.

Vapor density. Vapor density is the weight ratio of a volume of vapor compared to an equal volume of air. Most flammable vapors are heavier than air, so they may settle in low areas.

Explosive limits. To produce a flame, a sufficient amount of gas or vapor must exist. But too much gas can displace the oxygen in an area, making it unable to support combustion. Therefore, there is a window of concentrations for flammable gas concentrations where combustion can occur. The lower explosive limit (LEL) indicates the lowest quantity of gas required for combustion, while the upper explosive limit (UEL) indicates the maximum quantity of gas (Table 4). Gas LELs and UELs can be found in NFPA 325. LELs are typically 1.4 to 5 vol.%. As temperature increases, less energy is required to ignite a fire and the percent gas by volume required to reach the LEL decreases, increasing the hazard. An environment with enriched oxygen levels raises the UEL of a gas, and the rate of flame propagation. Mixtures of multiple gases add complexity, so their exact LEL must be determined by testing.

TABLE 1. SUMMARY OF THE MAIN REASONS FOR GAS MONITORING

Type of monitoring	Purpose	Hazard	Possible source of hazard
Personal protection	Worker safety	Toxic gases	Leaks, fugitive emissions, industrial process defects
Explosive	Worker safety and facility safety	Explosions	Presence of combustible gases and vapors due to leaks or process defects
Environmental	Environmental safety	Environmental degradation	Acid gas emissions
Industrial process	Process control	Process malfunction	Process errors

Source: MSA

TABLE 2. EXPOSURE DATA FOR SELECTED HAZARDOUS GASES

Chemical and formula	Properties	OSHA PEL (ppm)	IDLH (ppm)	LC_{50} (ppm)
Ammonia (NH_3)	Corrosive, flammable	50	300	4,000
Boron trifluoride (BF_3)	Toxic	1	25	806
Bromine (Br_2)	Highly toxic, corrosive, oxidizer	0.1	3	113
Carbon monoxide (CO)	flammable	50	1,200	3,760
Carbon dioxide (CO_2)		5,000	40,000	Not available
Chlorine (Cl_2)	Toxic, corrosive, oxidizer	1	10	293
Chlorine dioxide (ClO_2)	Toxic, oxidizer	0.1	5	250
Ethylene oxide (C_2H_4O)	Flammable	1	800	4,350
Hydrogen chloride (HCl)	corrosive	5	50	2,810
Hydrogen sulfide (H_2S)	Toxic, flammable	20	100	712
Methyl isocyanate (CH_3NCO)	Highly toxic, flammable	0.02	3	22
Nitrogen dioxide (NO_2)	Highly toxic, oxidizer	5	20	115
Phosphine (PH_3)	Highly toxic, pyrophoric	0.3	50	20
Sulfur dioxide (SO_2)	Corrosive	5	100	2,520

TABLE 3. PHYSIOLOGICAL EFFECTS OF OXYGEN DEFICIENCY BY DEGREE

Concentration of O_2 in atmosphere, vol. %	Physiological effect
19.5 to 16	No visible effect
16 to 12	Increased breathing rate; accelerated heartbeat; Impaired attention, thinking and coordination
14 to 10	Faulty judgment and poor muscular coordination; Muscular exertion, causing rapid fatigue; Intermittent respiration
10 to 6	Nausea and vomiting; Inability to perform vigorous movement or loss of the ability to move; Unconsciousness, followed by death
Below 6	Difficulty breathing; convulsive movements; death in minutes

Source: MSA

TABLE 4. EXPLOSION CONCENTRATION RANGES FOR SELECTED GASES

Gas type	LEL	UEL
Methane	5.0 vol. %	15 vol. %
Hydrogen	4.0 vol. %	75 vol. %
Propane	2.1 vol. %	9.5 vol. %
Acetylene	2.5 vol. %	100 vol. %

Source: MSA

References

1. U.S. Dept. of Labor, Occupational Safety & Health Administration (OSHA), 29 CFR 1910.1000 Table Z-1.
2. U.S. Centers for Disease Control and Prevention. National Institute for Occupational Safety and Health (NIOSH). NIOSH Pocket Guide to Chemical Hazards. www.cdc.gov/niosh/npg. Accessed March 2013.
3. Mine Safety Appliances Co., "Gas Detection Handbook" 5th ed. MSA Instrument Div., August 2007.
4. National Fire Protection Association. NFPA 325: Guide to Fire Hazard Properties of Flammable Liquids, Gases and Volatile Solids, 1994.

Show Preview

POWTECH 2013

April 23–25, Nuremberg, Germany

Malvern Instruments

Goudsmit Magnetic Systems

AKO Armaturen & Separationstechnik

A new pinch valve with a variety of connectors

New to this company's pinch-valve (photo) product range are the pneumatic pinch-valves type VMC, which are currently available in sizes DN10–100. These are multifunctional modular systems, which consist of a four-part construction: the housing (body), an elastomeric sleeve and two connection covers (socket ends). A variety of different end-connection types is available: flange; internal thread; Tri-clamp; weld-on ends; and threaded spigot (RJT connection). Special conductive models with separate grounding are also available for use in hazardous areas for Zones 1, 2, 21 and 22. The VMC Series of air-operated pinch valves meets all requirements of the Pressure Equipment Directive 97/23/EC. Hall 4, Stand 211 — *AKO Armaturen & Separationstechnik GmbH, Trebur-Astheim, Germany*
www.pinch-valve.com

Guided radar for level measurement of bulk solids

VEGA Grieshaber

The new guided radar sensor Vegaflex 82 (photo) features a new, automatic probe-end-tracking function, which enables the device to measure the levels of bulk solids — even those with dielectric constants of 1.1 and higher — with unprecedented reliability, says the company. When there is an increasingly unreliable product signal, the sensor switches automatically to the stable probe-end-tracking signal, which is always proportional to the level. This guarantees accurate, consistent readings, especially with critical products, such as plastic powder. Vegaflex 82 is suitable for level measurement in almost all bulk solids. It delivers reliable measurement data in process temperatures from –40 to 200°C and process pressures from –1 to 40 bars. Its applications range from large-volume bunkers to 75-m-high silos, from light, fine powders to coarse, heavy gravel. Hall 6, Stand 209 — *VEGA Grieshaber KG, Schiltach, Germany*
www.vega.com

Use this strong magnet to remove impurities from product

The recently developed, fast-cleaning clean-flow magnet (photo) reaches a magnetic field of 10,700 Gauss on the bars that are within the product. The magnetic system removes iron particles and weakly magnetic particles from powdery substances, such as powdered milk, sugar and flour. With the application of the new generation of Neoflux magnets, it is also possible to capture weakly magnetic particles, such as 304 stainless steel (SS). Because the grill of this type of clean-flow magnet can be drawn outward via a rail, the magnet ensures a smooth operation and safety for the operators. The casing is made of 304 SS and the entire machine has a smooth finish (Ra ≤ 0.8), thus complying with the HACCP/EHEDG requirements. Three standard sizes are available for handling capacities of 10 to 20 m^3/h. Hall 1, Stand 339 — *Goudsmit Magnetic Systems B.V., Waalre, the Netherlands*
www.goudsmit.eu

Many material-characterization instruments on display here

This company is exhibiting a number of instruments, including the new Zetasizer Nano ZSP, a system for measuring particle size, zeta potential, molecular weight and protein mobility, as well as for microrheology applications. The

Note: For more information, circle the 3-digit number on p. 68, or use the website designation.

Show Preview

Mastersizer 3000 laser-diffraction particle-sizing instrument (photo, p. 32I-1) will also be at the stand, as will the Insitec process analyzer for realtime monitoring. Finally, the company will show visitors how the Morphologi G3 can be combined with Raman spectroscopy to extend analytical capabilities. Hall 1, Stand 357 — *Malvern Instruments Ltd., Malvern, U.K.*
www.malvern.com

Lock Inspection Systems

These two inspection companies merge at Powtech
By joining forces, these two companies — Loma and Lock — will create one of the most diverse, technically advanced and service-oriented inspection companies. Among the products being exhibited together for the first time is the Insight PH (photo) for inspecting tablets and capsules. The unit is capable of accepting a high-output rate from tablet presses — up to 30,000 tablets or capsules per minute — and rejecting any minute metal contaminants with high efficiency. Also on the show floor is the Insight VF Vertical Fall metal detector, which inspects powders and granules at high throughput rates (up to 50 ton/h). Hall 6, Stand 237 — *Lock Inspection Systems B.V., Etten-Leur, the Netherlands; and Loma Systems, Carol Stream, Ill.*
www.lockinspection.com
www.loma.com

Filter bags and cartridges featuring patented microfibers
The Viledon Nexx and Hi-Nexx filter bags (photo), along with the sinTexx Plus filter cartridges have been designed for maximum efficiency in dust-removal applications. These new media feature the unique Evolon technology, which is based on a patented microfiber material that offers advantages compared to conventional needlefelt-type bags, says the company. Thanks to the fine microfiber surface featuring endless filaments, valuable recovered products will not be contaminated. Viledon Nexx and Hi-Nexx filter bags are up to 50% lighter than conventional needlefelt bags, which simplifies cleaning and installation. The Viledon sinTexx Plus Series of filter cartridges feature corrugated polyester nonwovens with a nanofiber coating, enabling the filters to handle dust and smoke, and an antistatic version is available for applications with potentially explosive dusts. Hall 5, Stand 235 — *Freudenberg Filtration Technologies SE & Co. KG, Weinheim, Germany*
www.freudenberg-filter.de

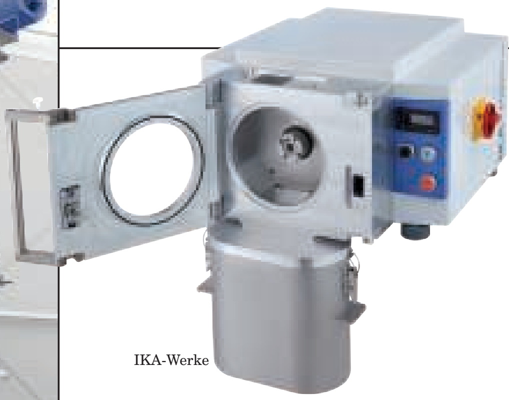
Doubling the production rate of personal-care products
This company has set a new milestone in mixing technology for large-

Freudenberg Filtration Technologies


Ekato Systems

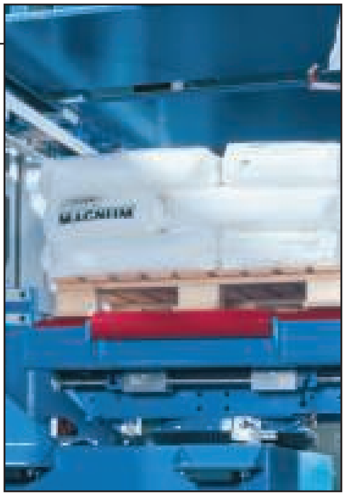
volume batch processing. The Paravisc agitator enables very fast wetting and the incorporation of structure-forming components, thereby allowing a very high powder-dosing rate to be achieved. The high-shear homogenizer S-Jet/V has the ability to incorporate thickener in high doses without the buildup of agglomerates. It exhibits a high circulation rate, even with high-viscosity media. Maximum production rate of existing 4,000–5,000-L equipment can now be more than doubled to 9,000–12,000 kg/h. The Unimix SRC 10,000 (photo) is said to be the world's largest batch-production machine. — Hall 6, Stand 410, *Ekato Systems GmbH, a subsidiary of the Ekato Group, Schopfhein, Germany*
www.ekato.com

One machine that combines two milling principles
The new universal mill Pilotina MU (photo) is said to be the ideal combination of a cutting and impact mill. This universal 2-in-1 dry mill combines the features and benefits of both a cutting and an impact mill into one machine. This dry mill's modular design allows for an easy transition from impact to cutting mill (and vice versa) within a matter of seconds. In addition, the mill features the highest milling quality as well as an adjustable circumferential speed of the tool to meet specific application needs. The machine is also equipped with special safety features. Hall 5, Stand 322 — *IKA-Werke GmbH & Co. KG, Staufen, Germany*
www.ika.com


IKA-Werke

This versatile, heavy-duty processor is self-cleaning
The Reactotherm (photo, p. 32I-5) consists of a cylindrical, horizontal shell

Buss-SMS-Canzler



Beumer Group

and a rotor with segmented discs and mixing bars. Stationary mixing hooks are attached to the inside of the shell. The close clearance between the mixing hooks, the segmented discs and the rotor results in a high mixing and kneading effect and largely self-cleaning properties of the rotor. Mixing bars are used to clean the inside of the shell, which prevents fouling of heat exchange surfaces and the formation of agglomerates. The shell, shaft and discs can be heated or cooled. The system can operate continuously or batch-wise under vacuum, atmospheric pressure and overpressure. Units are available with volumes from 160 to 10,000 L and 3 to 104 m^2 of heat-exchange surface area. Research and pilot units are also available with working volumes of 8 to 60 L. Hall 5, Stand 112 — *Buss-SMS-Canzler GmbH, Düren, Germany*
www.sms-vt.com

A system to accurately stack bags and package the pallet

Among the products and system solutions on display is the paletpac high-performance layer palletizer (photo) for palletizing bags filled with bulk materials. This flexible unit stacks polyethylene (PE) and polypropylene (PP) bags, layer-by-layer, on pallets of all common sizes up to a stack height of 2.4 m with geometrical accuracy and extreme stability. The user can quickly and easily set the required parameters using a multi-program. Depending on the product requirements, the paletpac is fitted with a rotary clamp or double-belt turning device that moves the filled bags quickly and gently into the required position. The stacked bags can then be packaged using the company's stretch hood. Hall 1, Stand 135 — *Beumer Group GmbH & Co. KG, Beckum, Germany*
www.beumergroup.com

This containment valve protects workers and the product

This company is presenting the new generation of the Müller Containment Valve (MCV), which is designed for the handling of toxic or highly potent products. The new series is suitable up to OEB (occupational exposure bands) Level 5 (occupational exposure level less than 1 μg/m^3), and not only safeguard the health of employees, but maintain the purity of the product and protect the environment. The locking device of the valve has been improved, offering advantages for the operator. The valve is sturdy, but lightweight, and the handling is simple. It can be used for applications under vacuum (–1 bar) as well as for those with overpressure (up to 6 bars). A new explosion-pressure-shock resistant version is also available offering safety against flame breakthrough for gases of explosion group IIB (up to 6 bars). Hall 1, Stand 345 — *Müller GmbH, Rheinfelden, Germany*
www.mueller-gmbh.com

Many new features incorporated in this electronic weighing scale

Like its predecessor of the MEC series, the new MEC 4 electronic weigher was designed in this packaging-machine manufacturer's development department for electronic weighing systems. The MEC 4 features a completely new family of hardware that allows the machines to keep up with the increasingly demanding requirements for more flexibility, speed and operator comfort. With its touch-


Haver & Boecker OHG


Gebr. Lödige Machinenbau

screen panel (photo), the MEC 4 offers a user-friendly interface, and is programmable with CoDeSys. Additional features include a high-speed processor, good electromagnetic compatibility and temperature stability up to 70°C. Up to 31 modules can be integrated for complex applications. Hall 1, Stand 533 — *Haver & Boecker OHG, Oelde, Germany*
www.haverboecker.com

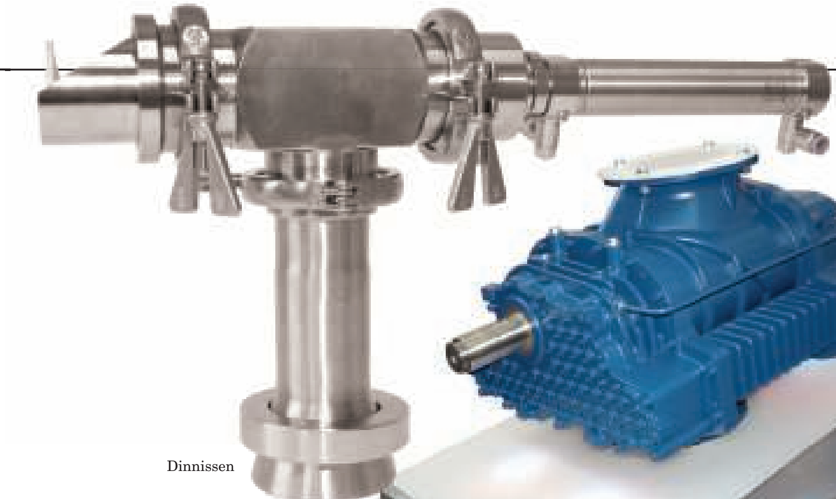
Reduce tablet coating times with this system

This fully optimized tablet coater (photo) is said to revolutionize the efficiency of coating processes and enables coating times that are 40 to 50% shorter than with conventional solutions due to its innovative drum geometry combined with a special configuration of the installed nozzles. The unit will be at the stand to demonstrate the possibilities for mixing processes and related technologies in the chemical, pharmaceutical, food and environmental industries. Also on display will be the horizontal Ploughshare mixer, which has a total volume of 900 L. A continuously operating Ploughshare mixer with the same volume will be used to demonstrate a multi-stage, continuous process that uses three continuous mixers of different sizes and designs. Hall 5, Stand 222 — *Gebr. Lödige Machinenbau GmbH, Paderborn, Germany*
www.loedige.de

Thin-film dryers gently process liquids, slurries and pastes


GIG Karasek

The thin-film drying technology (photo) from this company can be used for continuous drying of suspensions, municipal sludge, slurries, pastes, wet solids, filter cakes and chemical products. It is also suitable for heat-sensitive products, such as polymers, foods and pharmaceuticals. A thin-film dryer is a special type of wiped-film evaporator. The product to be dried is distributed over the circumference of the heating surface to form a downward-flowing thin film of liquid that is stirred by a specially shaped rotor system to create optimal turbulence. These dryers can produce dry powder from a liquid feed. They can be used either alone, or as a pre- or post-dryer combined with other equipment. Horizontal thin-film dryers are ideal for drying slurries and pastes, while their vertical counterparts are preferred for continuous drying of liquids to wet solids in a single step. Both types feature short residence times, low holdup, self-cleaning characteristics and low-energy consumption. Closed-system operation allows toxic and dangerous products, such as solvents, to be handled safely. Hall 6, Stand 328 — *GIG Karasek GmbH, Gloggnitz, Austria*
www.gigkarasek.at



Dinnissen

Aerzener Maschinenfabrik

Palas

This sampling system is completely automated

This new, fully automated sampling system (photo) complies with the most recent criteria of EHEDG (European Hygienic Engineering and Design Group) and can be used for the automated sampling of incoming, stationary and outgoing product flows. The system can be used for continuous and batch production processes based on dry powders, granules or granulates. The user simply selects the desired parameters (frequency, volume and weight) beforehand, and the system then takes the required samples quickly, accurately and efficiently, says the company. Sample capacity ranges from 25 to 1,000 g per sample. Hall 4, Stand 323 — *Dinnissen B.V., Sevenum, the Netherlands*
www.dinnissen.nl

More flowrate and power from this hybrid compressor series

The Delta Hybrid rotary-lobe compressor series has been extended by another size. The 14 sizes now cover intake volume flowrates of 110 up to 5,800 m^3/h, and overpressures of up to 1,500 mbar. The new sizes D 98 S and D 98 H are used in the upper volume flow range of 1,470 to 5,900 m^3/h, with drive powers up to 250 kW. The company is also preparing a version in the size D152S (photo), with volume flows of up to 9,100 m^3/h and maximum drive power of 400 kW. The multi-patented design of these compressors is said to improve energy efficiency, with energy savings of up to 15% compared to conventional designs. — *Aerzener Maschinenfabrik GmbH, Aerzen, Germany*
www.aerzen.com

Measure nanoparticles in gases with this condensation unit

The Universal Fluid Condensation Particle Counter (UF-CPC; photo) measures the number concentration of nanoparticles in air or gases by means of a condensation process that makes the particles large enough to be detected optically. The patented way that the working fluid is delivered in the UF-CPC enables the use of butanol, water or other environmentally friendly liquids. The modular design allows the unit to be optimized for single counting of droplets containing up to 10^6 particles/cm^3, and allows most maintenance tasks to be performed by the operator. For process control applications, the UF-CPC supports standardized interfaces (such as Modbus), remote access and data storage (internal network or internet). Hall 5, Stand 156 — *Palas GmbH, Karlsruhe, Germany*
www.palas.de

