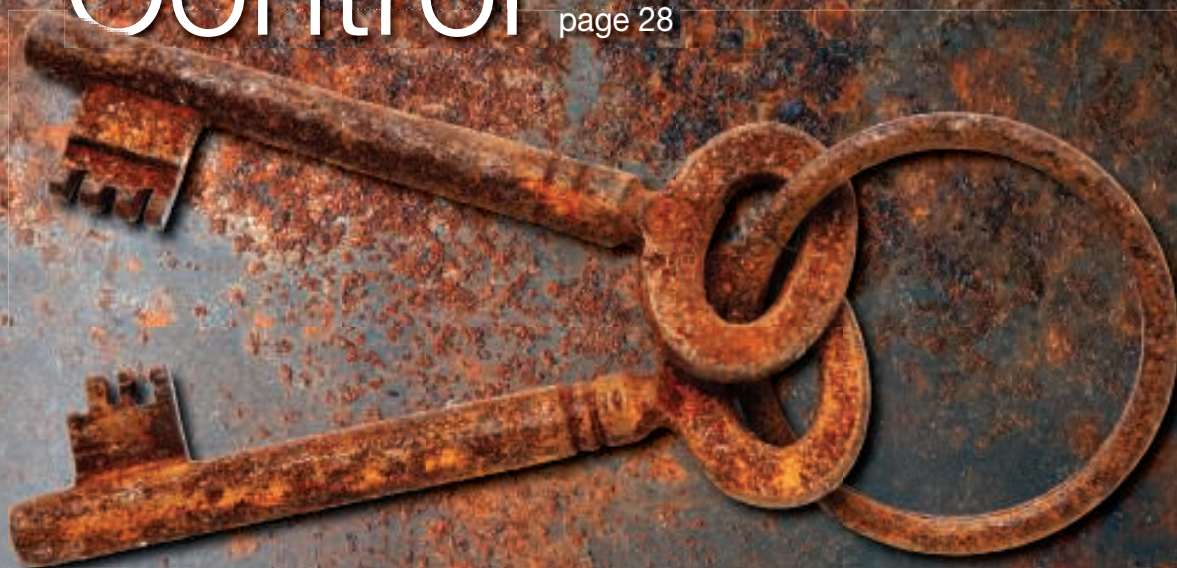




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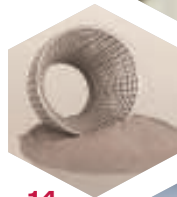
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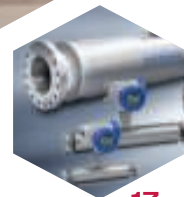
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The 2017 Kirkpatrick Award

Every other year, *Chemical Engineering* honors the most noteworthy chemical engineering technology that was commercialized anywhere in the world during the previous two years with the Kirkpatrick Chemical Engineering Achievement Award. The nominations for 2017 are now open.

The Kirkpatrick Award has been awarded continuously since 1933. In 2015, Dow Performance Chemicals received the award for its achievements with Intune olefin block copolymers. Honors were given to Newlight Technologies for its AirCarbon process; Clariant for HGM Technology for propylene dehydrogenation; AM Technology for the Coflore Reactor and CB&I for CDAIky alkylation technology. The 2017 winner will join this distinguished group and earlier winners that include milestones such as Genomatica's process for bio-based 1,4-butanediol (2013); Chevron Phillips Chemical for significant advances in alpha-olefins technology (2005); and Carbide & Carbon Chemical's petrochemical syntheses (1933). The full list of past winners can be found at www.chemengonline.com/kirkpatrick.

The nomination procedure

To submit a nomination, simply send an unillustrated nominating brief of up to 500 words to: awards@chemengonline.com. Any person or company, worldwide, can submit a nomination. The deadline for nominations is March 15, 2017.

In order to be considered, each nomination should include the following three items: 1) a summary of the achievement and novelty of the technology; 2) a description of the difficult chemical-engineering problems solved; and 3) a description of how, where and when the development first became commercial in 2015 or 2016.

If you are aware of an achievement that qualifies for nomination, but do not have enough of information to write a brief, contact the company involved, either to get the information or to propose that the firm itself submit a nomination. Companies are also welcome to nominate achievements of their own.

The selection procedure

After the deadline of March 15, the nominations will first be reviewed for validity. They will then be sent to department heads at accredited university chemical-engineering departments, who accordingly, constitute the Committee of Award. Each professor will vote, independently of each other, for a maximum of five best achievements.

The entries that collectively receive the most votes become the finalists in the competition. Each finalist company will then be asked to submit additional information, such as a description of the technology, performance data and examples of the teamwork that generated the achievement.

Copies of these more-detailed packages will then be sent to a Board of Judges, which will have been chosen from within the Committee of Award. The Board will judge the entries to select the most noteworthy. The company that developed that achievement will be named the winner of the 2017 Kirkpatrick Chemical Engineering Achievement Award and the other finalist companies will be designated to receive Honor Awards. The winner will be announced at the Chem Show in New York this fall. ■



Dorothy Lozowski, Editor in Chief

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A process for making longer carbon nanotubes

A new commercial manufacturing process for carbon nanotubes (CNTs) produces tubes in the range of 1–10 mm in length (5–12-nm dia.), two orders of magnitude longer than currently available CNTs, which typically have lengths from 5–20 μm .

“Despite attractive mechanical and electrical properties, CNTs have largely been a disappointment for ‘real-world’ applications, because it has not been possible to make them in formats that are useful for engineers,” explains Peter Antoinette, co-founder and president of Nanocomp Technologies Inc. (Merrimack, N.H.; www.nanocomptech.com), the developer of the process. Short CNTs do not readily form networks within other materials, unless used at very high concentrations.

The Nanocomp process revolves around a proprietary 1-m long heated reactor (photo) that contains a widely available iron catalyst and allows control of 23 separate process variables. Organic alcohols serve as the carbon source for CNTs. “By exerting tight control over the process conditions, we can manipulate the length and dimensions of the CNTs,” Antoinette says. The longer, polymer-like CNTs resulting from the process are commercially available as Miralon products, and they can be spun into “yarn” using equipment for textile fiber processing. Because of their length, the Nanocomp CNTs form bundles and networks that allow them to be more useful in macroscale materials,

Nanocomp Technologies



such as for lightweight structural materials.

“Our process allows CNTs to be made at high volumes with a cost structure that is similar to that of commodity chemicals,” Antoinette says.

Nanocomp CNTs can also be made into strong polymer-like sheets that can be used in firearm-protection armor. The U.S. Dept. of Defense recently awarded the company \$18.5 million to supply soldier and law enforcement body armor. The sheets can also be used as area heaters, Antoinette says, because they emit infrared radiation when low-energy power is applied.

Nanocomp currently produces its CNTs at a scale of 2 tons/yr and plans to triple its manufacturing capacity in 2017. Eventually, capacity could reach 20 tons/yr, Antoinette says, adding that the rapid growth has been helped greatly by support for nanotechnology manufacturing from the state of New Hampshire.

This process converts organic food waste to liquid fertilizer

Large supermarkets routinely waste 500 lb of food daily from past-due produce, deli and meat scraps and other sources. A new process developed by California Safe Soil (CSS; McClellan, Calif.; www.calsafesoil.com) converts the nutrient-rich waste food into a liquid fertilizer for farmers.

After a series of grinding steps, the waste food enters a ribbon-blender digester, where enzymes are added to break down proteins, carbohydrates and fats in the organic waste into amino acids, simple sugars and fatty acids. The mixture then undergoes mechanical emulsification and pasteurization processes. The liquid product, containing oil and water-based nutrients, is stabilized with phosphoric acid, and can be used to fertilize root systems of farm crops. The solid portion of the organic waste is used in pig feed.

“The liquid fertilizer, known as Harvest-to-Harvest (H2H), adds organic matter to the root zone of crops and stimulates the growth of beneficial soil microbes, which generates additional root growth. Plants take up more water and fertilizer, and increase flowering and fruiting,” explains CSS founder Dan Morash. “It also reduces the need for nitrate fertilizers on farms, which reduces farm runoff and algae blooms in nearby bodies of water,” Morash says.

Food waste is collected from supermarkets in insulated totes and buggies and is processed locally, because the CSS process generates no waste streams or nuisance odors, Morash points out. Liquid fertilizer has advantages over solid compost, because it can be dripped into the root systems using existing irrigation systems, rather than staying on the soil surface, he adds.

Edited by:

Gerald Ondrey

HVAC COOLING TOWER

A newly introduced cooling tower for building heating, ventilation and air conditioning (HVAC) has both its structural casing and fill made from high-density polyethylene (HDPE) resin that contains additives designed to prevent the growth of microorganisms and formation of biofilms within the cooling tower. The anti-microbial HDPE cooling tower was recently introduced by Delta Cooling Towers Inc. (Roxbury Township, N.J.; www.deltacooling.com) to address concerns over pathogenic microbes, such as the bacteria species that causes Legionnaire’s disease. Under certain conditions, the bacteria can be incubated and spread by water systems. The cooling tower’s design also avoids stagnant water areas, where microorganisms can grow, the company says.

PHOTOSYNTHESIS

Fujitsu Laboratories Ltd. (FLL; Kawasaki City, www.fujitsu.com) has developed a new process for layering thin films of inorganic photocatalysts onto a substrate. Using a proprietary nozzle, the catalyst precursors are sprayed onto a thin plate, and are fragmented into nano-sized particles that deposit on the substrate. Because of the enhanced (50-fold) increase in surface area compared to alternative methods, and the ability of the catalyst to operate over a broader spectrum of usable sunlight ($\lambda_{\text{max}} = 630 \text{ nm}$, compared to $\lambda_{\text{max}} = 490 \text{ nm}$ for existing catalysts), the new catalyst shows a 100-fold efficiency for the oxygen-producing step of photosynthesis.

FLL plans to further develop the technology, with industrial applications projected for 2025.

(Continues on p. 8)

COLD OIL SPILLS

Researchers at the U.S. Dept. of Energy's Pacific Northwest National Laboratory (PNNL; Richland, Wash.; www.pnnl.gov) have chemically modified sawdust to make it exceptionally oil-attracting and buoyant — characteristics that are ideal for cleaning up oil spills in Arctic waters. The nontoxic material absorbs up to five times its weight in oil and stays afloat for at least four months.

"Most of today's oil-remediation materials are designed for warm water use," says PNNL microbiologist George Bonheyo, who leads the modified sawdust's development from PNNL's Marine Sciences Laboratory. Beyond absorbing oil, it also enhances another approach to combatting oil spills — controlled burns. If changing weather or tides move spilled oil toward a sensitive area fast, oil can be burned before it can cause further harm. Called in-situ burning, the practice can significantly reduce the amount of oil in water and minimize its adverse environmental effects.

To modify the sawdust, researchers chemically attach components of vegetable oil onto the material's surface, making it hydrophobic. The final product is a light, fluffy, bleached powder. The team is also trying out adding tiny, oil-eating microbes — fungi and bacteria — to the powder's surface so any left-behind material could naturally break down oil over time.

NEW CATALYST

Haldor Topsøe A/S (Lyngby, Denmark; www.topsoe.com) has introduced a second-generation HyBRIM catalyst, which is said to boost profitability of diesel and hydrocracking units at petroleum refineries. Refiners can achieve longer cycle lengths, high-value products from low-quality feedstocks and increased volume swell with the latest HyBRIM nickel-molybdenum catalyst, with 25% improved activity for both nitrogen and sulfur removal, says the company.

Successor to TK-609 of the

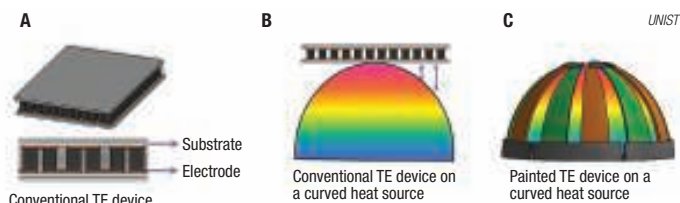
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Paint a thermoelectric device onto any shaped surface to recover waste heat

Scientists from Ulsan National Institute of Science and Technology (UNIST; Ulsan, South Korea; www.unist.ac.kr), led by Prof Jae Sung Son, have succeeded in producing high-performance, solid-state thermoelectric (TE) materials with liquid-like properties that can be easily brush-painted on surfaces of almost any shape.

The thermoelectric effect is the direct conversion of temperature differences to electric voltage and vice versa. This phenomenon can be used for heating or cooling, and for waste-heat-recovery systems. The output power of thermoelectric generators depends both on the inherent properties of the TE material and on the engineering of the device to minimize heat losses.

The power generating efficiency of solid-state TE devices can be estimated from a dimensionless parameter, $ZT = S^2\sigma T/\kappa$, where S is the Seebeck coefficient, σ is the electrical conductivity, κ is the thermal conductivity and T is the temperature). In real-world applications, the minimization of heat loss due to incomplete contact between the surface of the heat source and the TE module is no



less important than the TE material's power-generating efficiency.

In response to that challenge, the scientists developed a shape-engineerable TE painting (diagram, C) that prevents that heat loss suffered by conventional planar TE devices (diagram, A and B). They achieved this by using a molecule-level sintering process, which is described in detail in a recent issue of *Nature Communications*. They used the molecular Sb_2Te_3 -based chalcogenidometalate (ChAM) for n-type BiTeSe and p-type BiSbTe TE particles. Optimizing the process, they achieved the ZT values of 1.21 for p-type and 0.67 for n-type TE materials. They fabricated TE generators by applying TE paint on flat, curved or large-sized hemispherical substrates, followed by sintering at temperatures above 350°C. The process is said to be the most effective means of heat-energy collection from any heat source, and the output power density (4.0 mW/cm²) is the best value among the reported printed TE generators.

An inexpensive adsorbant for removing silver from wastewater

The release of silver from industrial wastewater has caused serious environmental problems. Many methods have been developed to remove silver ions from industrial wastewater, including chemical precipitation, ion exchange, electrolysis, replacement, membrane and reverse osmosis. Several adsorbents have been used to remove and recover silver from aqueous solutions or industrial wastewater. However, those adsorbents are expensive, and lately many low-cost materials have been tried, including waste wool, peanut shells, crab shells, soybean hulls and cotton. Now, a group from Gangneung-Wonju National University (Gangneung, South Korea; www.gwnu.ac.kr) led by professor Choong Jeon, has used recycled waste coffee grounds to remove silver from industrial wastewater directly as a zero-cost adsorbent.

From the Fourier-transform infrared (FTIR) spectra analysis, the group found that the

waste coffee grounds — which have a porous and homogeneous structure and are composed mainly of carbon (61.60%) and oxygen (38.40%) — have functional groups like COO^- and OH^- , which play an important role in Ag^+ adsorption. The existence of Ag^+ on the adsorbent was confirmed by scanning electron microscope (SEM) images and energy-dispersive x-ray (EDX) spectroscopy analysis. The highest adsorption capacity (46.2 mg/g) and removal efficiency (92.4%) were achieved at pH of 6. The adsorbed amount of silver ions decreased slightly with increasing temperature in the range of 15 to 45°C. Most of the adsorption was completed within 60 min.

The group believes the adsorption process using waste coffee grounds can be applied to the adsorption and recovery of silver ions in industrial wastewater-treatment systems, and that the process could replace conventional treatment processes, such as solvent extraction and ion-exchange resin.

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HyBRIM series, the new TK-611 HyBRIM catalyst offers new possibilities to either increase profitability of high-pressure, ultra-low-sulfur diesel production from heavy feedstocks or to guarantee a superior pre-treatment for the hydrocracking unit. Refiners can process more severe feeds, increase cycle lengths by several months, achieve larger volume swells or maximize unit throughput, says Topsøe. HyBRIM also supports profitability by substantially improving the cetane number of the final product, the company adds.

The improved activity for nitrogen removal improves overall hydrocracking-unit performance because it lowers the nitrogen slip from the pre-treatment step to the second stage in the hydrocracker. This minimizes inhibition of the hydrocracking catalyst, enabling higher conversion and selectivity.

The HyBRIM technology combines the well-proven BRIM technology with an improved catalyst-preparation step. This leads to optimal interaction between the active metal structures and the catalyst carrier, which increases activity while delivering the same high stability.

NEW ODHP CATALYST

Chemists and chemical engineers from the University of Wisconsin–Madison (www.wisc.edu) have discovered a new family of catalysts to drive the oxidative dehydrogenation of propane (ODHP) reaction. The catalyst family of hexagonal boron nitride and boron nitride nanotube catalysts is said to produce a greater proportion of propene during the reaction than traditional oxide catalysts. Whereas the traditional catalysts lead to the formation of CO₂ and other undesirable byproducts in addition to propene, the new catalysts instead produce ethane as a byproduct.

“Boron nitride catalysts are nontoxic, they don’t contain precious metals, and they reduce the temperature of the reaction, resulting in energy savings,” says UW–Madison graduate student Joseph Grant, first author of the new study published in a recent issue of *Science*. Additionally, the boron nitride catalysts may be used continuously without an intermediate regeneration step, as required in alternative dehydrogenation processes.

This work was supported in part by the Wisconsin Alumni Research Foundation (WARF) Accelerator Program. WARF has filed a number of patent applications on this technology.

MOF MAKES METHANOL FROM CO₂

Researchers at the University of Pittsburgh’s Swanson School of Engineering (Pa.; www.engineering.pitt.edu) are developing a new catalyst that enables the hydrogenation of CO₂ into methanol. The catalyst — a Lewis-pair functionalized metal organic framework (MOF) — was described in a recent issue of *Catalysis Science & Technology*, authored by postdoctoral associate, Jingyun Ye, and Karl Johnson, the William Kepler Whiteford Professor in the Swanson School’s Dept. of Chemical & Petroleum Engineering.

(Continues on p. 11)

Australians move to supply vanadium for redox flow batteries

Two Australian companies — Australian Vanadium Ltd. (AVL; www.austrianvanadium.com.au) and TNG Ltd. (both Perth; www.tngltd.com.au) — are now able to produce commercial-grade vanadium electrolyte for use in vanadium redox-flow batteries (VRFBs). Those batteries are increasingly gaining favor, primarily for grid-scale energy storage applications (*Chem. Eng.* September 2016, pp. 14–20).

AVL is sourcing its vanadium from its Gabanintha project in Western Australia. The company has just received a vanadium-electrolyte pilot plant from C-Tech Innovation Ltd. (Chester, U.K.; www.ctechinnovation.com), and is marketing VRFBs in Australia through a distribution agreement with Gildemeister Energy Solutions GmbH (Würzburg, Germany; www.energy.gildemeister.com).

TNG is sourcing its vanadium from its Mount Peake vanadium-titanium-iron project in the Northern Territory. The company has produced high-purity vanadium electrolyte for the first time using vanadium pentoxide (V₂O₅) from that project. TNG will

apply its Tivan process, designed primarily for extracting vanadium, preferably as vanadium pentoxide from a titanomagnetite ore body, which contains iron, titanium and vanadium. The process serves also to separate titanium and iron, preferably as ferric oxide and titanium dioxide. In the Tivan process, the vanadium is recovered entirely through a hydrometallurgical route incorporating leaching and solvent extraction. A benefit of this process is that within the same flowsheet hematite and titanium dioxide are separated and recovered as saleable byproducts in addition to vanadium pentoxide.

TNG says the Tivan process is unique in that existing processes cannot extract all three metals — iron, titanium and vanadium — as industrial-grade products. The company says the conventional method for extracting vanadium from titanomagnetite ore deposits is through a salt roasting energy-intensive, pyro-metallurgical process, which is suitable for only a narrow range of selected ore compositions, and a water leach route to recover a water-soluble vanadium compound.

Laminate packaging that is easier to recycle

A collaboration between ExxonMobil Chemical Co. (EMCC; Spring, Tex.; www.exxonmobilchemical.com) and Thanh Phu Plastic Packaging Co. (TPPP; Ho Chi Minh City, Vietnam; www.thanhphupack.com) has resulted in the development and commercialization of laminate packaging materials that can be collected and recycled in the same stream as polyethylene (PE) products. Due to their multi-component makeup, recycling laminated packaging is quite challenging, usually requiring a complex process to separate a nonpolar polyolefin layer from a polar laminate, such as polyethylene terephthalate (PET), polyamide (PA), ethylene vinyl alcohol (EVOH) or oriented polypropylene (OPP).

The collaboration combines TPPP’s proprietary film-conversion

process with PE performance polymers from EMCC’s Exceed and Enable product families. This full-PE alternative to conventional laminated products is comparable to traditional laminate products in terms of aesthetics and integrity, says EMCC. In similar products, the full-PE solution exhibited competitive secant modulus and bag-drop performance to PA-laminated at the same total film thickness, says TPPP. Leveraging TPPP’s Veloflex film-blowing technology, which is based on specific directional-orientation of polymers during the extrusion process, the new laminate packaging is being produced at global scales, and has been commercially deployed in the Asia-Pacific region. The full-PE laminate packaging is especially suited for use in pouches and medium-density sacks.

A very fast way to continuously synthesize zeolites

For a long time, it has been believed that the crystallization of zeolites is, by nature, a very slow process. The hydrothermal synthesis of zeolites is normally performed batchwise, requiring crystallization times on the order of days. Now, Toru Wakihara and Tatsuya Ohkubo at the Dept. of Chemical System Engineering, University of Tokyo (Japan; www.zeolite.t.u-tokyo.ac.jp) have demonstrated the continuous-flow synthesis of the industrially important zeolite, ZSM-5. Crystallization from the amorphous state to full crystallinity could be completed in just a few seconds, which demonstrates

that the time needed for crystallization is 3–4 orders of magnitude shorter than previously believed. The researchers say the fast synthesis offers a great potential for the mass-production of such materials, as well as deepening the fundamental understanding of zeolite formation.

The continuous flow reactor has millimeter-sized channels in which “well-tuned” precursors (at 90°C) are mixed with pressurized, preheated water at 370°C. This leads to the immediate heating of the precursors to 240–300°C, with subsequent, seed-free crystallization of ZSM-5 within tens of seconds, or fewer.

Japanese consortium synthesizes bifunctional oxygen-reaction catalysts

Researchers from the group of professor Ikuya Yamada at Osaka Prefecture University (Osaka, Japan; www.osakafu-u.ac.jp), in collaboration with the University of Tokyo, Japan Synchrotron Radiation Research Institute (Hyogo; www.spring8.or.jp) and Fuji Die Co. (Tokyo; www.fujidie.co.jp), have synthesized for the first time manganese-quadruple perovskites — $\text{CaMn}_7\text{O}_{12}$ and $\text{LaMn}_7\text{O}_{12}$ — which are compounds that both exhibit bifunctional electrocatalytic behavior for the oxygen evolution/reduction reaction (OER/ORR). The new catalyst systems are desirable for the development

of energy conversion technologies, especially in the field of next-generation secondary batteries, such as metal-air secondary batteries.

One explanation for the high OER activity of these new catalyst systems is the unique surface structure, which consists of corner-shared planar MnO_4 and octahedral MnO_6 units, which promote the direct formation of oxygen-oxygen bonds. Compared to existing manganese-oxide catalyst systems, the crystal structure of these new quadruple manganese perovskites could enhance the oxygen evolution catalytic activity by up to 30 times, say the researchers.

On-site carbon monoxide generation takes the next step

Gas Innovations (La Porte, Tex.; www.gasinnovations.com) has signed a 15-year, pay-per-use agreement with Haldor Topsøe A/S (Topsøe; Lyngby, Denmark; www.topsøe.com) for a second on-site carbon-monoxide production unit, using Topsøe’s electrolytic Carbon Monoxide solution (eCOs) technology. The first eCOs unit in the U.S. has been operating at Gas Innovations since January 2016. The new unit will have ten-times larger capacity — 96 Nm^3/h (3,650 std. ft^3/h) of CO at up to 99.999 vol.% purity — and is expected to be online at the end of 2017.

Topsøe’s eCOs is a new electrolysis cell technology that allows the safe, efficient, and cost-competitive production of CO directly at the site of facilities where the gas is needed.

The heart of an eCOs plant is a solid-oxide electrolysis cell (SOEC) operating at 700–850°C. The CO_2 “fuel” is fed to the unit, and is electrochemically reduced to CO, while O_2 is generated at the anode. Any remaining unconverted CO_2 is removed from the CO product gas using a combination of pressure-swing adsorption (PSA) and polisher units.

On-site carbon-monoxide generation is a significant development to the medical, pharmaceutical, metallurgy, electronics and specialty chemicals industries, which require CO in their processes. The eCOs technology ensures security of supply, eliminates the need to transport a hazardous gas and drastically reduce costs related to storage, rentals and connections, according to Topsøe. ■

The study builds upon Johnson’s previous research that identified the two main factors for determining the optimal catalyst for turning atmospheric CO_2 into liquid fuel. The research was conducted using computational resources at the University’s Center for Simulation and Modeling. Johnson and Ye focused on computationally designing a catalyst capable of producing methanol from CO_2 and H_2 utilizing MOFs. The MOFs could dramatically reduce the cost of carbon capture and conversion, bringing the potential of CO_2 as a viable feedstock for fuels closer to reality.

