CHEMICAL ENGINEERING

October 2012

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CONTINUOUS VERSUS BATCH PROCESSING

PAGE 34

Process Vacuum Systems

Dry Solids Mixing

Facts at your Fingertips: Fans and Blowers

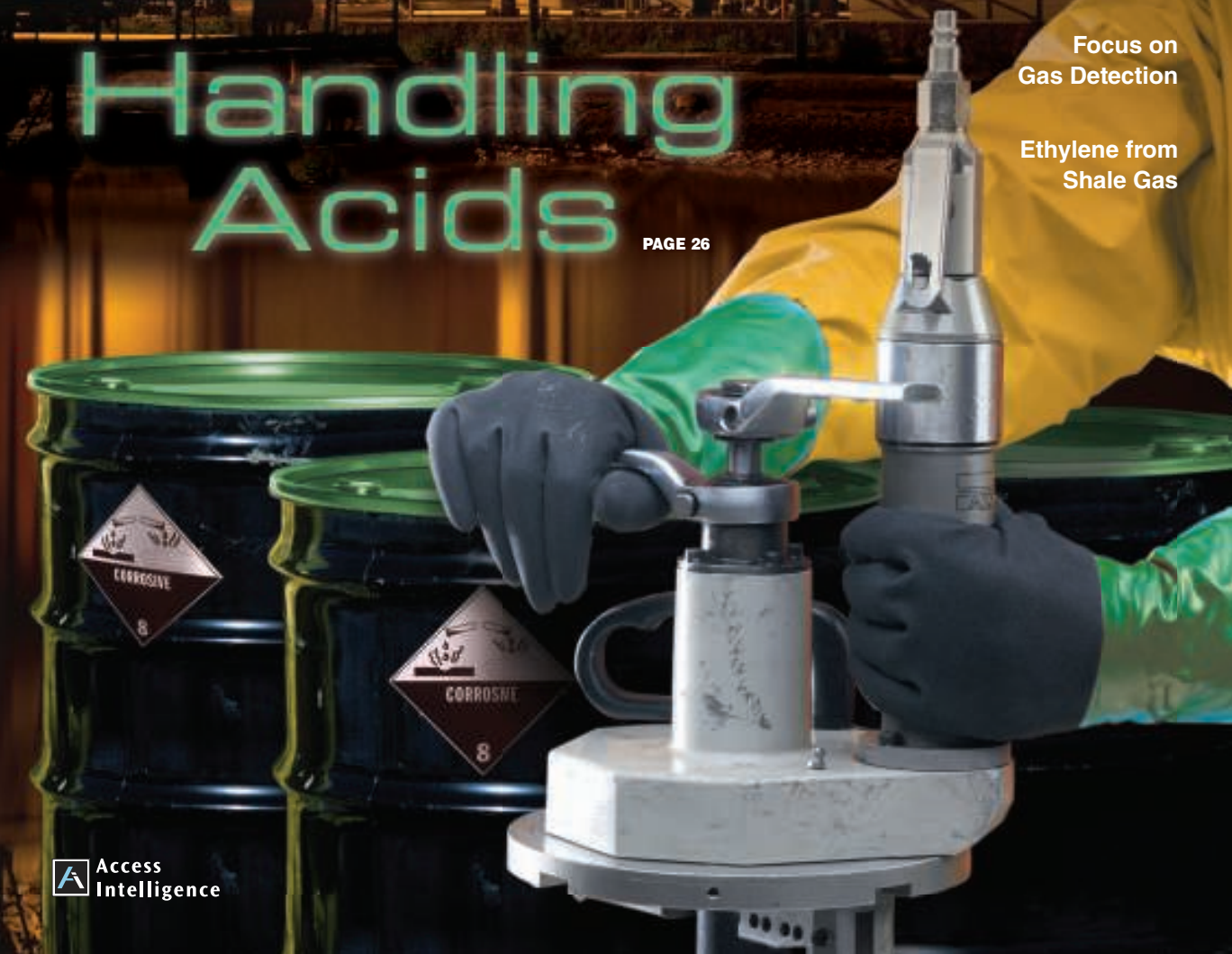
Steam Systems

Focus on Gas Detection

Ethylene from Shale Gas

Handling Acids

PAGE 26



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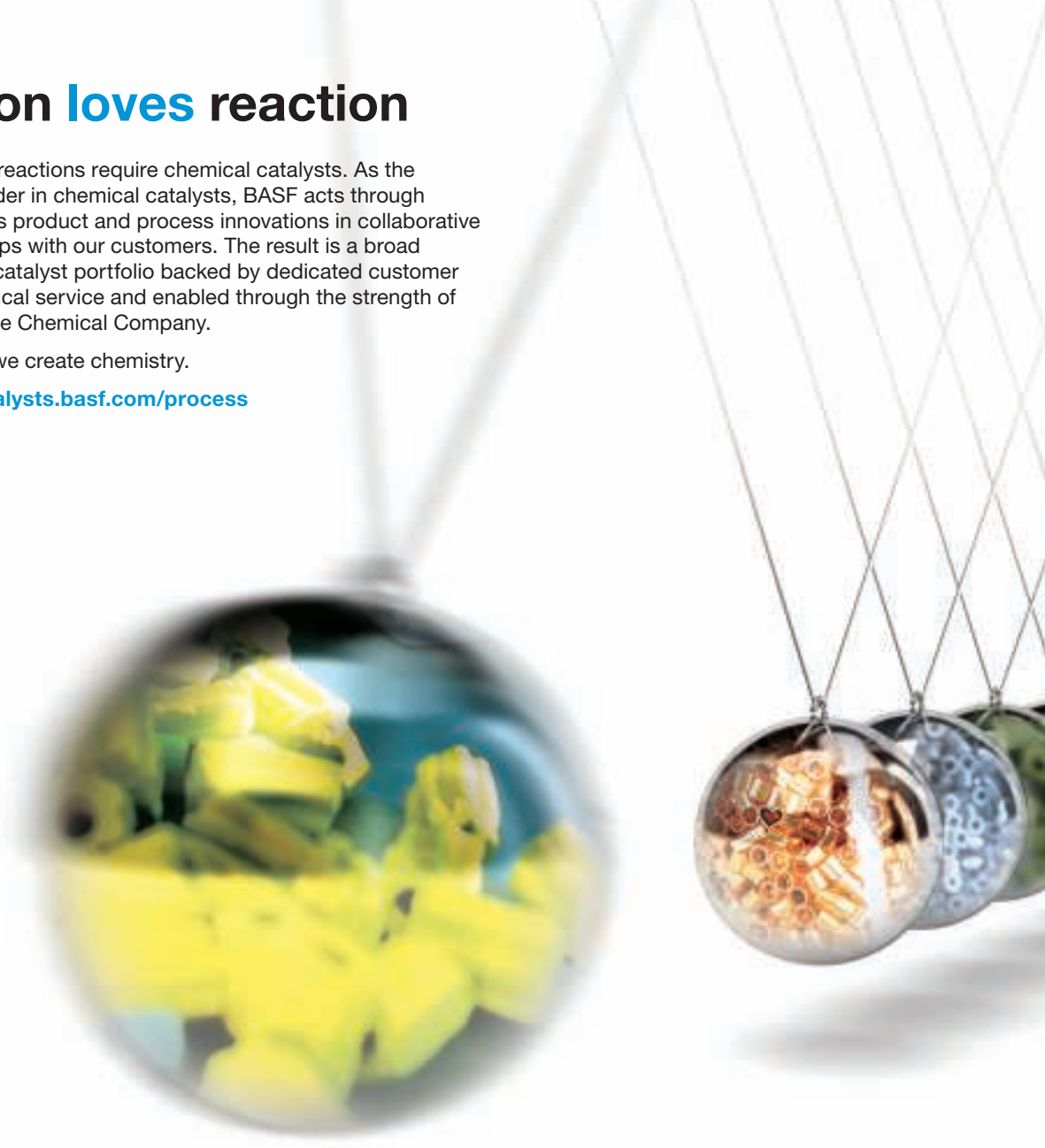

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Tel: 212-621-4900 Fax: 212-621-4694

EUROPEAN EDITORIAL OFFICES
Zeilweg 44, D-60439 Frankfurt am Main, Germany
Tel: 49-69-9573-8296 Fax: 49-69-5700-2484

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BPA

Editor's Page

Water and energy are linked

This month marks the 40th anniversary of the Clean Water Act (CWA), the primary federal law regulating discharges of pollutants into U.S. waters. As far as the developed world's water priorities go, a great deal has changed since 1972. For one thing, the top challenge back then was eliminating releases of toxic substances. Today, that challenge is arguably being met, and we are now getting more serious about minimizing the consumption of water altogether.

Despite that growing intention, a sometimes-overlooked fact is that water and energy consumption are interdependent. Whether the context is the chemical process industries (CPI) or much broader, the truth is this: The more water that is consumed, the more energy that is needed, and vice versa. The connection can be illustrated very clearly by looking at an everyday example from the U.S. Environmental Protection Agency (EPA; Washington, D.C.; www.epa.gov). Approximately 4% of electricity demand in the U.S. is devoted simply to moving and treating drinking water and wastewater. Conversely, it takes 3,000–6,000 gal/yr of water to power just one 60-W, incandescent light bulb for 12 h/d.

In the CPI, water requires significant energy input for pumping, heating, process uses and treatment (both on the front and back ends). So even if cost savings in water use are not incentive enough on their own, in many cases reductions in energy use could make up the difference, and sometimes more. Meanwhile, shifts in energy and water supplies promise to increase the stakes moving forward.

Consider for a moment that emerging energy supplies are not likely to require less water per unit of energy, and in some cases they will require much more. Take the practice of hydraulic fracturing, for instance, which uses vast volumes of water and is increasingly being applied to both oil and gas exploration. Meanwhile, many emerging biobased routes to both energy and chemicals — including those from crops or algae — are based on mechanisms that need water to grow. Perhaps innovation in these areas will improve this state of affairs, but it is doubtful that anything would result in a net decrease in global, or even regional, water demand.

Now think about the fact that as fresh-water resources become more scarce, the energy required to deliver a gallon of water could itself be on the rise. If demand increases enough to require that significantly more water be sourced through desalination processes, that would in itself magnify the energy drain. Desalination is power intensive.

With that in mind, it is time that the CPI start looking at water and energy bills together. In addition to reducing operating costs today, it can also get a head start on a brewing problem that is poised to get even more profound.

This month's Equipment News Roundup (p. 20), presents new innovations that can achieve big benefits in one of the most obvious connections between water and energy efficiency: steam.

Meanwhile, in the *CE* archives (www.che.com) there is a wealth of practical, how-to guidance on this subject. For instance, the article Wastewater Treatment: Energy-Conservation Opportunities* presents details on five specific proposals:

- Use variable frequency drives (VFDs) to adjust the speed of electric motors to meet process demand
- Replace old electric motors
- Maximize the production and use of biogas as a fuel
- Switch to a distributed effluent cooling system
- Optimize aeration and oxygen transfer ■

Rebekkah Marshall

* www.che.com/technical_and_practical/5404.html

More opinions on ChE education

I would like to contribute to the discussion on "practical" ChE education (*CE*, September 2012, p. 5–6). After graduation, I as well didn't know what a pipe flange was, nor a gasket, didn't know how to read a pump curve, didn't know how to size a relief valve and so on. I could have learned more that applies to the CPI [chemical process industries] in an operator training (process technology) curriculum that's offered at the local community college than I ever learned in college. The problem as I see it is that the faculty of universities are trying to train students to become like them. But very few actually go on to become teaching professors since the salaries are better in industry.

While I agree with Mr. Blanton's suggestion that internships are the way to go, opportunities are limited these days. Those lucky individuals who get an internship represent a tiny percentage of those who are eligible. The rest of us (like myself at the time) end up in manual labor summer jobs with no practical experience upon graduation.

I disagree with Mr. Bloss's assumption that the "engineering field is so broad that it is difficult for any university to meet the need of every industry." Isn't the ChE curriculum itself broad enough supposedly to meet the needs of all chemical engineers? Are you telling me that a centrifugal pump used in the plastics industry is different from one used in a biofuels plant? Common practical basics that can and should be taught to engineers bound for industry.

I also believe there is no reason why you can't combine engineering theory and practicality in one four-year curriculum. The amount of math and theory taught in a standard ChE program is far too excessive for those going on to industry, and there's no reason why you can't trim the fat. Like you say, eliminate the Laplace Transforms and the linear algebra that nobody's ever going to use. (I did apply a differential equation one time on the job, but that was early on and I still had to go back to the textbook to remember how its done.) For example, in the process controls class, devote one half of the semester to theory, and one half to a practical lab where one can do hands on tuning of control valves on a DCS simulation of a process plant.

I believe it is the responsibility of universities to provide more practical engineering options, and they are doing all of us a disservice by not offering an applied-ChE degree. I remember other articles discussing how it is difficult to pass the vast knowledge accumulated by retiring engineers onto the newly-hired, and a more practical degree would go a long way in shortening this gap. In some areas, new engineers could essentially "hit the ground running".

Jeff Kinsey, process engineer
Houston, Tex.