Piping for handling bulk goods, dust extraction and exhaust air

Pipe systems for bulk material handling as well as dust extraction and exhaust air units for environmental engineering (cooling and exhaust air) are the largest application areas for this company's modular pipe systems. The piping and components are available with 60–800-mm dia., or larger as a customized production, with 1–3-mm pipe wall thickness, and can be supplied as primed, galvanized or in stainless steel. The main applications are found in the chemical, pharmaceutical, plastics and food industries, and many more. Hall 5, Stand 348 — *Fr. Jacob Söhne GmbH & Co. KG, Porta Westfalica, Germany*
www.jacob-rohre.de

Bulk-material handling from components to systems

This company is presenting its latest generation of system components for bulk-material technology, including rotary feeders, double-flap gates and dome valves. The company's conveyor systems enable transport distances of over 1 km and capacities of over 100 ton/h. All systems are energy-optimized and tailored to the specific requirements of the user. Rotary feeders (including ceramic versions), pressure vessels, jet conveyors and screw sluices are used as feed-in units. In April, the company will also commission an in-house technical laboratory, which is equipped with industrial-scale bulk-material transport and storage systems that can reach a test capacity of up to 20 m^3/h and transport distance of 250 m. Hall 5, Stand 217 — *Kreisel GmbH & Co. KG, Krauschwitz, Germany*
www.kreisel.eu ■

Gerald Ondrey

Larson Electronics

APRIL
New Products

Anton Paar USA

These actuators are designed for large pressures

Designed for use on rising-stem globe control valves, the PL Series actuators (photo) provide up to 4,400 lb of force and up to 3.9 in. of travel. This increased up-and-down force, the company says, allows the use of globe valves for increased flow control in larger pipe diameters with higher close-off pressures compared to a corresponding butterfly valve with less flow control and lower close-off pressure. The force and travel of the PL Series actuators is said to be greater than competitive electric linear actuators. Key features of the PL Series actuators include: field-selectable travel stops, a customizable mounting system, precision proportional control, a manual override system and others. — *Promation Engineering Inc., Brooksville, Fla.*
www.promationei.com

Outsource high-throughput research with this service

Collaboration between an Italian university and an American company has resulted in a new service that will provide research services for outsourcing catalyst discovery and optimization to consultants and laboratory staff interested in high-throughput catalyst research. The service utilizes a range of high-throughput research tools for specialized polyolefin and organometallic catalysis investigation. The services are offered by a newly formed company spun off from a laboratory at the Federico II University of Naples (Italy), and are designed to extend the accessibility of high-throughput research. — *HTExplore*

Promation Engineering

s.r.l., Naples, Italy; and Freeslate Inc., Sunnyvale, Calif.
www.freeslate.com
www.htexplore.com

Use this light for boiler inspections

The recently launched WALBL-2 x 1000WMH Boiler Light (photo) is a telescoping light tower with a folding boom that is designed to allow versatile deployment and high-output illumination. The light features a right-angle extension arm, wheeled base, and a pair of 1,000-W metal halide lamps for effective illumination during the servicing and maintenance of boilers and tanks. The WALBL boiler light generates 220,000 lumens of light output in a wide flood pattern, and is easily maneuverable, the company says, allowing operators to position the light tower assembly to illuminate boiler interiors with maximum effectiveness. The telescoping arm can be shortened or lengthened, as well as angled, and an extra support leg on the boom provides extra stability. — *Larson Electronics, Kemp, Tex.*
www.larsonelectronics.com

Cover all applications with this versatile rheometer

The MCR 702 rheometer (photo) features this company's modular TwinDrive technology, and is said to be the only instrument capable of handling all rheological applications. The device has two synchronous EC (electrically commutated) motor units — one is permanent and one can be extracted and integrated when needed. The renowned dynamism and precision of the EC motors, the company says, allows users to take advantage of several options for meeting the requirements of their applications. In what is called 2EC mode, the rheometer employs both motors as drive units and torque transducers, rotating in either parallel or opposite directions. The double EC motors extend the scope of testing options. For example, the device can be used with counter rotation for enhanced rheomicroscopy. The lower motor can also be removed for use as a single-drive rheometer, enabling classic stress-controlled tests, the company says. — *Anton Paar USA, Ashland, Va.*
www.anton-paar.com ■

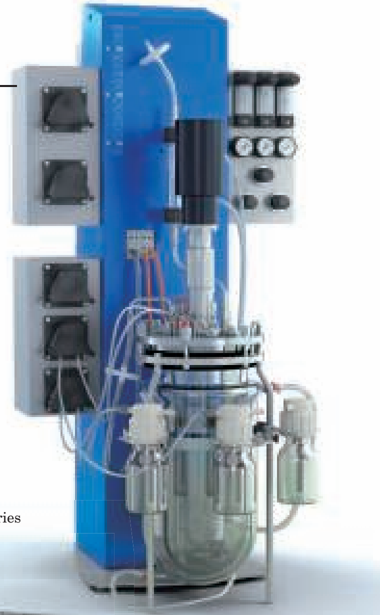
Scott Jenkins

Note: For more information, circle the 3-digit number on p. 68, or use the website designation.

INTERPHEX 2013

Bürkert Fluid Control Systems

M.O. Industries



Bioengineering USA

Fike

Aimed at the pharmaceutical and biopharmaceutical industry, Interphex 2013 will take place April 23–25 at the Jacob Javits Convention Center in New York. The conference program for the event features 52 sessions organized into five tracks, including supply chain, regulatory and quality control, product development, facility and process design, and manufacturing and packaging. Meanwhile, the exhibit floor will showcase over 1,000 product lines from more than 650 suppliers. The following descriptions represent a small sample of the products and services that will be highlighted in the exhibit hall.

This filter and valve system is integrated into one assembly

The company's IFV (integral filter valve; photo) system integrates a sterile filter assembly and adjacent valving into one integral block. Designed to increase yields and avoid dropped batches, the assembly reduces contact surfaces and hold-up volume while greatly reducing dimensional envelope constraints and installation time, the company says. The IFV comes with a universal filter-housing mount, two conventional hygienic diaphragm valves and one double-weir robolux hygienic diaphragm valve. Booth 3553 — *Bürkert Fluid Control Systems, Irvine, Calif.*
www.burkert-usa.com

This rupture disc technology has been expanded

The Atlas line of rupture discs (photo) is the latest to be manufactured with this company's G2 technology. G2 is a patented manufacturing process for rupture discs that avoids the use of hard-score tooling. The process builds rupture discs that are free of stress zones that can fatigue, says the company. Also the Atlas line offers higher pressure capability than was previously available. The product line is suitable for liquid or vapor service, and has a 95% operating ratio, backpressure resistance and high cycling capability. Booth 2236 — *Fike Corp., Blue Springs, Mo.*
www.fike.com

Transfer viscous products with this system

The Vispro (photo) transfers viscous materials from manufacturing sites to other locations, such as the packaging machine. The system uses air pressure to raise a plunger located inside the smooth wall of a stainless-steel drum. The viscous product is discharged through an outlet in the unit's lid. Booth 2725 — *M.O. Industries, Inc., Whippany, N.J.*
www.moindustries.com

Use these benchtop bioreactors for scaleup

RALF benchtop bioreactors and fermenters (photo) are designed for scaleup, and come with specialized software known as BioSCADA. This powerful software requires no programming knowledge, and allows users to benefit from the sophisticated tools of manufacturing experts in a straightforward, user-friendly way, the company says. The bioreactor control tower and vessels require minimal bench space, and offer superior accessibility to all components, as well as the ability to rotate a full 360 deg. The measurement and control components are durable and allow for thorough documentation. A two-year warranty is standard with all systems. Booth 1671 — *Bioengineering USA, Cambridge, Mass.*
www.mybioreactor.com

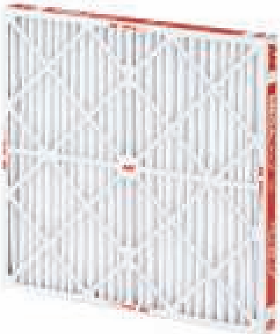
Use these pumps to control fluids without drift

Valveless, ceramic pumps from this company are designed for drift-free fluid control for pilot plants and benchtop operations. The pumps routinely monitor a wide range of substances, including monomers, catalysts, alternative fuels and water-treatment chemicals. These pumps also control additions of viscous fluids and slurries. The pump design eliminates the need for check valves, and can maintain accuracies of 1% or better for millions of cycles, the company says. — *Fluid Metering Inc., Syosset, N.Y.*
www.fmipump.com

Save energy with these air filters

The Megapleat M8 premium pleated air filter (photo, p. 35) is said to reduce energy costs by up to 20% compared to the leading MERV 8 pleated filter available in the market. It also lasts

Note: For more information, circle the 3-digit number on p. 68, or use the website designation.


American Air Filter


Metrohm NIRSystems

I Holland


30% longer and is 40% stronger than existing air filters, says the company. The Megapleat M8 filter has a patent-pending design that includes a heavy-duty, galvanized expanded metal support grid with an optimized pattern to reduce blockage to airflow. The metal cross-sectional area is 40% larger than other wire-backed designs. The strength of the grid, combined with the proprietary moisture-resistant glue formulation helps prevent blowouts and failures under the toughest environments, including high-moisture applications, the company says. Booth 3185 — *American Air Filter Co., Louisville, Ky.*
www.aafintl.com

This milling system delivers a narrow particle-size distribution

The ARTMiS (Automated Real-Time Milling System) features this company's Micronizer Jet Mill — a sanitary design, low-maintenance mill that delivers a narrow particle-size distribution, the company says, in the sub-micron size range. The ARTMiS is designed to simplify particle size reduction and maintain product quality despite fluctuating variables. The company says that the system can help pharmaceutical manufacturers increase yield and efficiency, improve batch quality, enable realtime decision-making, and help realize return on investment within 12 months. Booth 2267 — *Sturtevant Inc., Hanover, Mass.*
www.sturtevantinc.com

This cleaning system has a small footprint

The PH 810 cleaning system is designed for cleaning small pieces of pharmaceutical equipment and glass items, and is said to be the most compact pharmaceutical cleaning system in its class. With a width of 100 cm and chamber volume of 307 L, the PH 810 has an excellent ratio of payload-space to room-space. Specially suited to pharmaceutical production and research laboratories, the PH 810 cleaning system consumes 20% less water, energy and detergent, the company says. The system features items-dependent level control of detergent solution, and the company's gravimetric dosing system. Booth 3283 — *Belimed AG, Sulgen, Switzerland*
www.belimed.com

Use these NIR analyzers in the laboratory and in the plant

The XDS near-infrared (NIR) analyzer (photo) is designed for analytical performance with high sensitivity and precise instrument matching, says the company. The analyzers are capable of inline or online measurements and are available in single- and multiple-point configurations. They meet most electrical area classifications. Booth 2072 — *Metrohm NIRSystems (formerly FOSS NIRSystems), Riverview, Fla.*
www.metrohmusa.com

Apply semi-custom modifications to these transport containers

This company has a range of standard bins, drums, totes and hoppers that can be modified to allow users the optimal return on investment, and attain sustainable material-handling solutions. The reusable plastic containers for food, cosmetic and pharmaceutical applications can help users achieve efficiency increases and waste reduction, the company says. Booth 2162 — *Remcon Plastics Inc., Reading, Pa.*
www.remcon.com

This tool polisher has a larger capacity in the same footprint

The next generation in this company's MF product line (photo) is the MF40, a system for automated polishing of pharmaceutical tablet punches and dies. The MF40 has larger holders to allow more punches per polishing cycle, but fits into the same compact footprint of its predecessor. The MF40 polishing machine is part of a wider system for maintaining and storing tabletting equipment. Booth 3164 — *I Holland, Nottingham, U.K.*
www.iholland.co.uk

This separator is now available with a stainless-steel base

The Eco Separator is a spring-mounted grading separator that is suited to sizing, classifying or grading powders. The unit is now available with a fully stainless-steel base, making it ideal for applications in which the equipment is continuously washed down, and where painted equipment is not permitted, says the company. The Eco Separator has the ability to separate wet or dry materials on up to five fractions, and is available in six different sizes from 24 to 72 in. The grading sieve can be used in any sized plant, including laboratory-, pilot- or production-scale. Booth 2733 — *Russell Finex Inc., Pineville, N.C.*
www.russellfinex.com

Remove rust from stainless steel with these abrasive cloths

Abratec cleanroom abrasive cloths are designed to remove rust from stainless-steel aseptic processing equipment. Corroded surfaces are difficult to disinfect because the rough surfaces do not allow good contact in wiping and can provide bacteria with places to hide, the company explains. The Abratec cloths are made with various levels of abrasive grits, and are backed by the company's Quiltec fabric, which is said to be more durable than foam-backed abrasives. This allows the Abratec cloths to removed sanding debris without contributing additional


Schenck


The David Round Co.

particles to the remediation process. Abratec cloths are designed to work with solvents, including the water and isopropyl alcohol (IPA) mixtures that are typically found in sterile processing environments. Also, the cloths are irradiated with 25–50 kilograys of ionizing radiation to maintain sterile environments. Booth 1338 — *Contec Inc., Spartansburg, S.C.*
www.contecinc.com

This coating system uses ultrasonic vibration
This company has developed a new fluidized-bed coating system that uses an ultrasonic spray nozzle for gentle Wurster-type coating of solid-dosage pharmaceutical products. Rather than atomizing air pressure, the spray nozzle uses a tiny, ultrasonic "horn" that vibrates at a fixed frequency to break up droplets of coating material. The ultrasonic horn technology enables the nozzle to produce uniform droplet sizes in the range of 10 to 30 microns. The Wurster process involves suspending fine particulate substances in the fluid bed, while a nozzle applies the coating. The new ultrasonic nozzle system also features a controller for fine-tuning the spray pattern and shape, allowing users the ability to exert adjustable control over the size of the coating droplets. The system is designed for laboratory research and development applications. Booth 1542 — *Fluid Air Inc., a div. of Spraying Systems Co., Aurora, Ill.*
www.fluidairinc.com

This powder feeder is designed for pharmaceutical materials
The PureFeed AP-300 feeder (photo) was designed specifically for pharmaceutical processes and was engineered to have the following features: quick and easy disassembly, a dual-arm agitation system for maximizing material-handling versatility, and an FDA-compliant EPDM (ethylene propylene diene monomer) feed hopper that is disposable and recyclable. The design features allow simpler, shorter cleaning cycles and virtually no chance of cross-contamination when switching materials, the company says. The PureFeed AP-300 is available in volumetric and gravimetric configurations for pharmaceutical and nutraceutical applications with feedrates from 0.5 to about 150 kg/h. Booth 1843 — *Schenck AccuRate, Whitewater, Wisc.*
www.accuratefeeders.com

These mills are suited to wet grinding
Toothed colloid mills in the MZ series are suited to wet grinding in pharmaceutical, cosmetic and chemical applications of almost any viscosity. The milling system has a ring-shaped stator with internal teeth that slides over a high-speed rotor with external teeth. The rotor revolves in the stator around its own axis at speeds that can peak at up to 50 m/s. The unit has a choice of coarse, standard or cross-toothed tools and an individually adjustable milling gap. The shearing, rebound and crushing forces acting on the product can be precisely controlled because the rotor speed is continuously variable, the company says. The mill is available in several different versions, including laboratory models with a product throughput of 100 L/h to industrial types capable of handling up to 40,000 L/h, depending on the application. The design eliminates dead spaces, such as gaps, corners and slots from the interior to prevent material accumulation. Booth 3033 — *Romaco FrymaKoruma, Karlsruhe, Germany*
www.frymakoruma.com

Use these floor cranes inside cleanrooms
Stainless-steel portable floor cranes and strap hoists (photo) from this company are specifically designed for handling material in pharmaceutical cleanrooms. Design updates minimize contamination potential within the full line of the cleanroom material-handling equipment. The hoists and cranes are paint-free, and can be cleaned using methods that do not interfere with production schedules, the company says. Booth 2041 — *The David Round Co., Streetsboro, Ohio*
www.davidround.com

These single-use sensors make multiple measurements
This company's single-use sensors measure conductivity, pressure, temperature, and ultraviolet-absorbance, and can be used in numerous types of bioprocess operations. The sensors are offered in a wide range of sizes for inline measurement, and can eliminate several processes that would be required for reusable measurement devices, including parts cleaning, tracking, sanitation and possible re-sterilization. Booth 3565 — *PendoTECH, Princeton, N.J.*
www.pendotech.com

Hand-load small products with this packaging system
The Compact-4 vertical end-load cartoner erects cartons, pauses for hand-filling, and then glues or tucks each paperboard carton at speeds of up to 40 per minute. The unit is ideal for hand-loading vials, leaflets and other items into small cartons. The easy-to-operate machine features two ergonomic loading areas, and a quick-changing star-wheel design that allows for eight unique operations, including erecting, filling, closing flaps and sealing. Its footprint is 4 ft by 6 ft. Booth 3384 — *Adco Manufacturing, Sanger, Calif.*
www.adcomfg.com ■

Scott Jenkins

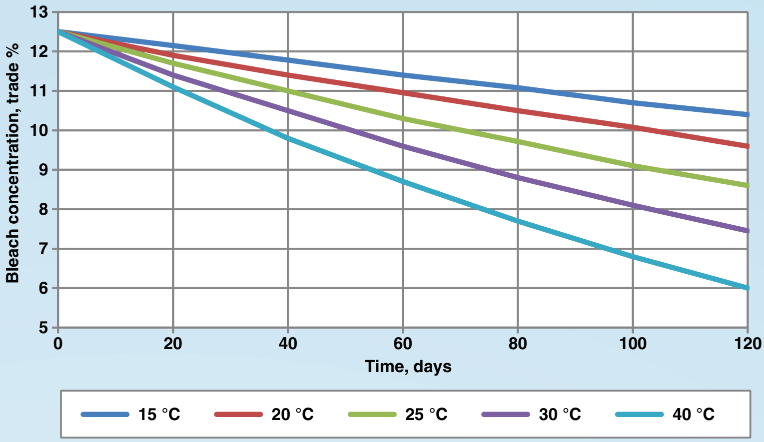
Technology Profile

Sodium Hypochlorite Chemical Production

By Intratec Solutions

Sodium hypochlorite (NaClO) is the active constituent in chlorine bleach, a strong oxidizer and bleaching agent. Increases in household bleach demand are driven mostly by population growth. In turn, population growth and its corresponding increases in water consumption — coupled with limited fresh water resources — makes water treatment the largest application for bleach, as well as the fastest-growing segment of bleach use.

Sodium hypochlorite chemical production is a well-established process in the industry, and the principle behind its operation is also employed for preventing chlorine emissions in chlor-alkali plants. The chemical process relies on the acquisition of chlorine and caustic soda (sodium hydroxide; NaOH) feedstock from external sources, in contrast with the electrochemical process for bleach, which also involves brine electrolysis.



The process

The chlorination of caustic soda to sodium hypochlorite is an exothermic reaction. The chemical production process depicted below is a widely used process, similar to the one employed by Solvay Chemicals (Brussels, Belgium; www.solvay.com), for example. The process is suited to producing both household bleach (5–6 wt.%) and industrial bleach (10–15 wt.%), and relies on the chlorination of caustic soda by chlorine gas within packed columns.

The industrial sodium-hypochlorite plant can be divided into two main sections: reaction and product discharge; and bleach filtration.

Reaction and product discharge. The chlorine absorption system can be divided into two parts: in the first, a packed column is operated with a safe excess of caustic to prevent reduction in pH; the second part, in turn, receives the liquor from the first column to be post-chlorinated, until the desired bleach concentration is reached. Prior to reaction, caustic soda is diluted with water in the first buffer tank, along with the first-column bottom stream. Chlorine gas is diluted with air and fed into the bottom of both columns. Flow to the second part of the system is established when the finished product is sent to the filtration steps.

Bleach filtration. Bleach filtration is necessary to meet product quality requirements and is often the last step before storage. Usually, the bleach filter system consists of pressure-leaf filters. After filtration, the product is sent to storage. Backwash water containing spent filter aid can be taken to a dry-cake pressure filter for further processing.