“Methanol synthesis has been extensively studied because methanol can work in existing systems, such as engines and fuel cells, and can be easily transported and stored, explains Johnson. “Methanol is also a starting point for producing many other useful chemicals. This new MOF catalyst could provide the key to close the carbon loop and generate fuel from CO_2 , analogously to how a [botanical] plant converts carbon dioxide to hydrocarbons.”

NEW PP PILOT PLANT

By the end of March, SABIC (Riyadh, Saudi Arabia; www.sabic.com) will start up a new pilot plant for developing next-generation polypropylenes (PP) in Sittard-Geleen, the Netherlands. The plant, which will use gas-phase polymerization technology, will support the production at nearby full-scale plants of superior materials that meet the needs of different industries, such as automotive, pipe, appliances and advanced packaging. SABIC plans to concentrate on the development of impact grades of PP, as well as random copolymers and homopolymers. It will also carry out experiments on advanced catalysts.

Zetron B.V. (Enschede, the Netherlands; www.zetron.com) has been contracted to design and build the plant. Zetron has developed a skid-mounted system that accelerates implementation times and allows full design flexibility. □

LINEUP

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GURIT
HONEYWELL
INEOS STYROLUTION
JACOBS ENGINEERING
LINDE
MEXICHEM
MITSUBISHI HEAVY INDUSTRIES
PRAJ INDUSTRIES
SASOL
SHOWA DENKO
SIGMA-ALDRICH
SOLVAY
TESORO
TOTAL
UMICORE
WESTERN REFINING
WOOD GROUP
ZACHRY

Plant Watch

Linde starts up CO₂ plant in Fort Worth

December 12, 2016 — Linde North America (Bridgewater, N.J.; www.lindeus.com) started up a new carbon dioxide plant in Fort Worth, Tex. The plant, which has a production capacity of 250 metric tons (m.t.) per day, is supplying CO₂ for food-and-beverage producers and chemical manufacturers.

AkzoNobel and Atul to jointly build MCA plant in India

December 9, 2016 — AkzoNobel N.V. (Amsterdam, the Netherlands; www.akzonobel.com) and Atul Ltd. (Valsad, India; www.atul.co.in) intend to jointly invest in setting up a production plant for monochloroacetic acid (MCA) at Atul's facility in Gujarat, India. Each partner will hold a 50% stake. From an initial capacity of 32,000 m.t./yr at startup, the plant has been designed for future expansion to produce 60,000 m.t./yr of MCA.

Praj Industries to build multiple bioethanol plants across India

December 9, 2016 — Praj Industries Ltd. (Pune, India; www.praj.net) has entered into an agreement with Indian Oil Corp. to set up three new second-generation (2G) bioethanol plants — one each at Panipat, Haryana and Dahej, Gujarat. These plants will each have capacity to produce 100,000 L/d of ethanol. Additionally, Bharat Petroleum Corp. has also selected Praj as technology partner for setting up a 100,000-L/d 2G bio-ethanol plant in Orissa.

Ineos Styrolution announces expansion plans in Mexico

December 6, 2016 — Ineos Styrolution, (Frankfurt am Main, Germany; www.ineos-styrolution.com) plans to expand the company's copolymer plant in Altamira, Mexico, which produces a variety of acrylonitrile butadiene styrene (ABS) and acrylonitrile styrene acrylate (ASA) solutions. The 20,000-m.t. capacity expansion will grow Ineos Styrolution's overall copolymer production capacity in Altamira to 180,000 m.t./yr. Startup is expected by the first quarter of 2018.

Showa Denko to construct plant for production of new high-purity solvents

December 6, 2016 — Showa Denko K.K. (Tokyo, Japan; www.sdk.co.jp) will build a purification plant to produce new grades of high-purity solvents at its Tokuyama Plant in Shunan City, Japan. Construction of the new plant will be completed in May 2017, and commercial operations will commence in June 2017.

Sasol announces polypropylene capacity expansion in South Africa

November 28, 2016 — Sasol Ltd. (Johannesburg, South Africa; www.sasol.co.za) has completed its C3 Expansion Project, which enables the company to increase production capacity of polypropylene by 103,000 m.t./yr at its Secunda Chemicals Operations in South Africa. The project represents an investment of around \$73 million.

BASF to implement stepwise capacity expansion for U.S. MDI production

November 16, 2016 — BASF SE (Ludwigshafen, Germany; www.basf.com) began engineering for a stepwise capacity increase of its methylene diphenyl diisocyanate (MDI) production facilities at the company's Verbund site in Geismar, La. Capacity will be increased incrementally from 300,000 to around 600,000 m.t./yr.

ExxonMobil to increase polyethylene production at Beaumont site

November 15, 2016 — Exxon Mobil Corp. (Spring, Tex.; www.exxonmobil.com) plans to add a new production unit at its Beaumont, Tex. polyethylene plant that will increase capacity by 65% — approximately 650,000 m.t./yr. Significant contracts were awarded to Jacobs Engineering, Wood Group, Zachry Group and Mitsubishi Heavy Industries for work on the Beaumont expansion project.

Mergers & Acquisitions

Umicore to acquire Eurotungstene from Eramet Group

December 12, 2016 — Umicore N.V. (Brussels, Belgium; www.umicore.com) has signed an agreement to acquire Eurotungstene from Eramet Group (Paris, France; www.eramet.com). Eurotungstene specializes in metal powders used in diamond tools and hard metal applications. Closing of this transaction is anticipated to occur in the first half of 2017.

Evonik acquires Huber's silica business

December 9, 2016 — Evonik Industries AG (Essen, Germany; www.evonik.com) has acquired the silica business of J.M. Huber Corp. (Edison, N.J.; www.huber.com) for \$630 million. For the 2016 financial year, Huber Silica was expected to achieve sales of close to \$300 million. Huber's silica business is especially oriented towards applications in the consumer goods sector.

Solvay to sell its cellulose acetate business

December 7, 2016 — Solvay S.A. (Brussels, Belgium; www.solvay.com) has reached an agreement to sell its cellulose acetate tow business, Acetow, to private equity funds



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managed by Blackstone. The transaction is based on an enterprise value of about €1 billion, and is expected to be completed in the first half of 2017.

Gurit acquires BASF's PET business

December 1, 2016 — Gurit Holding AG (Wattwil, Switzerland; www.gurit.com) has acquired BASF's PET (polyethylene terephthalate) structural foam business for an undisclosed purchase price. The transaction comprises BASF's PET operations in Volpiano, Italy, including staff, operating assets and intellectual property.

Mexichem acquires U.K.-based PVC compounder

November 29, 2016 — Mexichem S.A.B. de C.V. (Tlahuepantla, Mexico; www.mexichem.com) has acquired Vinyl Compounds Holdings Ltd. (VCHL; Chinley, U.K.), a manufacturer of technical polyvinyl chloride (PVC) compounds serving a broad range of industries. VCHL has annual revenues of approximately \$40 million.

BASF to divest Inorganic Specialties business

November 28, 2016 — BASF will sell its Inorganic Specialties business to Edgewater Capital Partners of Cleveland, Ohio. Inorganic Specialties includes a production site in Evans City, Pa., and the Specialty Alcoholates, Boranes and Alkali Metals product lines, which are produced at the site. The transaction is expected to be completed in the first quarter of 2017.

Honeywell launches new research chemicals business

November 28, 2016 — Honeywell (Morris Plains, N.J.; www.honeywell.com) launched a new business, Honeywell Research Chemicals, which will include several brands of solvent and inorganic chemical products that were acquired from Sigma-Aldrich (St. Louis, Mo.; www.sigma-aldrich.com) in December 2015. The Research Chemicals business is headquartered in Seelze, Germany.

Tesoro to acquire Western Refining in \$4.1-billion deal

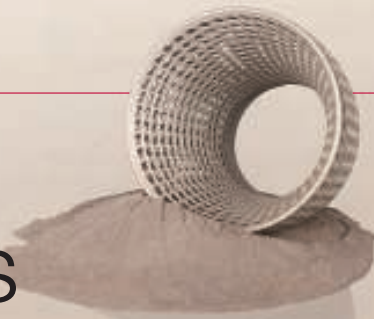
November 17, 2016 — Tesoro Corp. (San Antonio, Tex.; www.tsocorp.com) will acquire Western Refining, Inc. (El Paso, Tex.; www.wnr.com) in a transaction representing an equity value of \$4.1 billion. The transaction adds Western's petroleum refineries in Texas, New Mexico and Minnesota to Tesoro's existing refineries, resulting in a refining capacity of over 1.1 million bbl/d.

Total and Corbion form bioplastics JV

November 16, 2016 — Total S.A. (Paris; www.total.com) and Corbion (Amsterdam, the Netherlands; www.corbion.com) are creating a 50-50 bioplastics joint venture to produce and market polylactic (PLA) polymers. The two partners plan to build a PLA polymerization plant with a capacity of 75,000 m.t./yr at Corbion's site in Thailand. The JV will be based in the Netherlands and will launch operations in the first quarter of 2017. ■

Mary Page Bailey

3-D Printing for Finished Products



Moving 3-D printing beyond prototyping toward production of finished parts has driven innovation and spurred investment, but has also forced the industry to address a host of challenges

IN BRIEF

REALITY REPLACES HYPE

DESIGNED FOR 3DP

3D-PRINT SIMULATION

METALS AND QUALITY

SPEED AND POST-PRODUCTION

Industrial use of 3-D printing (3DP; also known industrially as additive manufacturing; AM) for research and development (R&D) projects and prototyping is widespread, but the focus of this potentially disruptive technology has shifted toward utilizing 3DP as a manufacturing platform to produce finished parts. Fully realizing the benefits of 3DP in the manufacturing space requires the industry to address a number of challenging questions about equipment capabilities, material properties and about the integration of 3DP into wider industrial processes.

"3-D printing is already a key R&D and prototyping tool, but a fundamental shift is ongoing right now to use it for the production of final parts," explains Terry Wohlers, a 3DP industry analyst and consultant with Wohlers and Associates (Fort Collins, Colo.; wohlersassociates.com). "This is where the most significant financial benefits are found.

3DP technology continues to advance in terms of printing equipment, availability of engineered raw materials, and software for designing and building 3D-printed components. Along with these advancements has come investment from companies seeking to position themselves to either supply the 3-D printing industry or to take advantage of the benefits offered by this manufacturing approach, which include design freedom, mass customization and the ability to reduce or eliminate assembly. Although the 3DP industry remains relatively small (estimated \$5.2 billion in 2015, according to the *Wohlers Report 2016*), it is growing rapidly (26% compound average growth rate in 2015; anticipated growth to \$26 billion by 2021, according to the report), and a number of indicators are signaling that 3DP is entering mainstream industry, including investment from both the public and private sectors.

Reality replaces hype

As AM's objective crystallizes around production of final parts, the frothy hype sur-

rounding industry a few years ago has given way to an optimistic, but sober reckoning of key real-world issues: chief among them are how to ensure the safety, quality and consistency of 3D-printed components and how to integrate AM equipment and digital attributes into broader manufacturing processes.

"For many industries, AM offers huge potential benefits, but as you move from prototyping to production, there are many questions that need to be considered carefully in order for these potential benefits to be made real," explains Chris Chung, head of strategic development and external affairs at Lloyd's Register (LR; Singapore; www.lr.org). "We need to get a good handle on all essential variables for AM processes, and develop standards for systems and personnel, so that there is a methodology to producing a series of AM-printed products, and the effectiveness of the printed parts can be demonstrated," says Andy Imrie, LR business development and global product launch manager. "We want to make sure that AM enters the mainstream in a safe way."

In addition to ensuring the quality and consistency of final parts from 3DP, the industry must also address questions of reliability and repeatability of the process itself. "When you are talking about mass production of finished parts, machine uptime is a critical aspect," says analyst Wohlers, as are the supporting processes, equipment and systems upstream and downstream of the printer. "The overall process steps need to be streamlined and automated for 3DP manufacturing processes," including powder handling before, for example, and surface finishing after. "Companies are still figuring out how to do this," he says.

In October 2016, the LR Foundation (www.lrfoundation.org.uk) released a "Roadmap for Additive Manufacturing," a consultation document that outlines the work needed to safely implement AM technologies for commercial production of final parts, especially in safety-

critical areas. Because the roadmap was created under the purview of the not-for-profit LR Foundation, it is in a position to examine longer-term, industry-wide concerns that individual companies might not consider, Chung and Imrie explain. The roadmapping process identified four principal challenges that the AM industry must resolve, including qualification of AM technology, supply-chain standards, workforce development and realizing safety benefits.

Designed for 3DP

A significant portion of 3DP technology is subject to patent protection, so in some cases, raw material costs are high where equipment vendors produce proprietary materials for use with specific printers. But the industry is gradually moving toward a freer model, where printer consumables can be made by third-party producers. The chemical process industries (CPI) have become involved in developing and supplying raw materials specifically for use in 3-D printers.

Polymers designed for use in 3DP require different properties than those for injection molding, because engineers need to consider the nature of the process, in terms of rheology, and the stability of the polymer at high temperatures. Temperature history is more important in 3DP due to the significantly longer cycle times, and while the pressure is generally ambient in 3DP, injection molding occurs at high pressures, controlling the packing and dimensional stability of the final part.

“To engineer a polymer specifically for use in 3DP applications, you have to think in parallel about the polymer chemistry and the equipment in which it will be used. For the material, you need to focus on the thermal management of the process system (that is, how heating and cooling are accomplished),” says Dominique Giannotta, Solvay’s manager of Sinterline nylon products for Solvay S.A. (Brussels, Belgium; www.solvay.com). Three general approaches exist for developing a polymer for 3DP, Giannotta explains: modify the material to work with existing equipment; start with the material and design equipment that works for that material, or a blend of

the two. Solvay is working mainly on the latter two approaches, he says.

For example, Solvay was a pioneer in developing the high mechanical- and thermal-performing range of polyamide-6 (PA6) powders, known as Sinterline, which are specially engineered for selective laser sintering (SLS), a prominent 3DP technology class (for more on 3DP techniques, see *Chem. Eng.* February 2015, pp. 20–23).

Solvay is joined by BASF, Eastman, Evonik and others in developing raw materials specifically for 3DP.

3D-print simulation

Solvay’s Sinterline PA6 is involved in a project that is an example of how 3D-printed finished parts could be developed. Specifically, the project, known as Polimotor 2, aims to demonstrate operation of a fully plastic internal combustion engine for a racecar. Solvay’s Sinterline material was used to 3D-print an air-intake plenum for the engine. For Solvay, the inclusion of a 3D-printed part is helping to change the prevailing mindset about the performance possibilities for such components, Giannotta says.

The Polimotor project is also illustrating the importance of simulation software in understanding the performance and properties of a plastic component before it is made. “Simulation in 3DP is improving in its ability and increasing in its importance, as companies are adapting simulation techniques currently used for injection-molded parts to model 3D-printed parts,” Giannotta says. “We are now assembling a raw-material database to allow designing a finished part from the beginning that will be able to withstand the demands of the application,” he remarks.

The Polimotor project represents the first time that Solvay’s new simulation tool (known as MMI Technyl) was used along with the Sinterline PA6 material to produce a 3D-printed final part. “In this case, experimental testing of the plenum confirmed what was indicated in the simulation work,” Solvay’s Giannotta explains, and the simulation helped to answer questions about how the part met engine specifications. “Simulations offer the ability to save time and money for experimental testing, and also allow the



FIGURE 1. Carbon’s CLIP process addresses speed and finish issues in 3-D printing of final parts

ability to modify the design prior to the start of production,” he says.

Metals and quality

Alongside engineered plastics, “the use of 3DP for manufacturing finished metal components is a super-hot area right now,” says Wohlers, and improved materials are becoming available. For example, Alcoa (Pittsburgh, Pa.; www.alcoa.com) recently opened a state-of-the-art facility for manufacturing metal powders specifically engineered for 3DP. Located at Alcoa’s Technology Center near Pittsburgh, the facility will produce proprietary titanium, nickel and aluminum powders optimized for 3D-printed aerospace parts. Alcoa also has invested in a range of technologies to further develop AM processes, product design and qualification, the company says.

Because product quality and consistency are critical in metal part production, the atmosphere inside the build chamber becomes very important. Many 3DP processes use high-purity inert gases, such as argon or N₂, to control the influence of oxygen and water during the fabrication of the part. However, the presence of traces of O₂ and humidity, due to incomplete purging, machine leakages, or from the metal powder itself, can negatively affect mechanical properties or chemical composition of the end product — for example, leading to oxidation of the metal, decreased fatigue resistance and sub-optimal performance. Industrial gas companies are recognizing this need and getting involved to support 3DP processes.

At European tradeshows last Fall, Linde Gases (Munich, Germany; www.linde.com) launched ADDvance O₂ precision, a first-of-its-kind measuring and analysis unit that enables metal additive manufacturers to analyze and control more precisely the level of O₂ and humidity within the printer chamber, the company says.

The new technology — developed in response to a need identified by aerospace company Airbus Group Innovations — can detect O₂ levels up to 10 parts per million (ppm) within the printer chamber and then modify the gas atmosphere by adjusting the level of argon or nitrogen. ADDvance O₂ allows for more accurate detection of oxygen and humidity without cross-sensitivity to other species in the complex mixture of impurities.

“Handling powders can also introduce some humidity, and this is known to increase porosity in the final part,” explains Pierre Foret, of Linde Gas. “ADDvance O₂ allows manufacturers to accurately and reliably measure oxygen in the build chamber and lower the levels by adjusting inert gas flow,” he notes. Currently with the instrument, a small pipe is installed on the printer and sucks a sample of gases from the build chamber and analyzes it externally for O₂ and H₂O content, Pierre explains, but in the future, the sensor should be integrated into the print chamber.

The ADDvance O₂ launch comes on the heels of Linde’s recent opening of a dedicated industrial gases laboratory specifically for AM in Unterschleissheim, Germany. The focus of the facility is to study the effects of various atmospheric gases and gas mixtures on the different metal powders used in AM to optimize the various layering processes, Linde says.

Speed and post-processing

Two significant obstacles to widespread adoption of 3DP for manufacturing finished parts have to do with its speed, and the fact that 3D-printed parts often have to undergo post-production finishing steps in order to be suitable for use. If these steps are not integrated well or are costly, it may make the particular part uneconomical for certain applications. A number of approaches and technolo-

gies are aimed at improving speed and streamlining the downstream processing of 3D-printed parts.

In some laser-based 3DP technologies, for example, unfused material may have to be cleaned off in a post-production process. Linde Gas has developed a post-production cleaning technology that claims to be faster, more cost effective and more environmentally friendly than existing alternatives, such as power-washing and sand-blasting. Tradenamed CryoClean Snow, the method uses small particles of dry ice (solid CO₂) for cleaning. The particles are formed on-demand by feeding liquid CO₂ into a specially designed snow chamber to form very hard dry-ice particles. The particles are then propelled against the component surface using compressed air. Uses for CryoClean Snow in the context of 3D-printed parts include removal of unfused powder from laser-fused AM parts and removal of surface oxides from steel, aluminum and other metals.

Printing technologies are also evolving to reduce post-processing. Rize Inc. (Woburn, Mass.; www.rize3d.com) has developed a printing technology, known as augmented polymer deposition (APD), that the company says achieves zero post-processing (such as filing or sanding) of parts and generates no toxic fumes. APD allows the extrusion of Rize’s proprietary engineering-grade thermoplastic simultaneously with the jetting of functional inks that can change the properties of the thermoplastic. The printer is capable of jetting these functional inks with each voxel (3DP version of a pixel) of material, so parts can be stronger, and downstream processing steps can be eliminated, the company says.

Another factor limiting the use of 3DP technologies for finished parts is their relatively slow speed. “Current technologies require many hours to days to generate parts, which is acceptable for prototyping applications, but not for manufacturing of finished parts,” says Jason Rolland, vice president of material for Carbon (Redwood City, Calif.; www.carbon3D.com), a company that recently introduced a printer designed to address both speed and finishing

issues in 3DP.


When selecting materials and printing technologies, there are often tradeoffs between mechanical properties and surface finish. “Light-based technologies (such as stereolithography) offer the best choice for surface finish and resolution, but the parts have poor mechanical properties, especially when compared to injection-molded thermoplastics, explains Rolland. “Heat-based technologies [like fused deposition modeling and selective laser sintering] offer better mechanical properties, but are limited with respect to surface finish and resolution,” he says.

Carbon recently introduced a unique technology, known as continuous liquid interface production (CLIP; Figure 1) that the company says addresses significant current gaps. CLIP is a photochemical-based process in which ultraviolet (UV) light is projected through an oxygen-permeable window into a reservoir of liquid UV-curable resin. The resin solidifies in a specific shape according to a series of UV images projected through the window. The built part rises out of the reservoir as the UV images induce resin hardening.

The company has also developed resins with both UV- and thermal-cure components, which allow improved strength after triggering secondary heat-activated reactions after printing. Carbon’s printer, known as the M1, can make parts with high resolution, engineering-grade mechanical properties and surface-finish properties like those of injection-molded parts, the company says. CLIP is faster than current stereolithography technology, Rolland says, and it does not involve layering of material, which, when coupled with digital optics to define part geometry, results in a clean surface finish.

“By expanding the available formulation space to include thermally curable materials, we open up a much broader range of chemistries and mechanical properties than ever before,” Rolland says. ■

Scott Jenkins

 **Editor’s note:** For more information on mainstream investment in 3DP, the LR Roadmap, next-generation 3DP, potential application areas for the CPI and more, please view the expanded online version of this article at www.chemengonline.com.

Smart Flow Monitoring for Better Process Control

Tighter control, increased safety and versatility result from improved flow-measurement technology

Flow monitoring and control are very important in the chemical process industries (CPI) because data gathered by flow-measurement equipment can pinpoint areas where a process can be improved and may help predict where and when a process is likely to fail. While intelligent self-diagnostic flow instruments are essential for repeatable and accurate results, the information obtained via today's "smarter" flow monitoring and control instrumentation may also be used to achieve tighter control of the process. In addition, flow equipment advances increase safety in the facility, allow more versatility and application in difficult situations.

As the CPI face the challenges of having fewer engineers and technicians in their facilities and a large portion of the knowledge and skills are being lost to retirement, processors require "smarter" equipment. "In terms of flow measurement, this results in greater reliance on instrumentation to give more information beyond a flowrate. It means that devices need to be 'smarter' and easier to use," says Sam Hassan, product manager, flow products, Yokogawa (Sugar Land, Texas; www.yokogawa.com). "Also, there are more safety, environmental and internal regulations requiring compliance. All these factors call for flow instrumentation with more advanced diagnostics to ensure measurements are reliable, accurate and informing the facility of any potential issues."



FIGURE 1. The Rotamass Total Insight (TI) Coriolis product provides insight into the process and throughout the lifecycle. The TI concept provides enhanced settings for customized set ups, pre-defined trend views, or multiple configuration sets of fast changeover, data mobility, increased safety and operational flexibility

Smarter instruments

Hassan adds that while there are few new flow technologies being developed, existing technologies are being enhanced to provide not only a better measurement, but also more measurement points and much more in-depth and enhanced diagnostics. "Insight into the process" is becoming very important to end users, notes Hassan. "Functionality, such as meter verification, is becoming more important and is being supported on a wider variety of devices. Self-diagnostic capabilities now go beyond just monitoring the flowmeter itself, but also monitor the flow in more detail. Knowing what's happening in a process besides just the basic flowrate is becoming more of a requirement," he explains. "Information about the process fluid, such as concentration, non-filled pipes or even aeration in the liquid are all available. These are just some of the examples of important functionality that are allowing the industry to have better insight and providing better control of the process."

Yokogawa's Rotamass TI (Total Insight) Coriolis product (Figure 1) provides this

IN BRIEF

SMARTER INSTRUMENTS

TIGHTER CONTROL

ENHANCED SAFETY

INCREASED VERSATILITY

CHALLENGING APPLICATIONS



FIGURE 2. The Brooks SLA 5800 sealed, digital thermal mass-flow controllers and meters feature high accuracy, high zero stability and high repeatability. They are engineered with advanced, configurable multi-gas/multi-range capabilities

type of insight into the process and throughout the lifecycle. To facilitate optimal processes and increase the efficiency of personnel, the company has simplified fundamental operating concepts with Total Insight. The Total Insight concept is built into the latest generation of Rotamass transmitters and provides enhanced settings for customized set ups, pre-defined trend views, or multiple configuration sets of fast changeover, data mobility, increased safety and operational flexibility.