Christine Banaszek
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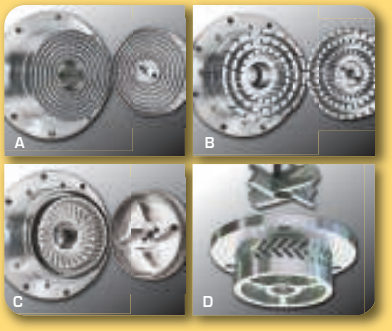

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Calendar

NORTH AMERICA

2012 Gasification Technologies Conference. Gasification Technologies Council (Arlington, Va.). Phone: 703-276-0110; Web: gasification.org
Washington, D.C. **October 28–31**

2012 AIChE Annual Meeting. AIChE (New York, N.Y.). Phone: 203-702-7660; Web: aiche.org
Pittsburgh, Pa. **October 28–November 2**

AFPM International Lubricants & Waxes Meeting. American Fuel & Petrochemical Manufacturers (AFPM; formerly NPRA) (Washington, D.C.). Phone: 202-457-0480; Web: afpm.org
Houston **November 1–2**

Carbon Black World 2012. Smithers Rapra (Shrewsbury, U.K.). Phone: 207-781-9618; Web: carbonblackworld.com
San Diego, Calif. **November 7–9**

1st International Forum on Commercializing Global Green: From Raw Materials to International Brands. Society for the Commercial Development of Industrial Biotechnology (SCD-iBIO, a SOCMA affiliate (Washington, D.C.). Phone: 202-721-4100; Web: scd-ibio.org
Philadelphia, Pa. **November 12–14**

2012 Aveva World North America User Conference. Aveva (Houston). Phone: 832-204-5623; Web: aveva.com
New Orleans, La. **November 12–14**

3rd Annual ChemInnovations Conference & Exhibition, co-located with Clean Gulf/Industrial Fire, Safety and Security, and Shale EnviroSafe Conference & Exhibitions. TradeFair Group, an Access Intelligence Co. (Houston). Phone: 713-343-1891; Web: cpievent.com
New Orleans, La. **November 14–15**

Silicon-Containing Polymers & Composites. American Chemical Society, Polymer Chemistry Div. (Washington, D.C.). Phone: 540-231-3029; Web: polyacs.net/workshops/12silicon/home.htm
San Diego, Calif.. **December 9–12**

SOCMA's 91st Annual Dinner. SOCMA (Washington, D.C.). Phone: 202-721-4165; Web: socma.com
New York, N.Y. **December 10**

Power-Gen International. PennWell (Dallas, Tex.). Phone: 888-299-8016; Web: power-gen.com
Orlando, Fla. **December 11–13**

EUROPE

7th World Mycotoxin Forum and 13th International IUPAC Symposium on Mycotoxins & Phycotoxins. Bastiaanse Communication (Blithoven, The Netherlands). Phone: +31-30-2294247; Web: wmfmeetsiupac.org
Rotterdam, The Netherlands **November 5–9**

Water Wastewater & Environmental Monitoring (WWEM) 2012. International Labmate Ltd. (St. Albans, Hertfordshire, U.K.). Phone: +44-1604-879-861; Web: wwem.uk.com
Telford, England **November 7–8**

New Horizons in Catalysis: The Art of Catalysis in Chemistry. Scientific Update (E. Sussex, U.K.). Phone: +44-1435-873062; Web: scientificupdate.co.uk
Prague, Czech Republic **November 19–20**

Valve World Expo 2012, 8th Biennial Valve World Conference & Exhibition. Messe Düsseldorf North America (Chicago, Ill). Phone: 312-781-5180; Web: mdna.com
Düsseldorf, Germany **November 27–29**

International Electronics Recycling Congress (IERC 2013). ICM AG (Birrwil, Switzerland). Phone: +41-62-785-1000; Web: icm.ch
Salzburg, Austria **January 16–18, 2013**

Interplastica 2013 — 16th International Trade Fair Plastics and Rubber. Messe Düsseldorf North America (Chicago, Ill.). Phone: 312-781-5180; Web: mdna.com
Moscow, Russia **January 29 – February 1, 2013**

ASIA & ELSEWHERE

Electronics Recycling Asia. ICM AG (Birrwil, Switzerland). Phone: +41-62-785-1000; Web: icm.ch
Guangzhou, China **November 13–16**

Industrial Pumps, Valves and Systems (IPVS) Trade Fair & Conference — India 2012. Orbitz Exhibitions Pvt. Ltd. (Mumbai, India). Phone: +91-22-2410-2801; Web: ipvs.in
Pune, India **December 14–16** ■

Suzanne Shelley

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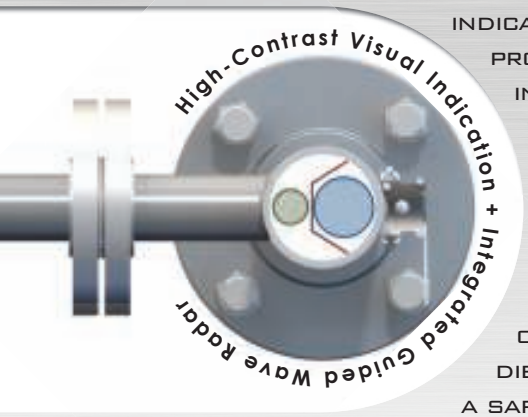

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A cryogenic turbo mill generates rubber powder from recycled tires

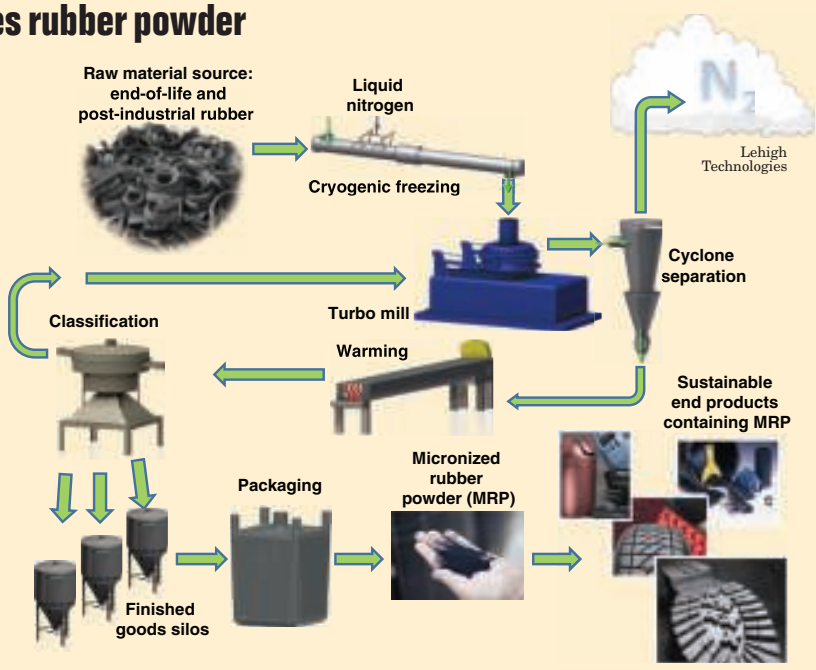
A process developed by Lehigh Technologies (Lehigh; Tucker, Ga.; www.lehightechnologies.com) for embrittling and milling rubber from end-of-life vehicle-tire material is capable of generating micron-scale rubber particles that can be used in a host of rubber, foam and plastic products to lower costs while maintaining performance. The process (flowsheet) depends on a specially designed turbo mill that Lehigh CEO Alan Barton likens to "a jet engine with teeth." Spinning at 2,000 rpm, the mill's turbine rotates rows of plates that are studded with small metal protrusions. These metal "teeth" impact cryogenically frozen rubber chunks that are fed into the mill.

Through a carefully orchestrated addition of liquid nitrogen, the recycled tire rubber is taken to a temperature below its glass-transition temperature (T_g), which renders the rubber chunks brittle, explains Barton. The frozen rubber is fragmented in the mill into micronized rubber powder (MRP), with an average particle size distribution in the range of 105–400 µm.

"We can shift the distribution to larger or smaller particle sizes, as well as make it narrower, by altering our proprietary liquid-nitrogen addition technique and by changing the pattern of teeth inside the turbine," says Barton. The MRP that comes off the turbine is classified using cyclones to obtain the desired particle sizes for particular applications, he adds.

The powder can then be introduced into customer processes, such as tire making and plastics production, to replace a percentage of virgin material, extending the normal feedstock and saving money. For example, the powder is used at levels from 3–7% in tire manufacturing, and from 5–40% in plastics production.

The main benefit of using MRP is feedstock cost savings (by displacing a portion of the original material), and in some cases, the MRP imparts improved properties. Barton cites examples where the powder makes plastic floor tiles softer and less slippery, and its addition to polyurethane foam for car interiors acts as a sound deadener. The rubber powder also produces a smoother, quieter ride when added to road asphalt.



Save energy (and more) with this hybrid compressor system for EOR

Last month, Lewa GmbH (Leonberg, Germany; www.lewa.de) and Burckhardt Compression AG (Winterthur, Switzerland; www.burckhardtcompression.com) signed an agreement to commercialize hybrid compression systems for enhanced-oil-recovery (EOR) applications. EOR — injecting high-pressure CO_2 into oil wells to both reduce the crude oil's viscosity and increase the underground pressure — has been used for decades to boost the yield of a reservoir to up to 60%, compared to 20–40% achieved with primary and secondary recovery. Nevertheless, EOR requires considerable energy to compress the CO_2 to the pressures required (up to over 400 bars).

The two companies have developed a hybrid approach that makes use of an intermediate step — gas liquefaction — to increase the overall energy efficiency of the compression. First, the semi isothermal compression is performed in multiple stages by reciprocating compressors from Burckhardt Compression. Then, at about 70 bars, the CO_2 is cooled and liquefied to a temperature of about 20°C. Finally, the pressure of

(*Continues on p. 12*)

Flexible aerogels

Researchers at the NASA Glenn Research Center (Cleveland, Ohio; www.nasa.gov) have developed new flexible aerogels that could eventually be used in highly insulating materials and linings. Aerogels, sometimes called "solid smoke," are superlightweight structures of silica reinforced with polymer to add flexibility. Traditional silica aerogels were brittle, and crumbled easily. Another type of aerogel was made from polyimide, a strong and heat-resistant polymer, braced with cross-links for added strength. The work was presented at the recent meeting of the American Chemical Society (ACS; Washington, D.C.; www.acs.org) in Philadelphia.

HART enhancements

Last month, the HART Communication Foundation (Austin, Tex.; www.hartcomm.org) unveiled five new enhancements to the HART Communi-

(*Continues on p. 12*)

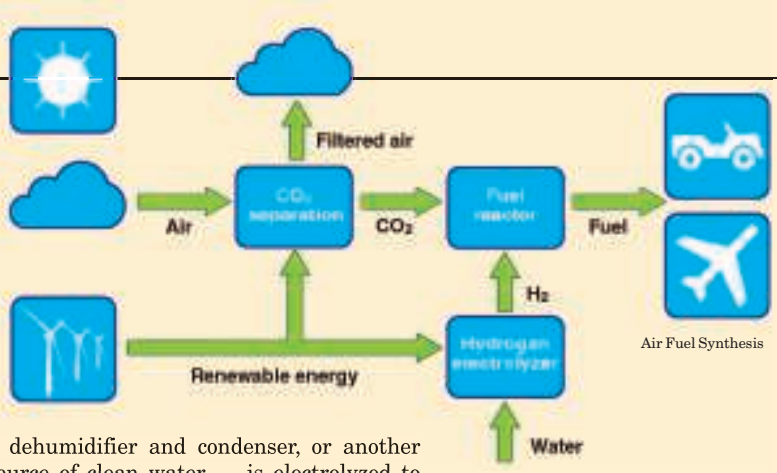
Note: For more information, circle the 3-digit number on p. 60, or use the website designation.

A concept for making gasoline from 'air'

Air Fuel Synthesis Ltd. (AFS; Darlington, U.K.; www.airfuelsynthesis.com) has recently demonstrated a process for producing carbon-neutral liquid hydrocarbon (HC) fuel from air-captured carbon dioxide and hydrogen. The proof-of-concept demonstration facility, located at Teesside, England produces 5–10 L/d of liquid HCs. The next step of the company's three-year (2012–2015), £1.1-million development program is scaling up to 1–10 metric tons (m.t) per day commercial unit, with the long-term goal of 10-million m.t./yr by the year 2025.

AFS's process combines well-understood chemical techniques. First, CO_2 is absorbed from air (fluegas or a fermentation process, such as in a distillery) in a sodium-hydroxide scrubber to form aqueous sodium carbonate. The Na_2CO_3 solution is then pumped to an electrolytic cell where the carbonate is reduced to CO_2, and the Na_2CO_3 solution is returned to the scrubber. Meanwhile, water — either recovered from air by means of a dehumidifier and condenser, or another source of clean water — is electrolyzed to produce hydrogen and caustic. The H_2 and CO_2 are then reacted to form synthesis gas (syngas; CO and H_2) via a reverse water-gas-shift reaction. The syngas can then be used to make liquid HCs via Fischer-Tropsch synthesis, or methanol, which can be converted into HCs via a mobile methanol-to-gasoline (MTG) reactor.

The process can be used for making diesel, gasoline and aviation fuels, as well as methanol and raw materials for plastics and construction materials. Economics for the process are being evaluated in the demonstration facility.



Air Fuel Synthesis

A polymersome that releases its cargo where needed

Several drug delivery systems have been studied involving the encapsulation of molecules in a suitable structure and their transport through the human body. In particular, polymersomes — tiny hollow spheres that enclose a solution — formed using synthetic block copolymers to form a vesicle membrane (with radii from 50 nm to 5 µm, mostly containing an aqueous solution in their core) have been used for encapsulating molecules such as drugs, enzymes, other proteins and peptides, and DNA. Those encapsulated molecules can be transported and released anywhere within the human body.

A group led by professor Kyoung Taek Kim from the School of Nano-Bioscience and Chemical Engineering, Ulsan National Institute of Science and Technology (South Korea; www.unist.ac.kr) has reported what it claims to be the first synthesis of the boroxole-containing styrene monomer and its controlled radical polymerization via the reversible addition-fragmentation and chain transfer (RAFT) method.

The group synthesized a series of sugar-responsive block copolymers that self-assembled to form polymersomes in water. It demonstrated that the polymersomes of these block copolymers could encapsulate water-soluble cargo, such as insulin, which could then be released only in response to the presence of monosaccharides in aqueous solution under physiological pH conditions. "To the best of our knowledge, there have been no reports describing boronic acid-containing block copolymers that form polymer vesicles and exhibit sugar-responsive release of cargo in water at physiologically relevant pH," says the group.

(*Continued from p. 11*)

cation Protocol standard. When implemented in new HART-enabled products, these enhancements will give users a simple device-status notfication and easy access to *Wireless*HART network performance.

The new enhancements are: Condensed Device Status Indicators, which provide new commands and standardized support for Namur NE107 diagnostics for operator notification of failure, out of specification, function check or maintenance required; Key Performance Indicators, which are additional standardized commands to support Namur NE 124 for host access to *Wireless*HART networks performance indicators; HART Over Internet Protocol, which specifies and standardizes the protocol between remote I/O systems, multiplexers and gateways and host systems; Discrete Functionality, which are new commands that enable enhanced support of several types of discrete and on/off type devices with diagnostics; and Infrared Device Access, a new wireless interface option for device configuration and maintenance that eliminates the need to open the device housing in hazardous areas.

HYBRID COMPRESSOR SYSTEM

(*Continued from p. 11*)

the liquefied CO_2 is boosted to the required pressure using a Lewa triplex diaphragm pump.

The overall power savings due to liquefaction can be up to 15%, says Lewa, because the power consumption for the compression of liquids is lower than for gases. Other advantages of this approach are: Corrosion problems associated with low-pressure CO_2 are avoided in the liquid phase; variable flowrates and gas compositions can be handled by the use of speed control; and changes in reservoir pressure are not disruptive because the system boosts the liquid to the final pressure in a single step.

The limit for piston compressors and diaphragm pumps is approximately 150 ton/h of acid gases, so one set of machines could handle the CO_2 emissions from a 200–300-MW, fossil-fueled power plant.

Progress for making smart membranes

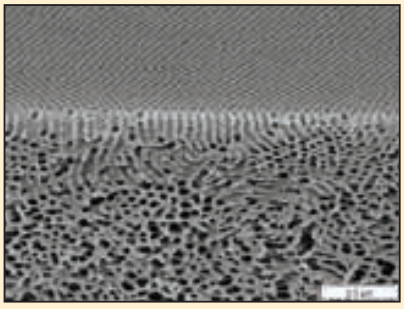
Last month marked the completion of the three-year, €3.6-million E.U.-funded research project, Self-assembled Polymer Membranes (SelfMem). Coordinated by the Institute of Polymer Research at the Helmholtz-Zentrum Geesthacht (HZG; Germany; www.hzg.de), with twelve partners from industry and academia in Israel, Canada and Europe, the SelfMem network studied self-organizing isoporous block copolymer membranes. For example, fine block copolymer membranes were produced with an upper surface layer that is highly ordered and uniformly permeated with pores, which serves as a filter, and a lower layer with a sponge-like structure that provides stability (photo).