Sodium hypochlorite solutions are very sensitive and special operations must be carried out to prevent its decomposition. Besides pH, other factors affecting degradation are the initial bleach concentration, exposure to ultraviolet light, presence of certain metals and elevated temperatures. The graph above shows temperature dependency for bleach degradation, starting from a 12.5 trade-percent solution. Trade percent is an expression of available chlorine, in units of grams per liter of available chlorine.

Economic performance

An economic evaluation of the process was conducted for three distinct locations — the U.S. Gulf Coast region, Germany and Brazil — and is based on data from the first quarter of 2012. The following assumptions are made for the analysis:

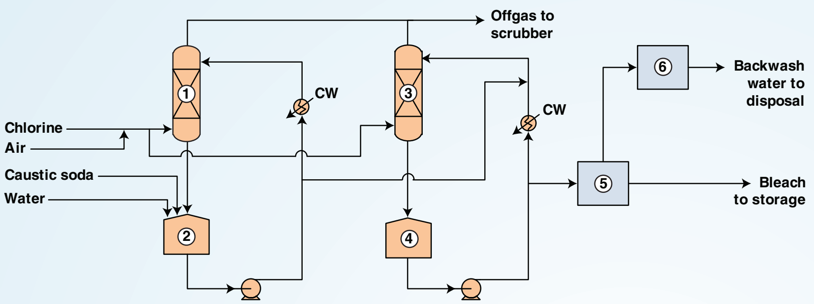
- A 250,000 ton/yr chemical production unit (NaClO solution, 12.5 wt. %) erected inside a chlor-alkali facility (all equipment is represented in the simplified flowsheet)
- Storage of products is equal to 20 days of operation, and there is no storage for feedstock

The estimated capital investment for such a plant on the U.S. Gulf Coast is about $35 million, the lowest among the regions compared. Germany presented a higher capital investment, at $40 million, and the highest operating costs — about $170/ton (compared to $140/ton in the U.S.)

Global perspective

Selling prices for bleach vary substantially, depending on supply and demand fluctuations of the chlorine/caustic market. Based on the estimated capital and operating costs, a U.S. Gulf Coast-based bleach manufacturing venture can reach an internal rate of return (IRR) above 15%, by selling bleach at an average price of $175/ton, a reasonable value. However, a venture in Germany would not be economically attractive at the same selling price. ■

Edited by Scott Jenkins



(1) 1st Absorption column
(2) 1st Buffer tank
(3) 2nd Absorption column
(4) 2nd Buffer tank
(5) Bleach filter system
(6) Backwash water filter
CW = Cooling water

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.

Filtration and Separation In Sulfur Recovery

Controlling contaminants is key to reliable, economical processing across all segments of industry

David Engel
Sulphur Experts, Filtration Division

Sulfur is a major contaminant in the oil and gas industry that exists in various forms and requires separation from both water and process streams. In recent years the removal of sulfur species has received increasing attention because of safety concerns, the need to reduce emissions of sulfur oxides (SOx), and the fact that sulfur harms the combustion properties of fuels. The "sulfur recovery trail" starts with hydrogen sulfide (H_2S) and mercaptans either present in raw hydrocarbon streams or arising in the petroleum refinery as a result of cracking or hydrogenation processes. The trail continues as H_2S and mercaptans are captured by methods such as amine treatment, caustic scavenging, or sour water stripping, and ends with the conversion of H_2S to elemental sulfur, sulfuric acid, salts or other materials.

In the many plants that make up the sulfur recovery trail, filtration and other separation processes are important in minimizing process upsets caused by the presence of contaminants. In fact, contamination control through separation is a key step in helping to maintain process control. Separation processes are often the first — sometimes the only — line of defense for avoiding many process variations and out-of-control episodes, and separation devices play critical roles in minimizing downtime and increasing systems reliability.

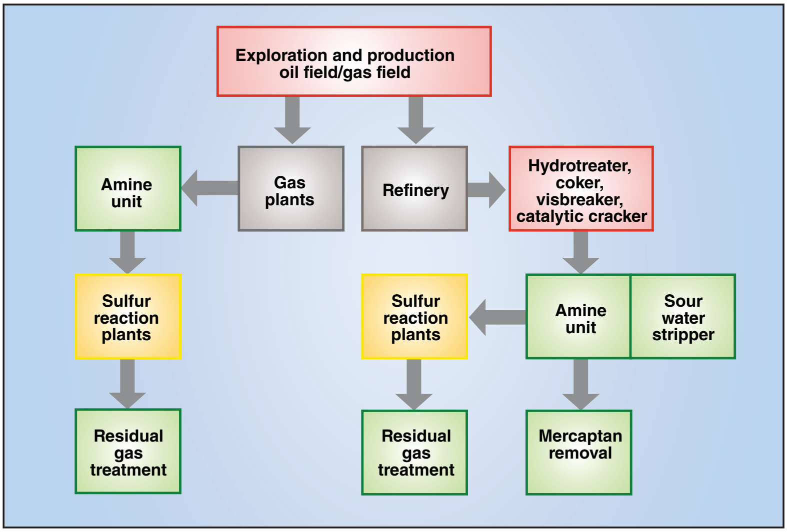


FIGURE 1. Typical stages of the sulfur recovery trail include (red) sulfur generation, (green) sufur capture, and (yellow) sulfur reaction

Studies at a number of plants worldwide reveal that many possess less than adequate separation and filtration systems, resulting in significantly higher operating costs. Many of the lessons learned along the sulfur recovery trail about the need for good separation are equally applicable to other parts of the chemical process industries (CPI).

SULFUR RECOVERY TRAIL

Sulfur in hydrocarbons has two major sources. It can be found naturally as covalent sulfides in hydrocarbon macrostructures, and it also occurs in other chemical forms in crude oil, connate water, produced water, natural gas and gas condensates. New sulfur species can also be generated when crude oil is processed. For instance, when water containing high levels of sulfate is used to aid crude oil production, sulfur reducing bacteria can turn sulfates into H_2S. In some cases, H_2S is produced by bacterial degradation of organic matter.

Removal of H_2S is accomplished without much difficulty by amine systems, solvent systems, redox (reduction-oxidation) reactions, precipitation and chemical scavenging. Mercaptans (also called thiols or, in chemical shorthand, RSH), are somewhat closely related to H_2S and are removed by fairly similar technologies. However, sulfur species that are covalently linked to organic systems are not as reactive as H_2S and need to be transformed to H_2S before they can be removed.

One of the most common ways to do this is through reduction with excess hydrogen, using catalysts, heat and pressure to drive the reaction to near completion. This is the basis of catalytic hydrogenation (also called catalytic hydrodesulfurization), in which sulfur residues (and to some extent nitrogen residues) in hydrocarbons are replaced by hydrogen. The sulfur combines with excess hydrogen to produce H_2S, while nitrogen yields ammonia.

The resulting H_2S-laden hydrocarbons are purified alongside hydrogenated hydrocarbons by amine units and other methods, followed, if necessary, by further processing to reduce mercaptan levels. H_2S in water streams can be removed using chemical methods such as resin beds or additives, or by volatilization in a stripper (oftentimes with co-separation of NH_3). In both cases the resulting gaseous H_2S is generally sent to a sulfur recovery unit. At the downstream end of the sulfur recovery unit, remaining trace sulfur species can be further removed using a SCOT-type unit or other tail-gas recovery technologies before venting to the atmosphere. Taken together, these processes form the basis of the sulfur recovery trail (Figure 1), in which sulfur is generated, captured and converted for further use, transport, storage or disposal.

Role of separation processes

The sulfur recovery trail is a complex interlocked array of different units. Optimal performance of the trail is dependent on each of the units operating at its highest efficiency. Among the most basic needs for any process, system and plant in the sulfur recovery trail is the control of contaminants. Contamination control is essential in that it allows effective process control and enables units to operate consistently at an optimized cost and while maintaining or enhancing throughput.

Separation systems play a dual role in the process. They not only remove the daily contamination that exists in a normal process, but also protect the process from upstream upsets. The most common separation technologies in the sulfur recovery trail include demisters, filters to remove suspended solids from liquids, adsorption (activated carbon) beds, gas coalescers, flash tanks and two-phase/three-phase separators. Other systems are also utilized, but are more specialized and less common.

To cover all separation systems in a single article would lead to a lengthy discussion, so for simplicity we will look at one specific unit — the amine unit — which encompasses most, if not all, of the basic separations utilized in the sulfur recovery trail. The amine unit arguably suffers the most from contamination instabilities that considerably affect the performance, stability and reliability of the whole sulfur recovery process, but the concepts discussed here apply for the most part to other units in the sulfur recovery trail, and can be extended to the rest of the CPI.

Even well-operated amine units have uncontrollable factors that cause process upsets. Some of these relate to contaminants entering the plant via the gas or liquid to be processed, which is generally liquefied petroleum gas (LPG, C3/C4). To better understand its ramifications, it is important to understand what kind of contamination might be present in the feed. In addition to the gaseous hydrocarbon containing the H_2S, the amine unit must handle many other components generated in the upstream oil production and transmission stages or in the various upstream refinery units.

Upstream gas production yields produced water loaded with a variety of water-soluble contaminants, and compressor lubricants with surfactant properties. Additives used to enhance production include demulsifiers, hydrate inhibitors, biocides, methanol and many others, and with the recent rise of shale gas availability we might expect new contaminants in both the upstream gas and the produced water. Clearly, proper separation technology is critical.

On the refinery side, contamination can take the form of coke fines, heat-stable salt precursors, corrosion products, water, additives (such as corrosion inhibitors and anti-fouling agents), carbon fines and compressor oil. Most if not all of these have some kind of surfactant activity or other detrimental effect. Together, these contaminants can cause a multitude of plant problems, such as amine contactor foaming leading to lower capacities, amine losses and low efficiencies. Additionally, some components can also cause issues such as amine degradation and formation of heat-stable salts.

Effective separation is especially challenging since all these contaminants can be in three possible states: solid, liquid and gas. In the solid state it is generally found that iron clusters predominate, along with carbon and coke fines, and in some cases sand, salts and oxides. Most of the liquid contaminants are lubricants, carried-over hydrocarbon products, carried-over amine solvent, cleaning products and water. This water can also contain soluble impurities, such as chlorides, sulfates and acetates among others. In the gas phase the predominant contaminants are substances such as hydrogen cyanide, oxygen and carbonyl sulfide. Other contaminants that can cause major plant upsets include methanol and BTEX (benzene/toluene/ethylbenzene/xylene).

EQUIPMENT IN DETAIL

Shortcomings of demisters

Inlet separation for gas streams is usually carried out using a knockout drum equipped with a demisting element, such as a mesh pad or vane pack, installed near or at the outlet of the vessel. These systems are typically horizontal in gas plants and vertical in petroleum refineries. The basic difference is that a horizontal drum offers increased residence time and holding volume, while a vertical drum has a smaller footprint. Some less-used technologies include conventional horizontal filter-separators as well as cyclonic separators.

However, conventional demisters are only adequate for removing large-diameter contaminant droplets. These separators were originally designed for bulk liquids removal (hence their alternative name of slug catchers). In addition, they are not designed for solids separation (usually done by a wet scrubber or a particle filter), with the exception of cyclonic systems that can remove large solid particles and some larger liquid droplets.

Most traditional approaches to separating liquid droplets, including demisters with mesh pads, vane packs or certain horizontal filter-separators, display rather low efficiencies when removing sub-micrometer aerosols from the gas streams. As a result, only a small number of plants have the necessary means to adequately condition sour gas entering amine plants.

The most challenging contaminants to separate in any gas stream are sub-micrometer liquid aerosols. These finely divided liquid droplets have diameters ranging from less than 0.1 µm to a few hundred micrometers. Droplets around the 0.1–1.0 µm range are the most difficult to remove due to the absence of a specific separation mechanism that yields high removal efficiency. Yet, as Figure 2 shows, about 50 wt.% of all liquid droplets in the gas stream are smaller than 1 µm, and 80 wt.% are smaller than 10 µm.

The lack of efficiency of most traditional separators relates to the aerosol droplet-size distribution, the flow configuration inside the separator, and the mechanism of liquid droplet interception. In other words, the separation medium is simply not capable of intercepting and coalescing sub-micrometer liquid droplets, followed by the liquid unloading required to maintain proper high-efficiency separation. As a result, most aerosol contaminants break out of the system almost intact, or are re-entrained in the gas flow.

Mesh pads suffer from flooding when excessive liquids are introduced and the mesh becomes saturated with liquid. This leads to efficiency losses through carry-over. These devices are also prone to fouling by solid particles, further reducing efficiency and causing considerable maintenance costs and pad failures. Movement of the mesh pad inside the vessel is somewhat common due to the difficulty of properly anchoring these devices to the vessel interior.

Vane packs offer better mechanical performance and lower differential pressure, but inferior separation efficiencies. Sub-micrometer liquid droplets in particular do not have enough momentum to properly contact the vane surface, so most small droplets are just carried with the stream.

In many vane packs and some mesh pads, one cause of inefficiency is the formation of interfacial layers. Vendors have tried to mitigate this through designs including double and single pockets, and also by combining vane packs with mesh pads. In general, however, none of these results in removal efficiencies adequate to protect sensitive equipment and processes.

Even when the separation medium is appropriate to the job at hand, the wrong vessel configuration can reduce separator performance. Gas routing inside the separator can be a source of significant inefficiency, and poor vessel design can actually shatter liquid aerosols into smaller sizes, adding more difficulty to an already challenging separation process. If the gas flow creates sufficiently large shear forces, large droplets will break up into successively smaller droplets until the distribution is stabilized by the balance of energy distribution, gravitational settling and shear.

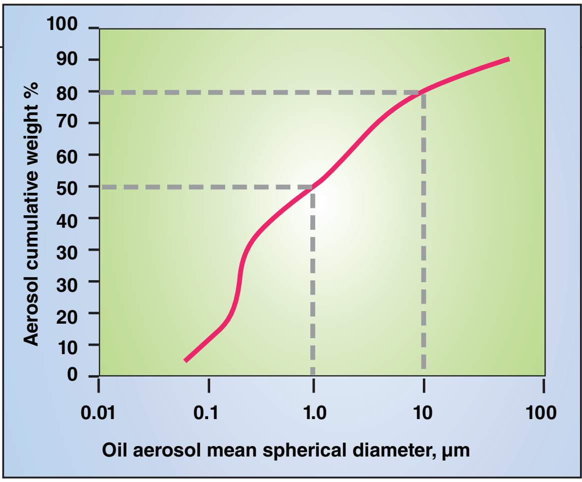


FIGURE 2. A typical oil aerosol size distribution at the outlet of a compressor shows that half of the oil load is in the hard-to-remove size range of 1 µm and below

Microfibers perform better

Demisters are often used in sulfur recovery units, where care has to be taken to properly design and size them because these factors directly impact contamination removal and plant performance. So if mesh pads, vane packs, and cyclones should only be considered for liquid aerosols with droplet sizes well above 10 µm, what is the answer for sub-micrometer aerosols? Today, the technology of choice is built around specially formulated microfiber media. Properly designed and installed, a coalescer separator incorporating microfibers should remove 99.98+ wt.% of all droplets larger than 0.1 µm — at least in laboratory tests.

Such a high-efficiency sub-micrometer coalescer is a specialized piece of equipment that needs to be carefully designed depending on the flow, pressure, temperature, gas composition and contaminants. Many fabricators say that their systems are capable of removing sub-micrometer liquid aerosols, but most do not correlate these claims with actual performance. Only a small number of companies possess the proper technology to supply sub-micrometer gas-liquid coalescers.

The sub-micrometer coalescer should be installed as closely as possible to the unit or process it is intended to protect. It should be protected in turn by an upstream separator designed to remove large droplets — typically a conventional mesh pad. This extends the on-line life of the coalescer and minimizes operational costs, as a mesh pad is much less expensive to replace than a coalescer element.

A typical amine inlet-gas separator therefore has two stages mounted one above the other. Gas flows upwards into the lower stage, where large droplets are removed by a mesh pad, or sometimes a vane pack or cyclone. The gas then flows upward to the second vessel, across the microfiber coalescing medium, and the purified gas exits from the top of the vessel. Each stage has a liquid removal system comprising a level control and drain valves.

Typical campaign times for gas coalescing elements vary from a few months to two years, depending on the amount of solids entering the coalescing stage and the nature of the additives present.

Flash tanks and separators

Separation technologies based on pressure decay, velocity changes and residence time are among the most common separation systems used in oil and gas operations. All have the common theme of using simple concepts to attempt to solve a separation problem. One such example is the amine flash tank. This device removes off-gases by reducing the pressure of the rich amine solution downstream of an amine contactor. If designed correctly, the flash tank also provides limited liquid-liquid separation capabilities for hydrocarbon removal in the event that the stream has a high hydrocarbon content. However, removal will only apply to free

FILTER EFFICIENCY: DON'T RELY ON THE LAB

Filtration efficiency has always been an area of nebulous and sometimes inconsistent recommendations. Experience shows that efficiencies measured in the laboratory under controlled conditions, using a single, uniform contaminant, might not be totally relevant to field conditions. The only way to ascertain "real" filter efficiency is through tests on operating systems: measuring particle compositions and size distributions at the inlet and outlet of the filter. This allows filtration to be optimized as necessary by changing the properties of the filter medium.

As a starting point for filter optimization, if possible, it is recommended to match the particle size distribution to the medium in terms of efficiency and micrometer sizes. In the absence of such information, it is best to start with a filter of lower efficiency and increase efficiency as needed based on periodic sample analysis. Filter lifetime and cost are also important to consider. ❑

hydrocarbons that separate from the amine solution over a timescale of minutes. Emulsified or dissolved contaminants are unaffected.

A number of different flash tank designs are available, but most feature poor design and short residence times. Some designs even incorporate metal mesh internals to promote coalescence and decrease residence time. Another reason why these apparently promising devices often provide poor to marginal results is their designers' poor understanding of highly fouling, rich amine streams. Detailed design of the internals with respect to hydrocarbon separation is also an area of weakness: many vessel fabricators do not consider proper location and sizing for the internal box or weir that serves to remove hydrocarbons.

Two- and three-phase separators are similar to flash tanks with the difference that they are often larger in size and in some cases do not have any mist elimination devices at the gas outlet.

Many two- and three-phase separators are sized based on the correct parameters, but with a lack of understanding of liquid and solid loading. To use coalescing mesh pads correctly, these should be designed not only according to the gas velocity across the pad using the modified Souders-Brown equation, but also considering liquid (that is, water versus liquid sulfur) and solid (iron sulfide gels versus coke fines) properties and internal flow geometry. Any disregard of these aspects will invariably lead to element flooding and liquid carry-over or fouling with a differential pressure increase.

Liquid-liquid coalescers

Less commonly used, but quite powerful as separation devices, are liquid-liquid coalescers. These are often used to remove water or hydrocarbons from process streams. In the sulfur recovery trail they typically are installed in rich amine streams, sour water feeds, mercaptan removal unit outlets and many others. In sour water strippers and rich amine streams, hydrocarbon removal is important for plant reliability and is critical in enabling acid gases to be processed in sulfur recovery units (by minimizing hydrocarbons in the gas stream).

Liquid-liquid coalescers fall basically into two categories:

- Low-efficiency systems with metal internals
- Systems with microfiber internals

Both perform rather differently and should be used for different objectives. While coalescers with metal internals are good for separating free liquids and macroemulsions (about 100 µm and larger), coalescers with microfibers are more in line to separate microemulsions (100 µm and smaller), almost down to solubility limits.

Most liquid-liquid coalescers should be protected by particle filters or other solids pre-separation devices. These not only protect the coalescing pads or microfiber elements from solids plugging, but also disrupt solids-stabilized emulsions.

Suspended solids filtration

Most of the filtration in the sulfur recovery trail occurs in the amine unit (the sour water stripper, due to its highly corrosive environment, is another unit where filtration is important). The lean-amine filtration system is better defined as lean-amine conditioning, because filtration is not the only event taking place. Lean-amine filtration generally comprises three separate components, each with distinct functions and requirements. None of these can be replaced, eliminated or by-passed, and all of them are necessary for proper lean-amine conditioning. Rich-amine filtration is the most cost-efficient way to purify the amine solution. Its objective is to protect the amine solution itself, safeguard the lean/rich heat exchanger from deposition, reduce instability in amine regeneration, decrease corrosion rates in the regeneration stage, and reduce the solids burden on the lean-amine filters. It also provides a way to control high iron concentrations, which can cause unexpected foaming in the absorber through sudden sulfide formation (soluble iron entering the absorber with the lean amine rapidly generates insoluble iron sulfide upon exposure to H_2S).