Rotamass's Process Guard feature manages advanced diagnostics. Nearly 200 different events can be dedicated and classified accordingly by the user. Events can also trigger data logging of the most important process and device variables. Sustainable and safe operation often requires continuous monitoring, especially of in-line meters. The Maintenance Manager function continually observes the key sensing elements and also allows a total health check during operation, minimizing interruption of processes.

Thomas McCulloch, senior global product manager, DP Flow Transmitters with Rosemount (Chanhassen, Minn.; www.emerson.com/en-us/automation/rosemount), a subsidiary of Emerson (St. Louis, Mo.; www.emerson.com) agrees that the modern, advanced diagnostic capabilities can now provide greater process



FIGURE 3. To better and more accurately control two-phase gas flows, Krohne's Entrained Gas Management (EGM) technology is available with the company's Optimass Coriolis mass flowmeters

insight. "Conditions like plugged impulse piping, wiring degradation and advanced warning of other problems can be detected and the information can be used to increase safety and efficiency by drastically reducing potential downtime."

He says Emerson's Micro Motion Model 5700 Coriolis flow transmitter has been designed to translate measurement data into meaningful insight and instruction. The transmitter also provides detailed measurement history for troubleshooting or optimizing the process. The measurement data are translated into operating insight through robust, time-stamped history files for process and meter health data, and logs for configuration changes and alarms. The result allows users to better leverage Coriolis measurement data in order to further understand and improve their operating environment and achieve new levels of productivity.

In addition to the assistance provided by advanced diagnostics, much of the information and process insight provided by this new, smarter generation of flow instrumentation can be used to provide more accurate information and tighter control of the process, as well as increased safety.

Tighter control

"One of the flow types that is really under scrutiny in today's chemical-processing environment is gas flow,

so this part of the process has to be monitored and controlled very carefully," says Howard Kramer, senior applications engineer with Brooks Instrument (Hatfield, Pa.; www.brooksinstrument.com). "Smart digital gas-flow control is necessary in these applications, as are the data the instruments provide. The more data you collect, the more likely you are to prevent product deficiencies, minimize failure and eliminate process shutdowns. Preventing issues such as these also helps to save downstream equipment, which is often affected by process anomalies."

For this reason, having a flow device that can not only compensate for inadequacies in the process, but also one that is capable of reporting upon the discrepancy is essential to avoiding process upsets. "The main benefit of reporting discrepancies is to know that there's a problem before the problem actually exists," says Kramer. "A smart thermal mass-flow controller is a great product for this application because it provides precision, as well as valuable information. The precision and compensation it provides can directly impact the process and it will show you that there's an anomaly that needs attention to prevent failure and the need for compensation in the future."

The Brooks SLA 5800 sealed, digital thermal mass-flow controllers and meters (Figure 2) feature high

accuracy, high zero stability and high repeatability. They are engineered with advanced, configurable multi-gas/multi-range capabilities. "Onboard control electronics control flow without any external input, except for user programmed commands," says Kramer. "This means users tell the mass flow controller what to do and it will not only provide the flow rates, but also compensate for any inadequacies in the process, such as by opening or closing a valve to compensate for a change in upstream pressure. This is a big deal because it means that the process can continue, but also because the instrument will alert the user to the fact that it had to compensate for an upstream issue. As soon as the user knows there's an anomaly in the process, he or she knows there is an issue that needs attending in order to keep the process going, which is a huge benefit in the chemical industry."

Also helping to better and more accurately control gas two-phase flows is the Entrained Gas Management (EGM) technology available on Krohne (Duisburg, Germany; www.krohne.com) Optimass Coriolis mass flowmeters (Figure 3), says Ralf Haut, industry manager for chemicals with Krohne. With the additions of Optimass 3400 and 7400, the entire series of instruments offers continuous and repeatable mass-flow or density measurement with two-phase flows such as liquids mixed with gas, slurries with gases or highly viscous fluids with gas entrainments. In the past, this presented a huge challenge for mass flowmeters: without gas entrainments, the measuring tubes in the Coriolis mass flowmeters have the desired regular oscillation. Gas entrained in the liquid dampens this and, as the gas content increases, it can come to a complete stop. To overcome this, Krohne developed control algorithms that allow the meter to maintain oscillation and continue to measure even with complex flow conditions. This is possible even during a complete transition from a pure liquid phase to a gas phase and back, allowing mass flow and density measurements to remain stable, continuous and repeatable. "This technology allows robustness and an advanced level of process control," says Haut. "The instrument won't stop working, production is more efficient and the output may increase, which all means the profit at the end of the process will also be greater, thanks to better control of the flow."

Enhancing safety

"In the chemical industry, many products are hazardous and processes use high temperatures and high pressures, so they must be properly monitored and controlled," says Art Womack, regional manager with Fluid Components International (FCI; San Marcos, Calif.; www.fluidcomponents.com). He says this mandates that processors use equipment that controls the process but also maintains certain safety limits demanded by internal processes, as well as by government standards. "Many applications and equipment are currently in place because they are a safe and reliable technology, so the focus becomes being able to demonstrate that the system meets a certain risk reduction factor and that equipment complies with current safety regulations," says



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FIGURE 4. With Endress+Hauser's Heartbeat Technology, the new Proline generation of flowmeters offers diagnostic coverage from the measuring tube to the outlet. This ensures a low residual risk of a passive protection failure

Steven Craig, international sales manager with FCI.

One of the most prevalent standards related to flow equipment is SIL safety, as outlined in various IEC standards, which look at the failure rate of equipment and manufacturing techniques of the instrument to ensure safety in applications. Process and plant engineers with operations that require IEC 61511 compliance can employ FCI's ST51A and ST75A thermal mass flowmeters for air/gas flow measurement applications. Both meet SIL 1 compliance standards. ST51A is an insertion-style flowmeter designed for use in pipe diameters larger than 2 in. and is optimized for flow measurement of air, compressed air, nitrogen, natural gas, digester gas and other biogases. ST75A is an inline instrument designed for pipe diameters from 0.25 to 2 in. for applications in air, all inert gases, natural gas and other hydrocarbon gases.

Nathan Hedrick, flow product marketing manager with Endress+Hauser (Houston; www.us.endress.com) agrees that safety is a challenge and that compliance with current industry ratings provides flow equipment users with the assurance they need that their instruments will operate reliably and safely in their applications. "Quality is a pre-requisite of both safety and availability. There must inherently be quality in the

design and manufacture of a device that follows good engineering practices and international metrology standards," he says. "Without this, a chemical processor cannot have confidence in the ability of the device to produce an accurate and reliable measurement, to operate as required in a safety critical application or to ensure maximum plant and process availability."

For safety applications, flowmeters must meet the highest requirements in terms of reliability. With E+H's Heartbeat Technology, the new Proline generation of flowmeters (Figure 4) offers diagnostic coverage from the measuring tube to the outlet. This ensures a low residual risk of a passive protection failure. Traceable factory calibration and redundant internal references complement the safety-by-design principle with minimal failure rates in accordance with IEC 61508.

In chemical and petrochemical companies, flowmeters must ensure maximum reliability. Although costly, proof testing is the established method for validating functional reliability. Heartbeat Technology provides the basis for minimizing the effort and exposure of personnel involved in these routine activities.

Increased versatility

In the real world, process changes come via plant upgrades, retrofits

and expansions. According to Dan Cychosz, global product management, DP Flow products with Rosemount, these can change flowrates and required material compositions from original specifications for both process and utility applications. "Using a flow instrument that has the flexibility to accommodate these changes without replacement reduces the cost of these projects," he says. "It also has the day-to-day benefit of providing accurate flow measurements during unintended changes such as process upsets."

Technological advancements such as Emerson's Rosemount Ultra for Flow allow traditional DP flow installations to accommodate a much wider range than older technologies. Rosemont's McCulloch says: "Being able to have a transmitter with an accuracy specified as a percent of reading is crucial in many applications. Users can standardize on one product for a variety of applications because this approach extends the range of accuracy over a broader turndown ratio. This helps customers eliminate the need to change out primary elements due to seasonal flow changes or eliminates the need for stacked transmitters due to varying flowrates."

Jesse Arenstein, lead applications engineer and engineering product manager with Alicat Scientific (Tucson, Ariz.; www.alicat.com) agrees that flow needs in the chemical industry change often, making it necessary to program gas mixtures on the fly. In an effort to eliminate what is sometimes called "disposable flow instruments" that don't accommodate changes, Alicat offers Gas Select Composer onboard firmware for its mass flowmeters and controllers. Composer is a gas-composition feature that allows users to define up to 20 custom gas compositions with up to five constituent gases per mix. Setting Gas Select to the appropriate gas that is flowing guarantees the NIST-traceable accuracy of measurement for that gas. The Gas Select functionality precludes the need for correction factors and also allows the user to change between gases, without needing to change

the full-scale range of the measurement device.

Challenging applications

"The chemical industry has challenges associated with hazardous and corrosive fluids, as well as high pressures and high temperatures, so finding flow instrumentation that contains all stainless-steel wetted components or exotic materials is important in these challenging applications," notes Mike Iverson, general manager at AW-Lake (Oak Creek, Wis.; www.aw-lake.com). In addition, he says, many users in the chemical and oil-and-gas industries are interested in the flexibility and reliability of Coriolis flowmeters, but the high pressures in some of these applications may have prevented use of this technology in the past. However, the company introduced the new high-pressure TCMH 0450 Coriolis flowmeter for high-pressure chemical injection applications, as well as gas-measurement applications. It

is available in three different pressure ratings: up to 15,200, 10,000 and 6,000 psi.

Another technology that is gaining popularity in the CPI because of its ability to be used in challenging processes is non-invasive ultrasonic transducers for mass flowmetering applications. PIOX S from Flexim GmbH (Berlin, Germany; www.flexim.com) measures from outside of the pipe. And, since the ultrasonic transducers are mounted onto the outside of the pipe wall, the measuring system is not exposed to any wear and tear by the medium flowing inside, allowing it to operate virtually maintenance-free. In addition to that, there is no need to open the pipe when installing the system, which means there is no production, processing or supply.

Bas Tammen, global project and international key accounts manager, with Flexim, says: "Due to these advantages, the use of this flowmeter is gaining popular-

ity when dealing with highly corrosive or toxic media and also in challenging applications, such as those with large diameter pipes. In many of these applications, we can reduce the cost of flow monitoring because clamping on our flowmeters does not require the use of special materials since we don't have contact with the fluid and we don't have to stop a process to install our technology."

He says this is allowing users to measure flow in areas that might have been expensive or dangerous to install metering technologies in the past. Some applications might include high-pressure processes in LDPE (low-density polyethylene) production and measurement of corrosive liquids such as sulfuric acid. The company has recently partnered with Emerson to bring its technology to more customers in these and other industries looking for a lower cost solution to measuring flow in challenging applications. ■

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Vertiflo Pump



This sump pump ensures unrestricted flow

The Series 900 Industrial Vertical Immersion Vortex Sump Pump (photo), available for service in industrial and municipal applications, provides an unrestricted flow since the impeller is not in contact with the solids being pumped. Applications include chemical slurries, fragile food-processing solids, pulp-and-paper solids, petroleum products, wastewater, sewage and more. It is designed for long life in severe services with heads to 170 ft., temperatures to 350°F, pit depths to 26 ft deep, and flows to 1,600 gal/min. Several materials of construction are available. — *Vertiflo Pump Co., Cincinnati, Ohio*
www.vertiflopump.com



ITT Pro Services

This pump has a dry-running, frictionless sealing system

The TCC DryRun submerged pump has a bearing and sealing unit that has no contact with the pumping medium. The axial bearing preload ensures sufficient minimum load without any hydraulic force when the impeller is not immersed in liquid, says the company. The pump's modular design provides operating flexibility; for instance, the basic version ensures an immersion depth of up to 2.40 m; by installing intermediate bearings, immersion depths of up to 5.50 m can be achieved. The pump has relatively low installation and maintenance costs and a maintenance interval of 3–5 years. — *Paul Bungartz GmbH & Co. KG, Düsseldorf, Germany*
www.bungartz.de



Milwaukee Tool

process insight and diagnostic data without the need for external sensors. This helps operators to optimize pump control strategies and reduce system design and operating costs, says the company. — *ITT Pro Services, Seneca Falls, N.Y.*
www.itt.com

Cordless, self-priming pump is designed for water removal

The M18 Transfer Pump (photo) is both cordless and self-priming. It pumps up to 480 gal/h and its flexible impeller helps to produce a powerful pressure differential that can produce 18 ft of lift and 75 ft of head, says the company. Its viewing window helps operators avoid blockages. Its cordless design eliminates the need for a.c. power and cords, while its self-priming design helps to eliminate steps during setup. Equipped with Redlink Intelligence, the Transfer Pump switches off when the application is complete and the tool begins running dry, protecting the motor. — *Milwaukee Tool, Milwaukee, Wis.*
www.milwaukeetool.com

This pump combines gentle handling and low energy use

The SRU rotary-lobe pump (photo) is designed to provide smooth, low-shear pumping and good energy efficiency. Its design minimizes the risk of contamination, making it well-suited for applications in the dairy, food, beverage, personal care, biotechnology and pharmaceutical industries that require hygienic, low-shear and low-pulse operation. The pump has clean-in-place and sterilize-in-place capabilities to support operators' rigorous validation requirements, says the company. — *Alfa Laval, Richmond, Va.*
www.alfalaval.com



Alfa Laval

Get realtime insight on pump flow, head and efficiency

The PumpSmart PS220 (photo) provides intelligent variable-speed pump control and protection for single and multiple pumps. It helps to improve pump reliability and safety by protecting against upset conditions, such as dry run, minimum flow and cavitation. It uses so-called Advanced Sensorless algorithms to critical performance parameters such as flow, head and efficiency in realtime, and provides valuable

Diaphragm pumps operate on natural gas or compressed air

This company's recently expanded line of Husky 1050 natural gas-operated, 1-in. (25-mm) double-diaphragm pumps (photo, p. 23) operate safely on natural gas or compressed air. They are designed

for the midstream and upstream oil-and-gas markets. These pumps can transfer low-, mid- and high-viscosity fluids, and are designed to withstand deadhead and dry-run conditions with no damage to the pump or surrounding equipment, says the manufacturer. They provide flowrates to 50 gal/min (379 L/min) and pressures to 100 lb/in² (6.9 bars). — *Graco, Inc., Minneapolis, Minn.*

www.graco.com

Miniature diaphragm gas pumps are versatile for OEMs

The Boxer 20K miniature diaphragm pumps, the KPV miniature gas vacuum pumps, and the KPMS (Square) miniature gas pumps combine small size, light weight and low cost. They are available to address the needs of original equipment manufacturers (OEMs) producing various analyzers, air-sampling devices and other monitoring devices. These miniature pumps range in length from 12 mm (0.47 in.) to 55 mm (2.2 in.). — *Clark Solutions, Hudson, Mass.*

www.clarksol.com

Progressing-cavity pump offers easy access for maintenance

The NEMO progressing-cavity pumps (photo) are known for their continuous, gentle and low-pulsation conveyance of almost any substance, including sludge. The FSIP (full service-in-place) version of the NEMO progressing-cavity pump (photo) gives operators full access to the pump's rotating parts, allowing them to open the pump cavity onsite, dismantle all rotating parts and install them again without removing the pump from the pipe assembly. This helps to reduce installation and maintenance time. The new iFD-Stator 2.0 features a two-part, reusable stator housing and replaceable stator. Compatible with all NEMO NM Series progressing-cavity pumps, the iFD-Stator 20 helps to reduce energy and maintenance costs, says the company. — *Netzsch Pumps North America LLC, Exton, Pa.*

www.pumps.netzsch.com/en

Robust air-driven liquid pump enjoys long lifespan

The AHL118 is a high-volume, double-ended, double-acting high-pressure pump (photo) that is designed for oil-and-gas, chemical, industrial and research applications. The pump

operates to pressures up to 23,000 psi and delivers 5.6 gal/min (25.5 L). The AHL118 is designed to be extremely robust, with an unscratchable, carbon-based coating on the plunger, making it three times harder than Stellite, according to the manufacturer. This, coupled with hydraulic parts that are manufactured from stainless steel, helps to extend the lifespan of the seals while reducing downtime, repairs and servicing, helping to improve mean time between maintenance (MTBM) and increasing safety. — *Parker Autoclave Engineers, Instrumentation Div. of Parker-Hannifin Corp., Erie, Pa.*

www.parker.com

Intelligent metering pump meets demanding standards

The Excel XR Intelligent Metering Pumps (photo) are designed to provide accuracy, reliability and process-control flexibility in a wide array of industrial and municipal water- and wastewater-treatment applications. They are available in two control configurations (manual and enhanced). They can communicate with digital or analog devices to administer a variety of control schemes, such as pulse input, timed events, or batch processes. Remote connectivity enables control and feedback in realtime. These pumps feature a backlit color display that allows for navigation in five languages. A series of training videos are available online. — *LMI, Iyland, Pa.*

www.lmipumps.com

Latest addition extends the flowrates in this pump family

The Qdos suite of high-accuracy chemical-metering pumps now includes the Qdos 120, which offers flows up to 31.7 gal/h at a maximum pressure of 58 psi, which is double the flow of existing Qdos peristaltic metering pumps. The new Qdos 120 has been designed to deliver continuous, smooth flow for optimal fluid mixing; its high accuracy provides savings and increased process quality. It has no valves or seals to clog, leak, gas-lock or corrode. The only consumable part on Qdos 120 pumps, the ReNu pump-head, can be replaced in 60 s to return the pump to its as-new state, says its maker. — *Watson-Marlow Fluid Technology Group, Wilmington, Mass.*

www.wmftg.com

Suzanne Shelley



Netzsch Pumps



Parker Autoclave Engineers



LMI

Endress+Hauser



This data-acquisition system is updated for EtherNet/IP

The Memograph M RSG45 Data Manager (photo), a data-acquisition system for small process-control applications, now features Ethernet/IP capability, providing a cost-effective way to integrate legacy 4–20-mA, RTD, thermocouple and HART-based instruments into a modern control system. The RSG45 can quickly integrate up to 20 individual instruments into an EtherNet/IP environment using a single cable. The EtherNet/IP connection also allows a plant to control batch start and stop activities from the control room. The RSG45 is able to accept up to 14 discrete and 20 universal/HART analog inputs from process sensors. It can then display this sensor data on its 7-in. multicolor screen, record the data internally, perform calculations and alarm checks and transmit the data to a PC or any control system. — *Endress+Hauser Inc., Greenwood, Ind.*

www.us.endress.com/rsg45



NEO Monitors

Compact combustion monitor based on TDL technology

The LaserGas III CO combustion analyzer (photo) is an Ex-d certified tunable-diode-laser (TDL) carbon monoxide (CO) analyzer that also measures methane, water and temperature. Suitable for combustion processes where increased explosion safety is a requirement, this analyzer is said to be the most compact CO combustion monitor on the market. Typical applications that can benefit from this device are fluid catalytic cracking (FCC) units, package boilers, process heaters, electrostatic precipitators, waste-gas recovery, incineration and so on. — *NEO Monitors, Skedsmokorset, Norway*

www.neomonitors.com



QED Environmental Systems

Air stripping for water decontamination

The E-Z Tray air stripper (photo) is a sliding-tray, stainless-steel air stripper used for removing volatile organic compounds (VOCs) from contaminated groundwater and waste streams. E-Z Tray's design achieves high removal efficiencies in an easy-to-maintain process unit that is readily accessible for process monitoring



Eisele Pneumatics

and inspection, even while in operation. The stripper is not prone to fouling and can be cleaned by one person with a simple pressure wash. It also offers a wide turndown range. As contaminated groundwater enters through the top of the E-Z Tray air stripper, millions of air bubbles are forced by blower pressure up through the perforated trays. This creates a turbulent froth zone with an extremely high air-to-liquid surface area for mass transfer of VOCs from liquid to air. Using the froth instead of a conventional tower packing delivers high VOC-removal efficiencies, even under fouling conditions. — *QED Environmental Systems, Ann Arbor, Mich.*

www.qedenv.com

Hygienic ball valves constructed from stainless steel

This company now offers its simple and robust hygienic ball valves with stainless-steel construction (photo), making them easy to flush and clean for compressed air, gases and liquids in the food processing industry. The ball valves were originally developed for use in painting stations, and are therefore completely free of dead zones and easy to clean. These qualities also make them suitable for hygienic applications in food processing. The ball itself is permanently enclosed and sealed by two polytetrafluoroethylene (PTFE) half-liners. The inside of the ball valve and the ball seats are completely free of dead zones and micro-machined to minimal roughness. The stainless-steel ball valves have 1/4-in. threads on both ends. This provides a dead-zone-free interface to the company's FreeLine connectors or enables direct mounting in installations and lines. — *Eisele Pneumatics GmbH & Co. KG, Waiblingen, Germany*

www.eisele.eu

Simplify instrumentation management in any location

The Field Information Manager 1.1 Handheld Edition software enables users to do configuration, parametrization and diagnosis of HART instruments in many locations — in the field, at the back of the panel or junction box or in the instrumentation laboratory. The Field Information Manager can be

installed on any Windows tablet, laptop or computer, meaning that multiple instrumentation programs can be installed on a single machine. It is very quick to install and saves time for instrument and service technicians. The Field Information Manager Store and Print device configuration allows easy transfer of parameters from one device to another and eliminates manual recording of parameters. — *ABB, Zurich, Switzerland*
www.abb.com/fieldinfo

Comprehensive protection for laboratory workers

Westex ShieldCXP is a protective fabric (photo) that defends the wearer against hazards from both flames and inadvertent chemical splash, providing laboratory workers with a single-layer protective solution. Interactions with hazardous chemicals and flames are frequent in laboratories, so an effective protective coat must be properly worn to serve as a solution to multiple risks. Because other fabrics may only offer



Milliken & Co.

flame resistance without protection against toxic or corrosive chemicals, users often are required to layer separate garments. Westex ShieldCXP is capable of both flame and chemical resistance, ensuring proper protection and simplifying purchasing decisions for laboratory managers. — *Milliken & Co., Spartanburg, S.C.*
www.milliken.com

This app streamlines commissioning and startup

The eStart mobile app (photo) is used during project commissioning and startup to digitally gather data and create loop folders to house all relevant instrument data and check sheets.



Maverick Technologies

Digital folder creation not only greatly reduces documentation time, but also keeps all electronic data at the technicians' fingertips. In addition, eStart's Near Me feature uses GPS technology to locate nearby instruments and identify in which stage of commissioning they are. Visibility within commissioning projects is crucial, and with eStart, every time a step is completed, the app automatically records the technician's name, date and time, providing realtime updates. This allows users to proactively make adjustments to get the project back on track. — *Maverick Technologies, Columbia, Ill.*
www.mavtechglobal.com

Mary Page Bailey and Gerald Ondrey

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Solids-blending processes

Department Editor: Scott Jenkins

Mixing and blending of bulk solids is common in pharmaceutical and food manufacturing, specialty chemicals, explosives, fertilizers, glass and ceramics, detergents and resin industries. This column provides information on batch versus continuous blending mechanisms.

Batch versus continuous

Batch blending processes typically consist of three sequential steps: weighing and loading blend components; mixing; and discharge of the blended product. In a batch blender, solids motion is confined only by the vessel, and directional changes are frequent. The retention time in a batch blender is carefully controlled, while for a continuous blender, this is generally not the case. Blending cycles can take from a few seconds with high-intensity units to 30 min or more where additional processing, such as heating or cooling, may be involved. Blender discharge may be rapid or take substantial time, particularly if the blender is used as a surge vessel to feed a downstream process.

In a continuous blending process, weighing, loading, blending and discharge steps occur continuously and simultaneously. Blending occurs during transport of the material from the in-feed point toward the mixer outlet. Unlike batch blenders, where product retention time is carefully controlled, material retention time with continuous blenders is not uniform and can be directly affected by blender speed, feedrate, blender geometry and design of internals (see Table 1).

Blending mechanisms

There are three primary blending mechanisms: convection, diffusion and shear.

Convective blending. Convective blending involves gross movement of particles through the mixer, either by a force action from a paddle or by gentle cascading or tumbling under rotational motion.

Diffusion blending. Diffusion is a slow blending mechanism and will pace a blending process in certain

TABLE 1. BATCH VERSUS CONTINUOUS BLENDING	
Batch processes are used when:	Continuous processes are used when:
Quality control requires strict batch control	A continuous, high-production-rate process is required
Ingredient properties change over time	Strict batch integrity is not essential
The blender cannot be dedicated to a specific product line	Combining several process streams
Production quantities are small	There is a need to smooth out product variations
Many formulations are produced on the same production line	
Advantages of batch processes over continuous	Advantages of continuous processes over batch
Lower installed and operating costs for small to medium capacities	Ease of equipment integration into continuous processes
Lower cleaning costs when product changes are frequent	Less opportunity for batch-to-batch variation caused by loading errors
Production flexibility	Automation can improve quality and reduce labor costs
Pre-blending of minor ingredients is easily accomplished	Higher throughputs are often possible
Control of blending time	

tumbling mixers if proper equipment fill order and method are not utilized.