In the beginning of the project, "we knew practically nothing about structure property relationships and the parameters that are necessary for the formation of membranes," says Volker Abetz, the project coordinator and HZG institute director. In 2011, membranes could be made from chemically different block copolymers for the first time. It was established that solubility is a key parameter. "Very slight variations in the composition of the solvent can suffice to prevent a membrane from forming," he says.

One achievement of SelfMem was the production of membranes with 20-nm-dia. pores that could be used, for example, for separation of hormones and pharmaceutical substances from wastewater. Other applications for such fine-pores membranes are for intensifying catalytic reactions and for gas separation.

HZG has applied for four patents, including one for "switchable" membranes in which the pore size can be adjusted by variations of temperature and pH.

Helmholz-Zentrum Geestacht



"Their functionality can also be extended by means of a subsequent coating of the membrane," explains Abetz. Polydopamine, for example, increases the hydrophobicity of the membrane, thereby inhibiting fouling.

The membranes are made by first pouring a solution of the block copolymers onto a fleece. As the solvent evaporates, cylinders are formed that grow vertically downward from the surface. A solvent exchange is then performed in a subsequent precipitation bath, which fixes the formed structures.

A nano cage made from a stack of benzenes

Professor Kenichoro Itami and his group at Nagoya University (synth.chem.nagoya-u.ac.jp) have synthesized a cage-type carbon nano-molecule, Carbon Nano-cage, composed of 20 condensed benzene rings — $C_{120}H_{78}$. The group used a procedure it developed four years ago to synthesize carbon nano-rings with the ability to eliminate the distortion of ring-type benzene structures during synthesis steps. First, they synthesized an unstrained cyclic precursor by assembling six L-shaped units [*cis*-di(p-bromophenyl)cyclohexane derivative] and two three-way units (1,3,5-triborylbenzene) by cross- and homo-coupling reactions. Then, they obtained carbon nano-cages through acid-mediated aromatization of the cyclohexane moieties in the precursor.

The nano-cage is a white solid that is soluble in almost all organic solvents and stable at temperatures up to 300°C. It has a 1.8-nm inner diameter, which could confine a guest molecule. In collaboration with the National Institute of Advanced Industrial Science and Technology, the researchers found that the carbon nano-cage has a large two-photon absorption cross section and a high fluorescence quantum yield of 87%. These properties are advantageous for such applications as organic electroluminescent and organic transistor materials, optical recording materials, for high-density light storage, fluorescence imaging of bio-molecules and light sensing (using a guest molecule). The structural characteristics of the $C_{120}H_{78}$ cage make it suitable as a backbone structure that could be applied for the bottom-up synthesis of branched carbon nanotubes, or as a junction unit of branched carbon nanotubes, which could be applied as miniscule transistors and logic gates.

A wall-less vessel

Scientists at the U.S. Dept. of Energy's Argonne National Laboratory (Ill.; www.anl.gov) have discovered a way to levitate individual droplets of solutions containing different pharmaceuticals — a technique that may help in the development of more efficient drugs. By eliminating the vessel walls — where compounds typically crystallize — the researchers are hoping to better understand the formation of amorphous solids as the solvent evaporates. Most drugs are crystalline, so they don't get fully absorbed by the body, says Argonne's Chris Benmore. who leads the study.

The scientists use an acoustic levitator to study how droplets evaporate without touching anything. The droplet is placed at the node of standing waves, which are generated by two opposed speakers. The acoustic pressure from the sound waves is sufficient to cancel the effect of gravity. The system allows *in-situ* analysis with the high-energy X-Ray beam at Argonne's Advanced Photon Source. ❑

A little gold can reduce the Pt loading in fuel cells

A challenge in the development of polymer-electrolyte membrane fuel cells is the durability and electrocatalytic activity of platinum-based electrocatalysts. The sluggishness of the oxygen reduction reaction (ORR) causes the fuel cell performance to be limited by the cathodic reaction, and a high Pt loading is required for the cathode catalyst to achieve good activity for the ORR.

Now researchers from the Institute of Bioengineering and Nanotechnology (IBN; Singapore: www.ibn.a-star.edu.sg), led by IBN executive director, professor Jackie Y. Ying, have discovered that by replacing the central part of the catalyst with a gold-copper alloy and leaving just the outer layer in platinum, a superior electrocatalytic activity and excellent stability toward the ORR are achieved.

The researchers reported the synthesis of core-shell AuCu@Pt nanoparticles by depositing Pt on preformed AuCu-alloy nanoparticles in oleylamine. The AuCu alloy core has a slightly smaller lattice parameter than Pt, creating a beneficial compressive strain effect on the Pt shell. In contrast, a tensile strain effect would be induced by depositing Pt on a core of Au — which has a larger lattice parameter than Pt — leading to a lower catalytic activity. In addition to the stabilization effect exerted by the alloy core on the Pt shell during the ORR, using the alloy also reduces the Pt loading in the resulting electrocatalyst.

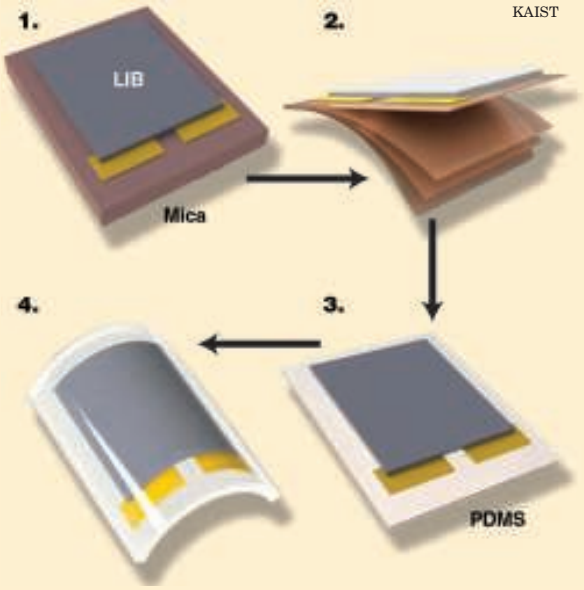
The catalytic activity of AuCu@Pt was 0.571 A/mg of Pt, which was more than five times higher than that of commercial Pt catalysts.

New catalysts for Baeyer-Villiger oxidation reactions

Kazuaki Ishihara and his group at Nagoya University (Japan; www.ishihara-lab.net) have established a new, environment friendly reaction technology — using Baeyer-Villiger oxidations — for selectively oxidizing ketones into esters. The reaction uses hydrogen peroxide as the oxidant, and has potential as a safer alternative to the conventional industrial route for making the nylon precursor ϵ-caprolactam from ϵ-caprolactone. Instead of H_2O_2, industrial routes have used acetyl hydroperoxide for making ϵ-caprolactam, which required very careful handling of explosive acetyl hydroperoxide, and also purification steps for removing byproducts and the corrosive, smelly residual catalyst after the reaction.

The new route uses hydrogen peroxide with an effective oxidation-acceleration catalyst system, both of which are easier to handle, and no byproducts are produced. The researchers found that two environmentally friendly catalysts — lithium tetrakis(pentafluorophenyl) borate or $Li[B(C_6F_5)_4]$, and calcium tetrakis(pentafluorophenyl) or $Ca[B(C_6F_5)_4]$ — can be used in both hydrophilic systems with H_2O_2 as the oxidant, and in lipophilic systems, including those with ketone substrates and ester products. These catalysts selectively oxidize the C=O group, even if the substrate contains groups that are sensitive to oxidation.

In laboratory trials, the researchers produced ϵ-caprolactone from cyclohexanone with 80% yield after 2 h agitation at 70°C, using H_2O_2 with $Ca[B(C_6F_5)_4]$ as the oxidation catalyst system. They also obtained the lactone from 2-adamantanone with 98% yield after 10 h agitation at 70°C using the oxidation catalyst system of hydrogen peroxide, $Ca[B(C_6F_5)_4]$, and oxalic acid as a promoter.



KAIST

This fully functional Li-ion battery is flexible

A team from the Korea Advanced Institute of Science and Technology (Kaist; Daejeon, South Korea: www.kaist.ac.kr) and Seoul National University (www.useoul.edu) led by Kaist's professor Keon Jae Lee, claims to have developed the first prototype of a fully functional all-flexible lithium ion battery (LIB). The LIB is based on all-solid-state materials with an energy density of 2.2×10^3 μWh/cm^3 at a current flux rate of 46.5 μA/cm^2 under polymer sheet wrapping. The Korean scientists claim this is the highest energy density ever achieved for flexible batteries.

The construction of the bendable thin-film battery starts with a standard fabrication process upon a brittle mica substrate (diagram). A cathode current collector, a lithium cobalt oxide cathode ($LiCoO_2$), high-temperature annealing of $LiCoO_2$ at 700ºC), a lithium metal anode, and protective encapsulation multilayers are sequentially deposited on the substrate (Step 1).

The next step is physical delamination of the mica substrate using sticky tapes (Step 2). The flexible LIB from substrate delamination is then transferred onto a PDMS (polydimethylsiloxane) polymer sheet (Step 3), where the surface bondability of the PDMS helps the stable settlement of the flexible LIB. The final step is capping of the fabricated flexible LIB with another thin PDMS sheet to enhance its mechanical stability (Step 4).

The thin film LIB is capable of a maximum 4.2-V charging voltage and 106-μAh/cm^2 capacity. The bendable LIB enables the fabrication of an all-in-one flexible LED display integrated with a bendable energy source. This novel approach can be expanded to various high-performance flexible applications, such as thin-film nanogenerators, thin-film transistors and thermoelectric devices. ■

CFATS administration needs improvement, testimony argues

The Society of Chemical Manufacturers and Affiliates (SOCMA; Washington, D.C.; www.socma.com) testified before Congress last month that poor implementation of the nation's chemical security regulations has created burdens on small chemical companies.

Testifying on behalf of SOCMA before the House Subcommittee on Environment and the Economy, Matthew Leary, corporate EHS&S manager for Pilot Chemical Co. (Cincinnati, Ohio; www.pilotchemical.com), discussed several issues impeding compliance with the Chemical Facility Anti-Terrorism Standards (CFATS), which are administered by the U.S. Dept. of Homeland Security (DHS; Washington, D.C.; www.dhs.gov). Specifically, Leary highlighted communications issues with DHS and difficulties budgeting for compliance. Leary said small companies cannot afford to spend on security in advance unless they are certain it will lead to compliance.

Also testifying was Timothy Scott, chief security officer at the Dow Chemical Co., who also represented the American Chemistry Council (ACC; Washington, D.C.; www.americanchemistry.com). Scott said that while the CFATS concept is sound, problems with implementing the standards — particularly in the area of personnel surety — are real.

Shell to construct world's first oil sands CCS project

Royal Dutch Shell (The Hague, the Netherlands; www.shell.com) says it will go ahead with the first carbon capture and storage (CCS) project for an oil-sands operation in Canada. The project will be built on behalf of the Athabasca Oil Sands Project joint-venture owners (Shell, Chevron and Marathon Oil) with support from the governments of Canada and Alberta.

The Athabasca Oil Sands project produces bitumen, which is piped to Shell's Scotford Upgrader near Edmonton. Starting in late 2015, the project will capture and store, deep underground, more than 1 million metric tons a year of CO_2 produced in bitumen processing. The project is the world's first commercial-scale CCS project to tackle carbon emissions in the oil sands, and the first CCS project in which Shell will hold majority ownership and act as designer, builder and operator. It will also form the core of Shell's CCS research program and help develop Shell's CO_2 capture technology.

The CO_2 will be transported through an 80-km underground pipeline to a storage site north of the Scotford site. Here, the CO_2 will be injected more than 2,000 m underground into a porous rock formation called the Basal Cambrian Sands. Sophisticated monitoring technologies will ensure the CO_2 is permanently stored.

STB SEEKS RATE PROTECTION FOR CAPTIVE RAIL SHIPPERS

The U.S. Surface Transportation Board (STB; Washington, D.C.; www.stb.dot.gov) announced two initiatives to explore ways to further protect captive shippers — those having no practical alternative carrier for transporting their goods — from unreasonable rail rates. First, the STB proposed to reform its rules on how it resolves rate disputes to ensure that all captive rail shippers have a meaningful way to challenge rates. Second, the STB is considering a proposal submitted by the National Industrial Transportation League (NITL) to increase rail-to-rail competition.

The centerpiece of the STB's rate-rules proposal removes the limitation on relief for cases brought under the Simplified Stand-Alone Cost (SAC) alternative. "Our goal is to encourage shippers to use a simplified alternative to a Full-[Stand Alone Cost] analysis that is economically sound, yet provides a less complicated and less expensive way to challenge freight rates by discarding the requirement that shippers design a hypothetical railroad to judge a railroad's real-world rates," the STB wrote in a recently issued decision.

Captive shippers have long stated that they do not bring rate disputes to the STB because of the high litigation costs associated with the Board's complex Stand Alone Cost test, traditionally used to resolve major rate cases. To provide rail customers with a lower-cost, expedited alternative to this test, STB simplified evidentiary procedures, but because the methods used are less precise than those used in full SAC cases, the Board capped the amount of relief available under them. ❑

ECHA seeks public consultation on substances of high concern

The European Chemicals Agency (ECHA; Helsinki; Finland; echa.europa.eu) has opended a public comment period on 54 potentially harmful substances, including 44 that are proposed to be identified as substances of very high concern (SVHCs) because of their classifications as carcinogenic, mutagenic or toxic for reproduction (CMR). The list of substances proposed for identification as SVHCs include four fluorodecanoic acid compounds, several phthalates and lead compounds, as well as diazene-1,2 dicarboxamide, methoxy acetic acid and N,N-dimethyl formamide.

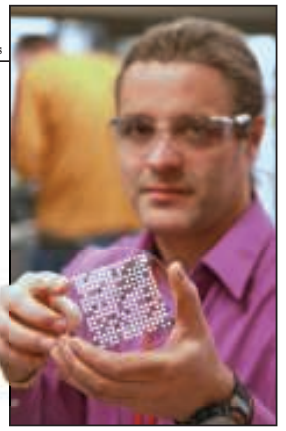
The European Commission has requested ECHA to prepare Annex XV dossiers for 37 of the substances on its behalf, and other countries have put forward proposals for the other 17 proposed substances.

Comments can be posted on the ECHA website until October 18, 2012. The Member State Committee will take comments into account when seeking agreement on the identification of all proposed substances as SVHCs.