Filtration can be applied to either the full rich-amine flow or to a side stream. If structured packing is used in amine regenerators or absorbers, full upstream filtration is the best way to protect the packing and minimize solids deposition. Column packings make great particle filters.

Most plants find that rich-amine filtration brings significant process improvement and enhanced reliability. Prior to embarking on rich-amine filtration, however, a cost/benefit analysis and return-on-investment study, taking into account the costs of maintenance and filter disposal, are recommended. For amine solution filtration, the best technology is still disposable cartridges. These are relatively low in cost, easy to use, reliable, and ensure that contaminants are actually removed from the refinery. Other systems, such as automatic filters, generate significant secondary impacts in the form of the solids-laden backwash stream, which is generally sent to slop tanks or cokers. Automatic filters do not require frequent maintenance, but their filtration efficiency is low and in some applications their overall operating cost can be higher than that of cartridge filters. Automatic filters or backwash systems should preferably not be used in streams with a high fouling tendency, such amine units and others with complex mixtures of adherent suspended solids and hydrocarbons.

Centrifuges are one of the most promising emerging technologies for amine purification. However, drawbacks include high capital costs and maintenance costs, limited fluid capacity, poor efficiency on small particles, and challenges in operating under the highly fouling conditions that characterize amine streams.

Adsorption beds

Adsorption materials used to remove soluble contaminants include molecular sieves, alumina, salts, activated carbon, sand and fruit shells. Contaminants removed include certain heat-stable salts ("residues") and their precursors, amine degradation products, certain dissolved hydrocarbons and foam-promoting species.

Activated carbon is perhaps the most common adsorbent. Many different types of activated carbon are available, with varying adsorptive powers for contaminant molecules of different types and sizes. Differences in the performance of different grades of activated carbon originate from the source of the material — coconut shells, wood, bitumen, fruit skins and many others — and the different activation processes used to enhance surface area and eliminate impurities from the pores. Some activated carbons also contain additives to enhance their mechanical strength.

Activated carbon for liquid streams is usually granular in form. Powder types are more suitable for some gas applications. Extruded carbons are now being used more widely; these tend to have slightly lower surface areas and somewhat higher differential pressures.

From a design perspective, activated carbon beds are simple, but require good understanding of their operation and failure modes. Fundamental aspects for efficient performance include correct carbon type, effective bed loading, proper liquid distribution to avoid channelling, adequate residence time, correct cross-sectional velocity, and a suitable ratio of bed diameter to length. Activated carbon beds are not filters (see box above): they are not designed to separate suspended solids, nor free or emulsified hydrocarbons. The presence of these types of contaminants will rapidly render the bed useless.

Activated carbon beds release carbon fines (small carbon particles) from fractured carbon granules, and it is necessary to capture these solids before they reach any downstream operation. For amine plants this is generally the absorber tower, where the presence of carbon particles will cause foaming, fouling, and depending on fluid velocity, erosion corrosion. Any of these can lead to low sweetening efficiency and amine losses. More often than not, carbon particles are also found in the rich amine stream, inside and downstream of the rich-amine flash tank, and even in the regeneration stage.

Amine recovery

Amine losses have become an area of great interest lately due to the considerable economic costs of amine replacement. Often overlooked is the fact that the lost amine will end up in a downstream process unit, wastewater treatment facility or slop oil tank. Any of these destinations will have a significant impact on equipment reliability, process stability and economics.

Methods of recovering lost amine include knockout drums with mesh pads or vane packs, water washes, coalescers and other more specialized technologies. Removal of amine from treated liquid-hydrocarbon streams is important due to the high emulsification rate of the amine in the hydrocarbon, in addition to its solubility and mechanical entrainment. In this case, water washes and other separation systems have been implemented with varying degrees of success. Conventional water washes tend to use large amounts of water and have low amine-recovery efficiencies. Today, there are newer and more technologically advanced systems called extractive separation technologies. These devices are capable of recovering amines much more efficiently and with lower costs, reduced water use and minimal footprint.

TYPICAL FLUID CONTAMINATION SCENARIOS

In principle, a well-operated amine unit has no need for filtration. Such an ideal system is seldom encountered, however. In the real world, filtration and related separation technologies may be the only line of defense against serious upsets in amine units, and in other plants too. Deploying the best and most advanced contamination-control devices helps to ensure process stability, equipment reliability, and enhanced throughput. Separation technologies mitigate a series of problems commonly found in the sulfur recovery trail, such as the following:

Fouling. This is deposition of solids and hydrocarbons along with other components to form a coating over equipment surfaces typically in hot, low-velocity locations, such as heat exchangers and reactor columns. Fouling has many mechanisms, but there is some agreement that there are two main routes: (a) a free radical polymerization, condensation or decomposition of dissolved species present in the stream, or (b) deposition of suspended matter present in the stream. Today, fouling is generally prevented by chemical means (free radical inhibitors such as hindered phenols) or by mechanical means, such as filtration. Both methods can be effective, depending on the fouling mechanism and process conditions. Fouling also leads to energy losses and flow reductions and is a source of major maintenance efforts.

Corrosion. Typically, where there is fouling, the next natural progression is electrochemical corrosion beneath the fouling deposit. This is caused by elevated local concentration of corrosion initiators and the formation of electrochemical cells. Usually if fouling is

A CARBON BED IS NOT A CARBON FILTER

The core of lean-amine conditioning is the activated carbon bed. It is critical to remember that carbon beds are not filters. They are designed to remove dissolved species via surface attraction forces (adsorption), and as such it is always necessary to keep the surface of the activated carbon clear of solids.

To achieve this, the carbon bed has to incorporate a suitable pre-filter designed to remove suspended solid particles, which would otherwise block the pores on which activated carbon relies for adsorption.

An activated carbon bed should show only a small pressure drop, if any. If the pressure drop is significant, the system is already saturated with solids or emulsions, with a high probability that the activity of the bed has been long extinguished. The activated carbon is commonly tested for lifetime and activity using empirical foaming tests at the bed inlet and outlet. More sophisticated spectroscopic tests require removing samples of the activated carbon.

The activated carbon bed also requires a post-filter designed to retain any fractured or residual activated carbon particles present at the outlet stream of the bed. Carbon residues can stabilize foam due to their small size and density, and can also cause deposition and erosion corrosion in pipes and equipment. ❑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minimized, corrosion rates will be lowered as well. Some plants also suffer from erosion corrosion, which occurs when hard, dense particles strike the material surface, removing passivation layers and the metal beneath. Corrosion mechanisms are often complex.

Heat-stable salts and amine decomposition. Heat-stable salts are amine salt contaminants that do not decompose under normal regeneration conditions. Despite the name, some "heat-stable" salts will break down at temperatures higher than those routinely found in the plant. It is believed that the formation of many of these salts is accelerated by the presence of suspended solid contaminants and mediated by dissolved metal ions such as iron. Solid surfaces possess many active sites for heterogeneous reactions and are also rich in metal species that can catalyze salt formation, which increases the concentration of heat-stable salts and promotes amine decomposition. In many cases, reducing suspended solids and hydrocarbons in the amine solution will decrease the rate of heat-stable salt formation.

Foaming. This is generally produced by the association of gases and liquids stabilized by surfactants, which lower surface tension at oil/water interfaces. These surfactants can be solids (such as micrometer-size iron sulfide particles) or individual molecules (such as compressor lubricant). Foaming invariably leads to amine loss and lower efficiency. Removal of solids and hydrocarbons greatly reduces foaming and hence the need for antifoam additives. In amine units, however, other factors can also lead to foaming, and these can have many root causes. Hence, proper plant evaluation (process, equipment, feed composition) is required to determine the source of foaming. Antifoam agents should be used with caution as in large doses they may actually promote foaming, and they may separate out in certain filtration systems. Remember that antifoam agents treat only the symptom, not the cause.

Regenerator protection and acid gas quality. Filtration and separation systems on the rich-amine stream are designed to protect the regeneration section of an amine plant and to protect the sulfur recovery unit by facilitating the delivery of good-quality acid gas. This is done by ensuring that the amine is free of contaminants that would foul the rich/lean heat exchanger, increasing the reboiler duty and generally causing corrosion at the bottom of the regenerator. More plants are now adopting rich-amine filtration in addition to lean-amine filtration. In fact, both stages are not only necessary but complement each other. Removal of contaminants, such as suspended solids and hydrocarbons, from the rich-amine stream ensures a better acid-gas quality with better, smoother and easier amine regeneration. The ingress of contaminants such as hydrocarbons into the sulfur recovery unit not only produces soot, but can also seriously compromise the catalyst's physical integrity. Hydrocarbons compete with H_2S in the initial oxidation stage, resulting in considerable variability and mismatched oxygen demand in the stoichiometry of the modified Claus reaction.

Low sweetening efficiency. Filtration is designed to remove suspended solids from the amine solution. It is known that lean amine with a high solids content is less able to transfer H_2S efficiently. This is caused by multiple layers of solids at the interface of the gas (or liquid) and the amine solution, essentially blocking mass transfer. Good-quality amine solutions with minimal contaminants perform much better, promote process stability, prevent equipment damage, save energy, and so lower the overall annual operational cost of the plant.

The following sections present a compendium of recommendations and guidelines for proper filtration and separation in the sulfur recovery trail. This is not a definitive recipe, because every plant is different, but it is a fairly comprehensive summary of experience gained in many years of field tests around the world. Many of the messages here will have application beyond the sulfur recovery trail.

CONTAMINATION SOURCES IN SULFUR RECOVERY

Hydrogenation plants. Inlet contamination, recycled hydrogen contamination and outlet contamination

Amine units. Inlet contamination, outlet carryover and amine solution contamination

Sour water plants. Inlet contamination, outlet contamination, and acid gas contamination

Sulfur reaction plants (including sulfur recovery units). Inlet contamination

Mercaptan removal plants. Inlet contamination, outlet contamination and caustic solution contamination

Filtration and separation systems considerations

- Knowing the end goal, and the constraints on plant operation and cost
- Protecting equipment from fouling or degradation
- Meeting fluid specifications
- Enhancing equipment reliability and stabilizing the process
- Ensuring environmental compliance, reducing waste and emissions
- Lowering maintenance effort and cost
- Reducing overall contamination removal costs
- Enabling the unit to increase throughput

Criteria for selecting filtration and separation systems

- Analyze the process stream for contaminant types and concentrations
- Understand particle sizes and shapes
- Sample over a period of time to detect process variations
- Understand the contamination sources; this may lead to different solutions
- Filter efficiency ratings form a reference point from which to start — perform adjustments with the unit operational and with on-line sampling
- Consider future expansions
- Carefully choose the location for installing the system
- Consider maintenance and environmental aspects

Filtration and separation systems failure modes

- Improper technology for the application
- Incorrect compatibility (chemical, thermal or mechanical)
- Deficient vessel design

- Inappropriate or deficient sealing surfaces
- Incorrect media and efficiency
- Lack of or incorrect maintenance
- Instrumentation deficiencies (or lack of necessary instrumentation)
- Change in feed conditions

Concluding remarks

Many years of field experience teach that a key step in process control is proper control of contamination. Most plants that do not take this step struggle with high operational costs and low reliability, in addition to many detrimental technical, economic and environmental aspects.

There is no significant disadvantage to implementing enhanced separation and filtration besides a marginal increase in capital cost. One might tend to believe that cost will be prohibitive, but experience shows that this is not the case, and operational costs can actually be lower. On the other hand, there are real and serious issues involved with neglecting separation and filtration systems, using systems with deficient designs, or simply not giving the proper attention to contamination control. Invariably, any capital savings from low-cost separation and filtration will ultimately lead to exponentially higher processing costs, low reliability and frequent unit upsets.

It is also important to understand that each plant and process has its own equilibrium point where the cost of contamination control is acceptable and the residual contamination level is tolerable. Users, engineering firms and suppliers have the responsibility to be involved in finding such balance, with the objective of supplying the right separation and filtration solution for each individual plant.

A holistic understanding of the case, and why a given separation system is required, is critical in designing, troubleshooting, optimizing, operating and maintaining systems. This will provide positive process and economic benefits, allowing each plant to take full advantage of the installed process capabilities and to maximize throughput. ■

Edited by Charles Butcher

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Boosting Energy Efficiency in Chemical Manufacturing: A Look at Cogeneration

Producing electricity and heat from a single energy source offers operational, environmental and financial benefits

Ray Ratheal
Eastman Chemical Co.

Source: U.S. EPA

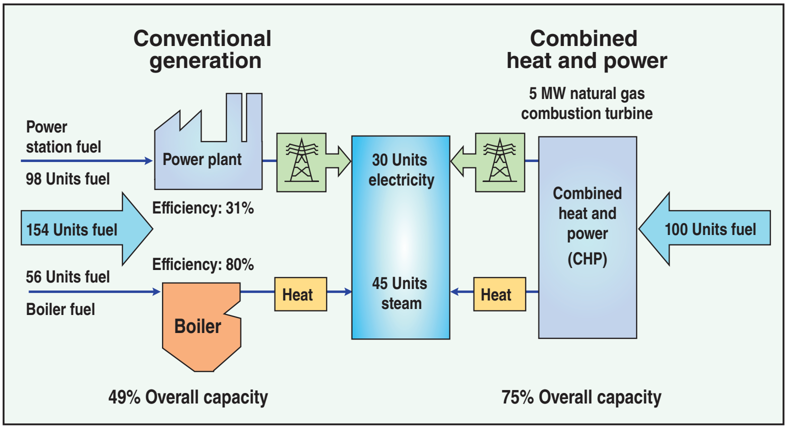

FIGURE 1. Generating both electricity and steam is a much more efficient way to extract energy from a fuel

Many chemical manufacturing processes require large amounts of energy, and chemical companies are faced with the constant challenge of determining how to use that energy as sustainably as possible. More and more chemical companies are prioritizing a range of energy efficiency measures aimed at decreasing energy use and costs while reducing environmental impact. One of the most promising energy-efficiency technologies being used in the chemical process industries (CPI) today is cogeneration — or combined heat and power (CHP).

Cogeneration offers financial, operational and environmental benefits by concurrently producing electricity and heat from a single energy source. It has the potential to convert more than 80% of the energy obtained from fossil fuel into power and steam (Table 1).

TABLE 1. POTENTIAL COGENERATION EFFICIENCY

Type	Efficiency, %	Type	Efficiency, %
Steam turbine	80	Gas turbine	75
Diesel engine	75	Fuel cell	73
Natural gas	75	Microturbine	70

Eastman Chemical Co. is one organization leveraging cogeneration to boost the efficiency of its manufacturing operations. The company today meets more than 90% of its global electricity needs with cogeneration, resulting in 40% less fuel used, and significantly fewer greenhouse gases (GHGs) emissions than with the conventional uses of fossil energy. At its Kingsport, Tenn. facility alone, the company saves the equivalent GHG emissions of taking more than 130,000 cars off the road each year by using cogeneration.

Adding to cogeneration's potential for the CPI and other energy-intensive manufacturing industries is the fact that the technology is adaptable, scalable and can be seamlessly integrated into existing manufacturing facilities with the right planning and expertise.

About cogeneration

The key advantage of the cogeneration process is its capture and recovery of "waste heat" that is normally rejected in a typical electricity generation cycle (for example, Rankine or Brayton cycles) to create additional "useful" energy. By recovering so-called "waste heat" from coal and natural gas cycles that would normally be released during electricity generation, cogeneration reduces the total quantity of fuel that would otherwise be used to produce heat or steam (Figure 1). According to the U.S. Environmental Protection Agency (EPA; Washington, D.C.), 50% of coal's energy is wasted if it is used conventionally, and more than 60% is wasted if coal is converted to electricity in a traditional power plant. Cogeneration effectively uses this wasted energy for chemical production plants' heating requirements, allowing manufacturers to boost efficiency and reduce the environmental impact of their operations.

Because many traditional CPI manufacturing plants purchase electricity and use boilers to make steam, they can be modified to include cogeneration capabilities. To implement cogeneration processes at a traditional chemical plant, an electricity generation step in the manufacturing process must be added prior to the export of steam. In

Source: ATCO Power

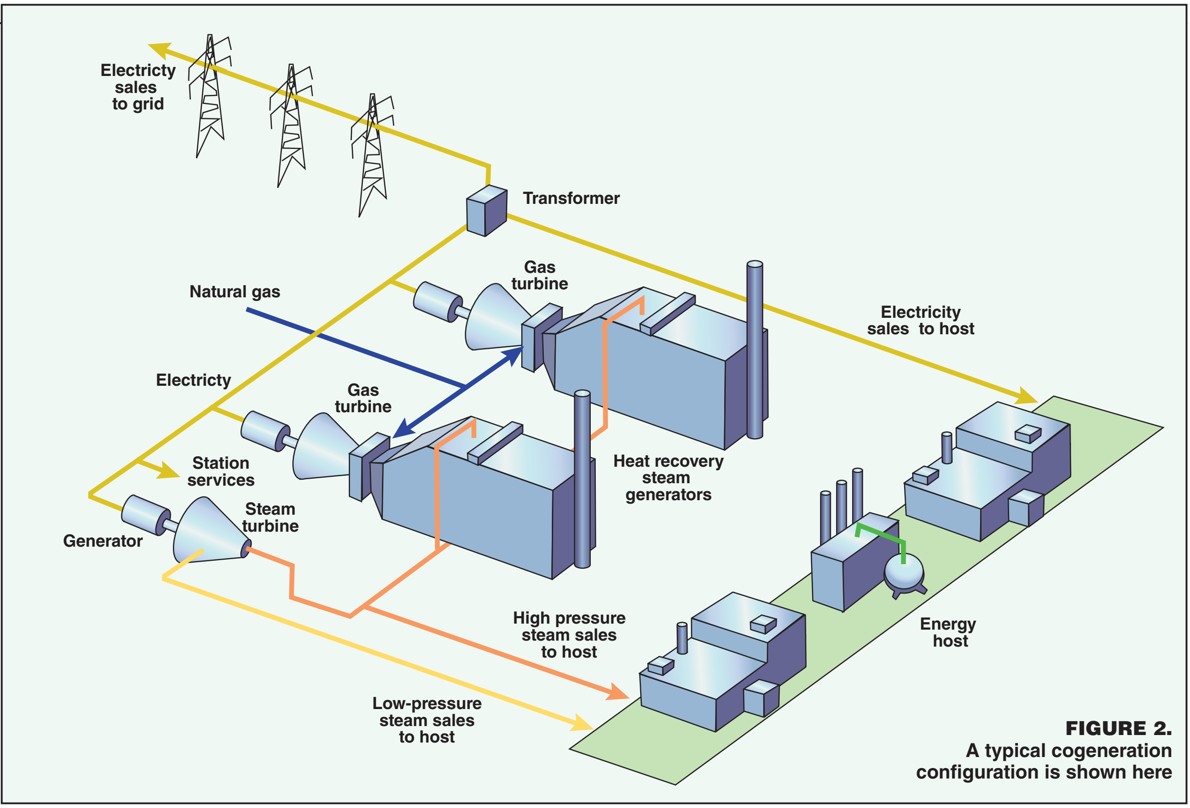

FIGURE 2. A typical cogeneration configuration is shown here

one common cogeneration configuration (Figure 2), natural gas is burned in a combustion turbine connected to an electric generator to produce electricity. The hot exhaust gases from the combustion turbine are routed into a heat recovery steam generator (HRSG), a steam boiler that uses the heat in the exhaust gases (sometimes supplemented with additional natural gas firing) to heat feedwater into steam. That steam can be used to power a steam turbine connected to another load (an electric generator that makes electricity for the plant, or to drive a large piece of equipment), or to supply steam directly for process heating needs, or both.

Another common example of cogeneration is the use of a traditional "topping boiler" to produce high-pressure steam that generates electricity in a steam turbine-generator, which then exhausts steam at lower temperatures and pressures to match the thermal needs of the manufacturing processes. In general, cogeneration processes can be configured in a variety of ways to accommodate the ratio of thermal-to-electric energy required, temperatures and pressures of steam needed by manufacturing or mechanical drive systems, the scale of the system, the types of fuels available, and a variety of site-specific technical and economic factors.