Shear blending. The shear mechanism of blending involves thorough incorporation of material passing along high-intensity forced slip planes in a mixer. Often these mixers will involve dispersion of a liquid or powdered binder into the blend components to achieve granulation.

Random versus ordered blend

There are two types of blend structures: random and ordered. A random blend occurs when the blend components do not adhere or bind with each other during motion through the blend vessel. In this case, dissimilar particles can readily separate from each other and collect in zones of similar particles when forces such as gravity, airflow or vibration act on the blend.

Ordered or structured blends are more common in industrial processes. This occurs when the blend components interact with one another by physical, chemical or molecular means and some form of agglomeration or coating takes place. The process of granulation involves this approach, whereby larger particles are created from smaller building-block ingredient particles, and each "super" particle has ideally the correct blend uniformity. A blend of perfect superparticles of identical size will not segregate after discharge

from the blender, which is clearly an advantage over a random blend.

Segregation and sampling

If particles are not mono-sized, then segregation by size may occur and induce problems with bulk density, reactivity or solubility in post-blend processing. There are cases where some ingredients have a tendency to adhere only to themselves, without adhering to dissimilar ingredients. This often happens with fine materials, such as fumed silica, titanium dioxide and carbon black. At times, a blend can reach "saturation," where minor fine components will no longer coat larger particles, and concentrations of the fine component will build (and segregate from the blend).

With achieving a uniform blend as the goal, defining uniformity strongly depends upon the scale. For instance, loading two components into a tumble blender does not guarantee blend uniformity across the range of sample sizes. If the entire quantity in the blender was analyzed, then uniformity may be present. However, taking smaller samples from either side of the blender will result in substantial differences, which clearly does not meet uniformity requirements. ■

Editor's note: This column was adapted from the following article: Maynard, E., "Fundamentals of Bulk Solids Mixing and Blending," *Chem. Eng.*, September 2013, pp. 66–71.

Technology Profile

Ethylene Glycol Production from Synthesis Gas

By Intratec Solutions

Ethylene glycol, also known as monoethylene glycol (MEG), is a major chemical commodity, widely used in the production of polyethylene terephthalate (PET) bottle-grade resins and polyester fibers. These materials, in turn, are used to manufacture textiles, soft drink and water bottles, tire cords and more.

MEG was first synthesized via the hydrolysis of ethylene glycol diacetate. Now, it can be made from multiple raw materials, such as coal, natural gas and ethylene. Globally, it is mainly produced from ethylene via an ethylene oxide intermediate. This process generates di- and tri-ethylene glycol along with MEG.

The process

In the process described here, ethylene glycol is produced from synthesis gas (syngas), a gaseous mixture of carbon monoxide (CO) and hydrogen (H₂). CO is first converted to dimethyl oxalate (DMO), which is then hydrogenated to form ethylene glycol (Figure 1).

Carbonylation. The CO and H₂ in the feed syngas are separated. The recovered CO is fed to the carbonylation reactors along with a recycled stream from the nitrite regeneration section (discussed below) that contains an intermediate (methyl nitrite). Methyl nitrite reacts with CO to produce the intermediate DMO and nitric oxide (NO). The product from the carbonylation reactors is partially condensed, generating a gaseous stream, rich in unconverted CO and NO, and a liquid stream, rich in DMO. The former is directed to the nitrite-regeneration section, and the latter is directed to the DMO hydrogenation section.



FIGURE 2. This graph shows the yearly average prices for MEG in the U.S.

DMO hydrogenation. The DMO-rich stream is fed to the hydrogenation reactors along with H₂ recovered from the syngas feed. DMO reacts with H₂ to produce the final product, ethylene glycol and methanol. A few byproducts from undesired side reactions also form. The product stream from the hydrogenation reactors is partially condensed, and the condensate is directed to the purification section. Uncondensed vapor (mostly H₂) is compressed and recycled to the hydrogenation reactors.

Purification. The purification system consists of a series of distillation steps to separate fiber-grade ethylene glycol from methanol and other byproducts formed during DMO hydrogenation. Methanol is recovered from an intermediate distillation column and is recycled to the nitrite-regeneration section.

Nitrite regeneration. The recovered NO stream from the carbonylation section is mixed with O₂ and contacted in a reactive absorber with methanol, which is recycled from the purification section, as well as from a distillation column downstream. These chemicals

react to produce methyl nitrite and water. The top product stream from the nitrite reactor is partially condensed to remove most of its water and the resulting methyl-nitrite-rich stream is recycled to the carbonylation section. The reactor bottom product is directed to a water-removal distillation column.

Economic performance

Variable costs (raw materials and utilities) for manufacturing MEG from syngas in the U.S., using data from Q1 2013, are estimated to be \$600/ton of product. Historical yearly average prices for MEG are shown in Figure 2.

This column is based on "Ethylene glycol production from Syngas - Cost Analysis," a report published by Intratec. It can be found at: www.intratec.us/analysis/ethylene-glycol-production-cost.

Edited by Scott Jenkins

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 are prepared on the basis of publicly available and non-confidential information. The content represents the opinions of Intratec only. More information about the methodology for preparing analysis can be found, along with terms of use, at www.intratec.us/che.

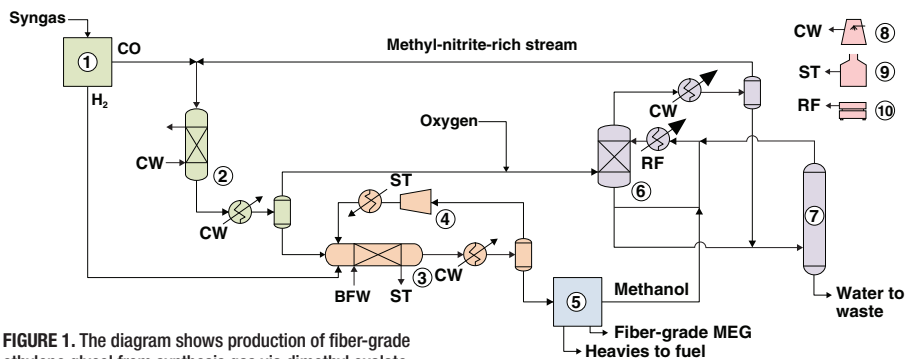


FIGURE 1. The diagram shows production of fiber-grade ethylene glycol from synthesis gas via dimethyl oxalate

Overcoming Corrosive Processes with High-Alloyed Stainless Steels

Advanced high-alloyed stainless steels with austenitic properties can address corrosion concerns in challenging phosphoric and nitric acid processes

Daniel Gullberg
Sandvik Materials
Technology

IN BRIEF

SELECTING
ANTICORROSIVE
MATERIALS

PHOSPHORIC ACID
PROCESSES

GRAPHITE VERSUS
METALLIC TUBES

THE EFFECTS OF HIGH
ALLOYS

ENVIRONMENTAL
CONTAMINANTS

SMALLER TUBE
DIMENSIONS

NITRIC ACID PROCESSES

CHROMIUM FOR NITRIC
ACID SERVICE

TESTING ALLOYING
ELEMENTS

RECOGNIZING HIGH-
CORROSION SITUATIONS

CLOSING THOUGHTS

The growing worldwide demand for fertilizer has created a significant increase in production levels. As a consequence, operators face mounting pressure to keep up with these trends, and this challenge is especially evident in the greater expectations that are being placed on materials for critical tubes and pipes in heat exchangers. Heat exchangers are essential to fertilizer applications. Not only must they ensure efficient heat transfer from one medium to another while minimizing resistance to fluid flow through the tubes, the heat exchangers should also remain inexpensive in terms of both design and maintenance.

This is where issues of corrosion set in, made worse by the fact that such problems may vary considerably from plant to plant depending on local service conditions. For these reasons, many materials traditionally favored by the industry — such as standard AISI 316L, 317L or 304L grades of stainless steel, depending on the application — may exhibit insufficient performance in acidic fertilizer processes. In order to solve these problems, it is extremely important to select the correct material for any given application. This article explains how advanced high-alloyed materials can offer an alternative to traditional material grades.

Selecting anticorrosive materials

Across all fertilizer applications, there are two primary considerations when choosing a re-

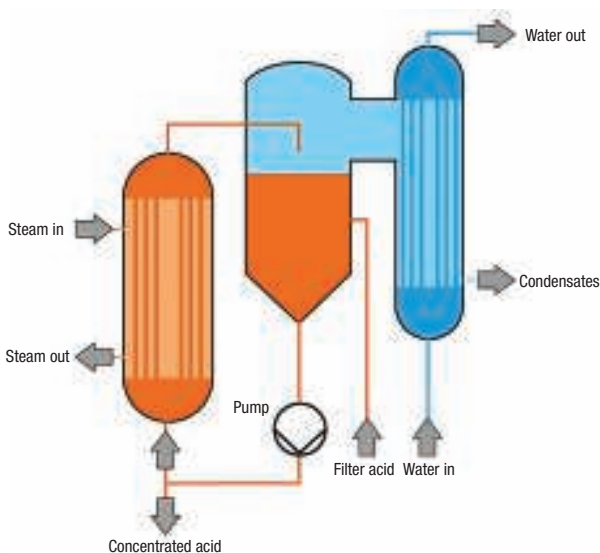


FIGURE 1. Forced-circulation evaporation is typically used in phosphoric-acid heaters for acid concentration

placement tube or pipe material — corrosion resistance and the presence of contaminants in the process stream.

First and foremost, processors must identify a material grade with an optimal content of chemical elements that promote good corrosion resistance. It is also important that levels of contaminants in the production environment are minimized, in order to avoid the formation of harmful segregations in the material's grain boundaries, which can initiate corrosion.

Phosphoric acid processes

It is estimated that around 90% of the phosphoric acid (H_3PO_4) manufactured worldwide is used for fertilizer production. Phosphoric acid is most commonly produced by extraction from rock phosphate (phospho-

rite) using “wet” process techniques. The wet method is favored above more expensive thermal processes, and entails dissolving the rock in sulfuric acid (H_2SO_4) to yield both phosphoric acid and calcium sulfate together. The concentration of phosphoric acid is most commonly achieved through forced-circulation evaporation, usually performed in a phosphoric-acid heater as shown in Figure 1. The sorts of corrosion phenomena encountered in wet phosphoric-acid processes can be quite complex and are dependent on several factors, such as the presence of certain impurities or phosphates, and how these substances react with the acid itself.

Composition of the phosphates may vary considerably depending on the source. For instance, phosphates from certain geographical locations are more corrosive than others. Also, varying temperature levels across different areas of the plant may affect composition. These changes result in large variances in corrosion parameters, as seen in Figure 2. Such variations in the content of the elements, and in the temperatures during the concentration of the acid, can make it quite difficult to estimate the corrosivity.

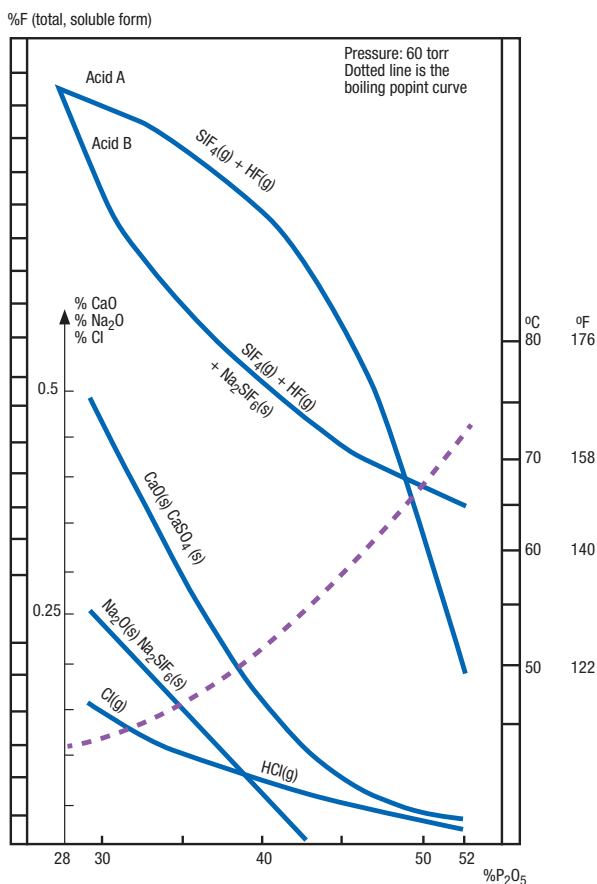
Graphite versus metallic tubes

Impervious graphite, either in the form of tubes or blocks, is the traditional choice of material for phosphoric-acid heaters. This is due to graphite's excellent corrosion properties. Nevertheless, it has subsequently been found that metallic materials exhibit some advantages over graphite. This is partly due to superior mechanical strength, which eliminates the risk for brittle fractures that can readily occur in graphite when cleaning scale, a common headache for phosphoric acid producers.

Metallic tubing is also not susceptible to the types of erosion that are otherwise common at the inlet of graphite-block heat exchangers. If the corrosion-resistant properties are sufficient, metallic heat exchangers can offer extended trouble-free service, yielding substantial economic advantages for operators.

This was demonstrated in one case where a doubling of the interval between cleaning operations was achieved with metallic tube. The increase from five to ten days (assuming a stoppage of 8 h for cleaning) resulted in 15 more days per year of access to the production line for the operator.

However, from the heat transfer point of



view, it should be noted that proper design of metallic phosphoric-acid heaters is vital for achieving these advantages, and also for ensuring that the tubes compare favorably with graphite exchangers.

FIGURE 2. A number of factors impact acid composition during the concentration process

The effects of high alloys

In light of the advantages that have been achieved with metallic heat exchangers over traditional graphite, stainless steels — particularly the standard grades AISI 316L and 317L — are now used extensively in phosphate plants. Furthermore, extensive tests and installations have also found that specially developed high alloys are required for the most severe conditions.

The corrosion resistance of a material is determined by its chemical make-up. In this context, chromium (Cr) is the most important alloying element in stainless steels, as established both in laboratory tests and through practical experiences.

This element is so essential because it imbues stainless steels with good corrosion resistance in wet-process phosphoric acid processes. The advantages of chro-

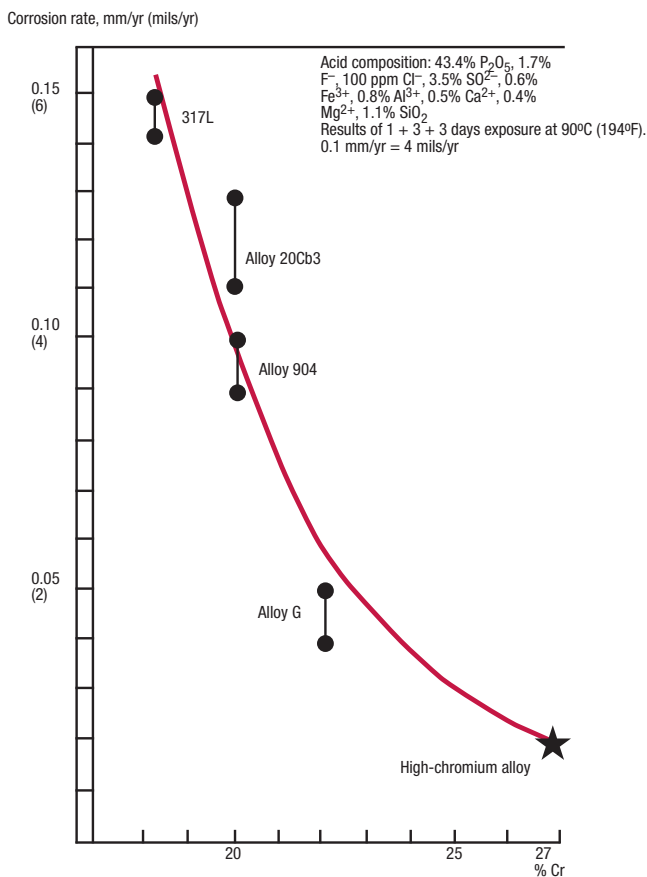


FIGURE 3. The corrosion rate of certain alloys are shown in relation to the chromium content

mium are twofold: it markedly reduces the rate of corrosion of a steel in the passive state, which is also important in the practice of preventing corrosion. Figure 3 shows the relationship between chromium content and corrosion rate. A high-chromium-alloy austenitic stainless steel that is designed for service in highly corrosive conditions is pictured alongside traditional graphite tube in Figure 4. The corrosion-resistant austenitic stainless steel shown in Figure 4 contains higher nominal levels (wt. %) of chromium than other common steel grades. As mentioned, this can reduce the material's corrosion rate in the passive state, thereby preventing corrosion.

High-chromium-alloy material also contains a higher level of molybdenum (Mo) than 316L,



FIGURE 4. A high-chromium alloy austenitic stainless steel tube (left) pictured alongside standard graphite tube (right).

though the value is equivalent to 317L. The element Mo also facilitates passivation, but does not have any marked effect in reducing corrosion in the passive state. This is likewise true of copper (Cu), which is included in this grade, but not the standard grades AISI 316L and 317L.

Further to our understanding of these elements, the addition of nickel (Ni) also plays a major role in corrosion resistance. Nickel is a strong austenitic former, which, in combination with chromium and the other elements, helps to imbue the material with the desired microstructure properties and also improved mechanical integrity.

For operators, a practical advantage of mechanical integrity is higher production time, as it becomes unnecessary to plug failed or broken tubes due to operational or cleaning damage. It is also possible to clean the material mechanically, if necessary, without risk of fracturing the heat exchanger tubes. It is clear from corrosion-rate data that, due to the enhanced high-alloying contents of chromium and nickel, a high-chromium alloy has considerably better resistance than standard stainless steels of type AISI 304, AISI 316L and 317L.

Environmental contaminants

As previously mentioned, levels of contaminants in the fertilizer production environment are a major concern when assessing the capabilities of replacement tubes. Figure 5 illustrates the strong negative influence of contaminating chloride and fluoride ions on a steel's corrosion resistance when combined at high temperatures. As shown, a maximum chloride ion level of around 700 ppm can be tolerated by a high-chromium alloy without risk of active corrosion. The significant influence of temperature upon the reaction is shown in Figure 6. An increase of 10°C (50°F) can nearly double the corrosion rate and, once again, the influence of a high chromium content can be seen.

Real-world example. A full-scale trial was conducted in order to assess the performance properties of high-chromium alloys compared to graphite tube. For the test, a heat exchanger was equipped with a high-chromium alloy grade and run parallel to a graphite block heat exchanger. A noticeable difference occurred during the service period when it became necessary to increase the steam pressure in the graphite unit but not in the high-alloyed unit. This difference can be attributed to the reduced scaling rate of the high-alloyed austenitic material. This, in com-

combination with a higher yield during the service lifecycle, resulted in a significant 20% increase in productivity over the graphite heat exchanger.

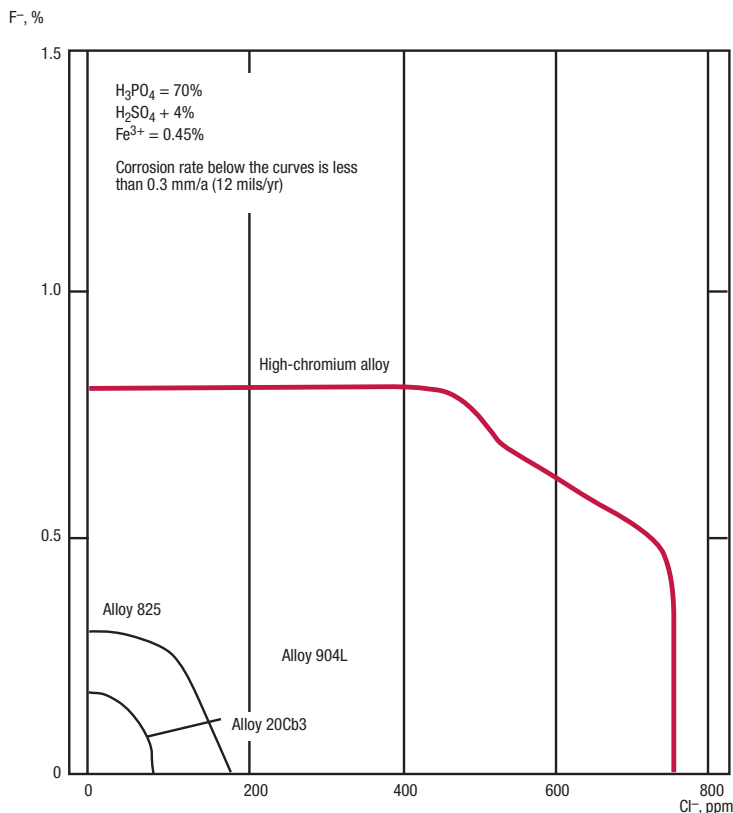
Smaller tube dimensions

The thickness of tube or pipe walls in phosphoric-, nitric- and sulfuric-acid applications is determined primarily by corrosion allowance, and less so by mechanical strength. This is because high pressures occur less in these processes than in other fertilizer-production methods, such as urea applications, where material strength is more of a factor. Whereas the brittle structure of graphite necessitates thick walls, high-chromium alloys make smaller tube dimensions possible due to the material's enhanced anticorrosion properties, thereby enabling more tubes and thinner walls in heat-exchanger applications.

It should be noted that redesign by a specialist is necessary in order to take full advantage of high-alloyed materials, such as high-chromium austenitic stainless steels. Although there have been a limited number of cases where high-chromium tubing has experienced increased corrosion, with proper design and operation of the heat exchanger, it has proven possible to diminish this concern.

Nitric acid processes

In nitric acid (HNO_3) processes, chromium also has a predominant effect on the corrosion resistance of stainless steels. Much of the nitric acid produced worldwide is used in the production of fertilizers, which the industry typically uses at a concentration



acid condensates, so almost all equipment that comes into contact with the process media is made from stainless steels.

The most commonly used stainless steel for the wetted parts of nitric-acid service is standard-grade 304L — often regarded as the “workhorse” of the industry. Despite the grade’s popularity, its performance in service can vary considerably between different producers, mainly because standard specifica-

FIGURE 5. The combined effect of chloride ion content and free fluoride ion content on alloys’ corrosion resistance at 100°C indicates the increased tolerance levels of alloys with higher chromium content

It should be noted that redesign by a specialist is necessary in order to take full advantage of high-alloyed materials, such as high-chromium austenitic stainless steels

of 60–65%. Nitric acid is a strong acid, yet only becomes extremely corrosive when it is processed in high concentrations or at high temperatures. During the nitric acid process (Figure 7), ammonia is oxidized to produce nitric oxide, then oxidized further to form nitrogen dioxide and then absorbed in water to finally give a solution of HNO_3 . Nitric acid service is very corrosive, due mainly to the presence of nitric

tions allow for very generous limits of critical impurities like carbon, sulfur and phosphorus. These impurities can segregate in grain boundaries in standard 304L heat-exchanger tubing and shells, thereby increasing the risk of intergranular corrosion. This has led to instances of costly downtime and maintenance in nitric acid plants. In order to avoid these consequences, good process controls are necessary. Controls can help to en-

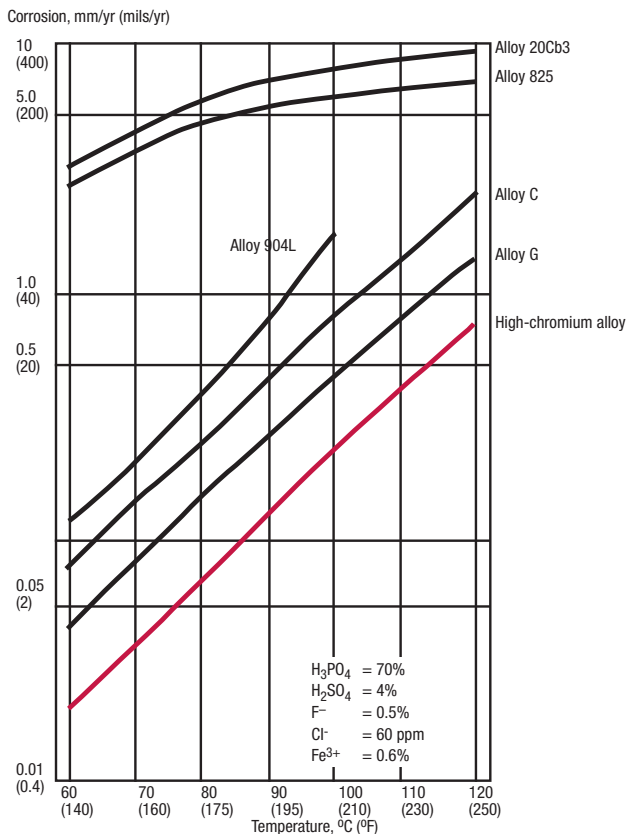


FIGURE 6. The influence of temperature on the corrosion rate of various alloys in contaminated phosphoric acid shows the effect of chromium content

sure the same quality is achieved in all steel melts, with very narrow tolerances for the chemical composition.