ECHA is also seeking information on the uses of the substances. This would include data on tonnages per use and exposures or releases resulting from these uses. Information on safer alternatives and techniques, and supply-chain structure, are also welcome. ■

SHALE GAS USHERS IN ETHYLENE FEED SHIFTS

Growth in North American ethane cracking has wider effects for the CPI, while some companies look to harness methane for ethylene



Siluria Technologies

FIGURE 1. Siluria Technologies has developed a catalyst for the oxidative coupling of methane (OCM), potentially opening the door to commercial-scale ethylene directly from methane in one step

Increasing production of natural gas from hydraulic fracturing of shale deposits has fundamentally altered the landscape of chemical production in North America. The higher margins on steam-cracking ethane to produce ethylene have resulted in significant feedstock shifts in the U.S. over the past two years. Numerous chemical producers are expanding ethylene capacity to take advantage of the increased availability of ethane from natural gas. The shift toward using more ethane as a feedstock for ethylene is generating a number of ripple effects for other chemicals, including tightness in the propylene and butadiene markets. Meanwhile, as manufacturers in energy-intensive industries take advantage of lower energy costs offered by shale gas, other companies continue the pursuit of an alternative route to ethylene using methane as the feedstock.

Higher shale-gas production has resulted in a significant and longterm change in the relationship between petroleum and natural gas prices, explains Russell Heinen, director of IHS Chemicals (Englewood, Colo.; www.ihs.com). "This change in price relationships has kept prices for ethane low relative to naptha, which is still the dominant feedstock for ethylene producers worldwide," he says (Figure 2). IHS is preparing a report, "The Game Has Changed: The Influence of Shale Development on the Global Chemical Industry," to be released this month.

Shale deposits vary greatly in the composition of natural gas. Some areas contain virtually no natural gas liquids (NGLs; ethane, propane and butane), and some are very "wet," containing from a few percent up to even 20% NGLs. Parts of the Marcellus shale in the eastern U.S. are very wet, for example, notes Heinen, while the Eagle Ford and Barnett shales in Texas produce dryer gas (more methane with less NGLs).

TABLE 1. NORTH AMERICAN ETHYLENE CAPACITY GROWTH

Company	Location	Capacity, thousands of ton/yr	Start up year
Dow Chemical	Freeport, Texas	1,906	2014–2017
Ineos	Lake Charles, La.	1,361	2018
CP Chem	Baytown, Tex.	1,134	2016–2017
Braskem/Idesa	Coatzacoalcos, Mexico	998	2015
Shell Chemical	Pennsylvania	907	2016+
Formosa Plastics	Point Comfort, Tex.	799	2015
LyondellBasell	Texas and Illinois	658	2012–2014
Dow Chemical	Hahnville, La.	363	2012 (4th Q)
Williams	Lake Charles, La.	272	2013 (3rd Q)
Westlake Chemical	Lake Charles, La.	104	2012
Ineos	Chocolate Bayou, Tex.	104	2013
Total		8,607	

Source: IHS Chemical

Ethane to ethylene

Driven by a wide range of derivatives, ethylene is the most-produced organic chemical in the world, with volumes expected to top 160 million tons in 2012, accounting for $150 billion in sales. Major polymers and chemicals made from ethylene include: low-density polyethylene (LDPE); linear low-density polyethylene (LLPDE); and high-density polyethylene (HDPE); as well as polyvinyl chloride; ethylene oxide; ethanol; ethylene propylene diene monomer (EPDM) and others. End-use markets include wire and cable insulation; consumer, industrial and agricultural packaging; woven fabrics; coverings, pipes, conduits and assorted construction materials; drums, bottles and other containers; and antifreeze, solvents and coatings.

According to an analysis by the American Chemistry Council (ACC; Washington, D.C.; www.americanchemistry.com), the additional output of chemical derivatives generated by a 25% increase in ethane production would translate to $18.3 billion worth of bulk petrochemicals and organic intermediates, as well as $13.1 billion worth of plastics resins and $1.0 billion of rubber.

New capacity

A major driver of the increasing ethane-cracking capacity is the ratio between crude oil and natural gas prices, which has been generally increasing since January 2009, and is at historically high levels (40 to 1 or higher).

"To date, most new capacity for ethylene has come through retrofits and expansions, but the bulk of new capac-

WIDER SHALE GAS EFFECTS

A host of additional effects of the shale gas boom is generating profound changes across the chemical industry and beyond, including rising methanol and ammonia production capacity in the U.S., says ACC economist Martha Gilchrist-Moore, who will be speaking about the implications of the shale gas boom at ChemInnovations 2012, along with IHS' Heinen (see p. 24D-1).

The availability of shale gas will also have a profound effect on manufacturing more generally. Energy-intensive sectors like iron and steel, plastics, glass, rubber, aluminum, fabricated metals and papers all stand to gain from inexpensive shale gas, she says. Also, combined heat and power cogeneration plants are even more economically attractive, she notes.

ity from newly built plants will start to come online in 2016 and 2017," says IHS's Heinen.

Since 2010, 450,000 ton/yr of ethylene production capacity have been added through retrofits, upgrades and expansions in the U.S., and producers have announced over 5 million ton/yr of new ethylene capacity that are scheduled to come online by 2018 (Table 1). And since the additional ethane potential would allow about 11 million ton/yr of ethylene, there is still plenty of room for new capacity to be added to balance potential supply with demands. That means more ethylene capacity is likely, Heinen says.

Industry faces several challenges associated with the added capacity, Heinen notes, including the ability to bring capacity online without delay, and the ability of ethylene producers to work out the supply logistics of handling nearly 300–400 thousand bbl/d of new ethane by 2020. Also, Heinen points out that other potential challenges are that U.S. ethylene supply increases could impact pricing; and environmental concerns related to shale-gas drilling could spur legislation restricting hydraulic fracturing.

Another challenge for ethylene producers is that some of the newer crackers are ethane-specific, giving up feedstock flexibility to focus on ethane cracking, so future growth might be hindered if economics change and flexibility is needed.

On-purpose propylene

The shift to lighter feedstocks for ethylene (using more ethane) is beginning to have ripple effects that are likely to grow going forward. When steam-cracking naptha or gas oil, propylene and other chemicals are formed as co-products alongside ethylene; but when ethane is the feedstock, ethylene is the primary product, with minimal co-products formed. So the shift to ethane as a feedstock has resulted in tighter supplies of three- and four-carbon chemicals, particularly propylene and butadiene, which is likely to raise prices for those chemicals, says IHS' Heinan (Figure 3).

Tightness in propylene supply has improved the cost-competitiveness of on-purpose propylene production, notes Felipe Tavares, director of Intratec (Houston; www.intratec.com). Several on-purpose propylene production alternatives exist, including those using NGLs — propane dehydrogenation (PDH) and propylene production via metathesis chemistry.

PDH is a catalytic process in which the hydrogen byproduct can be used as fuel for the reaction. Metathesis is a catalyzed reaction between butenes and ethylene where double bonds are reformed. Propylene technology is among the first topics covered in Intratec's "Knowledge Base," an online encyclopedia of chemical technology and economics. The tool can be found at: base.intratech.us.

Capacity growth for propylene from PDH is poised to grow significantly. The Dow Chemical Co. (Midland, Mich.; www.dow.com) has announced plans for a 750,000 ton/yr PDH plant in Freeport, Tex., using technology from UOP LLC (Des Plaines, Ill.; www.uop.com). Formosa Plastics and Enterprise Products Partners LP (Houston; www.enterpriseproducts.com) has also announced plans for PDH facilities on the U.S. Gulf Coast.

Lummus Technology / CB&I Co. (The Hague, the Netherlands; www.cbi.com) is currently the only licensor to offer an olefin metathesis process, known as olefin conversion technology (OCT), to make propylene from ethylene and butenes. In cases where only ethylene is readily available, Intratec's Tavares says the metathesis process could be combined with a dimerization plant, which converts ethylene to 2-butene.

Utilizing methane

While methane from natural gas will continue to play a prominent role as a cleaner-burning alternative to coal for power generation, its abundance (~10 times more than ethane in natural gas) and price (about half the price of ethane) is spurring a wide-ranging effort to utilize methane directly as a feedstock for ethylene and other chemicals, rather than burning it as fuel (Figure 1). Since so much natural gas is available, new demands for methane are needed, says Rahul Iyer, senior director of corporate development at Siluria Technologies (San Francisco, Calif.; www.siluria.com).

Dallas Kachan (Kachan & Co.; San Francisco, Calif.; www.kachan.com), an analyst and consultant in the clean technology industry, points out that the energy and capital intensity required for steam cracking of ethane provides an incentive to make ethylene in alternative ways. Commercially viable ways to make ethylene directly from methane, if successful, could be a watershed moment for the chemical and petroleum industries, Kachan recently wrote in a blog post.

Significant activity is ongoing toward commercialization of methane-to-ethylene technologies, with many startups — some announced and some "undercover" — working on various technologies, while university and government laboratories also attack the problem, according to research by Kachan & Co. The major integrated petroleum companies, including Chevron, ExxonMobil, Shell and BP hold a surprisingly wide-ranging portfolio of patents for methane-to-ethylene technologies, and several large chemical companies also hold some intellectual property in this area (BASF, Lubrizol, SABIC, GE, Honeywell and others), Kachan research found.

Methane-to-ethylene technologies are being considered for use in remote areas where natural gas associated with crude oil drilling is flared, or remote gasfields where no pipelines exist and transporting the gas is difficult. Smaller-scale gas-to-liquids (GTL) technologies are needed for areas with "stranded gas," says Sulkhan Davitadze, an investment director at Bright Capital (Moscow, Russia; www.bright-capital.com), a venture capital firm with a portfolio of companies in the energy and chemicals areas.

A number of smaller-scale GTL processes exist, some using a Fischer-Tropsch (F-T)-based approach and some taking different paths. All are

Source: IHS Chemical

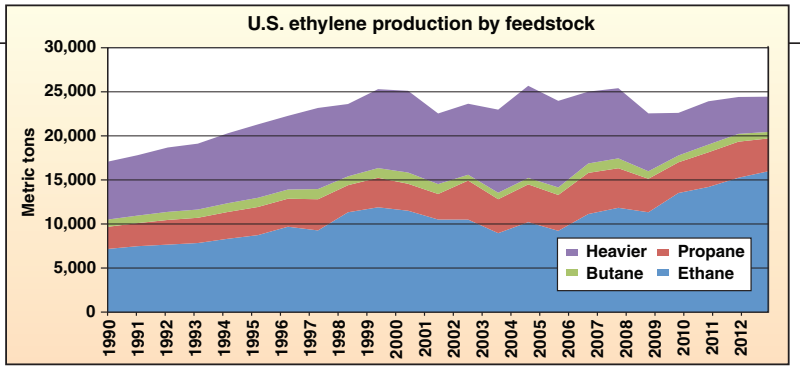


FIGURE 2. Shale gas has pushed natural gas and ethane production higher. Forecasts indicate the production will rise further

Source: IHS Chemical

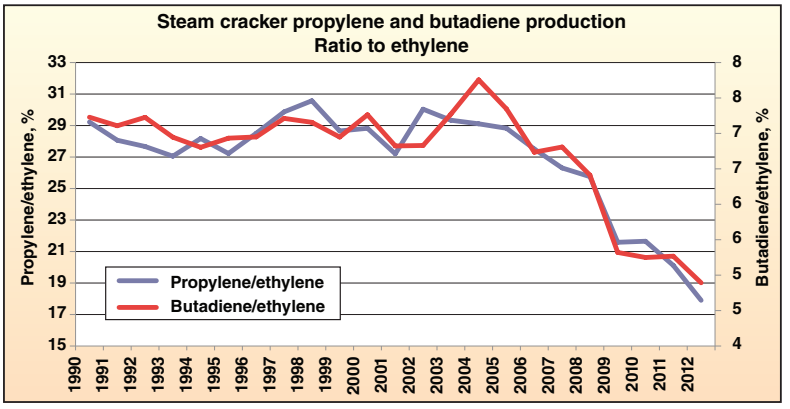


FIGURE 3. Propylene and butadiene production (shown as a ratio to ethylene) have fallen as a result of the shift to lighter feedstocks by ethylene producers

potentially useful, but it's not clear yet what the economics will turn out to be, Davitadze remarks.

SynFuels International (Dallas, Tex.; www.synfuels.com) is looking to make higher-value chemicals from methane without using Fischer-Tropsch chemisty. SynFuels has designed a non-catalytic, water-jacketed, fixed-bed reactor that pyrolyzes methane, generating a mixture of gases composed mostly of acetylene, as well as some carbon monoxide and hydrogen (Figure 4). The gas mix is fed into an absorber that separates the acetylene from the other gases.

Acetylene is then hydrogenated, while still absorbed, using some of the hydrogen produced in the initial pyrolysis step. A specially designed hydrogenation catalyst that tolerates high levels of carbon monoxide is used. The process, based on technology originally conceived at Texas A&M University (College Station, Tex.; www.tamu.edu), is capable of producing polymer-grade ethylene in 96% yields from acetylene. The ethylene can also be taken further to gasoline blendstock (a mix of C_4 to C_{12} hydrocarbons) through an oligomerization technique also developed by SynFuels. The company operates a fully integrated GTL demonstration plant in Bryan, Tex., capable of processing 50,000 ft^3/day of natural gas into gasoline blendstock.

Siluria Technologies is trying to commercialize a single-step methane-to-ethylene process. The company just announced plans to build a demonstration facility for its process, which is based on oxidative coupling of methane (OCM) chemistry. According to Siluria CEO Alex Tchachenko, construction on the demonstration plant will begin in 2013, and it will be capable of producing hundreds of thousands of gallons per year. Siluria's technology depends on a carefully made catalyst that promotes ethylene formation over non-selective oxidation reactions (*Chem. Eng.*, December 2010, p. 12).

Siluria's technology is attractive because it's a one-step process that is capital efficient as well as energy efficient, Davitadze comments.

Using a genetically engineered bacteriophage (bacteria-infecting virus) as a template for material growth, Siluria scientists developed a catalyst material that has a unique crystal structure and surface morphology. The structure

SynFuels International

FIGURE 4. SynFuels International uses methane in pyrolysis reactors to generate acetylene, which is then hydrogenated to ethylene

gives rise to catalyst active sites that can select ethylene formation over non-specific oxidation of methane. The catalyst is a doped metal oxide of transition metals that is designed for compatability with existing petrochemical-industry infrastructure.

The Siluria technology has a number of advantages over F-T approaches because it can be accomplished in one step, at lower temperatures, and works well with existing hydrocarbon processing equipment. Siluria's OCM technology is an exothermic reaction, so it requires less energy input, and the heat given off by the process can be harvested to drive the process. "We believe we are the first to develop a commercially viable, scalable process for OCM," Tchachenko says.

Siluria's catalyst-discovery engine has the potential to develop other catalysts to make different products. Siluria's Iyer says the company will be able to achieve the economics enjoyed by a world-scale plant in a much smaller facility, so significant capital-expense savings are possible.