Adapting and installing cogen

While most CPI plants can be configured to incorporate cogeneration processes, there are a few common hurdles that engineers must recognize and overcome when implementing the necessary technology.

Energy balance. The primary concern for engineers is to balance the ratio of thermal energy to electrical energy. This ratio can shift significantly depending on the facility's load profile and its appetite for exporting electricity to the grid, and is one of the key variables that will dictate the design engineer's choice of the prime mover. In general, a project with a high ratio of thermal-to-electric power (for example, 80% thermal to 20% electrical) will generally favor a topping boiler. Conversely, a project with a low ratio of thermal-to-electric power (for instance, 60% thermal to 40% electrical) will generally favor a combustion turbine with a HRSG — with or without a steam turbine generator.

The largest integrated cogeneration site of the author's company in Kingsport, Tenn. has operated independently from the electricity grid for decades by carefully matching the significant thermal demand growth with boilers and both turbine generators and mechanical-drive turbines. The blend of electric motors and mechanical-drive turbines resulted in a robust system with a variety of flowpaths to move steam between different pressure levels while ensuring both reliability and thermal efficiency. The company's integrated cogeneration site in Longview, Tex. uses a combustion turbine cogeneration system and takes advantage of the opportunity to export electricity to the grid while also supplying steam to manufacturing assets at the site.

Scale. Another key consideration for engineers implementing cogeneration processes is the scale of the project. The large capital cost of installing combustion turbines or topping boilers can be justified if large, stable energy demands exist (for example, tens or hundreds of megawatts of electric demand). However, a different kind of prime mover would be more suited to small energy demands. For example, a reciprocating internal combustion engine with heat recovery from the jacket cooling water may be appropriate for a system with a 500-kW peak electric load and a 15 psig steam demand.

Fuel available. The types of fuels available also influence the choice of prime movers (Figure 3). The availability and cost of natural gas, fuel oil, biomass or coal should be weighed against the operational, environmental and capital cost impacts of the prime-mover selection. For example, if a facility was co-located with a pulp mill that had an attractive source of hog fuel (bark), a topping stoker or fluidized-bed boiler would be the most appropriate choice, as opposed to a combustion turbine. Likewise, many manufacturing facilities recover useful energy from secondary materials generated at the site, which may be more readily fired in certain types of engines.

Exporting electricity. An additional variable for deploying cogeneration capabilities is the ability to sell power not needed by the manufacturing facility. A thorough understanding of the regional electricity grid (the electricity market) will help the designer and operator determine whether selling power to the grid is worth the incremental investment in capacity, and help optimize the configuration and the best operating rate. For example, if external electricity demand exists and the pricing is attractive, engineers could design the system to generate additional electric power during peak periods to maximize the economic value of the fuel in addition to fulfilling the thermal demand of the manufacturing operations. The additional capital required to create electricity for export beyond the site's electrical and thermal requirements (for example, combustion-turbine systems versus Rankine cycles) can be analyzed to see if the incremental return on invested capital justifies the investment. If analysis of the site's unique economic drivers are favorable, the designer can configure a cogeneration system that possesses the flexibility to satisfy internal thermal and/or electric needs while also capturing value from the electric grid.

It is well understood that it is generally more expensive and less efficient to move thermal energy than to move electricity. Most cogeneration systems are optimized when located in close proximity to the steam demand. However, if exporting power to the grid requires a new high-voltage transmission corridor, the engineer must weigh the relative costs and benefits of transporting both thermal and electric energy when choosing a site. This may require creative reconfiguring or the acquisition of new land or rights of way, and can be a key consideration for engineers implementing a cogeneration-powered plant.

It is also well understood that a cogeneration system is rarely the lowest-capital-cost approach to supplying energy. However, the additional gains in efficiency result in lower life-cycle energy costs to the site, and those systems that also export power to the grid can become a profit center. These factors vary widely depending on a variety of site-specific technical and financial considerations, but cogeneration is often a clear-cut winner when evaluated on a life-cycle cost basis. This can be especially true when a site's steam generators require expensive upgrades due to end-of-life considerations or environmental mandates. In that scenario, evaluating cogeneration as an alternative technology is similar to the decision to purchase a new car versus investing in extensive repairs to a worn-out older vehicle; while the new car carries a higher initial cost, the long-term benefits are realized and often justify the up-front cost.

Alternatively, when a site needs to expand its steam-generating capacity, cogeneration offers both higher efficiency and a smaller footprint compared to installing a steam generator without the capacity to generate electricity. Due to environmental constraints, cogeneration on a larger scale may offer the most cost-advantaged method of site expansion. Whatever the site-specific conditions, cogeneration often has an advantaged return on invested capital when evaluated on a life-cycle basis compared to replacing or upgrading older boilers.

Source: Cogen Europe

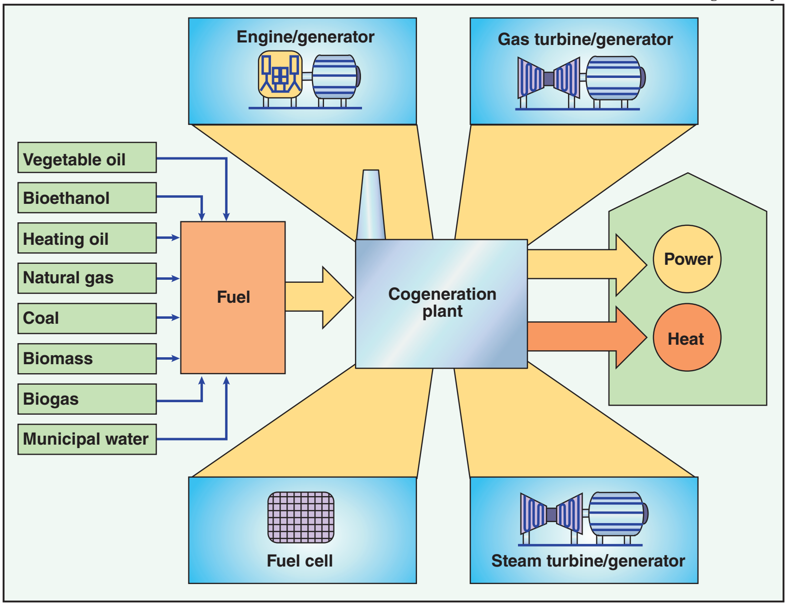

FIGURE 3. A wide range of fuels can be used for cogeneration. The availability — and cost — of a given fuel will determine the type of boiler required

Long-term benefits

Cogeneration provides meaningful environmental and economic benefits. With electric power and fuel/raw material pricing volatility, cogeneration is a steady and cost-effective thermal and electricity source. Many major environmental groups and policymakers concerned about GHG emissions strongly support cogeneration due to its lower GHG footprint compared to using fossil fuels to generate thermal energy and purchasing electricity from relatively inefficient electric utilities. The U.S. Department of Energy (DOE) cites cogeneration as one of the most promising energy-efficient technologies available, in part because it combines environmental effectiveness with economic viability and improved competitiveness. In fact, the DOE committed to investing nearly $25 million annually to improving technology for cogeneration programs.

Most recently, President Barack Obama signed an Executive Order on Aug. 30, 2012 to facilitate investments in industrial energy, including cogeneration systems. The Executive Order established a new national goal of 40 GW of new cogeneration processes by 2020, a 50% increase from 2012. According to President Obama's Executive Order, meeting this cogeneration goal would save energy users $10 billion per year, result in $40–80 billion in new capital investment in chemical manufacturing and other facilities that would create American jobs, and would reduce emissions equivalent to 25 million cars.

In addition to support from President Obama, the DOE and EPA, many chemical companies, including the author's, are working with the American Chemistry Council, Business Roundtable, International Energy Credit Association and Industrial Energy Consumers of America to promote policies that support implementing cogeneration processes so manufacturers can realize its environmental and economic benefits.

Now more than ever — with unprecedented attention paid to sustainability, energy use and bottom-line costs — chemical engineers should consider proven processes, such as cogeneration, to improve efficiency and environmental footprints. Moving forward, the CPI should set the standard for all energy-intensive industries by deploying cogeneration technology and uncovering additional energy-efficiency methods to complement and expand upon existing processes. ■

Edited by Gerald Ondrey

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Combined Heat and Power for the CPI: Modern Concepts

Some tips on maximizing the output from a cogeneration plant, as well as for maintaining proper HRSG chemistry

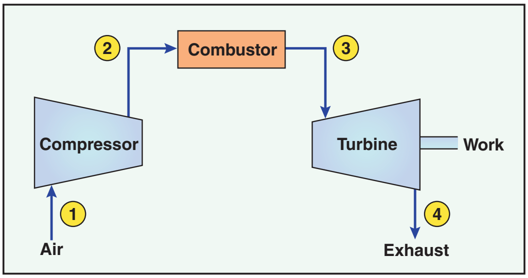


FIGURE 1. This schematic shows the basics of a combustion turbine that follow the four steps of the Brayton cycle (shown in Figure 2)

Brad Buecker
Kiewit Power Engineers

The continuing development of natural gas extraction from shale deposits, known simply as shale gas, is fueling the growth of simple- and combined-cycle power plants. Much coal-fired power is being replaced by these technologies. In addition, though, simple- and combined-cycle units are well suited for power and steam production at industrial facilities.

The combustion turbine

Back in the heyday of large powerplant construction, primarily from the 1950s through the 1970s, the most popular technologies were complex coal-fired or nuclear facilities, with hydro power providing much of the remainder. Concerns regarding nuclear safety, global climate change, and quite frankly, cost, have led to movement away from large steam generators to smaller units, and in many cases decentralized power. This includes chemical process industries (CPI) and other manufacturing facilities that produce process steam and at least some of their own power, rather than relying solely on electricity from utilities.

The core of many modern systems is the combustion turbine (CT), a simplified outline of which is shown in Figure 1.

A CT operates similarly to a jet engine via the following steps, which are part of a fundamental thermodynamic cycle, the Brayton Cycle (Figure 2).

1. Inlet air is compressed and injected into the turbine. The compressor is attached to the turbine shaft, and thus the compressor and turbine rotate in unison
2. Fuel, typically natural gas but occasionally fuel oil, is injected and ignited in the compressed air stream
3. The expanding gas drives the turbine
4. Hot exhaust, at 850°F or higher, exits the turbine

Like other energy-producing devices, most CTs are equipped with auxiliary features to improve efficiency, and we will examine some of these later in this article. For the time being, key points of a combustion turbine include very fast start times, low capital cost as compared to coal or nuclear, simplicity of fuel feed and minimal operations and maintenance issues. These benefits are quite important in the power industry, especially the fast start times during peak power periods when demand skyrockets. However, for industrial processes with continuous steam and electrical requirements, steady and efficient operation is required. Enter the combined-cycle plant.

The combined-cycle

The downside to simple-cycle operation is that the turbines are only about 35% efficient. Much energy escapes with the turbine exhaust. This

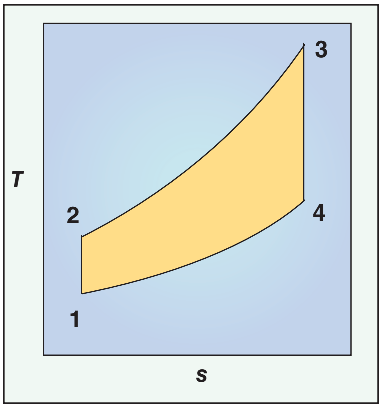


FIGURE 2. An ideal Brayton cycle is shown on this temperature versus entropy plot

is where the combined-cycle design, of which many readers are undoubtedly familiar, shines forth.

With combined-cycle, a heat recovery steam generator (HRSG) is placed at the exhaust of the combustion turbine or turbines to utilize the exhaust heat for steam production. While many HRSG designs are available, the most common is the multi-pressure, drum-type unit as depicted in Figure 3.

In this particular design, the condensate is split between the circuits, with a relatively small flow to the low-pressure (LP) steam network and the bulk of the flow to the intermediate-pressure (IP) and high-pressure (HP) circuits. Steam extraction may be

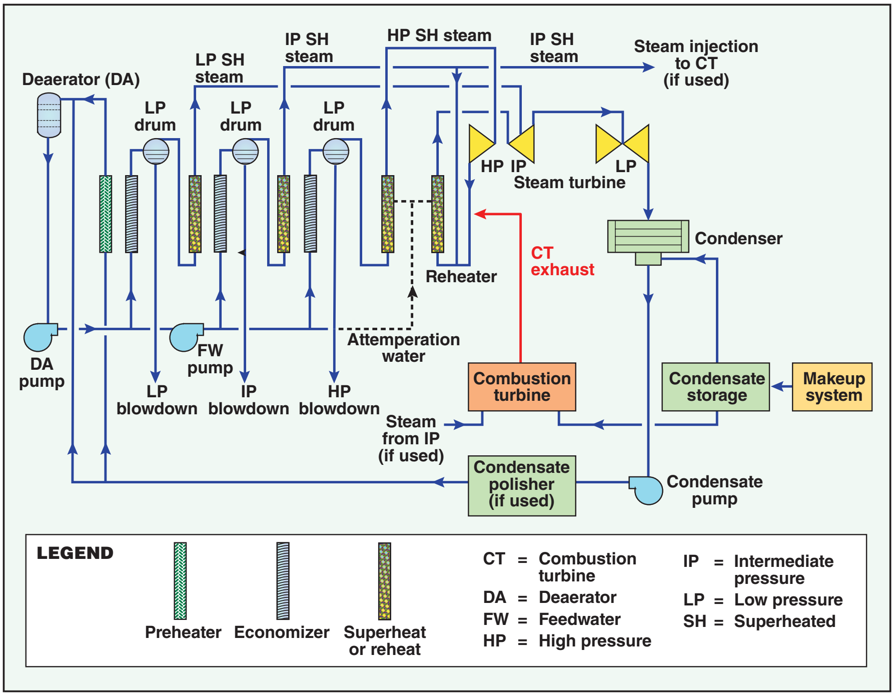
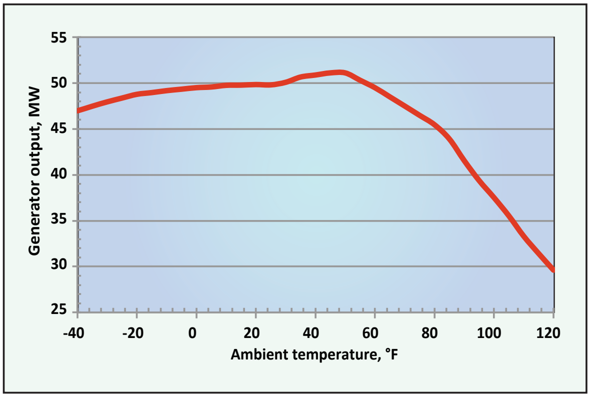


FIGURE 3. The most common type of HRSG is the multi-pressure drum type. This schematic is for a three-pressure HRSG

FIGURE 4. This graph shows the degradation of aeroderivative combustion-turbine efficiency with increasing ambient air temperature above 48°F



taken from any of the circuits, or, as is most efficient, from a non-condensing turbine. A less complex scenario that may be better for cogeneration applications is a combustion turbine with a single-pressure HRSG perhaps without a steam turbine, where the HRSG operation is less complex than with multi-pressure units. For example, a colleague and I recently prepared the water balance for a proposed power project in which one scenario called for two combined-cycle units, one with a CT and a three-pressure HRSG, and the other with a CT and a single-pressure HRSG that partly supplies steam for power augmentation. This example illustrates the flexibility that is possible with combined-cycle generation.

Net efficiencies of combined-cycle units for power production have closed in on 60%, while up to 80% efficiency is possible for co-generation. However, capacity and efficiency are greatly influenced by inlet air conditions — temperature foremost — but also humidity.

Maximizing capacity output

Figure 4 illustrates what happens to aeroderivative combustion-turbine capacity with increasing ambient air temperatures. Similar effects occur in larger frame units, albeit without a subcooling loss of efficiency.

To this point in time, the two most common methods for turbine inlet-air cooling have been mechanical chilling or water-fed evaporative cooling. With the former, parasitic power load may be in the 1 to 3 MW range. Evaporative coolers on the other hand, as their name implies, chill inlet air by evaporation of water spread upon an inlet media. Water requirements for these coolers may range from 30 to 80 gal/min. Also, evaporative coolers can only provide chilling down to a fixed approach to wet-bulb temperature.

A third method is gaining interest, absorption refrigeration turbine inlet air conditioning (ARCTIC), which mitigates or eliminates both parasitic power consumption and water requirement issues, while offering precise control over inlet air temperatures. Such control can be quite important for plants based in hot or humid environments. ARCTIC technology uses the extremely well-known process of closed-cycle, ammonia-based absorption refrigeration, with turbine exhaust heat as the energy source.

Figure 5 outlines the general flow schematic for ARCTIC technology. The process relies upon classic thermodynamic refrigeration. The turbine exhaust warms an aqueous ammonia solution in heat exchanger coils (HRVG on the diagram) located within the exhaust-gas path. The ammonia is then separated in the rectifier to produce a nearly pure vapor, which is condensed, reduced in pressure, and then allowed to expand within turbine inlet air-cooling coils (TIAC) located in the inlet air stream. The pure ammonia discharge from the cooling coils is blended with the aqueous ammonia bottoms product from the rectifier (a process that requires additional heat exchangers due to the exothermic reaction), and is re-pressurized for return to the HRVG. Thus, the process is a closed loop, with all equipment but the TIAC and HRVG coils located on a single skid.

A standard skid-mounted cooling unit can provide over 2,000 tons of chilling at 220 kW of auxiliary load.

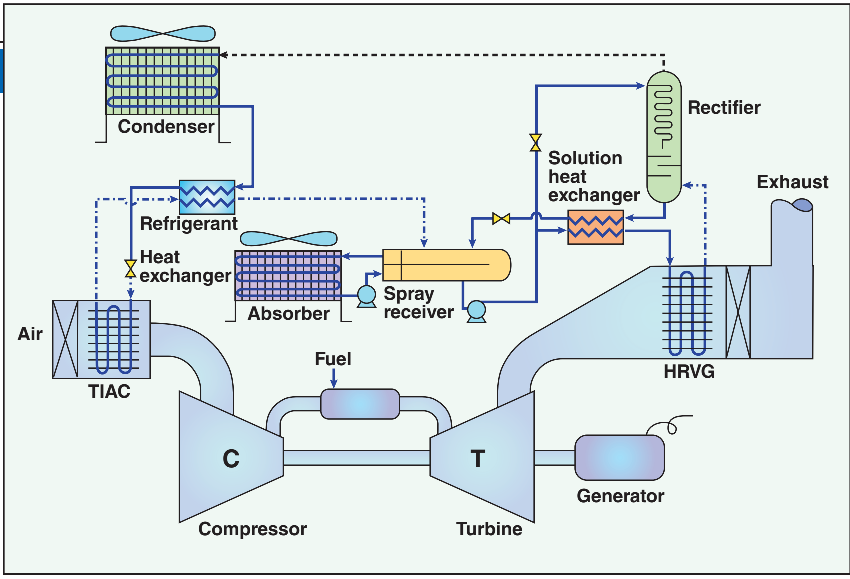


FIGURE 5. This generic flow diagram shows the operation of the ARCTIC system

The parasitic power load for a mechanical compressor to provide similar chilling ranges from 1.6 to 3.2 MW. During one summer day in 2011 at a plant in the southwestern U.S., an ARCTIC system maintained an inlet air temperature of 48°F (5°C) when the ambient temperature was 107°F (42°C). An added feature of this process, and one that is gaining interest in arid climates, is that cooling coils may be placed downstream of the HRVG to recover water from the fluegas. As is well known, the combustion process, and particularly that from gas turbines, produces a significant quantity of water due to the reaction of hydrogen in the methane fuel with oxygen.

$$CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$$

Recovery of fluegas moisture can potentially turn some plants from fresh water consumers to water producers. As an example, consider a combustion turbine fired with natural gas at a fuel flowrate of 87,000 lb/h. We will use three simplifying assumptions:

- Natural gas composition is 100% methane (CH_4)
- Complete combustion is achieved in the turbine
- The process recovers 100% of the water produced by combustion

Stoichiometric calculations show a water recovery rate of 390 gal/min. Given that natural gas is typically a very clean fuel, the recovered water could easily be returned to the inlet of the plant makeup-water system or to other processes. An obviously important issue with fluegas water recovery is the effect that chilling would have on the buoyancy of the fluegas stream, and in turn, how this might influence fan design and air-permitting issues.