Chromium for nitric acid service

All stainless steels contain chromium, and the element has a powerful effect on the corrosion resistance of stainless steel in nitric acid service, due to the influence it exerts on the material's fundamental electrochemical characteristics. This is illustrated in Figure 8, a schematic anodic polarization curve. Here, chromium lowers the maximum current density (i_{max}), which implies that the passivity is more

easily maintained.

Chromium also lowers the corrosion rate in the passive state and increases the transpassivity potential (E_{tp}). The latter is especially important, since the corrosion potential of stainless steel in nitric acid at high concentrations is often close to the transpassive state. Therefore, a steel with too low a chromium content, or a steel containing elements that will reduce the transpassivity potential, is likely to corrode at a high rate. An electrochemical study of the influence of various alloying elements on the transpassivity potential will indicate how a steel should be composed in order to obtain a good corrosion resistance. Table 1 measures the influence of various elements on the corrosion of stainless steels in HNO_3 both in quench-annealed (QA) and sensitized plus quench-annealed form. For QA, material is heat-treated followed by rapid cooling (quenching). Sensitized means that the material has been held at a temperature at which carbides can precipitate.

Table 1 clearly demonstrates that chromium is the most effective element in promoting the corrosion resistance of stainless steels in nitric acid concentrations of up to 65%, whereas carbon (C), silicon (Si), phosphorus (P), sulphur (S), molybdenum (Mo), titanium (Ti) and niobium (Nb) all have a negative effect. This shows that a high-chromium grade is needed for the most severe conditions. The reason is that good corrosion resistance depends largely on the ability of the stainless steel to form a protective oxide layer consisting mostly of chromium. Therefore, materials with higher chromium content have an enhanced ability to develop the protecting layer. The content needs to be about 10 wt. % in order for the oxide film to form at normal atmospheric conditions. This oxide layer could last indefinitely were it not for the corrosive effects of the environment and changing service conditions. These factors become more aggressive and cause the layer to lose its protecting properties.

Testing alloying elements

A simple means to evaluate the effect of alloying elements on stainless steels is the boiling nitric-acid test or Huey test, standardized as ASTM A262-Practice C. The Huey test is an intergranular corrosion test where a sample is boiled in nitric acid for five periods of 48 h each. Because it is performed in nitric acid, the test offers a very good indication of how well the material will perform in the plant by easily revealing susceptibility to intergranular corrosion.

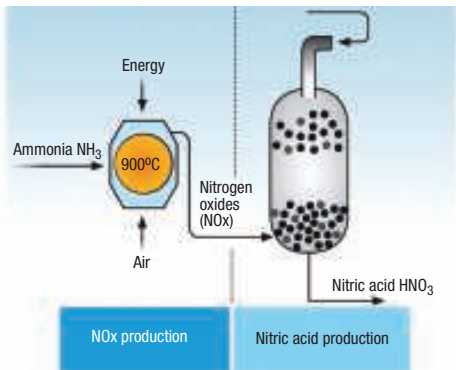


FIGURE 7. The typical process for producing nitric acid (HNO_3) is shown

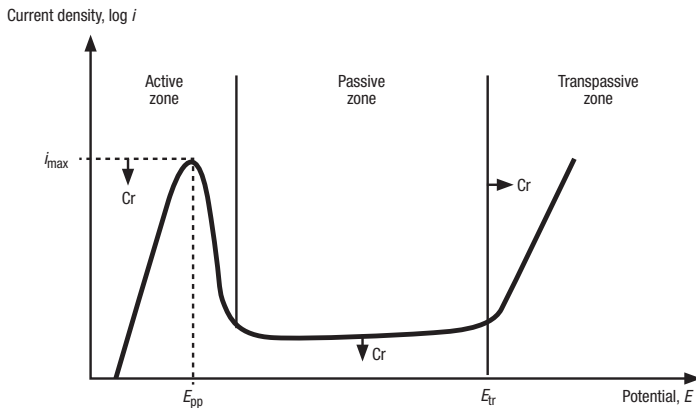


FIGURE 8. This schematic anodic polarization curve illustrates the effect of chromium on the electrochemical parameters of stainless steel. In nitric acid, the corrosion potential is near the transpassivity potential, E_{tr} .

By comparing the Huey test results of several materials, it is possible to rank the corrosion resistance of standard and special steels. Huey tests for various grades show that standard 304L is better in the quench-annealed state, but exhibits an unacceptable variation in the corrosion rate for the sensitized condition.

Huey tests also show that high-chromium stainless steel displays a slower corrosion rate on the order of 0.12 mm/yr, equivalent to 4.7 milli-inch (mils) per year. This can be attributed to the material's extremely low carbon content and its lower impurity content, as well as closer tolerances for impurities than similar, standard steels. These properties equip the high-alloyed nitric acid service grade with excellent resis-

tance to both corrosion in nitric acid and intergranular corrosion.

Recognizing high-corrosion situations

One of the locations in nitric acid plants where the most severe corrosive conditions are found is normally the device or unit that is used to concentrate the oxides of nitrogen (NO_x) from a gaseous to liquid state — usually a condenser or cooler (see Figure 7). Other very severe locations are the tail-gas preheaters and boiler feedwater heaters.

A common corrosion case occurs in coolers or condensers with process gas on the tube side. Corrosion takes place at the inlet where the first condensate is formed, the reboiling of which causes very severe corrosive conditions. Especially harsh conditions are found where condensation and evaporation take place (as in graphite block heat exchangers). This leads to the kind of attack illustrated in Figure 9.

It is useful to recognize the high-corrosion situations that may occur in equipment dur-

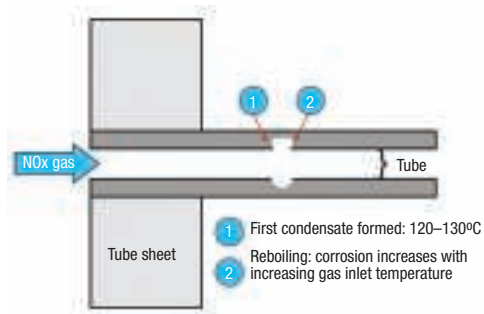


FIGURE 9. Hot-dewpoint corrosion can commonly occur in the coolers and condensers in nitric acid plants

TABLE 1. INFLUENCE OF ELEMENTS ON THE CORROSION RESISTANCE OF STAINLESS STEEL IN NITRIC ACID

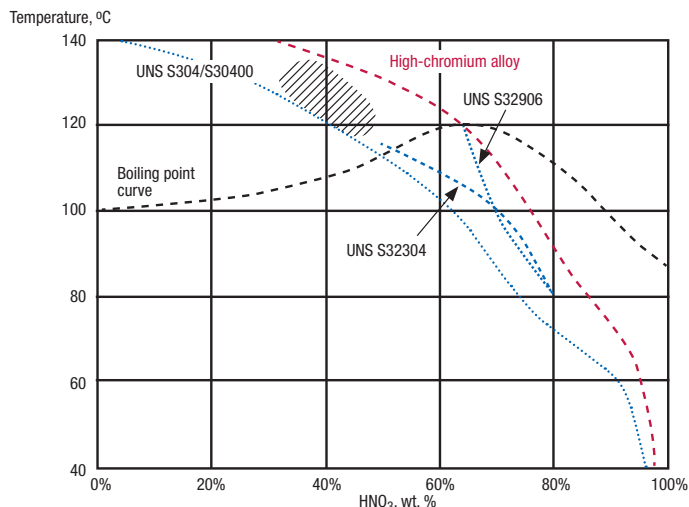
		≤ 65% HNO ₃	> 65% HNO ₃	> 65% HNO ₃
		QA/QA + Sensitized*	QA	QA + Sensitized*
Positive	Cr	x	x	
	Si		x (2)	
Negative	C	x	x (0.02)	x (0.01)
	Si	x	x (0.1)	
	P	x	x (0.01)	x
	S	x	x (0.01)	
	Mo	x (0.5)		
	Ti	x	x	x
	Nb**	x	x	x

* Quench-annealed (QA) and sensitization at 650°C for 1 h

** Pronounced end-grain attack

() denotes minimum level for influence

FIGURE 10. An isocorrosion diagram is a useful tool for identifying high-corrosion situations during the process-design phase



ing the design process. This can be achieved with isocorrosion curves, such as the one shown in Figure 10, which are produced using data from general corrosion testing whereby a test sample is exposed at a certain temperature and concentration. Figure 10 relates specifically to the cooler and condenser; the shaded area represents roughly the range of condensation of NO_x in high-pressure processes.

The curves show that a high-chromium austenitic stainless steel can be used at

higher concentrations and temperatures. This can be attributed to the material's extremely low carbon and impurity contents compared to the other grades measured: an austenitic stainless chromium-nickel steel with a low carbon content; a lean duplex (austenitic-ferritic) stainless steel; and a super-duplex stainless-steel seamless pipe.

Closing thoughts

In phosphoric-acid heat exchangers, more cost-effective performance is one reason why high-chromium alloyed austenitic stainless steels are proven as a suitable replacement for traditional graphite-reinforced tubing. Another reason is mechanical integrity, which can be attributed mainly to the grade's high-chromium alloy content.

For nitric acid service, use of high-chromium-alloyed austenitic stainless steel is also recommended when problems are identified with standard steels of type 304L due to condensation or evaporation of nitric acid droplets. Grades developed particularly for nitric acid service can withstand the corrosion problems that may vary from plant to plant because of local service conditions. High-chromium austenitic grades are also expected to increase the service life by several times compared to standard 304L.

For critical phosphoric or nitric acid service, it is vital to carefully select the material that is best equipped to withstand corrosion. While the initial cost may be higher, a later change to a more corrosion-resistant material will always be more expensive. The additional investment can also be recouped by increased production times that are enabled by more advanced material grades. ■

Edited by Mary Page Bailey

Author



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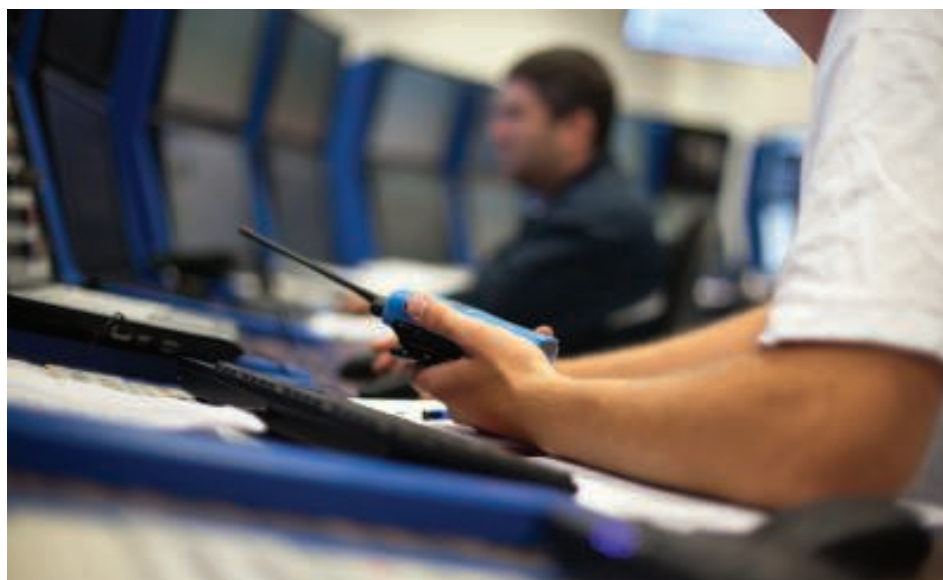
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What's Corroding Your Control Room?

Corrosion-induced failures are frequent in the electronics products used in control rooms, but proper environmental assessment, control and monitoring can help abate these concerns



Chris Muller
Purafil, Inc.

IN BRIEF

CAUSES OF CORROSION

CONTAMINATION
CONTROL PROCESS

ROHS AND RELIABILITY

LOOKING FORWARD

FIGURE 1. The critical electronic components in CPI control rooms are susceptible to corrosion, and protection mechanisms should be put into place to prevent failure.

Electronic process-control equipment — from small remote sensors and instruments to plant-wide distributed control systems (DCS) — make plants in the chemical process industries (CPI) capable of higher production rates, and keep them more competitive in the world market (Figure 1). Chemical, physical and environmental monitoring and control of processes, which previously was performed manually or through simple control systems, is now accomplished in real-time via sensors and actuators linked by a sophisticated DCS. This type of high-speed network enables manufacturers to produce goods economically at high rates and with high quality.

The key to proper control is a properly functioning DCS. This ensures correct sen-

sor function, communications and process control. The corrosive gases that are present in many industrial facilities will permeate into control rooms, DCS spaces and computer facilities, and will most likely degrade and eventually incapacitate critical electronic equipment. This scenario may, in many cases, lead to catastrophic failure in critical control and safety systems. The environment must be properly managed for safe and successful system operation and reliability. This article covers the ways environmental assessment, monitoring and control can be applied to avoid corrosion issues in control rooms.

To begin with, plant operators must manage several environmental species of airborne contamination, including the following:

- Liquids, including small levels of conden-

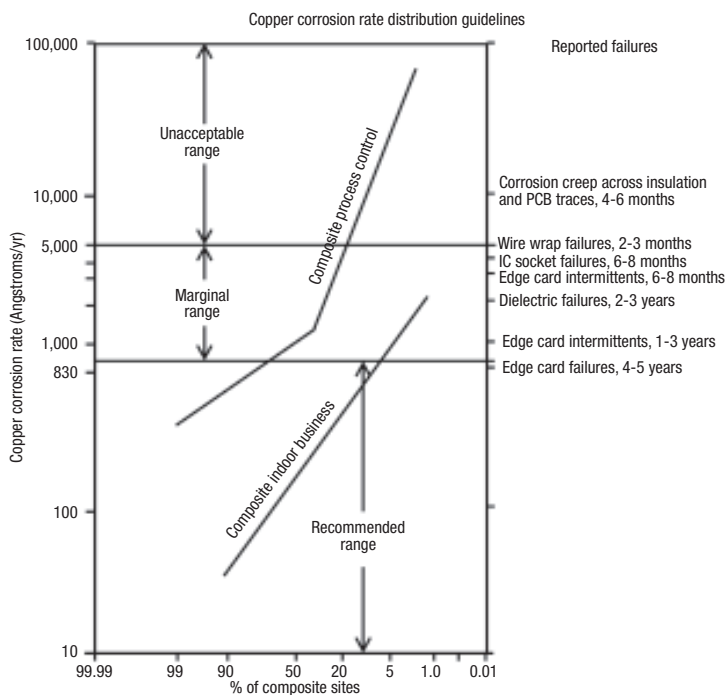


FIGURE 2. These corrosion guidelines were developed by various collaborating companies to protect control equipment against corrosion [3]

- sation and sea salt mist
- Solids, including grit, sand and dust
 - Gases, including active sulfur, sulfur oxides, gaseous chlorine, nitrogen oxides, hydrogen fluoride, ammonia, ozone and strong oxidants

One complicating factor that has arisen in the last several years has been the passage of the European Union (E.U.) Restriction of Hazardous Substances (RoHS) directive regarding the use of certain hazardous substances in electrical and electronic equipment [1]. The RoHS directive restricted the use of lead (Pb), mercury (Hg), cadmium (Cd), hexavalent chromium (Cr^{6+}), polybrominated biphenyls (PBB) and polybrominated diphenyl ether (PBDE) in the manufacturing of certain electrical and electronic equipment sold in the E.U. Although a misnomer, these regulations are most commonly referred to as “lead-free” manufacturing, due to the fact that essentially all electrical and electronic equipment to this point contained lead. This was the first of many regulations passed to control electronic waste by eliminating lead and other hazardous substances in electronic products. In its first iteration, RoHS provided exemptions for several types of critical electrical devices, including process measurement and control equipment, due to lingering reliability concerns when these regulations took effect in 2006.

The RoHS 2 directive (or RoHS recast) is an evolution of the original directive [2]. It became law in the E.U. in July 2011 and took effect in January 2013. RoHS 2 addresses the same substances as the original directive, while the new scope has been extended to all electrical and electronic equipment, including monitoring and control instruments, with full implementation required by July 2017. However, even with this extension, experience has shown that printed circuitboards (PCBs) made using lead-free materials can be more susceptible to corrosion in high sulfur environments, which typically have been associated with industrial applications.

Causes of corrosion

Corrosion of metals is actually a chemical reaction caused primarily by attack of gaseous contaminants, and is accelerated by heat and moisture. Rapid shifts in either temperature or humidity cause small portions of circuits to fall below the dewpoint temperature, thereby facilitating condensation of contaminants. Relative humidity (RH) above 50% accelerates corrosion by forming conductive solutions on a small scale on electronic components. Microscopic pools of condensation then absorb contaminant gases, which become electrolytes in the pools, where crystal growth and electroplating occur. Above 80% RH, electronic corrosive damage will occur, regardless of the levels of contamination.

In the context of electronic equipment, corrosion is defined as the deterioration of a base metal resulting from a reaction with its environment. More specifically, corrosive gases and water vapor coming into contact with a base metal result in the buildup of various chemical reaction products. As the chemical reactions continue, these corrosion products can form insulating layers on circuits, which can lead to thermal failure or short-circuits. Pitting and metal loss can also occur.

Corrosive gases. Specifically, we are only concerned with the three types of gases that are the prime culprits in the corrosion of electronics: acidic gases, such as hydrogen sulfide, chlorine and hydrogen fluoride, as well as oxides of sulfur and nitrogen; caustic gases, such as ammonia; and oxidizing gases, such as ozone. Of the gases that can cause corrosion, the acidic gases are typically the most harmful. For instance, it takes only 10 parts per billion (ppb) of chlorine to inflict the same amount



FIGURE 3. Corrosion classification coupons (CCCs) are used to establish baseline corrosion data

of damage as 25,000 ppb of ammonia. Each site may have different combinations and concentration levels of corrosive gaseous contaminants. Performance degradation can occur rapidly or over many years, depending on the particular concentration levels and combinations present at a site.

The “lead-free” transition. A typical failure mechanism of electronic systems in these environments is the reaction of atmospheric sulfur with exposed metals — particularly copper and silver. These metals are found in printed circuitboard traces, integrated circuit (IC) leads and device terminations. Copper sulfide (Cu_2S) or silver corrosion products can grow and creep across surfaces like IC packages and printed circuitboard substrates.

However, one failure mechanism caused by the lead-free transition was not foreseen by the industry — products with an immersion silver (ImmAg) surface finish will creep-corrode in what some electronic equipment manufacturers consider to be high-sulfur environments. The number and types of corrosion failures have increased since the implementation of RoHS. The most frequent failures are with the most common components, which include hard disk drives (HDD), graphic cards, motherboards, dual inline memory modules (DIMMs), capacitors and transistors.

Historically, the use of silver in electronic assemblies has been a reliability risk unless the silver is protected from the environment. Silver creep corrosion (electromigration) can occur quite readily in humid environments, especially in the presence of small amounts of atmospheric sulfur compounds and chlorides, which are common in many industrial environments. More than thirty years ago, DCS manufacturers, such as Digital Equipment Corp. (DEC), ABB and Honeywell, described their con-

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FIGURE 4. Atmospheric corrosion monitors (ACMs) provide realtime data on the performance of the corrosion-prevention measures in a control room, including chemical-filtration and air-quality systems

cerns regarding equipment corrosion in research documents, technical papers and site planning guides. These companies also provided guidance in terms of the environmental conditions necessary to protect their computer equipment (Figure 2) [3].

Manufacturers of industrial computer equipment still specify the control of corrosive gases in their site planning guides or in the terms and conditions necessary to maintain warranties and service contracts. Even in environments previously considered benign with regard to electronics corrosion, serious problems as a direct result of RoHS compliance are being reported.

Contamination control process

With the changes to process measurement and control equipment mandated by the various RoHS directives, process, facility and instrumentation engineers should include environmental contamination monitoring and control as part of overall site planning, as well as risk management, mitigation and im-

provement plans. This plan should consist of the following three steps:

1. Consider the assessment of the outdoor air and indoor environment with regard to corrosion potential. ANSI/ISA Standard 71.04-2013 (hereafter ISA 71.04) can be used to provide site-specific data on the types and levels of gaseous contamination in the amount of corrosion being formed. Corrosion classification coupons (CCCs, Figure 3) can be used as a survey tool to establish baseline data necessary to determine if and what type of environmental controls are needed.
2. Develop and specify a targeted contamination-control strategy. Corrosion in an indoor environment is most often caused by a short list of chemical contaminants or a combination of contaminants. The contaminants present in a specific area are highly dependent on the controls put in place to mitigate them. These efforts mainly involve the selection and application of the appropriate chemical filtration systems to clean both the outdoor air being used for pressurization and ventilation, as well as any recirculation air.
3. Establish a realtime environmental-monitoring program based on the severity levels established in ISA 71.04. Realtime atmospheric corrosion monitors (ACMs, Figure 4) can provide accurate and timely data on the performance of the chemical filtration systems as well as the room air quality.

The absence of gaseous contamination controls can be the result of a lack of knowledge and education. Often, the relationship between corrosion levels and hardware failures is overlooked or unknown. However, due to the continuing efforts of automation and control companies, this knowledge gap is shrinking, and successful corrosion monitoring and control programs are being developed and implemented, assuring reliable operation of process measurement and control equipment.

Environmental assessments. A simple quantitative method to determine the airborne corrosivity in a control room environment is by “reactivity monitoring” as described in ISA Standard 71.04-2013: Environmental Conditions for

TABLE 1. CLASSIFICATION OF REACTIVE ENVIRONMENTS

Class	Severity level	Copper reactivity	Silver reactivity	Comments
G1	Mild	<300 Å	<200 Å	Corrosion is not a factor in determining equipment reliability
G2	Moderate	<1,000 Å	<1,000 Å	Corrosion effects are measurable and corrosion may be a factor. Electroless nickel immersion gold (ENIG) and immersion silver (ImmAg) printed circuitboard surface finish failures
G3	Harsh	<2,000 Å	<2,000 Å	High probability that corrosive attack will occur. Organic solderability preservative (OSP) and immersion tin (ImSn) PCB surface finish failures
GX	Severe	≥2,000 Å	≥2,000 Å	Only specially designed and packaged equipment expected to survive

Process Measurement and Control Systems: Airborne Contaminants. Copper and silver sensors are exposed to the environment for a period of time and quantitatively analyzed to determine corrosion-film thickness and chemistry. Silver-reactivity monitoring must now be used as part of an assessment in order to provide a complete accounting of the types of corrosive chemical species present in the local environment and to establish environmental severity levels.

ISA 71.04 classifies four levels (Class G1, G2, H3 and GX) of environmental severity for electrical and electronic systems providing a measure of the corrosion potential of an environment (Table 1). The overall classification is based on the higher of the total copper and silver reactivity rates.

Corrosion monitoring. CCCs can be placed throughout the control room to determine compliance with air-quality specifications. Realtime ACMs are used in the controlled environment and on or in server cabinets to provide realtime data on corrosion rates and the effectiveness of corrosion control strategies. Proper assessment will require monitoring of the outdoor (ambient)

air and at various locations inside and outside the control center. Either monitoring technique may be used to provide the data necessary to troubleshoot and mitigate contamination issues inside the control center. There are many options that can be considered with respect to air-quality monitoring for industrial control-room applications, and there should be some consideration for room size and layout to determine the minimum number of CCCs and ACMs and their locations, to provide a statistically valid environmental assessment.

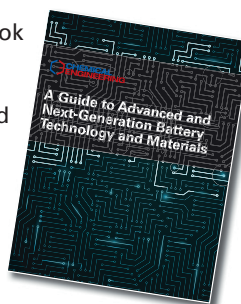
There is general confidence in being able to identify contaminant types, for instance active sulfur, sulfur oxides and inorganic chloride compounds, when using corrosion monitoring. This carries more weight from a scientific standpoint in that it can be verified using independent sources of environmental data, such as air-pollution indices or satellite data, to verify the results obtained from corrosion monitoring.