OCM is also of interest to the Polish national research laboratory Fertilizer Research Institute (Pulawy, Poland; www.ins.pulawy.pl). Scientists there have built a pilot plant for a methane-to-ethylene facility based on OCM. Also, UOP has reportedly proved, in the laboratory, a one-step process for directly converting methane to ethylene. The company is looking for partners to build a pilot plant for the process. UOP says its technology could potentially save 40% of the cost of ethane-based ethylene. ■

Scott Jenkins

Editor's note: An expanded version of this article, with additional background information and graphics can be found at www.che.com.

A STEAMY SITUATION

Short staffed and lacking expertise, many chemical processors don't realize the full potential of their steam systems

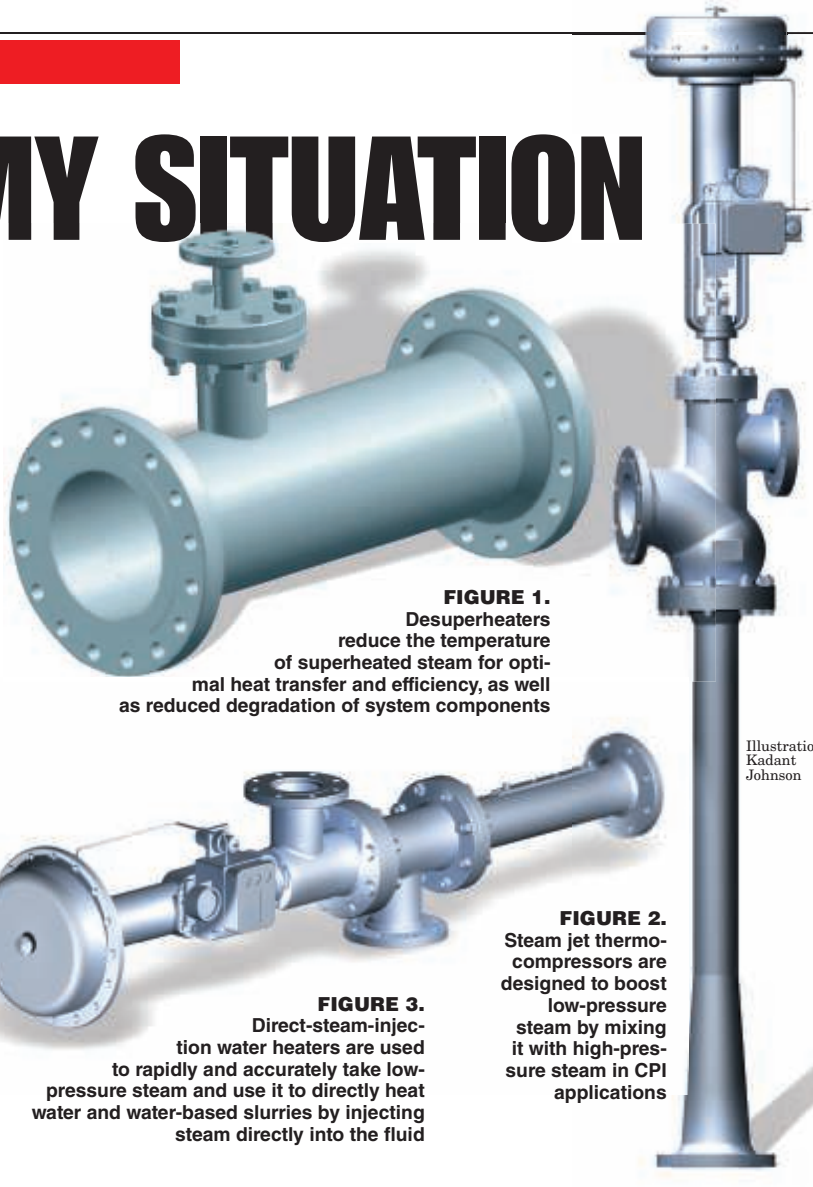


FIGURE 1. Desuperheaters reduce the temperature of superheated steam for optimal heat transfer and efficiency, as well as reduced degradation of system components

FIGURE 3. Direct-steam-injection water heaters are used to rapidly and accurately take low-pressure steam and use it to directly heat water and water-based slurries by injecting steam directly into the fluid

FIGURE 2. Steam jet thermocompressors are designed to boost low-pressure steam by mixing it with high-pressure steam in CPI applications

Illustrations: Kadant Johnson

Today's chemical processors must cope with rising labor costs, increasing global competition, escalating fuel costs and more-stringent safety and environmental regulations. To improve productivity and reduce costs in today's competitive environment, many facilities are operating leaner than even before. And, downsizing strategies have seen the reduction of maintenance staff, which often results in a decrease of in-house steam-engineering knowledge and experience.

"For today's chemical processors, maintenance and improvement of steam systems are low on the list of priorities," says Neil Davies, product marketing manager for Spirax Sarco (Blythewood, S.C.; www.spiraxsaco.com). While this is understandable, it is not necessarily wise. Not only do processors rely on their steam systems to lend process efficiency and uptime, but steam can also provide a versatile energy medium for heating and sterilization processes.

Although the modern steam system is very efficient, there is always room for improvement in existing systems and potential for waste steam to be recycled back into the process. However, lacking staff, experience and knowledge of today's technologies, many processors are ignoring these opportunities.

Tapping into available expert advice can be the ticket to getting the most efficiency out of an existing system, reducing carbon footprint and reducing costs associated with utilities, water and chemical requirements for treated water. For this reason, steam handling experts offer services such as audits, which often reveal not only where steam is being wasted, but also optimized solutions and proposals, with suggested business proposals to help demonstrate a return on investment.

"Even though [processors] may not realize it, steam utilization is one of the biggest areas processors need assistance with," says Mike Sneary, business development manager with Kadant Johnson (Three Rivers, Mich.; www.kadantsteamsolutions.com). "While some are beginning to see that they can make improvements that will increase efficiency and some understand that they can capitalize on the waste energy, they just don't know exactly how to do the things they need or want to do, especially regarding lost energy or heat recovery."

Working with an expert is indeed worthwhile. For example, Spirax Sarco audits hundreds of steam systems annually in the U.S. and has identified actions required to modernize plants, using best practices, technologies and products to save 20.7-billion lb/yr of steam energy and reduce carbon dioxide emissions by 1.4-million ton/yr.

And on an individual basis, says

STEAM CYCLE SOLUTION CAN OPTIMIZE PERFORMANCE FOR POWER PLANT BOILERS

Steam plant operators are looking for control solutions that will help them optimize boiler efficiency by reducing fuel consumption while reducing emissions, according to Bill Pezalla, global energy industry manager for GE Intelligent Platforms (Charlottesville, Va.; www.ge.com). High-performance plants under 300 MW can meet these needs with control solutions based on expertise in steam-cycle generation management, which can help reduce fuel usage while allowing boilers to run safely and efficiently.

For example, GE's Steam Cycle control solution is based on a flexible and scalable DCS, Proficy Process Systems, along with pre-packaged advanced combustion-control algorithms and strategies for fossil and biomass fuels to achieve operational benefits and mitigate risk.

These advanced combustion-control algorithms and strategies optimize the combustion process to reduce fuel usage 3 to 5%, which allows them to better meet stringent emissions regulations. In addition, the technologies keep equipment safe and protected by maintaining the water level in the boiler to prevent damage. Because the control algorithms and strategies are pre-engineered, they are capable of reducing system implementation time by 50 to 80%.

"Power plants are running longer with shorter maintenance outages," says Pezalla. "The advantage of this control system is that it is an open system that works with any type of boiler and can be implemented during a short maintenance outage leading to the realization of rapid fuel and water savings. These efficiencies need to be factored into the total cost of ownership when purchasing control systems."

"Adding boiler and turbine auxiliary-equipment control strategies provides a more comprehensive solution," notes Craig Thorsland, steam-cycle solutions leader for GE Intelligent Platforms. "Optimally, the overall control solution reduces the need for operator intervention. This stabilizes steam production and frees operators to focus on plant operations. With stable and consistent operations, the thermal stresses on the boiler and turbine are reduced, extending their lifecycles."

Safety is a concern and the Steam Cycle solution addresses that concern with drum-level control strategies that allow water levels to be properly maintained to avoid boiler failure. "It is critical to keep the water level in the boiler at the proper level," explains Thorsland. "If it falls too low, the boiler can be damaged, or worse yet, fail with problematic consequences. Control strategies can compensate for varying loads and the resulting increases or decreases in water level." ❑

Sneary, steam system improvements for many facilities can identify 50,000 to 100,000 lb of lost steam energy, which, if stopped, can result in a tremendous savings. But, he says, to reap the savings, processors need the assistance of an expert. "Often, folks in the facility just look at the wasted steam and think 'this is the way it's always been,' or they may not be aware of new equipment that could benefit their steam system. But, people from the outside can see the wasted energy, figure out ways to recover it and use it elsewhere and point out how modern equipment can assist with the process. So, it's really worth it to tap into this expertise."

The first step is to find a firm that has the experience to provide a range of services from onsite auditing to steam trap surveys to the ability to recommend and provide turnkey installations, as well as the knowledge and understanding to provide financial information that will justify any improvements.

From there, an audit, including a steam trap survey that reveals the condition of the steam traps and which ones have failed, closed or gone cold, is normally conducted. Following this audit, recommendations are made to repair or replace failed steam traps first, then cold steam traps and finally closed steam traps. And financial justification is often offered to increase the chances of investment in the system.

Once the expert fully understands the complete process that takes place in the facility, and if the processor chooses, the expert can then make recommendations on how to modify the system in an effort to move some of the wasted energy into another process. "The process is all about understanding their facility and their process and then trying to figure out a way to save them money," says Sneary. "And often we save them money by turning wasted steam into usable steam, using a wealth of equipment for this purpose that they may not yet know exists."

He says that currently products are available from many steam system providers, such as thermocompressors, desuperheaters (Figure 1) and water-jet heaters, to make heat recovery easier and more affordable than ever before.

For example, steam-jet thermocompressors (Figure 2) are designed to boost low-pressure steam by mixing it with high-pressure steam in the chemical process industries (CPI) in applications such as drying, filtration, distillation, absorption, mixing, vacuum packaging, freeze drying, flash cooling, deaerating and dehydrating.

Desuperheaters are intended to reduce the temperature of superheated steam for optimal heat transfer and efficiency, as well as reduced degradation of system components. The efficient geometry allows for direct installation into the steam pipeline with flanged connections.

And, direct-steam-injection water heaters (Figure 3) are used to rapidly and accurately take low-pressure steam and use it to directly heat water and water-based slurries by injecting steam directly into the fluid. They are used in CPI applications including boiler feedwater preheating, fibrous slurries such as pulp stock and biomass pre-treatment, sanitization and tank cleaning and anaerobic treatment of organic waste.

While just a simple steam-trap survey can yield hundreds of thousands of dollars a year in lost steam savings and energy reductions, it may provide the impetus to further investigate the possibility for heat recovery projects that could boost these savings even more. ■

STEAM-HANDLING PRODUCTS

Free float trap for facilities with corrosive environments

This free float trap (photo) is suitable for the food industry, plants with corrosive environments and petroleum refineries and petrochemical plants where steel materials are required. The J3X provides corrosion resistance through the use of high quality CF8 stainless-steel material. It delivers continuous, smooth, low-velocity condensate discharge as process loads vary. The float has an unmatched sealing performance and is designed for severe service operations. Rated for 1,740 psig hydraulic shock, the free float provides waterhammer resistance. The product also offers the possibility for inline repairs and a thermostatic air vent that automatically vents air for easier startups. — *TLV Corp., Charlotte, N.C.*
www.tlv.com

Steam generators and fluid heaters offered in many sizes

Offered in 18 sizes ranging from 25 to 1,000 boiler hp and 862 to 34,500 lb/h of steam, these controlled-flow watertube boiler units are built to appropriate national and local standards. Units are designed to burn natural gas, propane, light oil and heavy oil, as well as a combination of these fuels and are available with steam design pressures up to 3,000 psig. A Superheat version of the E-Series product, which provides superheated steam is available in a variety of sizes for a range of steam pressures and temperatures. — *Clayton Industries, City of Industry, Calif.*
www.claytonindustries.com

Thermostatic steam trap is unaffected by waterhammer

The Gestra MK 36/51 thermostatic steam trap with a corrosion-resistant membrane regulator (thermostatic capsule) is unaffected by water hammer. An integral strainer and standard capsule for discharge are available with virtually no backing up. The traps are suitable for draining saturated steam lines and steam tracers and can also be used for air venting.

They can be installed in any position, but if installed in horizontal pipes, it is important to make sure the cover is on top. — *Flowserve, Irving, Tex.*
www.gestra.com

This steam trap resists corrosion from clean and pure steam

The BTS7.1 stainless-steel clean steam trap is constructed to withstand corrosion from clean and pure steam applications. The unit is pressure rated to ASME standards and is designed to remove condensate from clean steam systems with minimal backup, making it suitable for the pharmaceutical, biotechnology and food-and-beverage industries. The self-draining trap operates close to steam saturation temperatures, reducing condensate backup and effectively venting air, ensuring full sterilization. The smooth surfaces provide a free-draining surface, reducing the risk of bacterial growth and product blockage. — *Spirax Sarco, Inc., Blythewood, S.C.*
www.spiraxsarco.com

A disc trap that performs with low-cost maintenance

Part of the PowerDyne thermodynamic steam trap family, the stainless steel P46SRN has a replaceable module that allows the unit to be serviced without removing the trap body from installation. With the same capacity, pressure and temperature specifications as the cast steel version, the P46SRN has a corrosion-resistant stainless-steel body. Designed for efficient performance, low-cost maintenance and durability, the product is a suitable for steam mains, tracers and coils. — *TLV Corp., Charlotte, N.C.*
www.tlv.com ■

Joy LePree

Honeywell Process Solutions

Gasmet Technologies

FOCUS ON Gas Detection & Handling

Trolex

Industrial Scientific

Manufacturer's warranty period extended to two years

This manufacturer of gas detection and environmental monitoring systems has extended its warranty period to two years, providing additional coverage for all of its equipment. The company also offers a service contract specifically tailored to requirements that cover the maintenance of its products (photo). When customers call for product support, they speak directly to an engineer, not a call center. — *Trolex Ltd., Stockport, Cheshire, U.K.*
www.trolex.com

PID detects VOCs and up to four more hazards

Rugged and durable, the GasAlertMicro 5 PID (photo) measures for photo-ionization detectable (PID) VOCs (volatile organic compounds), while simultaneously monitoring and displaying up to four additional atmospheric hazards, including oxygen, combustible gases and a range of toxic gases. The PID sensor adds photo-ionization detection capabilities. Compact and lightweight, with a concussion-proof, waterproof IP66/67-rated boot, it is well suited for harsh industrial environments. The portable gas detector provides multi-language support in English, French, German, Spanish and Portuguese and offers visual, audible and vibrating alarms. — *Honeywell Process Solutions, Houston*
www.honeywell.com