In addition to inlet air cooling, other techniques are available for maximizing power during peak-load conditions. One method, whose primary purpose is to control of oxides of nitrogen (NOx) formation, is injection of demineralized water into the turbine. The demineralized water lowers the combustion temperature, which is the driving force for NOx formation. But, the water adds mass to the combustion gases and improves capacity, albeit at a bit of an efficiency penalty. Another option that I have seen in a few recent power applications is steam injection to the combustion turbine. This process is primarily utilized for power augmentation (PAG) when an extra few megawatts are needed from the plant. One drawback to PAG and water injection for NOx control is that extra makeup water is necessary to produce the fluids for turbine injection. This requirement can increase the size of makeup-water treatment equipment or demineralized water storage capacity. The reader will note that I have emphasized demineralized water for these applications, as anything of lower quality injected into the combustion turbine would cause fouling and corrosion.

Don't neglect HRSG chemistry

In 2008, I discussed the importance of maintaining proper chemistry in CPI steam generators during regular operation and times of shutdown [*2*]. These principles also apply to heat recovery steam generators. The following list sums up many of the most important details of steam generator chemistry that have evolved over the past decades, and particularly within the past 20 years:

- Even seemingly minor contaminant in-leakage to condensates can result in major problems, most notably steam-generator tube failures that cause unit shutdowns
- For systems with water-cooled steam condensers, the condenser can be the most dramatic source of contaminant in-leakage. Impurity introduction via a condenser-tube leak has been known to cause boiler tube failures within weeks, and sometimes even days or hours
- Too often at CPI or other manufacturing plants, insufficient attention is given to condensate return from process equipment to the steam generator. The impurities introduced by contaminated condensate may be as harmful as those introduced by condenser tube leaks. Condensate recovery can be quite cost effective, but only if it is properly treated to prevent boiler contamination. Treatment may include filtration, ion exchange, or other techniques
- Unless the condensate/feedwater system of a high-pressure steam generator contains copper alloys, the use of oxygen scavengers is highly discouraged. These are now known to propagate flow-accelerated corrosion (FAC) in feedwater systems, economizers, and the low-pressure evaporator of HRSGs, among other locations (for more, see *Chem. Eng.*; March 2013, pp. 38–40). FAC-induced failures have caused a number of fatalities at power plants in the last 25 years. The same phenomenon will occur in CPI steam generators
- The most common treatment for IP and HP circuits in HRSGs is the program known as phosphate continuum, and most commonly at the low end of a 1 to 10 parts-per-million (ppm) phosphate range. This program was developed by the Electric Power Research Insti-

tute in response to problems with earlier phosphate programs, and calls for the use of only tri-sodium phosphate with perhaps a slight amount of caustic at startup. Alternatives include straight caustic treatment (at less than 1 ppm free sodium hydroxide) and all-volatile treatment, where only the ammonia or amine utilized for feedwater pH control provides the treatment for the steam-generator circuits

- For all of the above items, accurate and reliable online monitoring systems need to be in place to detect upsets. Operators of the steam generator must be properly trained to recognize off-specification conditions
- Improper boiler layup can cause excessive corrosion that not only damages components but generates massive amounts of corrosion products that travel to the steam generator and deposit on boiler tube surfaces. Under-deposit corrosion via several possible mechanisms is a primary cause of boiler-tube failures. Ideally, for short-term shutdowns where water is left in the steam generator, nitrogen blanketing can be utilized to protect the system from air in-leakage. If nitrogen blanketing is not an option, then other wet lay-up procedures should be established. Also, very reliable technologies now exist to remove dissolved oxygen from steam generator fill water. This is an important issue, particularly for units that face frequent shutdowns and layups, and where demineralized water and condensate are stored in atmospherically vented tanks [*3*]

Readers who are interested in learning more about steam-generator makeup-water treatment, chemistry, and layup issues are encouraged to attend the annual Electric Utility Chemistry Workshop (www.conferences.illinois.edu/eucw). ■

Edited by Gerald Ondrey

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The Direct Integration Method: A Best Practice for Relief Valve Sizing

The approach described here is easier to use, and provides more-accurate results, compared to leading valve-sizing methodologies

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BS&B Safety Systems

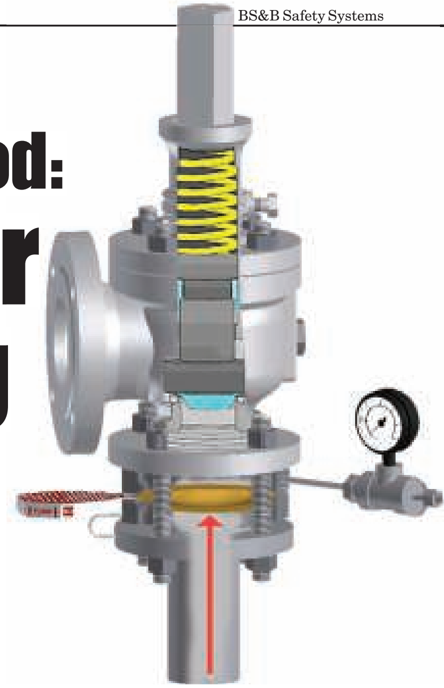

FIGURE 1. Today, with the help of spreadsheet programs and simulators, the once-cumbersome Direct Integration Method is easier than ever to use to size relief valves

What if someone were to tell you that there is one method available for sizing relief valves that applies to virtually every situation, including two-phase flow and supercritical fluids? And what if they told you that method is more accurate and easier to use than traditional methods or formulas? As it turns out, both of these statements are true. The approach described here — the Direct Integration Method — involves numerical integration of the isentropic nozzle equation [*1*].

From as early as 2005, the "method of choice" for determining the flow through a relief valve has been the Direct Integration Method [2]. API 520 has also sanctioned this method due to its general applicability to any situation where the fluid is homogeneous [*1*]. However, because this method is perceived to be difficult or time consuming, many engineers continue to opt for older, simplified methods, even though such methods can produce less-accurate results. For instance, without careful analysis, using the traditional gas-phase equation near a fluid's critical point can yield an undersized valve [*3*].

Fortunately, thanks to the widespread availability of process simulators and spreadsheet software, numerical integration of the isentropic nozzle equation is now easier, faster, and more accurate than other methods for determining the mass flux through a relief valve. This article discusses the use of process simulators to simplify the numerical integration method, and describes the advantages of numerical integration over other methods that may be used to calculate the required relief valve area.

Calculation methods

Isentropic Converging Nozzle Equation. The calculation of the theoretical mass flux for homogeneous fluids through a relief valve is generally accepted to be modeled based on the isentropic converging nozzle. The isentropic nozzle equation is developed from the Bernoulli equation by assuming that the flow is adiabatic and frictionless [*4*].

$$G_0 = \rho\left(-2\int_{P_0}^{P_n}\frac{dP}{\rho}\right)_{S,Max}^{1/2} \quad (1)$$

The required nozzle area of the relief valve is calculated using Equation (2).

$$A = \frac{W}{K_d G_0} \quad (2)$$

To use Equation (1), the fluid density must be known as a function of pressure at constant entropy over the pressure range encountered in the nozzle. To solve the integral analytically, an equation of state needs to be available for the fluid at constant entropy. However, for many fluids, such an equation is not available for density as a function of pressure. To overcome this limitation, various simplifying assumptions were traditionally made to allow the integral to be solved analytically, rather than by performing a numerical integration.

For instance, for non-flashing liquids, the density is assumed to be constant, and the integral is easily solved. The traditional vapor-sizing equation is obtained by assuming the vapor is an ideal gas with a constant heat capacity [*5*]. However, the assumptions required by these methods may introduce large errors under some conditions. In contrast, the Direct Integration Method has been shown to produce more-accurate results.

Direct Integration Method. The Direct Integration Method uses a numerical method to evaluate the integral in the isentropic nozzle equation [*2*]. API 520 proposes the use of the Trapezoidal Rule, shown below, to calculate the integral:

NOMENCLATURE[1]

G_0	Mass flux, lb/h • in.^{2}	P_n	Nozzle exit pressure, psi	K_d	Discharge coefficient, unitless
ρ	Density, lb/ft^3	A	Orifice area, in.^{2}	P_i	Pressure at stage i, psi
P_0	Relieving pressure, psi	W	Relieving mass rate, lb/h	ρ_i	Density at stage i, lb/ft^3

1. Unit conversion may be required, depending on the units selected.

$$G_0 \cong \rho\left(-2\sum_{P_0}^{P_n}\frac{2\left(P_{i+1}-P_i\right)}{\rho_{i+1}+\rho_i}\right)^{1/2} \qquad (\mathbf{3})$$

The method is performed by using a process simulator to generate data points for the fluid density at various pressures, utilizing an isentropic flash routine over a pressure range from the relieving pressure to the exit pressure. The simulation data are used to determine the theoretical mass flux at each point.

Using Equation (3), the maximum mass flux is determined by calculating the mass flux over incrementally larger pressure ranges, beginning at the relieving pressure, and observing where a maximum flux is reached. If the maximum occurs at the relief-valve exit pressure (built-up backpressure), then the flow is not choked. Generally accurate results can be obtained with pressure increments as large as 1 psi, but smaller step sizes can be specified if desired [*2*]. Once the mass flux is determined, the required relief valve orifice area* can be determined from Equation (2).

The value of the discharge coefficient, K_d, depends on the phase of the fluid and varies by the manufacturer of the relief valve. The discharge coefficient corrects for the difference between the theoretical flow and the actual flow through the nozzle. This value is determined empirically for liquid and vapor and reported by vendors for each make and model of relief valve. If vendor data are not available, an initial guess of 0.975 for gases, or 0.65 for liquids can be used [*1*].

For two-phase flow, the liquid-discharge coefficient should be used if flow in the valve is not choked and the maximum mass flux will occur at the relief-valve exit pressure. If the flow is choked, then the gas-discharge coefficient should be used and the maximum mass flux will occur at some pressure above the relief-valve exit pressure. This is called the choked pressure [*6*]

* While relief valves are designed with a nozzle, the area at the end of the nozzle is commonly referred to as the "orifice area".

Implementation

It is possible to fully automate the Direct Integration Method using a spreadsheet program (such as Microsoft Excel 2010) and a process simulator (such as AspenTech HYSYS 7.2) [*7*]. Users can automate the process to the point where all they would need to do is simply hit a button in the spreadsheet program and the numerical integration will be performed on an existing stream in the simulator using a VBA (Visual Basic for Applications) program.

First, the spreadsheet is set up to accept the pressure and density data for the numerical integration points. The inlet and outlet pressure points, pressure step size, and name of relief stream in the simulator are placed into specific cells in the spreadsheet, which are referenced in the VBA code. The VBA code instructs the simulator to create a new, ideal expander process block and associated streams in the simulator. The code then iterates across the pressure range and modifies the pressure of the expander product stream and automatically exports the pressure and density data to the Excel spreadsheet.

For each data point in the spreadsheet, the summand, cumulative sum, and mass flux are calculated using Equation (3) with typical spreadsheet formulas. When a maximum mass flux is reached, the spreadsheet uses this maximum flux value to calculate an orifice size, given the relieving mass rate and coefficients. Alternatively, the data can be collected using the "databook" feature in the simulator and copied into the spreadsheet using a simple copy-and-paste operation.

Two-phase relief scenarios

The existing single-phase vapor and non-flashing liquid methods are relatively easy to calculate and the resulting predictions are fairly accurate at conditions well away from the critical pressure. However, two-phase models are more difficult to implement. Existing two-phase flow models approximate the pressure-density relationship of the fluid in order to calculate the integral in Equation (3).

One of the simplest models, the Omega Method, assumes a linear pressure-density relationship, with the omega parameter (ω) representing the slope of the pressure-density curve. An analytical solution to the isentropic nozzle equation was developed using the omega parameter to solve the integral [*8*].

The TPHEM Method uses three pressure-density points to define coefficients for an empirical equation of state "model" [*9*]. The empirical equation is then used to evaluate the integral numerically. Pressure-density data for these models are often provided by a process simulator. If a simulator is available, then it is much simpler to use the Direct Integration Method.

The Direct Integration Method is fundamentally different from the other methods described here because it does not generate an explicit equation-of-state model to relate pressure and density. Instead, pressure and density data are generated using the full thermodynamic models available in the selected process simulator, and these data are then used to solve the integral numerically. Since there is no reliance on a curve-fit pressure-density model, the Direct Integration Method is more exact and reliable, assuming the simulator's thermodynamic model is accurate. Specifically, there is no chance for inaccuracies associated with the fluid equation of state "model" propagating through the rest of the calculations resulting in inaccurate mass flux estimations and ultimately an inappropriate relief-valve area [*8, 9, 10*].

Note that the Direct Integration Method assumes that the two-phase fluid is homogeneous, and that the fluid is in mechanical and thermodynamic phase equilibrium. The homogeneous assumption is valid for most two-phase reliefs due to high velocity in the nozzle, which promotes mixing


GE/Consolidated and Allied Valve

FIGURE 2. The Direct Integration Method is not only easy to use, but provides more accurate results when sizing pressure relief valves, since this approach does not rely on a potentially sensitive equation of state model

[2]. The mechanical equilibrium assumption is valid for flashing flows [2]. The thermodynamic equilibrium assumption is valid for nozzles with a length longer than 10 cm [4]. Most standard relief valves have a nozzle that is slightly longer than this [11].

Pros and cons

Advantages of this Method. The Direct Integration Method is not bound by the same constraints as many other models or methods. Using this approach, the same method can be used whether the flow is choked or not choked, flashing or not flashing, single or two-phase, close or far from the critical point, subcooled or supercritical. The only assumptions required for the Direct Integration Method are that flow through the relief valve is isentropic, homogeneous, and in thermodynamic and mechanical equilibrium, although it is possible to adjust the method to account for mechanical non-equilibrium or slip [6].

Although most other methods give unsatisfactory results near the thermodynamic critical point, the Direct Integration Method continues to function properly [12]. Additionally, many other concerns that come up when using relief-valve model equations, such as determining the heat capacity ratio or isentropic expansion coefficients, are no longer relevant since they are inherent to the simulator itself [3].

Downsides to this Method. The Direct Integration Method can produce overly conservative results in a couple of circumstances, which can lead to under-prediction of the mass flux and selection of an oversized valve. This appears to be an issue only when the fluid is in two-phase frozen flow (no flashing), or the relief valve has a short nozzle and there is flashing flow [2].

This potential limitation can be compensated for in both situations by applying a slip factor. However, at this time, there is insufficient literature available to provide accurate guidance on the value of a slip factor. The accuracy of the calculation is also limited by the accuracy of the physical property data in the simulator.

Closing remarks

Using a spreadsheet to import data from a simulator and to calculate the summation over a range of pressures is extremely easy and straightforward. One simply needs to simulate the relieving stream and perform a flash operation at each pressure and capture the required data. Not only is the Numerical Integration Method much simpler than the alternatives for two-phase flow, but it is also more accurate, since it does not rely on a potentially sensitive equation-of-state model. There is no need for a model because physical property data are generated for each data point directly from simulation. In addition, the Numerical Integration Method can be used for single-phase flow and choked or not-choked conditions. This versatility and ease of calculation makes Numerical Integration the obvious choice for any relief valve calculation where physical property data are available in a process simulator. ■

Edited by Suzanne Shelley

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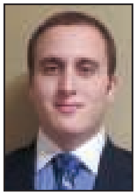
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Safety-Instrumented Systems: Focus on Measurement Diagnostics

WirelessHART and other new approaches help operators to achieve the needed safety at the lowest lifecycle cost

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This article provides guidance to the designer and operator of a safety-instrumented system (SIS), with an emphasis on conducting measurements. The use of best practices to ensure the strength of the overall system design, and the use of instrument diversity to combat common causes of failure, are covered briefly. Greater detail are provided on the use of new technologies — in particular, new smart-transmitter diagnostics and digital protocols, including wireless options.

Background

Best practice design of SIS has evolved over the past decade, prompted by the widespread adoption of the ANSI/ISA-S84.01-2004 Standard, which is itself based on IEC 61511. In their first article on this subject [*1*], the authors described how the new standard encourages a user to change from purely qualitative risk assessment to quantitative risk analysis. One benefit of the qualitative approach is that it is prescriptive and hence simpler to apply — the user is told what to do to achieve safety.

One potential downside is that the qualitative approach is designed to produce conservative results, which can result in over-design. The quantitative approach is more flexible — the user can use whatever approach minimizes lifecycle cost while still achieving the desired risk reduction. The designer must substitute quantitative data for qualitative descriptors; for example, a dangerous event previously described as "very likely" would now be assigned a probability of "0.1 events per year." That article guided users through the process of finding these numbers. This was found to be a particular challenge for field devices such as sensors. What is the risk that a given transmitter might falsely report a safe condition? While suppliers can provide safety statistics that are "certified" by third parties, those data are typically derived from white papers or laboratory analysis. Is the actual risk greater in a "real world" installation? Might the risk be significantly different for two identical devices that are in different installations? How can the user quantify these "installed" risks?

The focus of a follow-up article by the authors [*2*] was on strategies for minimizing, rather than quantifying, identified risks. Since users commonly employ redundant sensors in critical applications, special attention was paid to identifying "common causes,"which can impact both sensors in a redundant system. For example, if a user has identified that impulse line plugging in a given installation might cause the pressure transmitter to falsely report a safe condition, rather than trying to quantify that risk, a better approach is to make the risk so small that it no longer has a material impact. The user was advised to employ a strategy that aimed to improve strength, diversity and diagnostics:

- *Strength* — Change the installation by shortening and widening the impulse lines so they don't plug
- *Diversity* — Rather than using a second (redundant) differential pressure (dP) transmitter on a given orifice plate, use a vortex flowmeter. Choose a vortex design that is immune to the common cause of plugging
- *Diagnostics* — Select a transmitter that can detect that its lines are plugged

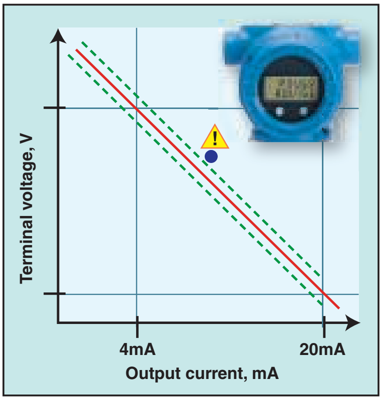

FIGURE 1. The transmitter characterizes the "normal" relationship between current and voltage at commissioning, then alerts the user to changes during operation, which might indicate a problem

Best practices

To maximize the strength of an overall SIS design users should employ the same best practices that apply to any process measurement application, whether used for safety or for basic process control. Note that best practices tend to be specific to a given technology, and evolve over time with technological advances. Some examples of best practices with particular relevance to safety applications include ensuring that:

- *The measurement uncertainty is smaller than the safety margin*. For example, if a process is operated within 5% of where it becomes dangerous, the measurement must be much better than ±5%. This sounds obvious, but is often not the case. While tools and methodologies exist [*3*] to quantify measurement uncertainty, many users do not routinely employ these

FIGURE 2. The Statistical Process Monitoring (SPM) screen shows that while the process' mean has not changed, its high frequency variability has increased

- *The thermowell is properly designed for the application*. Poor thermowell design has been cited in the 1995 failure of the Monju fast-breeder reactor, which caused a spill of radioactive sodium [*4*]. Although no radiation was leaked to the environment, the reactor was not restarted until September 2010 — 15 years after the incident. New standards, such as ASME PTC 19.3-2010, significantly improve the reliability of wake-frequency calculations and minimize the risk of thermowell failure due to flow-induced vibration and transient effects
- *The measurement is fast enough to detect the hazard*. Speed of measurement response is affected by the device, the installation and application conditions. Again, few users go through the effort to quantify response time in a given application except when response time is known to be critical (for instance, for compressor anti-surge [*5*]). Similar effects are seen with temperature measurements, although there the sensor and thermowell dominate the overall response time
- *Measure mass flow of gas and steam*. Use a multivariable flowmeter that compensates for changing density, or measure mass flow directly using a Coriolis flowmeter
- *In a top-down level measurement, significant changes in the vapor space are dynamically compensated*. For example, a radar level meter used for drum level control must compensate for changing density of the steam in the boiler drum
- *Proper materials are selected*. This is key to avoid effects such as hydrogen permeation, or stress corrosion cracking in environments with high vibration or pressure cycling

To the greatest extent possible, the user should maximize the consistency of devices and practices between the measurements that are used for the safety system and the basic control system. While the use of a new, unfamiliar device or practice solely for safety applications may yield some benefit in theory, in practice the user is more likely to make an error during design or maintenance, due to lack of familiarity [*6*]. In addition, failure statistics gathered from basic process control installations can be leveraged for safety system design if those installations are consistent.