CCCs are typically used for an initial survey of ambient air quality and the environment in which electronic equipment is located, and can be used on a continuing basis to



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TABLE 2. PULP AND PAPER MILL CCC DATA, IN Å/30 DAYS

Location	Area/room	Cu ₂ S	Cu ₂ O	Cu — unknown	Copper total	ISA Class*	AgCl	Ag ₂ S	Ag — unknown	Silver total	ISA class**
Austria	AHU room	0	180	0	180	G1	0	3,142	0	3,142	GX
	DCS room	182	78	0	260	G1	0	3,491	0	3,491	GX
	Electrical room	115	46	0	161	G1	0	1,582	0	1,582	G3
Brazil	Control room	192	101	0	293	G1	0	1,042	0	1,042	G3
	Operations room	205	88	0	293	G1	0	1,234	0	1,234	G3
	MCC	0	125	0	125	G1	0	948	0	948	G2
China	DCS room	182	104	0	286	G1	0	1,022	0	1,022	G3
	Wet end control	182	78	0	260	G1	0	1,118	0	1,118	G3
	Computer room	124	44	0	168	G1	0	5,467	0	5,467	GX
Finland	DCS cabinet	0	118	0	118	G1	0	1,636	0	1,636	G3
	MCC	0	106	0	106	G1	0	2,634	0	2,634	GX
	Control room	0	150	0	150	G1	0	2,796	0	2,796	GX
Holland	Mechanical room	0	68	0	68	G1	0	2,182	0	2,182	GX
	Server room	0	78	0	78	G1	0	1,964	0	1,964	G3
	Splicer room	0	105	0	105	G1	0	1,145	0	1,145	G3
Japan	Control room	0	173	0	173	G1	0	1,497	0	1,497	G3
	IPC room	0	231	0	231	G1	0	2,145	0	2,145	GX
	DCS room	215	62	0	277	G1	0	1,038	0	1,038	G3
Korea	DCS panel	187	61	0	248	G1	0	1,848	0	1,848	G3
	MCC room	204	56	0	260	G1	0	1,810	0	1,810	G3
	PLC panel	102	50	0	160	G1	0	1,155	0	1,155	G3
U.S.	Electrical room	172	34	0	206	G1	35	1,315	0	1,340	G3
	Wet end drives	196	81	0	277	G1	0	1,565	0	1,565	G3
	Control room	175	86	0	261	G1	0	5,073	0	5,073	GX

* Severity level according to ISA Standard 71.04-1985

** Severity level according to ISA Standard 71.04-2013

provide historical data. This is especially important where equipment warranties specify establishing and maintaining an ISA Class G1 environment. Realtime monitoring may also be used, but should be limited to the control-room environment. Where corrosion problems have been identified, it is recommended to start with ACMs in a number of locations in order to determine if contamination is widespread or limited to a specific area. Once a baseline had been established, some of the monitors could be later redeployed around the problem areas

to gauge the effectiveness of contamination control strategies that may be put in place. Once the environment is under control and meets the conditions set forth in the manufacturers' warranties, one can determine the best permanent ACM locations for specific needs.

RoHS and reliability

The requirement for corrosion control in industrial environments remains constant. However, more companies are now taking a much closer look at developing or updating

specifications due to the changes made by controls manufacturers to comply with RoHS restrictions on the use of lead. This includes specifying an ISA Class G1 environment for control rooms, where in the past, a Class G2 environment was considered acceptable. Specifications are also now emerging that require the measurement and quantification of both copper and silver corrosion rates according to ISA 71.04-2013, with environmental severity levels determined according to the higher of the two.

Although the amount of silver on printed circuitboards and other electrical components has been reduced by many control systems manufacturers, the use of silver is still required and will be for the foreseeable future. With this comes an increased concern over equipment reliability when using a copper-only environmental classification system.

Example: problems with RoHS-compliant equipment. A recycled-paper mill began a project to replace obsolete equipment that could no longer be supported with new DCS and programmable logic controls (PLCs). The original hardware had been installed for 15 years, and had proven to be reliable and robust. Many of the locations where this equipment had been installed were classified as ISA Class G1 for copper. However, the corresponding silver corrosion rates were up to 20 times higher, and essentially all of the silver corrosion reported from the analysis of CCCs collected over several years was due to sulfur corrosion. This presented some concern based on changes to the equipment due to RoHS compliance.

Within three months of replacing the old systems, the mill began experiencing frequent failures of the input/output (I/O) stations installed on the process fieldbus (Profibus). A great deal of work was put into making the fieldbus installations error-free, and the architectures were reengineered to follow the core rules precisely.

Since the problems were intermittent, and symptoms appeared related to Profibus communication or

installation, a full diagnostic process was implemented to capture any and all information about each failure. This included examination of the hardware, the hardware location, its position on the fieldbus, architectures and software settings. Through systematic investigation and analysis, several potential causes were eliminated and a root-cause analysis for the remaining issues



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was performed.

Mill staff recognized that the resistance of a choke installed on the base unit of the I/O station to eliminate signal noise was increasing, and on failed units was a high or open circuit. Ironically enough, this choke was only installed to meet other E.U. legislation pertaining to radio-frequency interference (RFI) emissions. The critical issue identified pertaining to this choke was that it was an electronic chip design with a very isolated thin layer of silver in compliance with the lead-free regulations of RoHS. Corrosion of this silver layer destroyed the choke's connection to the printed circuit, resulting in the high resistance. Consultation with the manufacturer confirmed the presence of silver on the chokes.

The mill was already in the process of installing air-cleaning equipment to remove corrosive gases from the air and reduce the rates of copper corrosion. However, the extremely short time to failure for these silver-containing components accelerated this effort in order to prevent additional failures. The staff was confident that these failures were purely the results of RoHS compliance and the use of silver. The lack of information and warnings by the supplier regarding potential issues related to their RoHS compliance programs is troubling, considering that even when presented with this evidence, they still concluded these failures were due to "installation errors."

Problems exist in many locations.

CCC data from several additional mills (Table 2) show that many locations exhibiting an ISA Class G1 environment for copper, including air-handling units (AHUs) and motor control centers (MCC), have corresponding silver corrosion rates that would now cause the environment to be classified as G2, G3 or even GX. This would indicate serious cause for concern for any electronic equipment with an ImmAg surface finish specifically or any silver or silver-plated components in general.

Another contributing factor to concerns over the increased use

of silver in industrial applications is that even with tightened control requirements for other environmental parameters (including temperature and humidity) and the positive effect this has on the rate of copper corrosion, silver can still exhibit high rates of corrosion, even in well-controlled environments. Closer examination of CCC data shows that in locations reported as ISA Class G1 for copper corrosion, the corresponding silver corrosion rate can be up to 10 times higher [4]. Furthermore, every CCC analyzed shows evidence of sulfur contamination (as Ag_2S). On average, the amount of silver corrosion measured is double that of the copper corrosion reported.

Contaminant gases containing sulfur, such as SO_2 and H_2S , are the most common gases that cause hardware corrosion in paper mills, petroleum refineries and chemical plants, and corrosion control is acknowledged as a requirement to assure electrical and electronic equipment reliability. One example of component failure is from sulfur gases entering a component package and attacking the silver, resulting in the formation of Ag_2S . The mechanical pressure created by the Ag_2S formation inside the package damaged its mechanical integrity and caused the device to fail. This and other failure mechanisms are becoming common occurrences when using RoHS-compliant electronic equipment and components produced using the ImmAg process, and to a lesser degree, the electroless nickel-immersion gold (ENIG) process.

To maintain a high level of equipment dependability and availability, it should be understood that a control room is a dynamic environment where many maintenance operations, infrastructure upgrades and equipment change activities occur on a regular basis. Airborne contaminants that are harmful to sensitive electronic devices can be introduced into the operating environment in many ways in addition to the ventilation system. For instance, chlorine can be emitted from PVC insulation on wires and

cables if temperatures inside the DCS cabinets get too high. However, it is still the outdoor ambient air used for cooling and pressurization that remains the primary source of corrosive contaminants, and this air should be cleaned before its introduction into the control-room environment.

With the changes to process control equipment due to the RoHS directives, plant managers and operators should include an environmental contamination monitoring and control section as part of an overall site plan, as well as plans for risk management, mitigation and site improvements.

Looking forward

RoHS regulations, along with the continuing reductions in circuitboard feature sizes and miniaturization of components necessary to improve hardware performance, makes today's electronic hardware more prone to attack by airborne contaminants. Increases in corrosion-related electronic hardware failures have led to new electronic equipment warranties that require environmental corrosion (reactivity) monitoring and control of airborne contamination where necessary.

Manufacturers have to maintain the reliability of their equipment, and therefore the need to control airborne contaminants and to specify their acceptable limits are now considered to be critical to the continued reliable operation of process-control equipment. ANSI/ISA Standard 71.04-2013 now includes silver corrosion monitoring as a requirement in determining environmental severity levels. Most manufacturers of process-control equipment currently reference this standard in their site planning and preparation guidelines, as well as their terms and conditions for warranty compliance. The addition of silver corrosion rates as a required metric serves to bridge the gap between ambient environmental conditions and the reliability of RoHS-compliant (lead-free) electronic equipment.

Ongoing research will serve to further refine Standard 71.04 both

quantitatively and qualitatively. This, along with continuing advancements in the monitoring and control of corrosive contaminants, will help to prevent costly and potentially catastrophic failure of critical electronic equipment.

The requirement for the control of corrosive gases in the CPI remains constant. Assuring longterm equipment reliability requires working with plant personnel to quantify the corrosive potential of an environment toward the various types of electronic equipment in use, providing engineered solutions for gaseous contaminant control and ongoing monitoring of the controlled environment to assure compliance with standards and specifications. The successful application of such a corrosion control program in CPI settings will protect critical process measurement and control equipment and assure profitable manufacturing operations. ■

Edited by Mary Page Bailey

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High Purity Chemicals: Processing Equipment Essentials

Some practical tips regarding the design and fabrication of piping systems and equipment for high-purity applications are presented here

W. M. (Bill) Huitt
W.M. Huitt Co.

IN BRIEF

THE PURITY QUOTIENT

ELECTRONICS
CHEMICALS

PERSPECTIVE

HIGH-PURITY
EQUIPMENT

CLEANROOM
CLASSIFICATIONS

DESIGNING FOR HIGH
PURITY

FINAL REMARKS

Chemical processing, and the chemical process industries (CPI) on the whole, have evolved lightyears beyond their very beginnings — beginnings in which chemistry was more or less a form of alchemy and the mixing of basic natural elements. From such humble beginnings, and more pointedly over the past several decades, the chemical industry has progressed dramatically from those early times to today's high-tech chemical processing, which include bioprocessing and the electronics industry. These are two industries that not only rely on what is referred to as high-purity, ultrahigh-purity, and ultra-pure chemistry, they could not exist without it. This discussion does not focus on the chemistry itself, but on the various nuances in the equipment used to manufacture, monitor and handle these chemicals.

The purity quotient

Since the topic of this discussion focuses on what is vaguely termed "high purity," "ultrahigh purity," and "ultra-pure" chemical processing, we need to provide some clarification with regard to those three terms. I mention clarification rather than definition of these terms, because there are no universally accepted definitions that quantify and delineate these three terms.

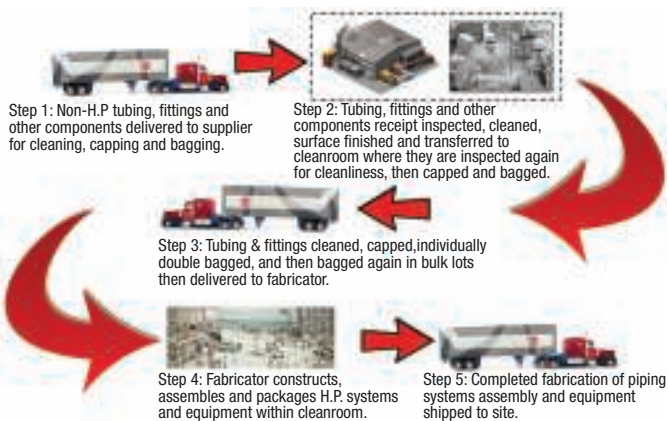


FIGURE 1. The evolution of high-purity equipment can follow the steps shown here

The three terms, as they pertain to chemical processing today are nominal, vague and self-defining at best — they mean only what the user of any of the three terms intends for them to mean. In other words, as previously alluded to, these terms currently possess no universally accepted or standardized real and specific value.

The bioprocessing industry relates, to a large degree, with the term high purity. But the semiconductor industry has an affinity for all three terms. These are terms that, with regard to the purity of chemicals, can mean whatever the user wants them to mean.

To get a feel for the scope of such a nominal term as "purity," Table 1 lists 47 various purity and chemical grade designations used throughout the CPI. Some designations are company trademarks and others find their source in various industry organizations. They do have one thing in common: there is no uniformity or conformity in the

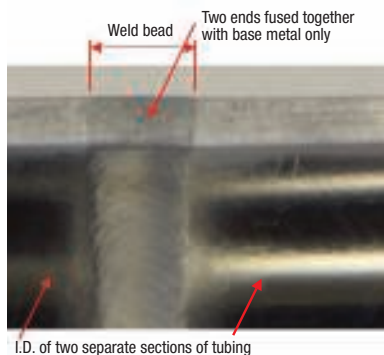


FIGURE 2. A magnified photo of a cross section of two stainless-steel tubes autogenously welded together

approach to, or the results of, their “chemical purity.”

Mosby’s Dictionary of Complementary and Alternative Medicine [1] defines “chemical purity” as, “the degree to which a substance is undiluted or unmixed with extraneous material, typically expressed as a percentage (%).” And a chemical’s purity is indeed expressed as a percentage, in which a chemical with a



FIGURE 3. This image shows a bead and crevice free (BCF) fusion weld of a tee section [3]

purity of 98% implies a content of 2% impurities. The purity of gases too, is defined in much the same manner, but is, in many cases, assayed and certified to more finite values. By assigning a purity factor, as an example, of 99.98% helium or 99.999% argon (referred to in industry jargon as “five-nines”), the purity of those gases is thereby quantified and specifically defined.

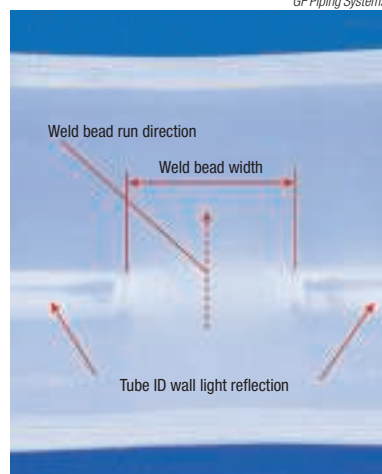


FIGURE 4. This image shows a section view of a BCF fusion weld [3]

Having such specific quantitative-purity values creates definable parameters for determining the degree of chemical purity that needs to be specified, procured and receipt-verified for use in electronics chemicals where purity is a critical factor.

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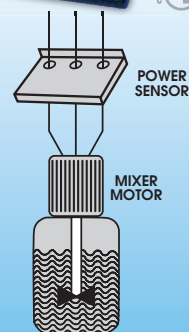
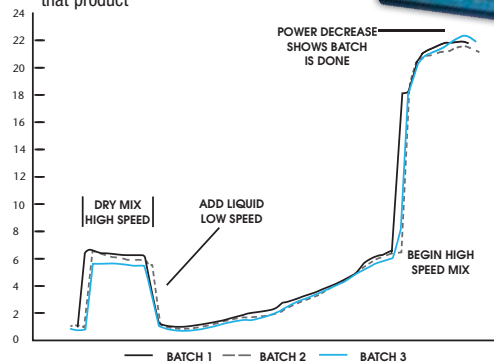
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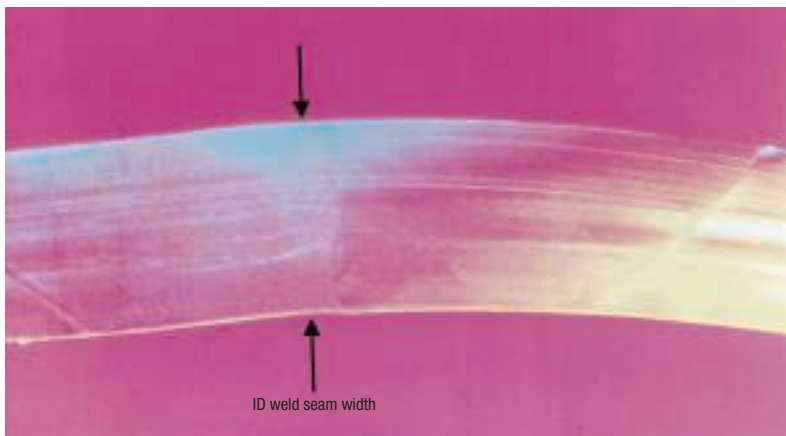


FIGURE 5. A closeup of a BCF fusion weld seam is shown here [3]

ultra-high purity, and ultra-pure, when they have no such specific values, then becomes almost euphemistic in their generality. The three terms are much like those of low-, medium- and high-pressure steam, which are not terms that hold universally accepted values, but are instead based on the relative steam pressure requirements of a particular facility.

As an example, a hydrolysis coal-gasification process uses saturated steam at 2,000 psig to break down the structure of lignite coal, causing the material to release its gases. In such a coal-gasification facility the 2,000 psig steam may be considered high pressure, with medium- and low-pressure values established relative to the high-pressure value.

On the other hand, in an active pharmaceutical ingredient (API) processing facility that produces and uses steam at a significantly lower pressure than that of the gasification process, a 100 psig steam system might be considered high pressure, with medium- and low-pressure values again established relative to the high pressure.

This analogy applies to high purity, ultra-high purity, and ultra-pure in which their application and implication, with regard to a purity quotient, is wide ranging in accordance with the chemical and the chemical's use. The specific meaning of purity will thus vary between chemicals, between manufacturers, and between applications of such chemicals. It therefore becomes much more pragmatic to simply segregate our three terms by industry and not by the non-specific

purity values.

The primary industries that use such purity terms, as mentioned previously, include the electronics and bioprocessing industries. The electronics chemicals industry, which includes the semiconductor sector, typically refers to its purity needs and requirements in utilizing each of the three purity terms. Whereas the bioprocessing industry, which includes the pharmaceutical sector, typically refers to its purity needs and requirements simply as high purity. As we move forward with this article we will be focusing more specifically on electronics chemicals. So rather than continue to make reference to all three terms in a somewhat indiscriminate manner we will, for this discussion, simplify things by grouping all three terms into the single catch-all term high purity.

Electronics chemicals

The electronics industry covers the manufacture of a wide range of sophisticated components and end products, including silicon wafers, integrated circuits, printed circuit boards, compound semiconductors, optoelectronics (a subset of photonics), solar panels and flat-panel display products. In discussing this topic I will make every effort to avoid being drawn into the many sidebar subjects this topic harbors. We will instead stay focused on essential criteria and considerations necessary for various chemical processing equipment used in the manufacture of electronics chemicals.

TABLE 1. INDUSTRY ACCEPTED STANDARDS FOR CHEMICAL PURITY AND GRADES

No.	Purity or Grade
1	Absolv (<i>Tedia designation</i>)
2	Accugen (<i>Anachemia trademark</i>)
3	Acculute (<i>Anachemia trademark</i>)
4	Accusolv (<i>Anachemia trademark</i>)
5	Accutin (<i>Anachemia trademark</i>)
6	ACS (<i>American Chemical Society</i>)
7	Anhydrosolv (<i>Tedia designation</i>)
8	AR (<i>MBI trademark</i>)
9	AR Select (<i>MBI trademark</i>)
10	AR Select Plus (<i>MBI trademark</i>)
11	BIO (<i>Tedia designation</i>)
12	CP (<i>Chemically Pure</i>)
13	ChromAR (<i>MBI trademark</i>)
14	DriSolv (<i>EMD trademark</i>)
15	Environmental Grade (<i>Anachemia trademark</i>)
16	Environmental Grade Plus (<i>Anachemia trademark</i>)
17	FCC (<i>Food Chemicals Codex</i>)
18	GenAR (<i>MBI trademark</i>)
19	Guaranteed Reagent (<i>GR; EMD trademark</i>)
20	HPLC/Spectro (<i>Tedia designation</i>)
21	HR-GC OmniSolv Grade Solvents (<i>EDM trademark</i>)
22	Laboratory
23	Laboratory Grade
24	Nanograde (<i>MDI trademark</i>)
25	NF (<i>National Formulary</i>)
26	OmniSolv Biosynthesis (<i>EMD trademark</i>)
27	OmniSolv Grade Solvents
28	OmniSolv HPLC Grade Solvents (<i>EMD trademark</i>)
29	OmniTrace Grade Acids (<i>EMD trademark</i>)
30	OR (<i>MBI trademark</i>)
31	Practical
32	Primary Standard
33	Purified
34	Reagent
35	Residue Grade Solvents
36	ScintillAR (<i>MBI trademark</i>)
37	SilicAR (<i>MBI trademark</i>)
38	SpectrAR (<i>MBI trademark</i>)
39	StandARd (<i>MBI trademark</i>)
40	Standardized Solutions
41	Suprapur Grade Acids (<i>EMD trademark</i>)
42	Technical
43	Tracemetal (<i>Tedia designation</i>)
44	Tracemetal Plus (<i>Tedia designation</i>)
45	UltimAR (<i>MBI trademark</i>)
46	USP (<i>U.S. Pharmacopeia</i>)
47	USP/GenAR (<i>MBI trademark</i>)

Electronics chemicals are, in many cases, considered "specialty chemicals" and are intended for use in the microscopically sensitive manufacture of the above mentioned products. Products that use atmospheric and specialty gases, photoresists, various ancillary chemicals, wet processing chemicals, CMP (chemical mechanical planarization) slurries, thin-film met-

TABLE 2. EXAMPLES OF ELECTRONICS CHEMICALS

Acids	Bases	Solvents	Performance
Acetic	Ammonium hydroxide	1,2-Ethylenediamine	Acetic Copper Clean
Hydrochloric	Potassium hydroxide	Acetone	Ammonium fluoride
Hydrofluoric	Sodium hydroxide	Cyclohexanone	Hydrogen peroxide
Nitric		Ethylene glycol	Proprietary etching formulations
Phosphoric		Hexamethyldisilazine	
Sulfuric		Isopropanol dilutes	
		Methylethylketone	
		Methanol	
		<i>n</i> -Butyl acetate	
		<i>N</i> -Methylpyrrolidone	
		Negative resist developer	
		PBR	
		Propylene glycol	
		Methyl ester acetate	
		Xylene	

als, copper-plating chemicals, and precursor materials for low-k and high-k dielectrics. The low-k and high-k of the latter referring to the low and high dielectric constant, k, inherent in such materials.

Examples of specific chemicals used in the electronics industry can be found in Table 2. In order to be acceptable for use in the electronics chemicals industry, these chemicals are sometimes required

to go through redundant purification steps using processes such as distillation, ion exchange, gas adsorption and filtration.

Just as liquid chemicals need to meet the high-purity demands required by the electronics industry, so too do the gases used in the manufacture of electronics. Table 3 represents a sampling of the types of gases that are typically used in the electronics industry.

Perspective

The semiconductor industry, back in the early 1970s, was creating chip-design pattern etchings, or fabrications, at the 10 micrometer (μm) level. In working at such microscopic levels, the required chemical purity, while it had to meet a higher-than-normal standard of purity, was not a major issue. However, in today's semiconductor world, they are working at the 10 nanometer (nm) level. And by 2021, it is expected that the semiconductor industry will be working at the 5-nm level.

The implication here is that any secondary debris or erroneous particulate matter entrained in a liquid or gas at even the 10-nm size coming in contact with a fabrication can take a work-piece out of specifications, making it rejectable. And this is where the demand for high-purity chemicals comes from. Creating the ability and sophistication to manufacture such components brings with it the need for other related industries, such as the chemical industry, to up

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TABLE 3. EXAMPLES OF ELECTRONICS GASES

Inert	Flammable	Halocarbons	Hydrides	Hydride/inert	Corrosive	Other
Argon	Butane	Carbon tetrachloride	Ammonia	Arsine (1–10%)	Boron	Acetylene
Helium	Cyclopropane	Hexafluoroethane	Arsine	Germane (1–10%)	Chlorine	Carbon dioxide
Krypton	Deuterium	Perfluoropropane	Germane	Phosphine (1–10%)	Dichlorosilane	Carbon monoxide
Neon	Ethane		Phosphine		Hydrogen bromide	Dimethyl ether
Nitrogen	Hydrogen		Silane		Hydrogen fluoride	Nitric oxide
Xenon	Methane				Silicon tetrachloride	Nitrous oxide
	Propane				Boron trichloride	Oxygen
					Trichlorosilane	Sulfur hexafluoride

their game as well.

Not only does this require that chemical processing systems have the technology and ability to refine chemical products down to such high-purity levels, but it also requires instrumentation with the ability to detect, monitor and validate at those levels. It also requires process contact material of construction (MOC) for such equipment and instrumentation to handle these chemicals without contributing nano-sized particles to the fluid stream. And finally, the fabrication and handling of process contact equipment and components has to meet specific demands with regard to welding, surface finish, mechanical joint design, system cleaning and system cleanability.