Detector now approved for underground mining

The lightweight and highly configurable Ventis MX4 (photo) is capable of detecting from one to four gases, including oxygen (O_2), methane (CH_4) and any two of the following toxic gases: carbon monoxide (CO), hydrogen sulfide (H_2S), nitrogen dioxide (NO_2) and sulfur dioxide (SO_2). The Ventis can be configured with CH_4, CO, and O_2 sensors for many everyday mining applications, or it can be configured with CH_4, CO, O_2 and NO_2 sensors, making it ideal for mines using diesel equipment. In fact, the Ventis MX4 has recently received MSHA approval for underground mining. The Ventis without pump has been certified by MSHA under Title 30 CFR, Part 22 when used with the standard or extended runtime lithium-ion battery packs. The company anticipates that the Ventis with pump will receive MSHA approval later this year. — *Industrial Scientific Corp., Pittsburgh, Pa.*
www.indsci.com/ventis

Measure thousands of gases in the field

The DX4040 (photo) is the second generation of what is said to be the world's first truly portable FTIR (Fourier transform infrared) spectroscopy analyzer, a technology that brought laboratory-grade gas analysis into the field. Up to 25 gases can be displayed simultaneously when operating the DX4040 with a handheld PDA. However, the most important development lies in the ability of the device to identify unknown gases in the field (at the touch of one button) when using a new version of the unit's Calcmet software on a Windows tablet. The DX4040 is unique because it is able to identify both organic and inorganic compounds, while also storing sample spectra for post-measurement analysis using a chemical library of over 5,000 compounds. There are no sensors that might need future replacement. No sample preparation is required, and calibration is simply a zero check with nitrogen or air. — *Gasmet Technologies Oy, Helsinki, Finland*
www.gasmet.fi

O_2 analyzer approved for hazardous-area use in North America

XTP601 Oxygen Analyzer (photo, p. 24) has attained CSA certification, making it approved for use in hazardous areas in the U.S. and Canada. The instrument already carries certification from ATEX and IEC Ex, and the addition of CSA broadens its global coverage. The XTP601 uses thermo-paramagnetic measurement technology to make accurate and stable measurements of oxygen in background gases such as hydrogen, nitrogen and carbon dioxide. It is capable of measuring oxygen ranges from 0–1% up to 0–25%. Typical hazardous applications include monitoring of inerting or blanketing gases in

Note: For more information, circle the 3-digit number on p. 60, or use the website designation.

Oldham Gas

General Monitors

Michell Instruments

Sierra Monitor

petroleum refining, chemical and pharmaceutical industries. The analyzer, which is housed in a tough flame-proof and explosion-proof housing, is available in three versions: a blind transmitter; a unit with status LEDs or a full-display unit. This last version has a touch-screen interface, which allows through-the-glass interaction, eliminating the need to remove the analyzer lid (except for service). All versions are supplied with two 4–20-mA outputs, two concentration alarms Modbus RTU over RS485 protocol and application software as standard. — *Michell Instruments, Ely, Cambridgeshire, U.K.*
www.michell.com

Fixed gas detector is protected against dust and water

The OLCT IR infrared fixed gas detector (photo) has been tested according to IEC/EN 60529 and gets the IP67 protection degrees. As a result, the OLCT IR is totally protected against dust and against the effect of immersion in up to 1 m of water, putting the OLCT IR ahead of competitors' products, says the manufacturer. With a stainless-steel 316L enclosure, dual source, four-beam technology, and heated optics the OLCT IR is said to be one of the most reliable infrared (IR) gas detectors on the market. A low-temperature version allows operation down to –50°C for the harshest industrial environment. The mean time between failures (MTBF) and the probability of failures on demand (PDF) both calculated by INERIS are respectively 28 years and $1{,}6 \times 10^{-3}$ with a one-year interval test. — *Oldham Gas, Pittsburgh, Pa.*
www.oldhamgas.com

CO_2 detector protects against dangerous leaks

Offering reliable gas detection performance with low maintenance, the rugged Model IR700 Carbon Dioxide Point Detector (photo) requires no routine calibration and provides complete control room status and control capability for monitoring 0–5,000 ppm. The Model IR700 features a precision IR point-sensing element that offers reliable protection against the hazards of carbon-dioxide gas leaks. While CO_2 is nonflammable, exposure to atmospheric concentrations above 5,000 ppm is considered unhealthy, making careful monitoring crucial at lower levels. The IR700 features a true fail-to-safe design for dependable gas detection performance, heated optics to eliminate condensation, and a dirty optics indicator informs the user the device must be cleaned before the lenses are entirely blocked, thereby reducing downtime. The Model IR700 IR detector features microprocessor-based technology to continuously monitor CO_2. It features multiple communication outputs, a 4–20-mA signal, proportional to 0–100% full-scale, Modbus and HART. — *General Monitors, Lake Forest, Calif.*
www.generalmonitors.com

Wireless communication improves installation and trending

Said to be an industry first, ELDS Version 1.2 Open Path Gas Detectors (OPGDs) feature Integrated 2.4 GHz Wireless Communications between the ELDS Transmitters/Receivers and the vendor's installation and commissioning tools. The ELDS is easy to install, with 2.4 GHz wireless alignment and access to event logs, self check results, and system diagnostics. This eliminates hard-wired Class I Division I or II connectors and cables, or area de-classification for technician access to detectors via open junction box terminals for commissioning. ELDS Version 1.2 Wireless is particularly beneficial for OPGD systems installed high in the air or under floor grates. Version 1.2 also features expanded gas applications for high-level H_2S detection at 0–15,000 ppm in FPSO Vent Gases, low-level ppm ammonia OPGD, low-level ppm HF OPGD for refinery alkylation processes, percent LEL HVAC OPGD cross-duct methane detection, and percent LEL cross-duct continuous methane monitoring for coal-bed combustion. — *Senscient, Inc., League City, Tex.*
www.senscient.com

Detect oxygen deficiency without high maintenance burdens

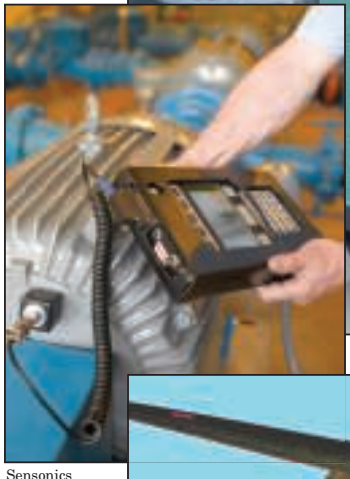
The 4501-03, Oxygen Deficiency Gas Detector (photo) is a two-wire, loop-powered gas detector that boasts lower cost and easier installation and maintenance. This eliminates the need for separate power runs and associated power distribution and circuit protection for each device. Meanwhile, maintenance costs are low due to 4–20-mA, two-wire loop-powered design, a six-month calibration interval and the ability to calibrate without declassifying the area. — *Sierra Monitor Corp., Milpitas, Calif.*
www.sierramonitor.com ■

Rebekkah Marshall

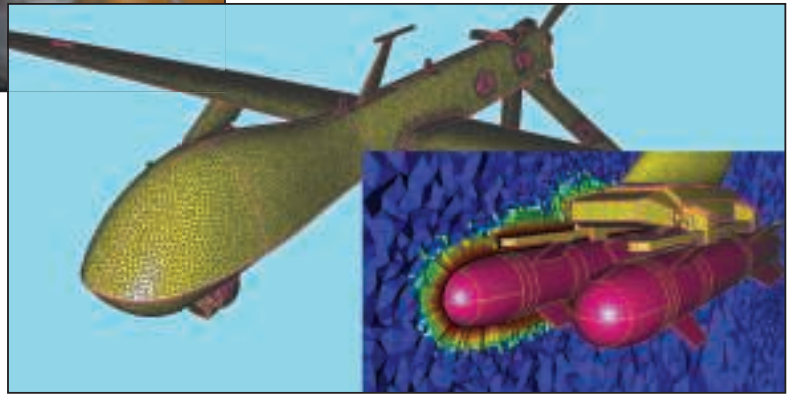
OCTOBER
New Products


Emerson Process Management

Siemens


Sensonics

Pointwise



Let this company collect and analyze machinery vibration data

This condition-monitoring specialist company has introduced a vibration data collection-and-analysis service (photo) for industrial plant operators that aims to minimize downtime and maximize productivity. Where no fixed machine condition-monitoring system is available (or is impractical) for critical plant, this company recommends that periodic vibration data collection should be implemented to monitor and trend the operational condition of machinery. Offline analysis by the company's vibration experts can then identify faults and impending failures that enable scheduling of maintenance. No upfront capital investment is required to add regular monitoring of all a company's machinery and plant equipment. — *Sensonics Ltd., Berkhamsted, U.K.*
www.sensonics.co.uk

Power supplies with large current outputs

The Sitop smart family (photo) has been extended with three single-phase, 24-V power supplies with rated output currents of 2.5, 5 and 10 A, as well as two 12-V power supplies with 7- and 14- A outputs. The output voltage of the new PSU100S power supply (photo) is adjustable over the range of 24 to 28 V for the 24-V versions, and up to 15.5 V for the 12-V models. All devices can supply 1.5 times the rated current for 5 s/min to ensure problem-free starting for all loads that draw a high current. — *Siemens Industry Sector, Erlangen, Germany*
www.siemens.com/sitop

New features offered in this latest CFD software

Last month saw the release of V15.18, the latest version of Gridgen software for computational fluid dynamics (CFD) mesh generation (photo). Gridgen V15.18 is said to increase the recovery of prism cells from the layers of extruded tetrahedra in the boundary layer. It offers the option to use a Green-Gauss formulation for computing prism volume that is "more forgiving" than the default formulation, and includes two improvements to the algorithm's ability to smoothly extrude more tetrahedral layers from concave regions. V15.18 also includes several "bug" fixes and other features. — *Pointwise, Inc., Fort Worth, Tex.*
www.pointwise.com

This wireless vibration transmitter now has ATEX approval

With ATEX Zone 0 and Class I, Div. 1 ratings, the CSI 9420 Wireless Vibration Transmitter (photo) can now be installed directly in hazardous areas, such as chemical, petrochemical and offshore facilities, as well as other ex-

Note: For more information, circle the 3-digit number on p. 60, or use the website designation.

New Products

plosion-classified environments. The safety ratings, which are in addition to existing hazardous-area ratings to the IECEx and Brazilian standards, further extends the benefit of wireless technology to new areas of the plant. The CSI 9420 connects quickly, easily and economically to any machine, and provides key insights to the condition of pumps, fans and other assets located in hazardous areas, without the expense of running cables. — *Emerson Process Management, Baar, Switzerland*
www.emersonprocess.com

A safety relay that is certified for SIL 2

The Safety Relay Module (SRM; photo) is said to be a versatile relay repeater module that has been certified by Exida for single use in safety-instrumented systems (SIS) up to SIL 2. The SRM is part of the company's FS Functional Safety Series and accepts single-contact closure inputs from logic-solver trip outputs, including the STA Safety Trip Alarm and the SPA2 Programmable Limit Alarm Trip. With three contacts per alarm input, the SRM allows for the addition of alarm contacts to safety processes without special installation or configuration. — *Moore Industries International, Inc., North Hills, Calif.*
www.miinet.com

Moore Industries International

Omega Engineering

Measure soil dampness with one hand

The new HSM50 Series of digital handheld moisture meters (photo) is easy to operate, and enclosed in a compact ABS-plastic housing for measuring moisture in soil or similar material. The microprocessor-based device displays data quickly and accurately on an LCD screen. The unit has a measurement range of 0 to 50% moisture content with an accuracy of ±5% of full scale, and operates over a temperature range of 0 to 50°C. — *Omega Engineering, Inc., Stamford, Conn.*
www.omega.com

This O_2 analyzer is certified for hazardous areas

This company has attained $_{C}CSA_{US}$ certification for its XTP601 Oxygen Analyzer (photo), making it approved for use in hazardous areas in the U.S. and Canada. The instrument already carries certification from ATEX and IEC Ex. The XTP601 uses thermoparamagnetic measurement technology to make accurate and stable measurement of O_2 in background gases, such as H_2, N_2 and CO_2, over ranges from 0–1% and 0–25%. Housed in a flame- and explosion-proof enclosure, the instrument is available in three versions: a blind transmitter, a unit with status LEDs or a full-display unit. All three versions are supplied with two 4–20-mA outputs, two concentration alarms, Modbus RTU over RS485 protocol and application software. — *Michell Instruments, Ely, U.K.*
www.michell.com

A Web-based application for generating valve assemblies

The new RapidDraw3D Valve-model Generator is a free automated valve-assembly model generator that allows users to quickly generate realistic, to-scale three dimensional (3D) valve assemblies and export them to any CAD program for use in plant piping diagrams. RapidDraw3D is said to be easy to use, and can be accessed for free upon registering at www.rapiddraw3d.com. The 3D rendering can be downloaded from the Internet for import to the chosen CAD program, such as PDF, AutoCAD, SolidWorks, DXF, STEP IGES Parasolids and more. — *Metso Automation Inc., Helsinki, Finland*
www.metso.com

Yokogawa

Mettler Toledo AutoChem

Syrris

Clamp on this process meter to measure currents

Launched last month, the CL420 Clamp-on Process Meter (photo) measures 0–120-mA d.c. process-control signals without breaking the loop. Typical applications include distributed control systems, programmable logic controllers, pressure and temperature transmitters and loop-powered isolators and indicators. The device offers a 0.2% accuracy and 0.01-mA resolution from 0 to 20 mA d.c. Other features include milliampere measurement and percent-of-span for 4–20-mA, dual LCD numeric display, LED torch light and data-hold function and a 6-mm-dia. clamp. An analog output is also available. — *Yokogawa Corp. of America, Newnan, Ga.*
www.yokogawa.com/us

A high-pressure reactor system for process development

The EM20-100-HC and EM60-100-HC 100-mL Hastelloy Pressure Reactors (photo) are designed for use with this company's EasyMax synthesis workstation. These reactors expand the use of EasyMax to both autoclave-like organic synthesis and high-pressure process characterization. The 100-mL reactor's design enables quick setup and fast results from low- to medium-pressure reactions (to 100 bars) using the built-in touch-screen and reactor combination like an autoclave; this simplifies mixing, temperature and pressure control and allows measurement of mass flow and gas uptake. Accessories, such as the iControl software, enable evaluation of gas consumption, mass transfer, kinetics and heterogeneous catalysis. The reactor covers the temperature range between 20 and 180°C. — *Mettler Toledo AutoChem, Inc., Greifensee, Switzerland*
www.mt.com/easymax