Employ diversity

Common causes of failure usually dominate safety risk in installations with redundant transmitters. Consider the case of a pressure or displacement-style level transmitter used to ensure that a vessel does not overflow. For these installations there are two main risks:

- *The transmitter electronics will fail dangerously and provide the wrong output*. Consider a case in which this probability of failure on demand (PFD) is 0.05, which means that if 20 transmitters are called on to shut the dangerous process down, one will fail to do so (Note: There are no units for PFD because it represents the probability that a safety system or component will fail to respond to a demand)
- *The density of the fluid in the vessel will change, so the output of the transmitter does not reflect the true level*. It may be reasonable to assume a PFD of 0.04 for this risk

$PFD_{Total}=PFD_{Transmitter}+PFD_{Density}=$
$0.05 + 0.04 = 0.09$

Making the device redundant:

$PFD_{Total} = (PFD_{Transmitter})^2 + PFD_{Density}$
$=(0.05)^2 + 0.04 \approx 0.04$

This is of course a simplified calculation, but it should be apparent that continuing to add redundancy — for instance, making the transmitter triply redundant — will yield little benefit, since the common cause of density variation will continue to dominate total system risk. Instead, the user should install a different (diverse) technology that is not affected by density variation.

Rather than trying to find a best practice to apply to a given technology, the user should consult a technology selection guide, widely available from technical societies such as the International Soc. of Automation (ISA). A truly objective guide avoids characterizing any given technology as "better or worse," but, instead it identifies relative strengths and weaknesses of different technologies. For example, a level selection guide [7] should explain that radar-based level-measurement devices — both contacting and non-contacting — will be immune to variation in fluid density.

Such a description is not to suggest that radar is necessarily "better" than a level-monitoring device based on differential pressure (dP). It just suggests that radar is resistant to the identified common cause, and thus should be suitable as a backup to the chosen primary technology. Similarly, an advantage of dP-level measurement over radar-based options is that the former

Exida

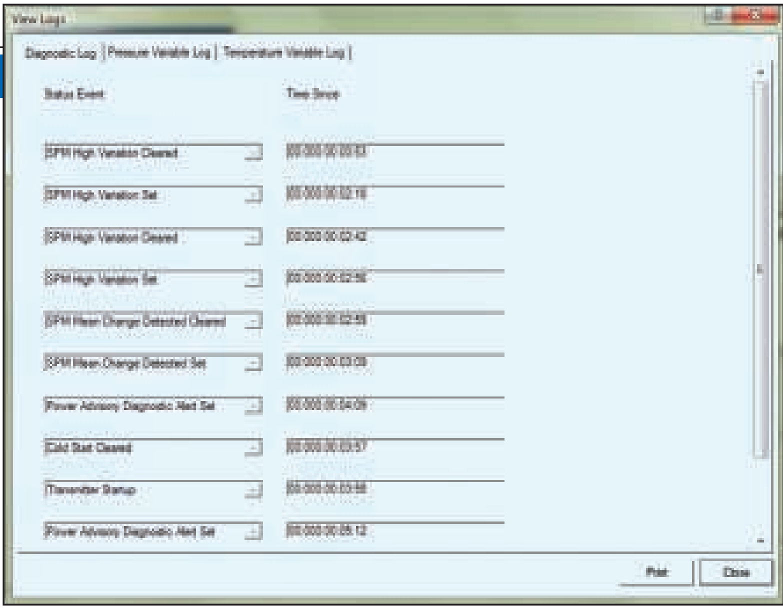

FIGURE 3. After an upset or near-miss, the user can review the diagnostic logs of attached transmitters to see if anything unusual occurred just prior to the upset. In this case, high variability was observed

can ignore the effects of vessel internals that reduce the signal-to-noise ratio. For instance, reflections from agitators or baffles can increase noise and foam, or vapor can absorb radar energy and reduce signal strength. In an installation that relies on radar as the primary measurement technique, if the user identifies internal vessel effects as a significant common cause risk, then dP-level monitoring devices would be a suitable backup option.

Complementary strengths and weaknesses can be found in other measurement applications. For instance, in temperature measurement, RTDs are accurate and stable, while thermocouples are physically robust. In a waste-gas flow application, a Coriolis flowmeter provides higher turndown and is less affected by changing gas composition, while a dP-flowmeter is more stable in low gas pressures and introduces less permanent pressure loss. These guidelines evolve over time. Modern Coriolis meters can be sized to provide acceptable accuracy at lower pressure drops compared to older designs, and modern dP-flowmeters now offer higher overall turndown. For this reason, the user should use the most up-to-date technology selection guide.

Diagnostics improve safety

The most direct way for a user to obtain a "process diagnostic" is to add a measurement, or upgrade a switch to a transmitter. While a switch can freeze in position, a transmitter that fails in position can be detected by an alert operator. Even where the new transmitter is not connected to the safety system, a deviation between the new transmitter and the existing safety system transmitter can alert the user to a problem. Conversely, agreement between the two transmitters can help the user to justify extending the proof test interval.

Internal transmitter diagnostics became available with the earliest smart transmitters, and have become more sophisticated over time [*8*]. They can detect internal faults — for example, the microprocessor can discover that its non-volatile memory has become corrupted by an electrical or magnetic disturbance, or that an internal totalizer or cyclic counter has stopped updating. The safety benefit of these internal diagnostics are accounted for in the Failure Modes and Effects Diagnostic Analysis (FMEDA) safety data, which are provided by the supplier, and explains why newer transmitters provide superior safety statistics compared to older devices.

Some diagnostics require user activation. For example, diagnostics are available to detect power supply problems. A loop might be unable to reach an output higher than 18 mA because the power supply is browning out or cannot keep up with load from all the devices in the loop. Or, water might have leaked into the housing, causing an output shift. As shown in Figure 1, newer transmitters characterize the relationship between current draw and transmitter terminal voltage at commissioning, and alert the user during subsequent operation to changes that would indicate a problem. As with internal diagnostics, the safety benefit of power diagnostics are accounted for in the supplier's safety data, with further improved transmitter safety statistics. An additional benefit of this diagnostic is that the user can eliminate the need for periodic manual proof testing of the loop's high- and low-alarm limits.

Temperature transmitters are configurable to accept either thermocouple or resistance temperature detection (RTD) inputs. The transmitter obtains temperature from changes in voltage of the thermocouple, or changes in resistance of the RTD. This means that a transmitter connected to a thermocouple has unused resistance circuitry, which it can use to characterize "normal" resistance of the thermocouple and associated wiring, and alert the user to changes that indicate temperature-measurement errors and impending failure. Again, this improves safety, and can extend the schedule for temperature-sensor proof testing.

The most advanced smart transmitters contain microprocessors that read the sensor 20 times or more per second. While the logic solver or control system can only handle an "average" signal two or three times per second, the high-speed, unfiltered signal is useful for characterizing high-frequency process variability within that average.

The meaning of a given increase or decrease in variability depends upon the process. For a pressure transmitter, a decrease in variability could indicate plugging of the sensing line or coating of the diaphragm seal. In a flow application, an increase in variability without a corresponding increase in mean — shown in Figure 2 — could signal that the liquid flow is becoming entrained with gas, or that the steam flow is becoming "wet" with liquid water. Either can cause measurement error, and also damage to mechanical components. In most tem-

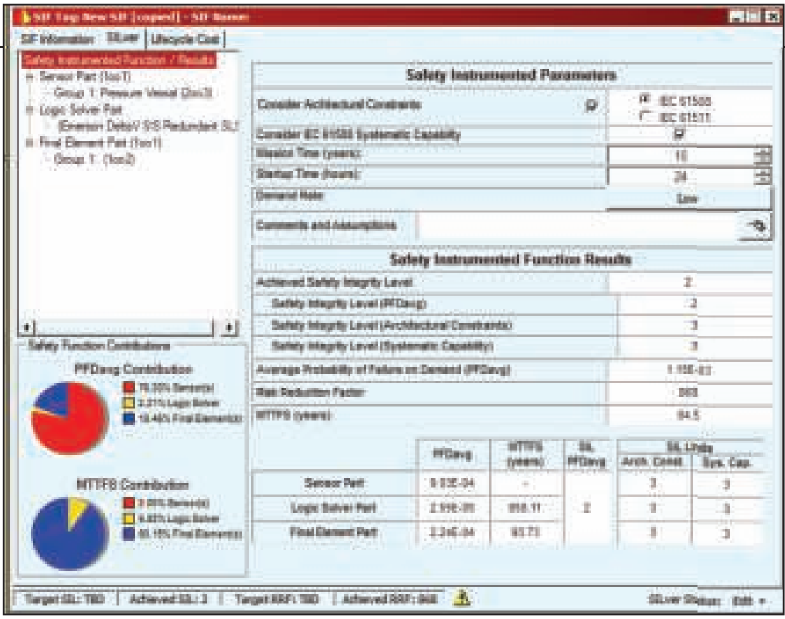

FIGURE 4. With a risk of line plugging, the sensor dominates system risk at 78% of the total PFD, and limits the system to a risk reduction factor of 868 (SIL-2)

perature and level applications, the rate of change is limited by the physics of the process. A large, sudden change is not physically possible, and usually reflects some external influence like electrical noise. Rather than damping the signal, which slows response time to a genuine process upset, the smart transmitter will ignore the spike or dropout for a scan or two, holding the last value for the logic solver.

It's not always obvious in advance how a change in variability should be interpreted. In practice, the user lets the diagnostics "learn" the process. If the process unit is upset — which may or may not engage the safety system — maintenance can later review the date-and-time stamped historical logs (essentially, the transmitter's non-volatile "black box") to see if any of the transmitters connected to the unit had observed a significant change in variability prior to the upset. As shown in Figure 3, review of the log might reveal that several minutes prior to the furnace flame blowout, pressure variability increased to "*x%*". Process engineering would confirm that this correlation makes sense — and is not just a coincidence — so the "furnace pressure variability greater than *x%*" warning could be used to prevent future blowouts.

To take credit for these process diagnostics, the user quantifies, in each application, the likelihood that the failure will occur, and the likelihood that the diagnostic will detect it. Values are derived from operating experience, in the same or similar applications. Until the user has gained experience with a given diagnostic, the user should operate the diagnostic in "open loop", so that a diagnostic alert causes the operator to investigate further. That means that the diagnostic coverage must be de-rated to account for operator response time.

Once the user gains confidence that the diagnostic does not cause false alarms, the diagnostic can be turned "closed loop", so that the transmitter output goes to the failsafe position when the condition is detected. Even then, with redundant transmitters a single alarm should not cause shutdown, but should alert the operator to investigate further. Figure 4 shows a safety analysis for a transmitter in an application with a risk of line plugging. The sensor contributes 78% of the probability of failure on demand (PFD), limiting risk reduction factor (RRF) to 868. Figure 5 shows the same analysis if the user implements a plugged-line detection diagnostic and takes credit for 70% coverage. RRF improves to 2077. Implementation of the diagnostic therefore significantly improves safety – from Safety Integrity Level (SIL) 2 to SIL 3 with no added hardware or proof testing.

Diagnostics — benefits

The most severe diagnostic condition is a "failed" status, which means the transmitter output can no longer be trusted, even if it remains onscale between 4 and 20 mA. The device that detects this type of failure immediately provides an offscale output — higher than 20 mA or lower than 4 mA, configurable by the user— to advise the logic solver that the process is operating dangerously.

Properly implemented, measurement diagnostics provide additional benefits. First, the user can be alerted to lower levels of severity preceding "failed." A "maintenance required" status informs the user that the measurement, while probably correct now, will eventually become invalid — and cause a failure — unless the user performs some remedial action. Many failures develop gradually, so the user can be informed of an "impulse line starting to plug," a "thermowell starting to coat," or a "power supply starting to brown out." The "water in terminals" diagnostic signals small amounts of condensate that cause a measurement shift, but if ignored will eventually — in weeks or months — cause corrosion through the terminals and device failure. A downward trending signal-to-noise ratio for a radar level meter indicates that the antenna of the meter is gradually becoming coated by condensing fluids in the process. If ignored, the signal will eventually become unusable, and the transmitter will report a "failed" condition. The diagnostic allows the user to ignore the antenna until and unless alerted to the need for cleaning. This predictive maintenance has dual benefits — it eliminates routine inspections for reduced maintenance cost, and prevents future failures for higher uptime.

Failure diagnostics force a potentially dangerous process to shut down and so are valuable by themselves. Predictive maintenance diagnostics are most useful when promptly and clearly communicated to maintenance personnel, and logged in a historical record. This requires enabling technologies in addition to the device diagnostics themselves — asset management systems and digital communication protocols.

A centralized asset-management system (AMS) is useful for several

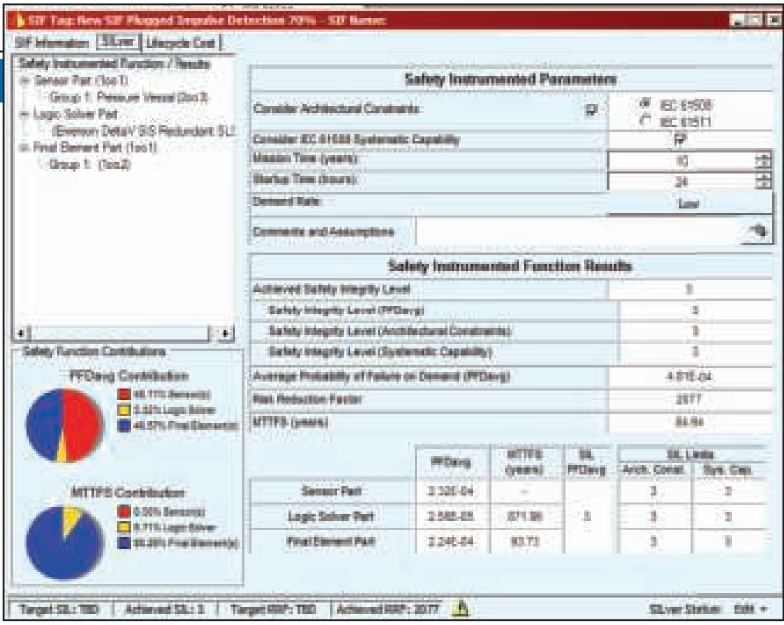

FIGURE 5. With a diagnostic that detects line plugging 70% of the time, sensor risk falls to 48% of the total PFD, allowing the system to achieve a risk reduction factor of 2,077 (SIL-3)

reasons. First, it provides early warning of impending failures in remote field devices, so maintenance can remedy the problem before it causes failure. Second, for devices that do fail, a detailed description of the failure and context-sensitive remedial action can speed trouble-shooting. Finally, regular use of the AMS helps to ensure consistency, for routine maintenance and proof tests and the collection of failure statistics.

It was explained earlier that a key challenge in safety system design is obtaining relevant data. The AMS automatically collects failure statistics for all connected devices, allowing the user to quantify the probability of specific failures under actual operating applications and conditions. So the user can better estimate risks of specific real-world interface failures, including those previously referenced (line plugging, RTD breakage, antenna coating and so on) This also applies to time-based conditions — if the as-found/as-left audit trail shows that a device did not require any maintenance during the last few proof tests, the user can better justify extending proof test intervals.

Digital communications

A digital communications protocol allows the user to communicate additional information from the transmitter, including diagnostic information, to the AMS, without additional wiring. Users tend to prefer open standards, which ensure access to competitive pricing and best-in-class technology, and minimize the risk of obsolescence. Although all-digital Fieldbus protocols such as Foundation Fieldbus and Profibus PA are widely used in basic process control, they have not seen adoption in process-industry safety systems beyond small demonstrations.

This is probably because the key benefit of the all-digital protocols — multi-drop capability to reduce wiring cost —is not realized in safety applications [*9*]. Combining process and safety applications, fieldbus transmitters account for a growing, but still small mix of new device installations. The vast and growing majority — nearly 75% of new transmitters shipped in 2011 — use HART [*10*].

HART is a "hybrid" protocol, communicating diagnostics and other information in a digital stream superimposed at 1,200 Hz on a 4-20-mA analog signal. Newer control systems and logic solvers can use both signals at the same time — the analog signal for control and safety, and the two-way digital HART information for configuration and diagnostics. An older logic solver can also use a HART transmitter, because it simply filters out and ignores the high frequency HART noise while using the analog signal. To obtain the information in the digital stream, many users have installed "HART multiplexers", which strip off the HART information and send it to a parallel AMS.

Not surprisingly, since it leverages existing supplier and user experience, the dominant protocol for wireless transmitters is WirelessHART, also referred to as IEC-62591 [*11*]. Like wired HART, WirelessHART allows users to add new transmitters for improved process visibility, plus provide access to complete diagnostic information from existing or new transmitters, from any supplier, for any control system or logic solver. Better, it minimizes cost and physical space, in both green and crowded brownfield installations, by eliminating the need for new infrastructure — wires, junction boxes, multi-conductor cables, conduit and wire trays, marshalling cabinets and logic solver input cards, racks and power supplies.

WirelessHART uses a self-organizing mesh (Figure 6). Each transmitter contains a smart radio frequency (RF) radio. While RF is a line-of-sight technology, it can work through some walls and gratings, and around smaller pipes and motors. In a mesh network, some devices can communicate directly with the gateway, and some devices can only see other devices. A data packet will "multi-hop" as necessary until it gets back to the gateway.

Once the system reaches sufficient density, the user can usually assume that any new device will be able to communicate with several other devices, so failure of any one device will not affect network reliability. The network is self-organizing, meaning that each device automatically forms multiple connections to the gateway, which reform dynamically as new devices and obstacles appear. This ensures high reliability with minimal engineering, and avoids costly site surveys.

As shown in Figure 7, the wireless signals are not used directly by the safety system. New or existing safety system transmitters connect into the logic solver via traditional HART/4-20-mA wiring. When the legacy logic solver does not support HART input, diagnostic information can be communicated in parallel to the AMS using smart antennas. Additional "process visibility" transmitters needed to improve availability, compliance and

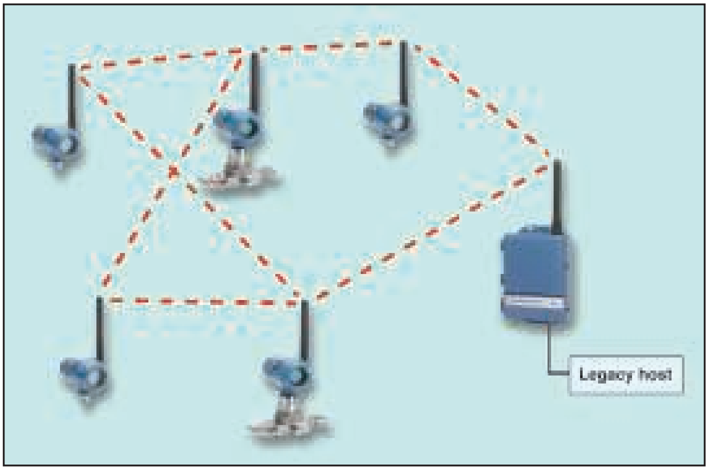

FIGURE 6. WirelessHART uses a self-organizing mesh. Data packets will take whatever path is necessary to get back to the host, providing reliability comparable to wired communications

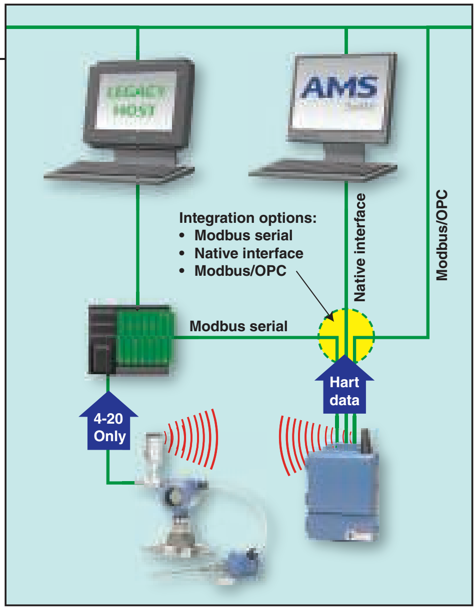

FIGURE 7. Process variables are communicated using traditional 4-20-mA connections to the logic solver, while diagnostic information travels via the WirelessHART to the maintenance terminal

efficiency communicate wirelessly to both the basic process control system and the AMS, and the user can choose to compare these new wireless transmitters to the wired safety-system transmitters to improve diagnostic coverage. Installation costs are minimized when the devices are battery powered, though the need to install and maintain devices in hazardous areas limits battery capacity. Although devices can be configured to communicate every second, with current battery and radio technology users are achieving 3- to 10-year battery lives with 4- to 30-second update rates.