High-purity piping

Referring back to Mosby's definition of "chemical purity," we can make the claim that purification, as it relates to fluids, is the physical aspect of separating a chemical substance from that of any resident foreign substance considered a contaminant or impurity. Achieving and maintaining a high degree of purity for any fluid, gas or liquid, requires that all process contact surfaces within the high-purity envelope, or boundary limits, meet or exceed a set of stringent requirements pertaining to materials of construction and fabrication of processing equipment, filtration, instrumentation and distribution systems.

Such specific criteria, as mentioned previously, involves proper material selection, unique welding requirements, mechanical joint design, product contact-surface finish, piping and equipment installation parameters, and a dogmatic approach to quality management throughout

the entire process of equipment manufacture through and including installation and maintenance.

Processing equipment used in the manufacture, handling, monitoring and distribution of electronics chemicals have to not only perform processing functions at a painfully high degree of accuracy, they have to do so without being a source contributor of impurities to the fluid stream. These are impurities that can slough off of poorly cleaned process contact surfaces, or microscopic matter that can form in a welding flaw and periodically slough off contaminants. It can also be a process contact material that may not be sufficiently compatible with the process fluid and become susceptible to corrosion, adding particulate matter to the fluid stream.

These are all issues that confront the design, manufacture and operation of equipment used to manufacture electronics chemicals. The manufacture or fabrication of such equipment and piping systems is an evolution guided by ever more stringent requirements and verifying documentation every step of the way.

A typical, simplified example of the steps to required to fabricate high-purity (H.P.) piping and equipment systems is shown in Figure 1. Such a process begins with the manufacture and delivery of non-high purity (raw) product components. The steps are described in more detail as follows:

Step 1. Manufacture and delivery of the basic raw material tubing, fittings and other components needed for finishing and preparing for use in high-purity fluid services.

Step 2. The raw material products are thoroughly examined upon receipt to affirm that they meet the required material specifications. The

components and material, having been received with various mill surface finishes, will then be cleaned in preparation for having the inside diameter (I.D.) surface or the outside diameter (O.D.) surface of the raw products mechanically polished or electropolished to meet specified roughness average (Ra) surface finishes. As the products complete the final steps of the surface-finishing process, they will transition into a cleanroom environment. Such cleanrooms will typically be environmentally controlled to an ISO 4 or 5 classification (see Table 4 for cleanroom classifications). Upon further examination, ensuring that the product meets all specifications for chemical properties, mechanical properties and surface finish, as well as for weld-end fittings, end preparation (prep) for autogenous welding, the, now considered H.P. product should have all openings capped or covered, get individually double bagged, then bagged again as a lot-of-material. A lot is defined by either the product finisher or the user/buyer. Each individually bagged item will contain its own material test report (MTR), including a certificate of conformance (C of C) pertaining to the surface finishing process.

Step 3. The H.P. components and material are then shipped to a fabricator or manufacturer who will fabricate and assemble the H.P. components and material into their final product form or assembly.

Step 4. The H.P. products will be receipt-verified by the fabricator. This may or may not entail un-bagging samples for inspection. Otherwise, all of the bagged material should remain bagged until such time as it is needed in fabrication. At the time the material is needed for its part

TABLE 4. CLEANROOM CLASSIFICATIONS¹

Classifications		Maximum number of particles by size (per m ³)					
ISO 14644-1	FS 209E ^{2,3}	0.1 µm	0.2 µm	0.3 µm	0.5 µm	1.0 µm	5.0 µm
ISO 1		10	2				
ISO 2		100	24	10	4		
ISO 3		1,000	237	102	35	8	
	Class 1	1,236	247	106	35		
ISO 4		10,000	2,370	1,020	352	83	
	Class 10	12,360	2,649	1,059	353	35	
ISO 5		100,000	23,700	10,200	3,529	832	29
	Class 100		26,485	10,594	3,531	353	35
ISO 6		1,000,000	237,000	102,000	35,200	8,320	293
	Class 1,000				35,314	3,531	353
ISO 7					352,000	83,200	2,930
	Class 10,000				353,140	35,314	3,531
ISO 8					3,520,000	832,000	29,300
	Class 100,000				3,531,400	353,140	35,314
ISO 9					35,200,000	8,320,000	293,000

Notes:

1. A comparison between ISO 14644 and FS209E standards for maximum allowed particles by size
2. Federal Standard (FS) 209E, "Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones" was withdrawn on November 29, 2001
3. The FS209E micron class values have been transposed from ft³ to m³

in the fabrication, it will be moved into a de-bagging area similar to a gowning/de-gowning area adjacent to the cleanroom, or through a pass-through opening. The cleanroom environment will again typically meet the environmentally controlled cleanroom classification of ISO 4 or 5. All fabrication within the cleanroom is carefully controlled to minimize or eliminate contamination of what will become the final product contact surfaces of the piping system or equipment being fabricated. Once fabricated, all welds — particularly the process contact surface of welds, including the heat affected zone (HAZ) surrounding the weld proper — will be thoroughly examined against a rigorous set of weld-acceptance criteria.

Once fabrication is approved, the process contact surfaces of the assembled piping systems and equipment, depending on the item's MOC, will be cleaned and passivated to aggressively initiate a stainless steel's natural tendency to create a passive and protective chromium-oxide-surface layer. If it is required that the assembly be qualified in accordance with a factory acceptance test (FAT), it would take place at this time. Once this process has been completed, the process contact surfaces are then cleaned, rinsed, dried and packaged for shipment.

Step 5. The H.P. packaged piping system or equipment is then shipped to the installation site

where, if it is set up as an on-time delivery with the fabricator, it will be moved directly into its permanent location. If not, the item should be placed in an area of the site in which both construction traffic and the environment are controlled. If it is required that the assembly be qualified in accordance with a site acceptance test (SAT), it would take place at any time after the assembly is installed in its permanent location, unless otherwise agreed to.

Cleanroom classifications

Cleanroom requirements and classifications were initially issued on September 11, 1992 under FS (Federal Standard) 209E. This program was accepted and adopted on an international basis for cleanroom standardization up until November 29, 2001. On that date, the U.S. General Services Administration (www.gsa.gov) issued, in part, the following notice regarding FS Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones: *"Federal Standard 209E dated September 11, 1992 is hereby canceled and superseded by International Organization for Standardization (ISO) Standards. International Standards for Cleanrooms and associated controlled environments, ISO 14644-1 Part 1: Classification of air cleanliness; and ISO 14644-2 Part 2: Specifications for testing and monitoring to prove continued compliance with ISO*

14644-1." [2]

With that notification, ISO 14644-1 and 14644-2 became the de facto international standard for cleanroom requirements. Cleanroom classification designations thereby changed from the 10, 100, 1,000, and so on classifications to the ISO 1, 2, 3, 4, and so on classifications, as reflected in Table 4. This table compares and contrasts both the FS and ISO standards, because there are many companies, catalogs, and specifications that still specify the classifications referred to in the old superseded FS 209E standard.

Designing for high purity

What separates the design of high-purity process equipment from that of typical industrial-type process equipment can be found in the consideration given to integrating design attributes of cleanliness into an overall design concept. The term "cleanability," can therefore be defined, for the purpose of this discussion, as, "adoption and application of specifications that render welded joints, mechanical joints, process-contact surface finishes, internal appurtenances, and pipe-runs as being designed to facilitate, to a satisfactory degree, their drainability by gravity or with the assist of pneumatic pressure (blow-down)."

What that definition implies is that equipment or piping designed for cleanability shall have equipment process contact internals designed

with no flat surfaces, an internal material surface finish specified to reduce interfacial tensions between a liquid and the process contact surface, and piping systems installed for drainability with slope, no pockets, and an internal material surface finish also specified to reduce interfacial tensions between a liquid and the process contact surface.

Welding. Aside from establishing an appropriate surface finish for process contact surfaces of equipment and piping, the joining of piping becomes a critical design element in the cleanability aspect of a system. While mechanical-type joints are necessary, they are inherently more prone to capture and release impurities in a system than that of a properly made welded joint. And for that reason, whether for metallic or nonmetallic material, the welded joint should be the default type joint, and mechanical-type joints used very selectively where breakout sections or equipment connections demand otherwise.

Welding metallic materials. In selecting a metallic material for piping and equipment, the welding method most widely used and selected for high-purity systems is the autogenous automatic GTAW (TIG) method performed with an orbital welder. Both the ASME BPE standard and SEMI standards F78-0304 – “Practice for Gas Tungsten Arc (GTA) Welding of Fluid Distribution Systems in Semiconductor Manufacturing Applications” and F81-1103 – “Specification for Visual Inspection and Acceptance of Gas Tungsten Arc (GTA) Welds in Fluid Distribution Systems in Semiconductor Manufacturing Applications” provide criteria for weld acceptance as well as guidance for examinations of the welds.

An autogenous weld is one in which no filler material is introduced to the weld. The weld, as shown in Figure 2, is made by fusing together only the base material of two components.

This is done by programming the welder with essential elements of the tubing that is to be welded. Once the essential parameters are programmed into the welding machine, a weld coupon is made and examined. Upon approval of the

weld coupon, production welding can begin. As long as the same size, wall thickness, tubing material and purge gas does not change, welding production can continue until any of the following occurs:

- Shift change
- Change in purge gas source
- Change in welding machine assembly (that is, weld head, weld head extension, tungsten and so on)
- Change in the power supply source
- Change in diameter or wall thickness, or both diameter and thickness

Figure 2 is a magnified photo of the cross section of two 3-in. O.D. \times 0.065-in. thick wall 316L stainless-steel sections of tubing autogenously welded together by means of an automatic orbital welder. Notice the smooth transition of both the O.D. and, more particularly, the I.D. of the weld bead as it transitions between the base material of the two sections of tubing and the weld bead between them.

An acceptable weld for high-purity fluid service is one that, upon examination, can meet the requirements found in the ASME BPE standard or the requirements found in SEMI Standard F81-1103, paragraph 7.

Welding nonmetallic materials. In selecting a nonmetallic material for piping and equipment, the welding method best suited for nonmetallic piping is BCF (bead and crevice free) fusion welding, also referred to as beadless, infrared (IR) welding, or non-contact welding. This is a nonmetallic version of automatic autogenous welding of metallic tubing. It is a multi-step process in which the ends of the components to be welded together are cleaned prior to being clamped into place in the welding machine.

After being clamped into the welding machine, the ends of the two sections of tubing are prepared by squaring the ends with a facing tool so that the end face of the two tubes are square to one another and the O.D. and I.D. of the tubes line up in accordance with specified tolerances. Butting the two ends together will then allow the machine to check fit-up alignment of the tube ends.

Prior to beginning the fusion pro-

cess, a bladder is placed inside the tubing at the joint to be fused. The expandable bladder maintains sufficient force on the I.D. of the joint, keeping the fused material, while in its liquid state, from extruding into the I.D. of the tubing. The result being an I.D. joint surface that is flush with the I.D. of the tube itself. And finally, fusion of the two sections is performed, all while the tubing sections remain blocked and clamped in the welder.

The finished joint, as seen in Figures 3, 4 and 5, is difficult, if not impossible to discern from the actual tubing. Figure 3 is a cutaway at the branch of a tee. The fusion bead width is pointed out because it is extremely difficult to discern from the base material. Due only to disruption of the base material in fusing the two components together, causing a slight variance in light reflection between the base material and the weld seam, can the seam be detected.

The same holds true in Figure 4. In looking down at reflected light coming from the invert of a sectioned piece of tubing, the seam is actually undetectable once out of reflective range of the overhead lighting. If the graphic did not indicate which way the weld seam was running it would be impossible to tell, either from the photo or viewing the piece directly.

Figure 5 is a close-up of a BCF fusion welded seam. The magenta or fuchsia color comes not from the tubing material itself, but from the reflective attitude of errant overhead lighting. If not for the disturbance of melting and re-solidifying the base material, which appears as though a wet brush had just glided over the surface of the tubing interior, the weld seam would be barely noticeable.

Standards to consult

In order to properly specify, design, procure, fabricate and handle high-purity piping, tubing, fittings, other wetted components and equipment, it will be necessary to comply with or adopt a set of standards that meet your specific needs. To do that, it is recommended that the following standards be consulted:

ASME Bioprocessing Equipment (BPE) Standard. BPE is a standard that is focused on the high-purity re-

quirements of bioprocessing, but much of its content can be utilized across the wide-ranging applications of other high-purity needs. Information on the standard can be found in Ref. 4. **The International Organization for Standardization (ISO).** This organization publishes the group of 14644 standards mentioned earlier, along with many others that can be found in Ref. 5.

Semiconductor Equipment and Materials International (SEMI). This organization has a wide range of standards and specifications that cover the high-purity manufacture of silicon wafers, integrated circuits, printed circuit boards and flat panel devices. Such standards can be found in Ref. 6, but the most relevant of those standards pertaining to our discussion here are those identified in the following:

- Process chemical standards carrying the prefix C: among other things, these standards provide guidance and specifications for the selection and use of liquid chemicals used in the semiconductor industry
- Gas standards carrying the prefix C: as with process chemicals, these standards provide guidance and specifications for the selection and use of gases used in the semiconductor industry
- Facility standards carrying the prefix F: these standards not only provide guidance for airborne molecular contaminant levels in clean environments they also provide guidance and specifications for allowable particle specification for gases, guidance on electrostatic charge, and many other criteria for pipe and tubing.

Institute of Environmental Sciences & Technology (IEST). This organization has a listing of standards and recommended practices focused on high purity that can be found in Ref. 7.

Final remarks

Manufacturing and fabricating high-purity components and equipment is a highly specialized field that requires technically qualified and disciplined personnel and procedures at all levels. It is suggested that when looking

for providers or submitting request for quotation (RFQ), rather than segregate and determine providers on price alone, the decision process should address the thoroughness of a company's written quality management system (QMS) and adherence to that program. It is also recommended that the provider's experience be a factor in the selection process. The ability and craftsmanship needed for a manufacturer or fabricator to perform and control such high-performance work is not a craft that is learned overnight. ■

Edited by Gerald Ondrey

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Author



William M. (Bill) Huitt has been involved in industrial piping design, engineering and construction since 1965. Positions have included design engineer, piping design instructor, project engineer, project supervisor, piping department supervisor, engineering manager and president of piping consulting firm W.M. Huitt Co. (P.O. Box 31154, St.

Louis, MO 63131-0154; Phone: 314-966-8919; Email: mhuitt@aol.com; URL: www.wmhuittco.com), which he founded in 1987. His experience covers both the engineering and construction fields crossing industry lines to include work on petroleum refining, chemical, petrochemical, pharmaceutical, pulp & paper, nuclear power, biofuel and coal gasification. In addition to writing numerous specifications, guidelines, papers and magazine articles on the topic of piping design and engineering, he has also authored "Bioprocessing Piping and Equipment Design — A companion guide for the ASME BPE Standard," which is considered the companion guide on the ASME Bioprocessing Equipment standard. Huitt is a past member of ISPE and CSI, and is a current member of ASME. He is a member of the B31.3 section committee, B31.3 Subgroup H on High Purity Piping, a member of four ASME-BPE subcommittees and several Task Groups, ASME Board on Conformity Assessment for BPE Certification where he serves as vice chair, a member of the API Task Group for RP-2611, and he serves on two corporate specification review boards. Huitt also authored the training program and provides training to ASME consultants wishing to audit applicants for ASME BPE Certification.

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The CPI Construction Boom: Project Delivery in a New Landscape

Companies need more creative ways to deal with complex and ever-changing market and technical challenges

Clay Gilge
KPMG

Low natural gas prices and high global demand for hydrocarbon-based chemicals have resulted in resurgence in the construction of new and existing chemical process plants in the U.S. Despite the positive growth environment, companies are taking a much more strategic approach to their capital investments as fluctuating consumer demands and commodity pricing make the future uncertain. As a result, CPI operating companies are considering a wide variety of options to remain competitive and agile, including geographic expansion and diversification of product lines.

These developments have directly impacted the planning, design and construction of new facilities. In the face of product innovations, geographic expansion and more flexible and adaptive facilities, CPI facility projects have become more complex and expensive than ever before. Gone are the days where an engineering, procurement and construction (EPC) project approach could successfully deliver a turnkey plant with minimal delay and cost overages. Companies need creative ways to meet both market challenges and the traditional challenges of any major capital project, and owners undertaking new plant projects need to address issues that weren't top risks even five or ten years ago. These include:

- *Division of responsibilities for project oversight and management.* Project activities and controls must be assigned appropriately to the individuals who are most qualified to manage and perform the task
- *Management of high volumes of transactional activity and documents.* Today, the ability to pro-

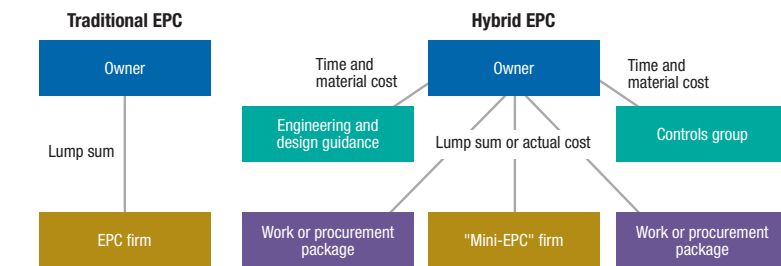


FIGURE 1. The diagram highlights the differences between these two delivery approaches

cess and analyze project information related to the project at a rapid pace is essential to leverage the performance trends to make informed decisions

- *Fraud and misconduct from project team or vendors.* Vigilance is needed to reduce the risk associated with potential exposure to inappropriate activities from internal or external project stakeholders, such as bribes, embezzlement, bid rigging and more

Addressing these items will help to ensure that any new high-quality facility is delivered on time, on budget and without legal or reputational impact for the owner.

Hybrid project strategy

To achieve success in this ever-changing market, companies need to explore transitioning from the traditional, more hands-off EPC approach, to a delivery method that is much more direct — a hybrid approach, whereby the owner assumes increased responsibility for project performance and monitoring. Additionally, today's operating companies must integrate technology solutions to support the project's key control functions, such as cost and schedule reporting, forecasting, contracting, change control, materials management, payment administration, inspections and commissioning. Un-

like the traditional EPC approach, a hybrid strategy (Figure 1) increases the number of direct vendor connections, purchase orders, contracts and management activities that are handled directly by the owner's project-management department (as opposed to being handled directly by the EPC firm on the project).

With the hybrid approach, the owner takes over many core project-management functions from the EPC contractor to maintain tighter control over project outcomes and to achieve greater cost savings. Resulting cost savings are primarily generated from the owner's ability to leverage internal positions (existing or new) to offset the need for additional fulltime equivalents that would have been provided by the EPC firm. Additionally, with owner resources administering the key cost controls, specific focus can be maintained on shielding the entity from any unwarranted or unallowable cost escalation.

Using a hybrid approach to CPI plant projects, the owner is more involved throughout the life of the project and maintains a greater connection to project activities. As a result, the owner is able to make adjustments and modifications throughout the project lifecycle to achieve improved outcomes and reduce costs.

Although changes to a project are possible using a traditional EPC ap-

proach, modifications typically take more time and have a larger cost impact than in a hybrid approach. By segmenting work packages such as site work, process piping, and others in a hybrid approach, owners can maintain flexibility and address scope changes related to new equipment selections or fluctuations in labor and material pricing. Owners can also take advantage of lower-priced options for select elements of the project.

Historically, global chemical firms and other operating companies in the CPI have traditionally aligned themselves with top EPC firms to manage their large, capital-intensive construction and expansion projects. This traditional approach can alleviate many concerns and reduce risk to a degree, but it can also be costly. By contrast, the hybrid approach discussed here allows CPI operating companies to shape the project team and take advantage of lower pricing by the following:

- *Utilizing local engineering and construction resources.* This allows the owner to engage contractors and consultants who are familiar with the regional construction requirements, workforce and customs
- *Purchasing major equipment directly without incurring EPC contractor markups.* This allows the owner to leverage the firm's internal procurement resources to negotiate favorable pricing and terms on many large mechanical components (such as turbines, pumps and compressors)
- *Supplementing the EPC contractor's project-management team with internal staff or lower-priced, third-party resources.* When the project owner is able to assemble an appropriate project team comprised of both internal and third-party contracted resources, they are often able to maximize efficiencies, reduce costs and ensure independent oversight

Managing risks

While the hybrid delivery model discussed here offers distinct advantages, increased owner involvement also invariably leads to greater exposure to many aspects of project performance. Without experienced project-management resources and strong project-delivery leadership, the owner's potential risk exposure can grow. For instance,

the hybrid approach will likely result in the owner holding multiple supplier contracts, including some with cost-reimbursable compensation terms for labor, overhead, materials, expenses and more. For these types of contracts, the owner must specifically review and validate costs, track progress and reconcile mark-ups and allowances. By necessity, this will require engagement with more internal and external resources to prevent unexpected cost overruns.

New tools and strategies

While technology is transforming (and has in some cases completely transformed) industries from retail and media to auto manufacturing, the CPI-related construction industry is in many ways still very similar to the way it was 20 years ago. Operating companies that are successfully able to rethink the entire project-management lifecycle and better understand how data are utilized on a project can achieve competitive advantage by improving the integrity of design, increasing the efficiency of construction processes and reducing operational lifecycle costs for the assets they build and operate.

Optimizing the use of staff and resources. Determining the level and type of owner or contractor resources required at each phase of a traditional major project is typically based on project manager estimates, project staffing projections or availability of project personnel. This is not only inefficient, but it often fails to leverage valuable resource and project staffing data from current and historical projects. This often occurs because current and historical data may not be readily available, or because the effort may require extensive data normalization, and there may not be internal personnel with data-analytic capabilities who can help develop staffing models and projections based on regression and other types of data analyses.

However, if structured properly and integrated into the project-resource-planning process, such data can help organizations to better plan, manage and forecast resources to align with project and organizational goals and targets. For instance, CPI project owners are often able to help make more-informed decisions regarding staffing levels and estimated project-

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TABLE 1. SIMILARITIES AND DIFFERENCES BETWEEN THE TWO APPROACHES

Traditional EPC role	Hybrid EPC role	Hybrid model differences
All design performed by the EPC	Detailed design performed by the EPC	Conceptual design, design quality assurance and quality control performed by separate, independent engineering firm retained by owner
EPC procures all equipment and materials	EPC manages the procurement of the majority of materials and small equipment and control instrumentation	Owner procures certain major equipment work packages directly
EPC manages and provides oversight for the entire project.	EPC manages and provides oversight over their contracted scope of work.	Owner engages internal or third-party control group to monitor the project and report out directly to the owner
EPC performs design and construction for all work packages	EPC performs design and construction for their contracted scope	Local design and construction firms contracted directly for specific work packages
EPC is contracted on a lump sum turnkey basis for the entire project	EPC is contracted on a lump sum turnkey basis for contracted scope of work	Pricing outside of the EPC varies between lump sum, time and material, and actual cost

resource costs when they are able to better use relevant data, implement standardized project-resource coding, and develop a consistent project taxonomy so that project attributes can be tracked and reported more effectively.

Ideally, if all projects within an organization were to utilize a common taxonomy of project attributes, and if all project team members were coded by function and time were tracked by project phase and activity, the following project metrics could be evaluated, benchmarked and utilized for real-time project estimating, forecasting and decision making:

- Workload capacity
- \$/Full time employee (FTE); for example: \$5 million/FTE/yr
- FTEs/project by project type per \$100 million
- Staffing ratios
- Vendor breakdown by category (for example, % architect/engineer, % contractor, % subcontractor), project-delivery method, region and project type
- Peak head count versus average head count by phase (for instance, by project-delivery method, by region or by project type)
- Functional staffing ratios (for instance, by % engineering, % project management, % administration, % project controls)
- Workload breakdown
- Percent of project hours by phase (for instance, by % planning, % design, % construction, % commission and closeout)
- Average hours by project activity (for instance, by project delivery method, by region, by project type)

Such detailed insight can then be utilized to rationalize staffing requests and project estimates. Additionally, companies can evaluate project-staffing curves with the ultimate goal of decreasing average project duration, improving estimating accuracy

and reducing average FTEs on a project to free up resources and increase overall output.

Improving project reporting, documentation and overall project efficiency. Implementing a project-management information system (PMIS) on a major CPI construction project can contribute significantly to its overall success. The PMIS allows project teams to make effective decisions and accurately report project status to stakeholders by maintaining constant, realtime access to project information and key performance indicators. It also addresses document- and records-management needs that result from environmental regulations and building codes. The increasing volume of construction data, the need for immediate access to it, and the challenges of maintaining confidentiality and security make the PMIS for construction a standard practice on large, capital-intensive CPI projects.

By converting to a digital standard for project documentation, project teams can automate document distribution, management and retention, process reviews and approvals. A key consideration for implementing this new standard would be to ensure that all project vendors and consultants are supportive of the preferred PMIS and will fully leveraging it with their work streams.