Continuous L-L extraction — no shaking required

The Asia FLLEX (flow liquid-liquid extraction) module (photo) is a flow chemistry equivalent to a separating funnel, yet involves no shaking, nor does it rely on gravity. It is suitable for the preparation of samples prior to analysis, further synthetic steps or purification. The Asia FLLEX is said to be the only commercially available, laboratory-scale flow aqueous extraction system, and can be used with a wide variety of organic solvents that are either more or less dense than water. Membrane technology enables separation of the widest possible range of phase mixtures, including tetrahydrofuran (THF) from aqueous phase, and controls bubble formation, thereby limiting the formation of emulsions that commonly occur using traditional liquid-liquid extractions. — *Syrris, Royston, U.K.*
www.syrris.com

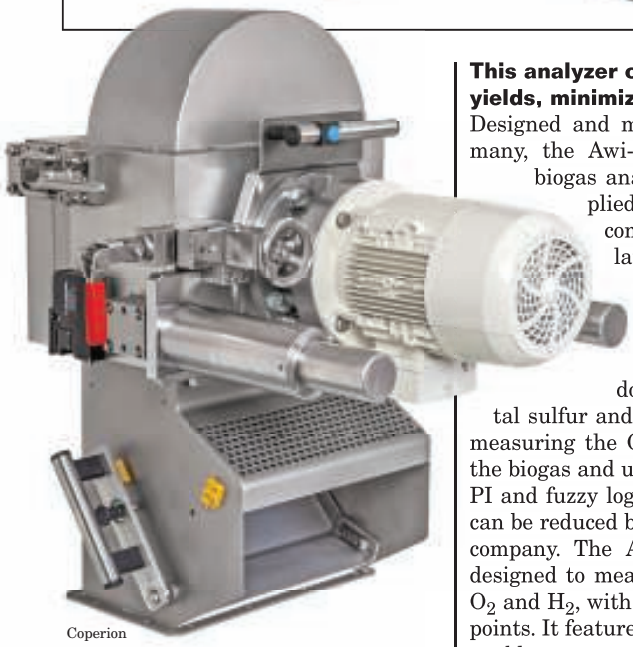
Lights that can be seen even when it's already bright out

Five new EZ-Light high-intensity indicators have been introduced to provide clear, long-range visibility in intense sunlight or brightly lighted indoor conditions. All lights use advanced LED technology to provide longer life and lower energy consumption than fluorescent or incadescent light sources. Custom capabilities include lights with five color combinations selectable from nine different colors. — *Hans Turck GmbH & Co. KG, Mülheim an der Ruhr, Germany*
www.turck.com

This extruder cooks ingredients and makes a healthy snack

The ZSK MEGAvolume Plus twin-screw extruder, featuring 54-mm-dia. screws, has been custom made by this company for a new production line for the food industry that was recently commercialized by Dinnissen B.V. (Sev-

Allison Engineering



Coperion

enum, The Netherlands). Tradenamed Magi-N.ext (photo), the new production line permits the production of a wide range of preserved and healthy convenience foods. The starch components of different recipes are gelatinized by means of an extrusion cooking process in which the shearing action of the twin screws rapidly causes the temperature of the mixture to rise to 100°C. In conjuction with the water from the content in the product, the native raw starch is gelatinized. A degassing step removes a large portion of the moisture via a vacuum system, creating directly expanded, easily manageable products obtained at the pelletizing stage by a centric pelletizer, ZGF 70. — *Coperion GmbH, Stuttgart, Germany*
www.coperion.com

This analyzer optimizes biogas yields, minimizes H_2S formation

Designed and manufactured in Germany, the Awi-Flex multifunctional biogas analyzer (photo) is supplied with a small air compressor that regulates air injection into the digester (above the sludge level) to between 0.5 and 1 vol.% for breaking down H_2S into elemental sulfur and water. By accurately measuring the O_2 and H_2S levels in the biogas and using a combination of PI and fuzzy logic control, H_2S levels can be reduced by up to 80%, says the company. The Awi-Flex analyzer is designed to measure CH_4, CO_2, H_2S, O_2 and H_2, with up to five measuring points. It features an integral PC that enables easy access to recorded data, sensor parameters and system diagnostics, with 4–20-mA, Profibus and Ethernet communications. — *Allison Engineering, Basildon, U.K.*
www.allison.co.uk

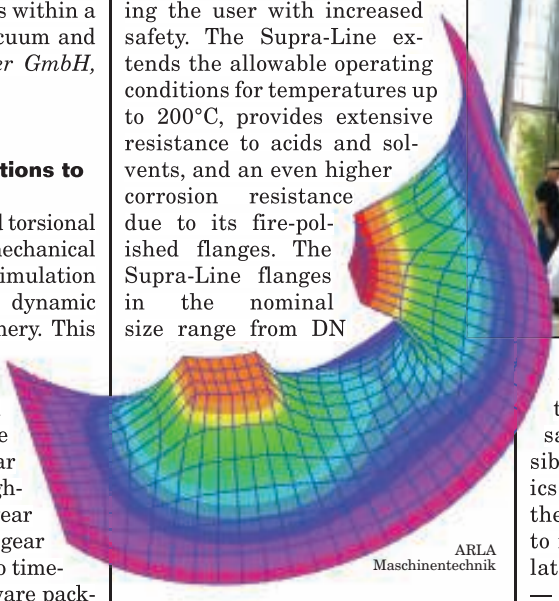
Separate components from gases with this desublimator

The Pluto mini-desublimator is used to separate substances in applications such as for tank breathing, thereby reducing emissions to the environment, for recovering raw materials or for protection of downstream vacuum generators. Gas comes in contact with cooled lamellae in the Pluto unit; here, the gas component to be separated desublimates at or below its triple point and adheres to the cold surface in crystalline form. The

Pluto unit is split into several sectors to prevent deposits from clogging the gas inlet. As soon as the desublimator is loaded, the solid product is melted off. Separation efficiencies of over 95% are achieved, says the manufacturer. The Pluto unit can be supplied in all weldable materials, and can be used across the temperature range from –20 to 350°C, as well as within a pressure range between vacuum and 10 bars. — *GEA Luftkühler GmbH, Bochum, Germany*
www.gea.com

Software simulates vibrations to keep machinery healthy

In order to detect unexpected torsional and lateral vibrations in mechanical drive systems, computer simulation is crucial to determine the dynamic behavior of rotating machinery. This company offers a new, revised software package to simulate both torsional and lateral vibrations while also considering nonlinear characteristics (such as high-elastic coupling elements, gear couplings and backlash in gear stages), steady state and also time-transient conditions. A software package for a comprehensive rotordynamic analysis, including fluid-film bearings (photo), is also available and integrated into the rotordynamic analysis simulation system ARMD 5.7 G2. — *ARLA Maschinentechnik GmbH, Wipperfuerth, Germany*
www.arla.de



ARLA Maschinentechnik

Gas cylinder storage simplified with Website Wizard

This company's updated Website now features a Gas Cage Wizard that makes choosing the right gas cylinder storage option simpler. Users enter the height, diameter and the number of cylinders that need to be stored, and the Wizard instantly calculates and suggests the most suitably sized cage from a standard range of cages. In addition to a wide range of mesh cages, enclosures and cabinets, the Website also provides essential, up-to-date U.K. Health and Safety Executive (HSE) information and FAQs. — *Gas Cage Direct, Crawley, U.K.*
www.gascagedirect.co.uk

A smart flange system for increased safety

The QVF Supra-Line is compatible with two proven flange systems — the QVF/WPR2002 and the Schott/KF systems — and thus reduces the number of technically equivalent components required while providing the user with increased safety. The Supra-Line extends the allowable operating conditions for temperatures up to 200°C, provides extensive resistance to acids and solvents, and an even higher corrosion resistance due to its fire-polished flanges. The Supra-Line flanges in the nominal size range from DN 15 to 300 require only one seal per nominal diameter, regardless of the shape of the flange. The flange connection features a patented clamping system with a ringed grounding element within the groove of the flange instead of screwed-on lugs. — *De Dietrich Process Systems GmbH, Mainz, Germany*
www.qvf.de

An economical way to distill large volumes of products

The patented Plate Molecular Still (photo) is capable of distilling large quantities of product (10 ton/h and more), under medium- or high-vacuum conditions. The system is suitable for applications involving temperature-sensitive substances, such as oils, fats, pharmaceuticals, vitamins, methyl esters and more. The product is fed through a distribution system to the outer surface of the evaporator plates within a cylindrical vessel. In this arrangement, the heated and cooled panels lead to a high evaporation and condensation surface in the smallest possible space. As a result, the economics are more favorable when using the Plate Molecular Still compared to more expensive short-path distillation processors, says the company. — *GIG Karasek GmbH, Gloggnitz, Austria*
www.gigkarasek.at

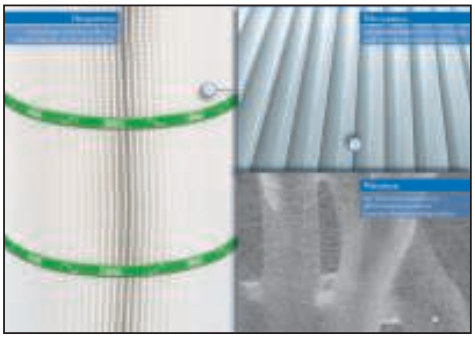
GIG Karasek

Level control is simplified with non-steady-state tuning

This company's software now includes non-steady-state (NSS) modeling features for integrating control loops. These tools are said to provide fast, simple modeling and tuning of level controls. Unlike temperature, pressure and flow, level controls have different dynamics, and these new tools make it simple to tune level controls, even while the level is moving, says the company. NSS tuning integrates seamlessly with the company's PlantTriage and PID Loop Optimizer software. These tools also provide capabilities for both tight control and surge-tank scenarios. NSS modeling is included with PlantTriage Version 11 and higher, and with PID Loop Optimizer Version 21 and higher. — *ExperTune Inc., Hartland, Wisc.*
www.expertune.com

Freudenberg Filtration Technologies

Herbold Meckesheim



Granulate and recycle plastics waste for less energy

Among the products being exhibited at the Fakuma Plastics Recycling Trade Show (Friedrichshafen, Germany; October 16–20) is the Granulator SML 45/60 SB (photo) with forced feeding (patent pending). This unit has a special granulator design that can considerably reduce its energy demand, with a 30 to 50% lower connected load compared to a traditional machine. Pre-shredded waste and hollow bodies can thus be recycled in a very economic way. — *Herbold Meckesheim GmbH, Meckesheim, Germany*
www.herbold.com

New piping software slashes time for modeling workflows

With the new release, EdgeWise MEP for Autodesk Revit, users can now, for the first time, bring extracted pipes, conduits and other cylindrical mechanical, electrical and plumbing (MEP) elements directly into Revit. This new MEP software leverages the powerful feature-extraction technology of EdgeWise to automatically extract pipe solids from laser scan-point clouds. These pipe solids can be automatically connected into pipe runs, annotated with EdgeWise MEP, and then brought directly into Revit as fully functional Revit pipe objects. All data, such as diameter, length, elbow bend radius and more, are also transferred into Revit. Beta testers report modeling-time savings of up to 85% over previous workflows, says the company. Future releases will handle square ducting, cable trays and other planar objects. — *ClearEdge3D, Herndon, Va.*
www.clearedge3d.com

Efficient filter cartridges that require no precoating

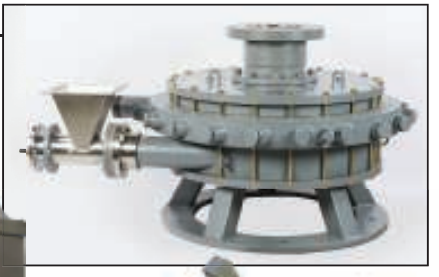
Installed into Viledon sinTexx filter cartridges, this new corrugated polyester medium with nanofiber lining (photo) is said to achieve optimum performance when dealing with fine and difficult-to-handle dust and smoke. Compared to conventional corrugated polyester materials and ePTFE (expanded polytetrafluoroethylene) membranes, the Viledon sinTexx media with nanofiber lining offers several advantages, says the manufacturer: Higher collection efficiency, even during initial operation; lower flow resistance, which reduces power and compressed-air consumption and extends their useful lifetime; and customary precoating is not required. — *Freudenberg Filtration Technologies SE & Co. KG, Weinheim, Germany*
www.freudenberg-filter.com

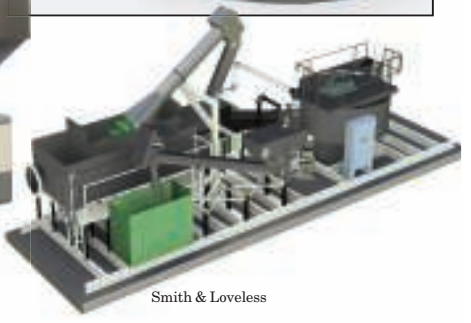
No membranes required for this dissolved oxygen sensor

The new 2610 Optical Dissolved Oxygen Sensor utilizes optical technology to measure dissolved oxygen with high repeatability and accuracy while reducing maintenance and energy costs, says the company. This device eliminates the need for replacement membranes and reference solutions associated with traditional galvanic or polarimetric measurement technologies, and therefore has reduced maintenance costs. The 2610 has

Flottweg

The Jet Pulverizer





Smith & Loveless

an optical cap sensor that is factory calibrated — no field calibration is necessary — and built-in ModBus RS485 and 4–20-mA current-loop outputs. The sensor can be combined with the new 9900 transmitter, and covers the range from 0 to 20 mg/L, 0–200% saturation with accuracies of ±0.1 mg/L (for 0–8 mg/L) or ±0.2 mg/L (for 8–22 mg/L). — *GF Piping Systems, Tustin, Calif.*
www.gfpiping.com

Process more beer with this larger clarifier

To be introduced next month at Brau Beviale 2012 (Nuremberg, Germany; November 13–15), the AC2500 beer clarifier (photo) processes up to 600 hL/h of beer, making it suitable for big breweries. The AC2500 can be used flexibly for pre-clarification before filtration, green-beer clarification and for turbidity adjustment in the case of cloudy beer, such as wheat beer. Like its smaller predecessors, the AC2500 features the SoftShot bowl discharge system, in which yeast is separated from beer and collected in the solids chamber of the bowl. The system automatically recognizes, by turbidity monitoring, when the bowl is completely full. The feed system, valves and discharge system have been optimized to minimize the impact of shear forces. — *Flottweg SE, Vilsbiburg, Germany*
www.flottweg.com

A pelletizing system for smaller-scale production

With a production flowrate of up to 600 kg/h, the Sphero 50 underwater pelletizing system can be applied in testing plants and smaller production plants with pelletizing options for virgin polymers, masterbatches, compounds, recycling, organic and wood-based polymers, and more. The entire system is mounted on a mobile frame with lockable wheels, and can be easily moved and adapted to different extruders. All components, such as startup valves, die plates, water basins and drives are suspended from a T-bar, can be shifted horizontally and are easily accessible. Special attention was given to the new design of the cutter head in order to ensure the production of evenly shaped, high-quality pellets, says the manufacturer. — *Maag, Oberglatt, Switzerland*
www.maag.com