Security is important because wireless data and devices can be accessed from outside the plant fence, bypassing the usual plant security. All data should be sent with encryption, so someone listening in will not be able to decode the message and steal the data. Related is authentication/verification — only valid devices, and not hackers, can gain access to the system. Finally, even the most secure design can be defeated by poor password/code management — human error. To add a new device to the network, the user manually inputs the network name and "join key" using the familiar HART handheld, but only the system-generated, encrypted rotating key is broadcast over the network.

Wrapping up

The safety system designer's first approach to minimize risk should be to improve strength through best practices. What cannot be eliminated should be avoided via a diverse technology that is resistant to common cause failures. What remains should be diagnosed, using a combination of new wired and wireless measurements, and device diagnostics. Diagnostics are available to detect problems in the device, wiring, process connections and in the process itself. For maximum benefit, information should reach operators and maintenance personnel in real-time, with context-sensitive remedial action. Logging all diagnostics and maintenance action from a central asset management system will ensure consistency, and simplify collection of failure statistics. Historically, users used multiplexers to obtain this diagnostic information. Open protocols such as WirelessHART now provide the same benefits at a much lower installed cost. ■

Edited by Suzanne Shelley

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Fractionation Column

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We need NASA

Richard Belzer, of the television program Law and Order, authored a book entitled "UFOs, JFK and Elvis: Conspiracies You Don't Have to be Crazy to Believe." I remember reading and enjoying the book. The author contended that there are five-sided pyramids on Mars and that NASA (the U.S. National Aeronautics and Space Administration) has purposely hidden that fact from an easily upset global populace.

When I saw that Dr. Doug McCuistion was going to speak at the recent ARC Forum (February 11–14, in Orlando, Fla.; www.arcweb.com), I was excited. McCuistion was the director of the very recent, and very successful, Mars Curiosity mission. I was hopeful that he would have close-up photographs of the pyramids, and a detailed explanation of how they were built. On that front, the director's presentation was a disappointment — on every other front, the audience's highest expectations were achieved.

Mars missions, including data collections, are extremely difficult. The Curiosity rover was equipped with many scientific instruments, including three gas chromatographs. Those chromatographs were capable of determining chemical compositions, but not crystalline structures. Carbon, for example, can exist as coal, graphite or diamond. The best way to determine crystalline structure, especially 200 million miles away, is by using X-ray diffraction. Prior to the Curiosity mission, a typical X-ray diffractometer was the size of a refrigerator. The NASA scientists and engineers successfully created a shoebox-size version. Back during my Northwestern University days, an X-ray diffraction apparatus filled a small laboratory that included a dark room for reading diffraction patterns on photographic films. (My M.S. thesis involved the straining of polymer films to see whether the amorphous-crystalline structure of the films was changing. Specifically, I was pulling on the plastic webbing that holds six-packs together to determine why the plastic films are easy to stretch but very difficult to break.) On Mars, the shoebox diffractometer was capable of differentiating minerals like feldspar, pyroxenes and olivine. Some of the minerals on Mars proved to be very similar to those that are found near Hawaiian volcanoes.

NASA issues an annual publication entitled Spinoff, which lists NASA inventions that have impacted daily life. To date, there have been at least 1,700. The list is diverse and amazing and includes the following: infrared ear thermometers, freeze drying, silicon solar-power cells, cochlear implants, grooved highway shoulders, improved radial tires, baby food nutrients, memory foam and the computer mouse. And of course, NASA satellites track dangerous weather for us. NASA is, however, falsely credited with inventing Tang, Velcro and Teflon. What NASA did achieve is the popularization of these products. We need NASA! ■

Mike Resetarits

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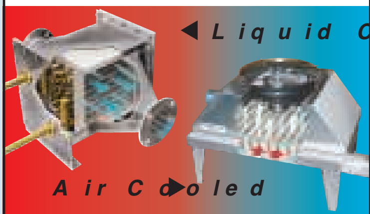

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58 Materials of Construction
59 Compressors

1	16	31	46	61	76	91	106	121	136	151	166	181	196	211	226	241	256	271	286	301	316	331	346	361	376	391	406	421	436	451	466	481	496	511	526	541	556	571	586
2	17	32	47	62	77	92	107	122	137	152	167	182	197	212	227	242	257	272	287	302	317	332	347	362	377	392	407	422	437	452	467	482	497	512	527	542	557	572	587
3	18	33	48	63	78	93	108	123	138	153	168	183	198	213	228	243	258	273	288	303	318	333	348	363	378	393	408	423	438	453	468	483	498	513	528	543	558	573	588
4	19	34	49	64	79	94	109	124	139	154	169	184	199	214	229	244	259	274	289	304	319	334	349	364	379	394	409	424	439	454	469	484	499	514	529	544	559	574	589
5	20	35	50	65	80	95	110	125	140	155	170	185	200	215	230	245	260	275	290	305	320	335	350	365	380	395	410	425	440	455	470	485	500	515	530	545	560	575	590
6	21	36	51	66	81	96	111	126	141	156	171	186	201	216	231	246	261	276	291	306	321	336	351	366	381	396	411	426	441	456	471	486	501	516	531	546	561	576	591
7	22	37	52	67	82	97	112	127	142	157	172	187	202	217	232	247	262	277	292	307	322	337	352	367	382	397	412	427	442	457	472	487	502	517	532	547	562	577	592
8	23	38	53	68	83	98	113	128	143	158	173	188	203	218	233	248	263	278	293	308	323	338	353	368	383	398	413	428	443	458	473	488	503	518	533	548	563	578	593
9	24	39	54	69	84	99	114	129	144	159	174	189	204	219	234	249	264	279	294	309	324	339	354	369	384	399	414	429	444	459	474	489	504	519	534	549	564	579	594
10	25	40	55	70	85	100	115	130	145	160	175	190	205	220	235	250	265	280	295	310	325	340	355	370	385	400	415	430	445	460	475	490	505	520	535	550	565	580	595
11	26	41	56	71	86	101	116	131	146	161	176	191	206	221	236	251	266	281	296	311	326	341	356	371	386	401	416	431	446	461	476	491	506	521	536	551	566	581	596
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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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Classified Index April 2013

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APRIL WHO'S WHO


Eichentopf


Aust


Sesselmann


Grenier


Allen

Ralf Eichentopf is now part of the management team at **GEA Bock GmbH** (Frickenhausen, Germany), where he will be responsible for manufacturing. *Erik Aust* is the new director of sales for the market segment Mobile Applications (compressors for buses and trains).

Bernhard Nick becomes head of the Strategic Planning & Controlling Div. of **BASF SE** (Ludwigshafen, Germany). *Friedrich Seitz*, president of Process Research & Chemical Engineering, will succeed Nick as head of BASF'S Verbund Site Management Europe Div. *Peter Schuhmacher,* SVP of strategic planning, will succeed Seitz as president of the Process Research & Chemical Engineering Div.

Sarah Sesselmann joins **Toray Plastics (America), Inc.** (North Kingstown, R.I.), as an account representative in the Torayfan Polypropylene Film Div.

Spectraseis (Houston), a provider of seismic monitoring, welcomes *David Grenier* as sales director, based in Houston. *Richard Marcinew* joins the company as an engineering advisor, based in the Calgary, Alta. office.

Terry Allen joins **Perten Instruments AB** (Springfield, Ill.) as vice president of sales for North America.

Marjan Oudeman is leaving her position as executive committee member responsible for organizational development and human resources at **AkzoNobel** (Amsterdam) to become president of Utrecht University. ■

Suzanne Shelley

BUSINESS NEWS

PLANT WATCH

AkzoNobel Chemical Island starts supply to world's biggest single-line pulp mill

March 7, 2013 — AkzoNobel's (Amsterdam, The Netherlands; www.akzonobel.com) new €90-million Jupiá Chemical Island in Brazil has begun supplying the Eldorado Brasil Celulose pulp mill, which is the biggest in the world. The Jupiá facility, one of AkzoNobel's largest investments in Latin America, will supply, store and handle all chemicals for the 1.5-million ton/yr eucalyptus pulp mill under a 15-yr agreement, and will provide permanent jobs for around 60 people.

BASF and Markor plan for BDO and polyTHF in China

March 5, 2013 — BASF SE (Ludwigshafen, Germany; www.basf.com) and Xinjiang Markor Chemical Industry Co. (Markor; Korla, China) have signed agreements for two joint ventures to produce butanediol (BDO) and polytetrahydrofuran (PolyTHF) in Korla, Xinjiang Uygur autonomous region, Northwest China. The plants are planned to go on stream in 2015, with capacities of 100,000 ton/yr of BDO and 50,000 ton/yr of PolyTHF.

Lanxess invests €80 million in S-SBR production in Brazil

March 4, 2013 — Lanxess AG (Leverkusen, Germany; www.lanxess.com) says it will convert production of emulsion styrene butadiene rubber (E-SBR), as used in standard tires, to solution styrene butadiene rubber (S-SBR) used in high-performance "green tires" at its site in Triunfo (Rio Grande do Sul) in southern Brazil. Lanxess is the first company to carry out such a conversion. The technology switch will cost €80 million and will leave production capacity unchanged, at 110,000 m.t./yr.

Toyo awarded ammonia project in Indonesia

February 27, 2013 — Toyo Engineering Corp. (Toyo; Chiba, Japan; www.toyo-eng.co.jp) has won a turnkey contract to build a 2,000-ton/d ammonia plant in Luwuk, Central Sulawesi, Indonesia. The client is LPG producer PT Panca Amara Utama (PAU), a subsidiary of PT Surya Esa Perkasa Tbk. (SEP). The plant will use technology from KBR (Houston; www.kbr.com), and is scheduled for completion in the third quarter of 2015. Toyo will carry out the project together with local Toyo group subsidiary PT Inti Karya Persada Tehnik (IKPT).

Stamicarbon lands Chinese urea contract

February 26, 2013 — Stamicarbon B.V. (Sittard, The Netherlands; www.stamicarbon.com), part of the Maire Tecnimont group (Milan, Italy; www.mairetecnimont.it), has signed two contracts for a new urea plant with Jiangsu Linggu Chemical Industry Co. (Yixing, China). The plant will have a capacity of 2,700 m.t./d of prilled urea and will be located in Yixing, Jiangsu Province, China. Engineering work will be performed by China Chengda Engineering Co. Startup is planned for 2015. The plant will use Stamicarbon's Urea2000Plus technology.

Grupo Idesa and CyPlus to build cyanide plant in Mexico

February 22, 2013 — Grupo Idesa S.A. de C.V. (Mexico City, Mexico; www.grupoidesa.com) and CyPlus GmbH (Hanau, Germany; cyplus.com), a subsidiary of Evonik Industries AG (Essen, Germany; www.evonik.com) have announced that they are close to signing a joint venture (JV) agreement for the construction of a sodium cyanide plant in Coatzacoalcos, Mexico. The 40,000 m.t./yr facility is expected to go on stream in the first quarter of 2015.

UOP technology selected to modernize refinery in Kazakhstan

February 21, 2013 — UOP LLC (Des Plaines, Ill.; www.uop.com), a Honeywell company, has won an upgrade contract from the largest petroleum refiner in Kazakhstan, KazMunaiGas. The 100,000 bbl/d Pavlodar Oil Chemical Refinery will use a range of UOP processes and services to modernize its facility in north-east Kazakhstan, allowing it to meet Euro-5 standards aimed at reducing motor vehicle pollution. Kazakhstan is expected to produce 1.6 million bbl/d of oil this year.

New bitumen project awarded to Pörner by Indian Oil

February 20, 2013 — In January, Pörner Ingenieurgesellschaft mbH, (Vienna, Austria; www.poerner.at) concluded a contract with Indian Oil Corp. (IOCL; www.iocl.com) for engineering and construction of a 100,000 m.t./y bitumen plant at Barauni Refinery, near Patna in the east of India. Startup is scheduled for 2014. Feedstocks will be vacuum resid or heavy vacuum gas oil. This plant is Pörner's 46th license for its Biturox bitumen oxidation technology, which provides 50% of India's bitumen production.

Dow and Davy license LP Oxo Technology to Sinopec

February 20, 2013 — Davy Process Technology Ltd. (DPT; London, U.K.; www.davyprotech.com) and The Dow Chemical Company (Dow; Midland, Mich.; www.dow.com) have announced that China Petrochemical International Co., a subsidiary of China Petroleum & Chemical Corporation (Sinopec), has selected LP Oxo Technology to produce 2-ethylhexanol, *n*-butanol and isobutanol in a plant in Anqing City, Anhui Province, China. The new LP OxoSM unit will be built by Sinopec Corp. Anqing Co. with a capacity of 100,000 ton/yr 2-ethylhexanol, 115,000 ton/yr *n*-butanol and 23,000 ton/yr isobutanol.

MERGERS AND ACQUISITIONS

Outotec grows its maintenance services by acquiring Scanalyse

February 28, 2013 — Outotec Oyj (Espoo, Finland; www.outotec.com) has signed an agreement to acquire Scanalyse Holdings Pty Ltd. (Bentley, Australia) for an undisclosed amount. Scanalyse, which has operations in Australia, Brazil, Chile and the U.S., produces software to monitor the condition and performance of process equipment.

Bilfinger Water Technologies links Passavant-Geiger and Johnson Screens

February 27, 2013 — Passavant-Geiger GmbH, a wholly owned subsidiary of Bilfinger SE (Aarbergen, Germany; www.bilfinger.com), has acquired Johnson Screens Inc. (New Brighton, Minn.), a global leader in screening products for water infrastructure, wastewater treatment, hydrocarbon processing and general filtration. The resulting new Bilfinger Water Technologies Group will have a turnover of more than €300 million, roughly double the size of both Passavant-Geiger and Johnson Screens.

Metrohm and FOSS NIRSystems form analytical alliance

February 19, 2013 — Wet-chemistry specialist Metrohm AG (Herisau, Switzerland; www.metrohm.com) and FOSS NIRSystems Inc. (Laurel, Md.; www.foss-nirsystems.com) have formed a strategic alliance in which Metrohm will become the sole global distributor of FOSS NIR instruments for the chemical, petrochemical, pharmaceutical and environmental sectors. FOSS NIRSystems will become a division of Metrohm USA and will be renamed Metrohm NIRSystems. ■

Charles Butcher

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

April 2013; VOL. 120; NO. 4

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FOR MORE ECONOMIC INDICATORS, SEE NEXT PAGE

Economic Indicators

2011 2012 2013

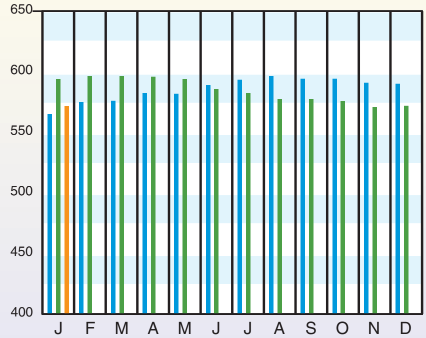
DOWNLOAD THE ***CEPCI*** TWO WEEKS SOONER AT ***WWW.CHE.COM/PCI***

CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	Jan.'13 Prelim.	Dec.'12 Final	Jan.'12 Final
***CE* Index**	571.4	571.9	593.6
Equipment	692.9	693.6	726.8
Heat exchangers & tanks	630.2	634.7	686.7
Process machinery	657.7	657.6	676.1
Pipe, valves & fittings	890.4	895.8	924.9
Process instruments	416.6	415.7	427.8
Pumps & compressors	913.9	899.6	911.6
Electrical equipment	513.9	511.4	511.6
Structural supports & misc	741.6	734.5	776.1
Construction labor	319.8	321.3	321.1
Buildings	531	526.8	520.4
Engineering & supervision	327.1	326.9	329.9

Annual Index:

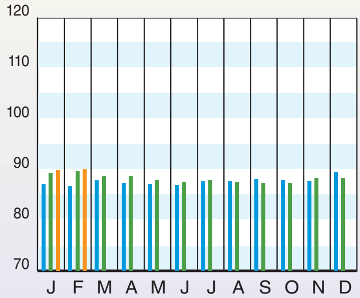
2005 = 468.2
2006 = 499.6
2007 = 525.4
2008 = 575.4
2009 = 521.9
2010 = 550.8
2011 = 585.7
2012 = 584.6



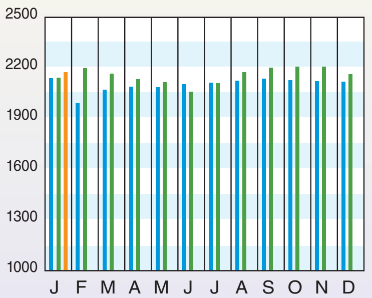
CURRENT BUSINESS INDICATORS

	LATEST			PREVIOUS						YEAR AGO		
CPI output index (2007 = 100)	Feb.'13	=	90.0	Jan.'13	=	89.9	Dec.'12	=	88.3	Feb.'12	=	89.7
CPI value of output, $ billions	Jan.'13	=	2,174.2	Dec.'12	=	2,164.3	Nov.'12	=	2,208.7	Jan.'12	=	2,143.7
CPI operating rate, %	Feb.'13	=	77.4	Jan.'13	=	77.4	Dec.'12	=	76.2	Feb.'12	=	77.5
Producer prices, industrial chemicals (1982 = 100)	Feb.'13	=	314.2	Jan.'13	=	299.7	Dec.'12	=	299.7	Feb.'12	=	314.9
Industrial Production in Manufacturing (2007=100)	Feb.'13	=	96.5	Jan.'13	=	95.7	Dec.'12	=	94.8	Feb.'12	=	94.6
Hourly earnings index, chemical & allied products (1992 = 100)	Feb.'13	=	155.2	Jan.'13	=	155.0	Dec.'12	=	153.9	Feb.'12	=	157.3
Productivity index, chemicals & allied products (1992 = 100)	Feb.'13	=	106.6	Jan.'13	=	107.1	Dec.'12	=	105.8	Feb.'12	=	108.7

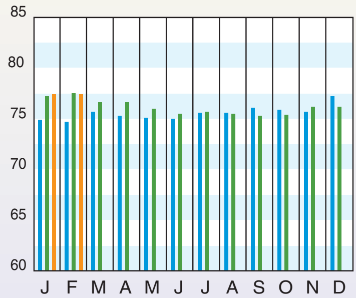
CPI OUTPUT INDEX (2007 = 100)



CPI OUTPUT VALUE ($ BILLIONS)



CPI OPERATING RATE (%)



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

CURRENT TRENDS

The overall *CE* Plant Cost Index (CEPCI; top) for 2012 was 0.19% lower than the end-of-year average from 2011. Preliminary data from January 2013 CEPCI (the most recent available) indicate that capital equipment prices declined from December to January, by 0.09%. When compared to year-earlier numbers (from January 2012), the January 2013 preliminary Plant Cost Index stands at 3.74% lower. Meanwhile, the Current Business Indicators from IHS Global Insight (middle), show that the latest CPI output index value (Feb. 2013) edged slightly higher from the previous month, and producer prices for industrial chemicals were modestly higher. ■