Using cloud and mobile technology to improve the way data are captured and utilized on a project to improve performance. Project teams throughout the CPI are also beginning to leverage cloud and mobile technologies to capture and share data in realtime and quickly analyze, assess and report on projects. Cloud and mobile technologies are becoming more cost-effective and permit even remote project sites to connect with teams at corporate headquarters. For example, the use of computer tablets to

capture and record field inspections, site walks, progress and permit status can provide realtime tracking and scheduling monitoring for all stakeholders, regardless of their location. If this technology is coupled with building information modeling (BIM) and regularly maintained progress photos, then stakeholders can get a true sense of construction activity and troubleshoot problems or questions as they arise without necessarily being physically present onsite.

CPI companies that have implemented cloud-based PMISs with mobile capabilities have experienced many benefits, including:

- Better integration of cost and schedule reporting
- Improved change-order management
- Reduced cycle times
- Secured and centralized management of project information
- Automation of information requests, submittals and the solicitation processes

Reducing project risk and operating costs with data analytics. Reviewing a baseline schedule (which can easily involve 40,000 activities) for a CPI plant-construction project in a matter of hours is impossible without the help of automated data-analytic tools. Schedule-related tools have enabled project teams to quickly assess the overall quality of schedules submitted by contractors and subcontractors. This not only simplifies the project team's understanding of large, mega-scale project schedules, but it also helps to reduce overall project risk. For instance, with the help of available data-analytic tools, schedule discrepancies can be addressed early in the project lifecycle, before larger, more costly issues arise.

The use of data-analytic tools can also help project owners to manage and mitigate complex construction

claims. Identifying claims-related costs at the end of a project can be a monumental effort riddled with assumptions and exclusions if impacts are not monitored from the outset of a potential claim. CPI operating companies with extensive claims experience understand the value of continuous cost tracking and monitoring of project schedules to manage associated claim impacts with improved analytics.

Gaining competitive advantage by implementing a project-data strategy to take advantage of current and historical project data.

The project team should consider the following to make the best use of cross-project data:

- How data will be captured and used
- Whether an integrated PMIS will be utilized to track, manage, report, share and archive project documents and records
- Whether there will be a common data taxonomy, project coding and work-breakdown structure leveraged across the entire project
- How to leverage past data to build a predictive model that proactively alerts you when performance is likely to be impacted
- How technology requirements and model results will be shared and used by contractors and subcontractors
- How remote users will access project documents, and what

level of security protocols will be employed

Although operating companies have historically been reluctant to take full advantage of technological advances, it is clear that many of these industries are poised for transformation. Leveraging historical and current project data while making better use of cloud and mobile technology can help to unlock the potential of project-management information systems for other business uses, as well. For instance, improved data analytics will help to improve project-management processes, allowing for better program-wide decisions to be made, providing improved visibility into project- and portfolio-level impacts, and improving capital project and portfolio performance.

CPI companies that are planning to embark on a facility construction project often face hurdles that can prevent successful project execution. However, many of today's leading companies are mitigating this with innovative approaches to project delivery, and by leveraging technology. This is helping them to adapt to market demands while improving project visibility and control.

Specifically, CPI operating companies that have embraced the changing landscape of project delivery have benefited significantly through improved project performance, increased cost predictability and an improved ability to identify and miti-

gate risks before they are able to have a significant impact on the project. Selecting the most appropriate delivery structure and project-system infrastructure can help to set any capital-intensive project on a track for success, and help to ensure that the firm is appropriately equipped to deliver the project to completion.

Although establishing a hybrid delivery model and PMIS system requires a higher upfront investment cost compared to the status quo, these investments are very likely to produce a return on investment that will greatly exceed the initial investment. With an adequately structured and equipped project-delivery model, numerous risks will be avoided and efficiencies gained. ■

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Eye on Flare Systems

Proper gas sampling is essential to meet operating and regulatory objectives

Michael Bequette
SOR Controls Group, Inc.

Flares are used extensively in chemical process industries (CPI) facilities and petroleum refineries, to combust flammable hazardous gases. When designed and operated properly, they provide a proven mechanism to handle upset conditions in a safe manner. For instance, flares are typically used to handle the unwanted gas streams produced during upset conditions and to reduce overpressure conditions (for instance, when a pressure-relief valve or rupture disk is used to help reduce natural gas pressure in a line). Similarly, during oil-and-gas production, when natural gas is extracted with the petroleum, flaring the gas stream may be the only alternative if the infrastructure to pipe the gas to another location is not in place.

When flares are used to combust a waste stream from a process, the

composition of the post-combustion exhaust gases will depend on the composition of the inlet gas received by the flare. To protect workers, neighboring communities and wildlife around the plant, and to reduce long-term impacts on the environment, a variety of state and federal requirements are in place to regulate the emissions stream. Such regulations seek to control the type and amount of specific air pollutants that are discharged from flares, and they typically require operating facilities to quantify the amount of pollutants in the exhaust stream that is ultimately discharged.

Hazardous air pollutants (HAPs; referred to as toxic air pollutants), create significant environmental



FIGURE 1. Flares are widely used to destroy unwanted pollutants, but proper sampling is required to ensure that their own emissions meet regulatory limits

concerns and potential health risks. HAPs have been associated with cancer and neurological, respiratory, and reproductive problems. In addition to health concerns, such emissions contribute to acid rain, smog, water supply pollution and climate change. The U.S. Environmental Protection Agency (EPA) currently lists 187 compounds as HAPs, many of which are commonly generated during CPI process operations [1].

Most HAPs are defined by the mode of production; for instance mobile sources (exhaust gases from vehicles), stationary sources (HAPs produced by petroleum refineries, power plants and other CPI facilities), and indoor sources (this includes HAPs produced by activities such as cleaning).

Regulatory framework

In 1990, the U.S. Clean Air Act (CAA) was amended to drive compliance by establishing actionable dates to achieve the reduction in the amount of HAPs released. As a result of the 1990 CAA Amendments, a new requirement of technology-based standards emerged for so-called major sources and certain area sources. Major sources are stationary sources that produce (or have the potential to produce) 10 ton/yr or more of any

TABLE 1. COMMON SOURCES OF LEAKAGE AND FUGITIVE EMISSIONS [3]

Equipment	Source of leaks
Pumps are used to move fluids from one point to another. Two types of pumps extensively used in petroleum refineries and chemical plants are centrifugal pumps and positive displacement, or reciprocating pumps	Leaks typically occur at the seal
Valves are used to either restrict or allow the movement of fluids. Valves come in numerous varieties and with the exception of connectors, are the most common piece of process equipment in the CPI	Leaks usually occur at the stem or gland area of the valve body and are commonly caused by a failure of the valve packing or O-ring
Connectors are components such as flanges and fittings used to join piping and process equipment together. Gaskets and blinds are usually installed between flanges	Leaks commonly result from gasket failure and improperly torqued bolts on flanges
Sampling connections are utilized to obtain samples from within a process	Leaks usually occur at the outlet of the sampling valve when the sampling line is purged to obtain the sample
Compressors are designed to increase the pressure of a fluid and provide motive force. They can have rotary or reciprocating designs	Leaks most often occur from the seals
Pressure-relief devices are safety devices designed to protect equipment from exceeding the maximum allowable working pressure. Pressure-relief valves and rupture disks are examples of pressure-relief devices.	Leaks can occur if the valve is not seated properly, operating too close to the set point or if the seal is worn or damaged. Leaks from rupture discs can occur around the disk gasket if not properly installed
Open-ended lines are pipes or hoses open to the atmosphere or surrounding environment	Leaks occur at the point of the line that is open to the atmosphere; they are usually controlled by using caps, plugs and flanges. Leaks can also be caused by the incorrect implementation of the block-and-bleed procedure

single chemical or 25 ton/yr or more of HAPs; area sources are stationary sources that do not follow the definition of a major source.

For these major sources of HAPs, EPA has established metrics for emissions that will determine the effectiveness of the control technology — commonly referred to as Maximum Achievable Control Technology or MACT standards [2]. Meeting the MACT standards doesn't necessarily require the addition of costly emissions-control systems, such as scrubbers and thermal oxidizers; rather, in some cases, the MACT standards can be met through modifications to processes and methods that can both determine and limit the quantity of HAPs being produced.

These MACT standards constrain users to specific objective emissions parameters. MACT standards are set by EPA (for each source category) based on the emissions performance that has been achieved by the best-performing processes in that category. This level is then set as the baseline throughout the in-

dustry. The MACT standards establish the so-called National Emission Standards for Hazardous Air Pollutants (NESHAPs), which specifically exist to safeguard against emissions of HAPs from major sources.

Leakage from valves, flanges, connectors and pumps are some of the leading sources for the release of HAPs and other volatile organic compounds (VOCs). According to EPA's Leak Detection and Repair (LDAR) Best Practices Guide [3]: "A typical refinery or chemical plant can emit 600–700 ton/yr of VOCs from leaking equipment, such as valves, connectors, pumps, sampling connections, compressors, pressure-relief devices and open-ended lines." Two methods are typically used to reduce emissions from leaking equipment: the use of "leakless components," and implementation of LDAR techniques. LDAR refers to a methodology that reduces emissions from leaks by identifying the leak as it occurs and repairing it within regulated time frames. Table 1 shows common sources of leaks.

Sampling systems for flares

To comply with LDAR, CPI operators must quantify the amount of pollutants being released. In order to properly and safely measure pollutants from flares, a sample of flare emissions must be taken. Automatic sampling systems provide a safe and environmentally friendly way to extract needed samples. However, if not properly installed and maintained, sampling systems themselves can be a source of leaks and they too must comply with LDAR.

Sampling systems come in many different styles and configurations, and no two sampling systems are exactly alike. For example, some sampling systems can take a process sample with operator interaction, while others can automatically collect samples without any operator interaction. Each approach has its pros and cons.

Manual sampling. Manual operation allows for surveillance of the process at random (unscheduled) times. In some instances, due to variability in the operating conditions

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FIGURE 2. Proper sampling location and stream conditioning can help to ensure the best results

(for instance, those that are not accounted for in the early design of a sampling system), automatic sampling may in fact not allow for a true representation of the process emissions. In these cases, manual sample-collection capabilities can help to address such variability.

For example, randomly sampling when the temperature has dropped at night may show differences in samples compared to those drawn at some automatic interval. Manual operation also allows for repeated samples to account and correct for special circumstances that may have inappropriately influenced the sample collection (making it not representative of the source). For example, if a process has a new blending agent, achieving consistent operation may take several attempts to get the desired mixture. Thus, several manually drawn samples may be needed in order to verify correct measures.

Automatic sampling. On the other hand automatic sampling systems (which require no operator involvement) can look at non-standard flare events and quantify pollutants such as NO_x, SO₂ and other chemicals. Such systems allow operators to set up a system for sample collection to coincide with a timed schedule or anticipated events. In addition, automatic sampling systems meet the regulatory requirements of 40 CFR 50.4 [4], in terms of capturing regular timed samples for SO₂ sampling in a defined time period. Appendix A of those federal regulations [4] define the acceptable requirements for sample probe, absorber, moisture trap, flow control and measurement, particulate-mat-

ter filters, temperature control, vacuum pump, and sample bottles that ensure that the sampling system and procedures do not negatively interfere with (and thus invalidate) sample collection.

Functions of a sampling system

Sampling systems for flares typically have the following six functions:

1. To take a representative sample that is based upon the specific needs of the application
2. To condition and treat a sample so that it can be used with an analyzer. This may include removing solids, moisture, or providing temperature control
3. To switch sample streams in order to get multiple reference samples of a process
4. To handle caustic, hazardous, and extreme environmental conditions that could adversely impact the operator or environment when attempting to extract the sample
5. To allow transport of the sample for analysis
6. To allow for a mechanism to dispose of the sample

As noted, sampling systems typically are of a time-based, flow-based, or volume-based scheme, with samples triggered off of events associated with these schemes. These schemes can be single action or a combination of events.

- *Time-based sampling* will attempt to fill a cylinder over a pre-determined amount of time, although the sample period within any given time-based sampling can vary. Time-based sampling systems are usually used when there is a continuous flowrate or the material composition is thought to be constant
- *Flow-based sampling* is designed to take samples in proportion to the flowrate. These systems have sample rates that are dependent on the flowrate and may increase or decrease in relation to flowrate, unlike time-based systems
- *Event-based sampling* systems take samples when a specific event triggers the operation; these may include an overpressure condition, changes in density, opacity or some other monitored variable

Many may argue: Why not just use a continuous analyzer to accomplish sampling requirements? As noted

earlier, flares typically handle upset conditions. Therefore, under normal conditions, the flowrates, pressures or caustic concentrations may be measurable in accordance with design conditions — but when compared to an upset condition where flowrates, pressures, and so on are very high, accurate measurement may not be attainable.

Providing a mechanism to analyze the stream continuously is very costly and technically challenging. In particular, most analyzers cannot be calibrated to handle the high flow conditions while still operating at the very low range and staying in calibration. This dynamic range condition of most flares challenges many instrument manufacturers. Manual grab-sampling systems help to address these challenges. However, not all grab-sampling systems on the market today are able to comply with MACT, LDAR and NESHAP standards.

Choosing a sampling system

Historically, manual grab-sampling systems were constructed with an open tap (two-way valve) or Strahman-style valve. This approach effectively provides no vent capture or fast loop to prevent operators from taking a bad sample or exposing the sample to the environment. By comparison, today's improved sampling systems that comply with the regulatory standards address this earlier design deficiency as part of their modern design. Specifically, effective sampling systems that ensure compliance to MACT, LDAR, and NESHAP requirements have a closed-loop design and a closed vent. Meanwhile, for operators, the following industry best practices can help to ensure the most appropriate sample collection, to ensure regulatory compliance:

- Ensure there is no condensation or other issues that could interfere with the sample
- Understand material compatibility to the process stream to ensure that the system is inert to the process
- Ensure an adequate purge system to remove residual sample material from the system
- Understand remote locations in terms of how sample collection will tie into the process line

- Choose a placement location in the process line that will get a truly representative sample
- Filter to separate liquid mixtures
- Make sample lines as short as possible
- Design to prevent pressure drop across valves and fittings
- Design to use as few fittings as possible to prevent potential leak points, and use weld fittings as needed

Sampling systems are typically custom designed and engineered at the time of order. They can involve a very complicated design in order to guarantee that they will get a true representative sample from a process. Depending on the application and the nature of the gases in the emissions stream, phase changes may occur, and if appropriate precautions are not taken, damage to expensive analyzers could result.

Meanwhile, if the samples that are collected for analysis are not truly representative of the process, inaccuracies will occur. This will lead to erroneous or misleading readings,

which can have serious ramifications in terms of increased environmental pollution, and increased costs to the end user, such as loss of product or inefficient process conditions.

Minimize sample contamination

Handling the sample can be a potential point of contamination. Common errors that can lead to sample contamination (and thus misleading readings by the analyzer) result from the following activities:

- Opening the valve on the cylinder to determine if the cylinder is still under vacuum
- Opening the valve on the cylinder to verify whether there is a sample in the cylinder
- Improper selection of valves and other system components

Meanwhile, some components may not be designed for use with vacuum and thus can result in leakage that brings in outside air. In addition, placement of the sampling system and its interface into the process can introduce tremendous variability. ■

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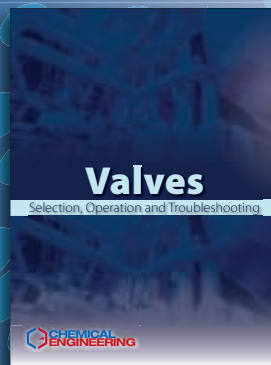
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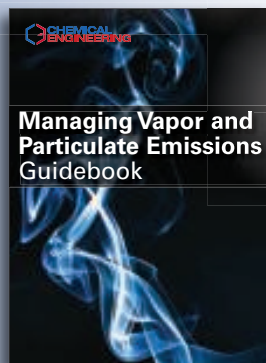


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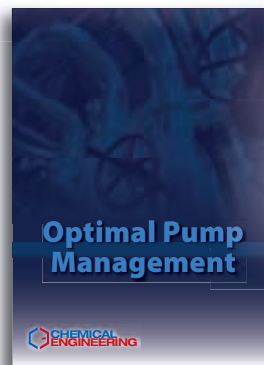
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- 22 Engineering
- 23 Research & Development
- 24 Safety & Environmental
- 26 Other

EMPLOYEE SIZE

- 28 Less than 10 Employees
- 29 10 to 49 Employees

- 30 50 to 99 Employees
- 31 100 to 249 Employees
- 32 250 to 499 Employees
- 33 500 to 999 Employees
- 34 1,000 or more Employees

YOU RECOMMEND, SPECIFY,

PURCHASE

(please circle all that apply)

- 40 Drying Equipment
- 41 Filtration/Separation Equipment
- 42 Heat Transfer/Energy Conservation Equipment
- 43 Instrumentation & Control Systems
- 44 Mixing, Blending Equipment
- 45 Motors, Motor Controls
- 46 Piping, Tubing, Fittings
- 47 Pollution Control Equipment & Systems

- 48 Pumps
- 49 Safety Equipment & Services
- 50 Size Reduction & Agglomeration Equipment
- 51 Solids Handling Equipment
- 52 Tanks, Vessels, Reactors
- 53 Valves
- 54 Engineering Computers/Software/Peripherals
- 55 Water Treatment Chemicals & Equipment
- 56 Hazardous Waste Management Systems
- 57 Chemicals & Raw Materials
- 58 Materials of Construction
- 59 Compressors

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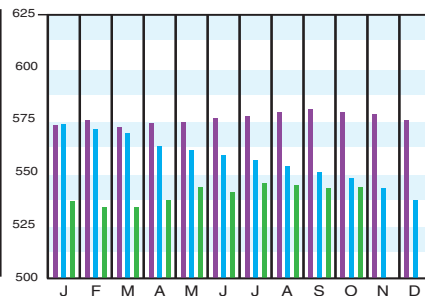
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CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	Oct. '16 Prelim.	Sept. '16 Final	Oct. '15 Final
CE Index	543.3	542.8	547.2
Equipment	647.7	647.4	654.9
Heat exchangers & tanks	557.2	556.7	575.4
Process machinery	653.3	653.6	655.0
Pipe, valves & fittings	811.0	813.6	808.6
Process instruments	390.2	390.1	390.1
Pumps & compressors	966.0	966.0	956.4
Electrical equipment	511.5	510.9	508.2
Structural supports & misc	710.4	705.9	723.6
Construction labor	329.8	328.3	325.8
Buildings	546.8	547.3	540.4
Engineering & supervision	314.7	314.4	317.7

Annual Index:
 2008 = 575.4
 2009 = 521.9
 2010 = 550.8
 2011 = 585.7
 2012 = 584.6
 2013 = 567.3
 2014 = 576.1
 2015 = 556.8

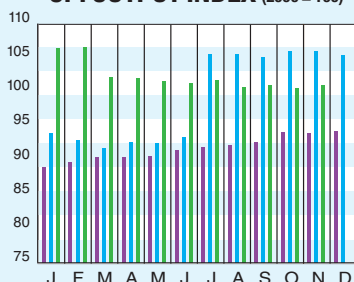


Starting with the April 2007 Final numbers, several of the data series for labor and compressors have been converted to accommodate series IDs that were discontinued by the U.S. Bureau of Labor Statistics

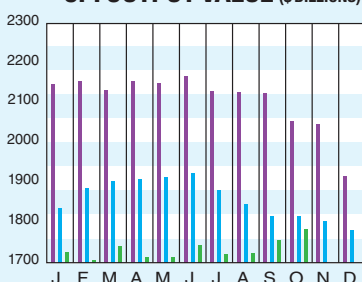
CURRENT BUSINESS INDICATORS

	LATEST	PREVIOUS	YEAR AGO
CPI output index (2012 = 100)	Nov. '16 = 101.0	Oct. '16 = 100.7	Sept. '16 = 100.4
CPI value of output, \$ billions	Oct. '16 = 1,785.2	Sept. '16 = 1,757.8	Aug. '16 = 1,731.2
CPI operating rate, %	Nov. '16 = 74.1	Oct. '16 = 74.0	Sept. '16 = 73.7
Producer prices, industrial chemicals (1982 = 100)	Nov. '16 = 239.8	Oct. '16 = 233.4	Sept. '16 = 230.0
Industrial Production in Manufacturing (2012=100)*	Nov. '16 = 103.2	Oct. '16 = 103.2	Sept. '16 = 102.9
Hourly earnings index, chemical & allied products (1992 = 100)	Nov. '16 = 169.4	Oct. '16 = 170.9	Sept. '16 = 169.8
Productivity index, chemicals & allied products (1992 = 100)	Nov. '16 = 100.7	Oct. '16 = 99.6	Sept. '16 = 100.2

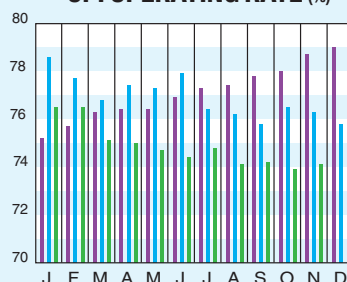
CPI OUTPUT INDEX (2000 = 100)†



CPI OUTPUT VALUE (\$ BILLIONS)



CPI OPERATING RATE (%)



*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.
 †For the current month's CPI output index values, the base year was changed from 2000 to 2012
 Current business indicators provided by Global Insight, Inc., Lexington, Mass.

CURRENT TRENDS

The preliminary value for the October CE Plant Cost Index (CEPCI; top; the most recent available) edged slightly higher than the previous month's value, reversing the small decline since July. The Equipment, Construction Labor and Engineering & Supervision subindices all ticked upward, offsetting a small decrease in the Buildings subindex. The preliminary October 2016 CEPCI value stands at 0.7% lower than the corresponding value from October 2015. Meanwhile, the latest Current Business Indicators (CBI; middle) for November 2016 showed a slight increase in the CPI Output Index and the Productivity Index. The October 2016 number for CPI Value of Output also increased compared to the previous month.



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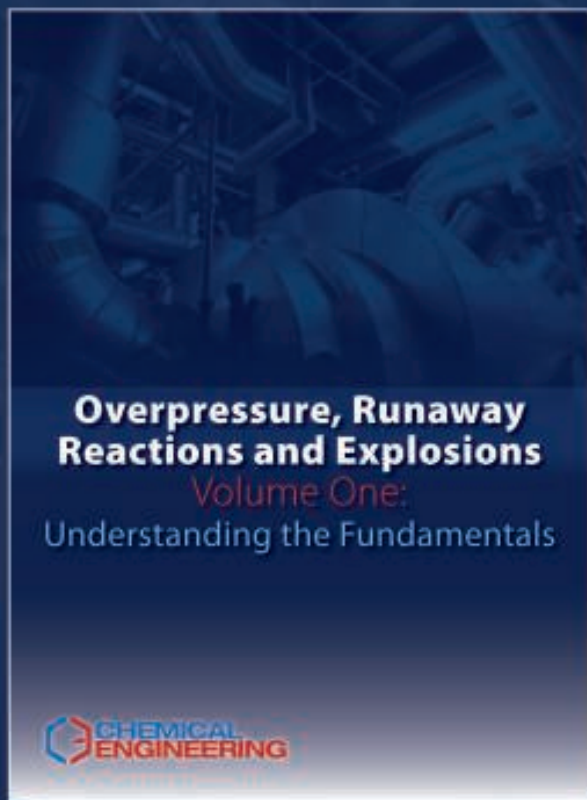
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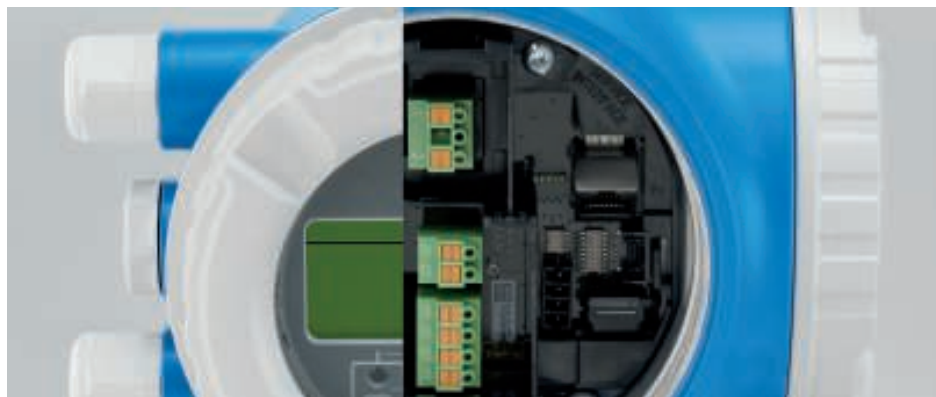
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