This large-capacity mill has design features of smaller ones

The new 36-in. Orbital Bottom Side Feed Mill (photo) is designed to process 3,000–6,000-lb/h capacities, and combines design features normally found only in smaller mills, says the manufacturer. The combination of the bottom side feed and orbital-mill design elements is said to offer best-in-class performance, efficiency and durability. The orbital distribution chamber offers multiple injection points, ensuring even distribution of material into the grinding chamber, which results in tighter product distribution and higher processing rates. The bottom side-feed modifications allow for greater process clarification of product, while reducing wear on the mill. Traditional C-clamps have been replaced with bolts to add rigidity, flat seals have been replaced by O-rings for tighter sealing, and a new feed-injector nozzle design allows for easy adjustments during processing. — *The Jet Pulverizer Co., Moorestown, N.J.*
www.jetpulverizer.com

Four headworks processes preassembled into one package

The complete Pista Works headworks system (photo) combines several headworks processes — screening, grit removal and grit washing — into one skid-mounted package. Each system is preassembled and shipped directly to the job site on one truck, thereby significantly reducing field installation costs while allowing for a compact footprint. All components are constructed of stainless steel and utilize patented technologies, including grit-removal technologies with V-Force Baffel, S&L Pista Turbo Grit Pump and the Grit Washer featuring Tri-Cleanse Technology. The system comes with a PLC-based control system with touch-screen, color human machine interface (HMI) and NEMA 4X panel to operate the entire system. Pista Works operates peak system flow capacity of 0.5–7-million gal/d, depending on the model. — *Smith & Loveless, Inc., Lenexa, Kan.*
www.smithandloveless.com

This concentrate pump operates with steam, air or more

The new Pivotrol PTF4 is a high-capacity, pressure-powered condensate pump that covers a wider motive-pressure range. This new version can be operated by steam, air or other pressurized gases, and covers a complete pressure range of up to 200 psig. The PTF4 incorporates dual Pivotrol pumping mechanisms with PowerPivotol technology, and is backed by a 3-million cycles or five-year extended warranty, which is available as an option. Patented vent-assist valves release pressure in the pump during the exhaust stroke, for faster filling or overall cycling, leading to higher capacities. — *Spirax Sacro, Blythewood, S.C.*
www.spirax.com ■

Gerald Ondrey

CHEMICAL ENGINEERING
FACTS AT YOUR FINGERTIPS
Department Editor: Scott Jenkins

Fans and blowers are common in air-handling and movement systems used in the chemical process industries (CPI). Two major uses of fans and blowers are in pneumatic conveying systems and in ventilation and air-pollution-control systems. Powering fans and blowers can contribute significantly to costs, and their efficient operation can be affected by the way plant operators select and run their systems. Included here are considerations for selecting and operating fans and blowers in dilute-phase pneumatic conveying and air-pollution control applications.

Air pollution control

Air-pollution control systems must protect plant workers from the process, while also meeting all emissions standards set forth in local and national regulations. The system should also seek to minimize energy costs as well as replacement costs (such as filter bags and neutralizing chemicals).

The power required for an air handling system is computed using the following factors: Volumetric flowrate (Q) in ft^3/min; total pressure (in. H_2O); resistance due to friction in ducts, hoods and ΔP of control device; density factor of the gas being collected (df), dimensionless; and efficiency of the fan (η), dimensionless. The air power equation is the following:

$$Power\ (hp) = \frac{(Q)(TP)(df)}{(\eta)(6{,}356)} \quad (1)$$

Any increases in flow, system total pressure, or density will raise power requirements. Increases in fan efficiency will reduce power requirements. A system handling twice as much air will require twice the power to control emissions if all other factors are equal. Twice the volumetric airflow also requires larger ducts, control devices and fans.

Careful manipulation of the factors in the equation, including fan efficiency, can have cost-saving effects throughout the system's life. However, reaching the desired flowrate and pressure requires that fan-system effects be considered. Inefficiency due to these fan-system effects could result because of poorly designed hoods, short-radius elbows, branch-entry angles of more than 45 deg, abrupt contractions and interferences at fan inlets and outlets.

Fan efficiency

The denominator of the power equation includes a factor to reconcile the units with fan efficiency. Efficiency affects power inversely. However, there are limits on what fans can deliver. The design of a fan and its blade type can greatly influence efficiency. Meanwhile, the fan's peak point of efficiency measured in a laboratory may not necessarily be the most stable point of operation. If peak efficiency coincides with the peak of the pressure curve, then there may be operational problems, as volumetric flowrates vary with only tiny changes in system pressure. The designer must consider both curves when choosing the best fan and operating point to optimize reliability and power usage.

A fan's peak point of efficiency may not be the most stable point of operation. Both the fan and system curves must be considered when choosing the best fan and operating point to optimize reliability and power usage (Figure 1).

System pressure is affected by hood and duct resistance as a function of velocities in the system and the inefficiencies of flow (fan-system effects).

Pneumatic conveying

Dilute-phase pneumatic conveying systems generally use centrifugal fans and positive displacement blowers for supplying conveying air to the system. Compressors are used for dense-phase, high-pressure pneumatic conveying.

Two key factors for pneumatic conveying systems are airflow and air delivery pressure. The relationship between the two dictates the selection of an air mover for these systems.

Fans. Fans are usually used in low-pressure conveying systems where the pressures are less than 2 psig, or where both large air flows and low pressures are required. Fans used in these situations are usually of the centrifugal type, rather than axial.

The main disadvantage of fans is their relatively steep-sloping performance curve, wherein even a small change in discharge pressure results in a significant change in the air volume delivered.

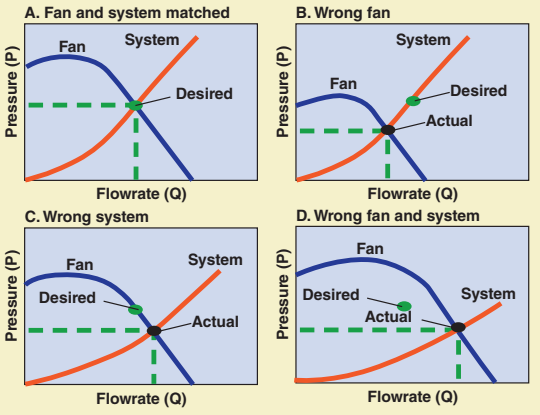


FIGURE 1. Both the fan and system curves must be considered when choosing the best fan and operating point to optimize reliability and power use

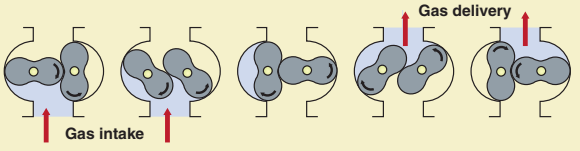


FIGURE 2. Air trapped between the rotors and blower housing is transferred from inlet to outlet without compression

A fan's volumetric output depends on its discharge pressure. The output of the fan decreases as the pressure increases, and increases as the pressure decreases (an inverse relationship). Fans' output air volume changes with a change in discharge pressure.

The best application of fans is a system in which conveying conditions (such as conveying rate, distance and material type) are relatively constant parameters, and the resulting conveying system pressure is not expected to change much.

For fans, airflow reductions correspond to air pressure increases, and airflow stops when the conveying line plugs. Horsepower decreases as air pressure increases. Fans are used mostly in applications that require high air volume at low pressures. The main advantages of fans are their low cost, low noise levels and avoidance of the need for close clearances.

Blowers. Blowers for dilute-phase pneumatic conveying systems are generally rotary, positive displacement, lobe-type blowers (Figure 2).

These blowers are considered positive displacement type because they deliver a constant volumetric airflow regardless of the discharge pressure. Compression ratio for these blowers is about 2 to 1, which means that if the inlet air is atmospheric pressure (14.7 psia), the blower can handle discharge pressures up to two times atmospheric pressure, or about 15 psig. Capacity is directly related to blower speed, and has an upper limit because of the maximum allowable tip speed of the timing gears (~4,500 ft/min).

To optimize the performance of blowers in pneumatic conveying systems, consider the following points:

- Blowers should be oil-free
- Blowers should be operated at the middle of equipment operating range
- Blower speed should be 1,800 rpm
- Volumetric efficiency should be high
- Mechanical efficiency of the drive should be as high as possible
- The pressure drop for the inlet filter should be as small as possible

Editor's note: The content for this edition of "Facts at your Fingertips" was adapted from the following sources: Lanham, G. "Energy Efficiency: Optimizing the Air-Power Equation." *Chem. Eng.*, June 1, 2007, pp. 50–53; and Agarwal, A. "Moving Air in Pneumatic Conveying Systems, *Chem. Eng.*, Sept. 2011, pp. 38–42.

Acids Handling

General guidelines on materials, storage, pumping and other concerns for the proper and safe handling of acids

Alberto Baumeister
Sebastiano Giardinella
Mayhell Coronado
Ecotek

Inorganic acids play a major role in the chemical process industries (CPI). They are used as raw materials, catalysts or finishing and pH control agents in the manufacture of a wide range of chemical products, from fertilizers to detergents, and even foods. Given their widespread use, a major issue in the CPI is the proper and safe handling of the acids, the adequate materials selection for the pieces of equipment, piping and fittings used in the process, and the correct storage and even disposal of these materials.

These are important factors that need to be taken into account from the design phase throughout the operating life of a facility, in order to ensure there will not be integrity problems that may negatively impact project economical turnover, personnel safety or the environment.

This article covers the most important inorganic acids: sulfuric acid (H_2SO_4), nitric acid (HNO_3), phosphoric acid (H_3PO_4), hydrogen chloride (HCl) and hydrochloric acid, and hydrogen fluoride (HF) and hydrofluoric acid; providing general guidelines on their physical properties, safety data, appropriate materials, storage, pumping and other common issues encountered when handling such fluids in the CPI.

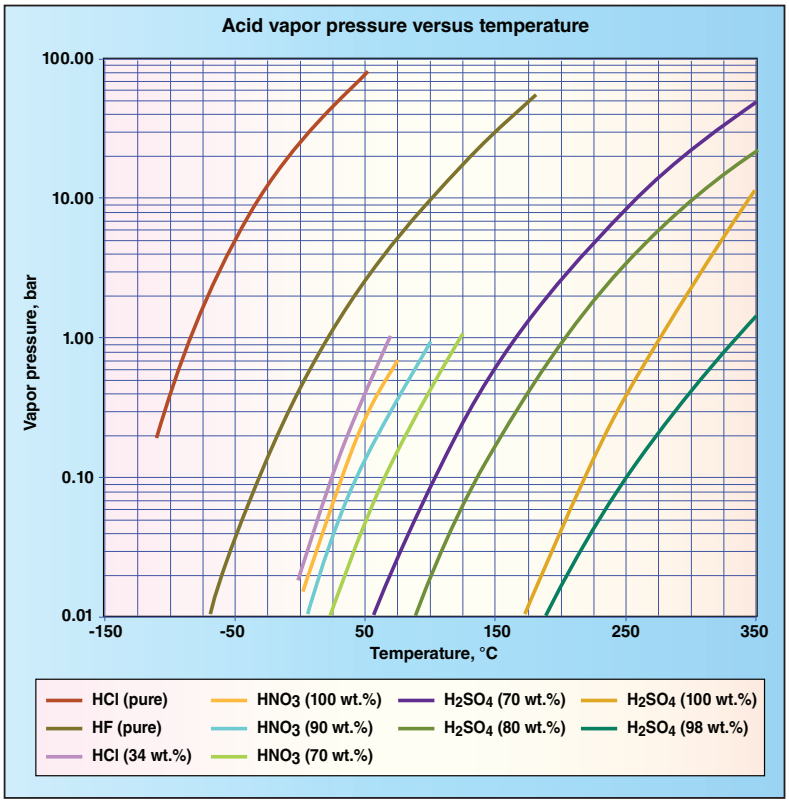


FIGURE 1. The vapor pressure as a function of temperature for the acids covered in this article

Physical properties

Some acids are naturally present as liquids (H_2SO_4), some are solids at ambient conditions (anhydrous H_3PO_4), and others are gases (HCl, HF). Acids are very soluble in water and thus also widely available as aqueous solutions at different concentrations. Some of these solutions are also enhanced by dissolving additional compounds (for example, fuming sulfuric acid is made by dissolving SO_3 in sulfuric acid).

Given that there are several available grades, the knowledge of physical properties for each one is important in order to avoid freezing, the formation of hazardous fumes, or other problems when storing and handling these materials. The physical properties of the acids covered in this article are briefly presented here. These properties for common available grades are presented in Table 1. Figure 1 plots their vapor pressures at different temperatures.

Sulfuric acid. Sulfuric acid is the single most important inorganic chemical in tonnage produced and in use. H_2SO_4, can be described as a colorless, oily, hygroscopic liquid with no odor; it is the largest inorganic chemical manufactured and one of the most widely used inorganic chemical in the manufacture of many other products. By the year 2004, North America and Asia were the biggest producers of sulfuric acid, recording almost 60% of world total production. Sulfuric acid

TABLE 1. PHYSICAL AND CHEMICAL PROPERTIES OF ACIDS [*1–5*]

	Units	H_2SO_4				H_3PO_4	HNO_3			HF			HCl		
CAS Number		7664-93-9				7664-38-7	7697-37-2			7664-39-3			7647-01-0		
Molecular weight	g/gmol	98.079				97.994	63.01			20.01			36.46		
Grade		Concentrated	Fertilizer	Tower	Fuming, 65% oleum	Pure	WFNA	Strong	Common	Gas (anhydrous)	Aqueous	Aqueous	Gas (anhydrous)	Aqueous	Aqueous. Technical Grade 22°Be
Concentration	wt.%	98	78–80	62–70	35 H_2SO_4, 65 SO_3	75–85	100	90	68	100	48–51	40	100	50	33
Physical state		Liquid	Liquid	Liquid	Liquid	Solid	Liquid	Liquid	Liquid	Gas	Liquid	Liquid	Gas	Liquid	Liquid
Color		Colorless	Colorless	Colorless	Colorless	Colorless	Colorless	Colorless	Colorless	Colorless	Colorless	Colorless	Colorless	Colorless	Colorless
Odor		Odorless	Odorless	Odorless	Odorless	Odorless	Pungent odor	Pungent odor	Disagreeable (can cause choking)	Pungent odor	Acrid Odor	Acrid odor	Pungent odor	Pungent odor	Pungent odor
Boiling point (760 mmHg)	°C	340	200	155.85	151.1	212.8	86	120.5	121	20	108	112.2[F]	–85	100	83
Vapor density (air = 1)		3.4				3.4		2.2	2.5[D]	0.7	1.97		1.3	0.62	1.267
Specific gravity (H_2O=1)		1.83	1.72	1.57	1.56	1.87	1.50	1.50		1.50	1.17	1.14		1.03	1.16
Liquid density (at 20°C)	g/mL	1.8361	1.7272[B]	1.6105[C]	1.5533	1.579[A]	1.5129	1.4826	1.4048		1.202	1.159		1.251	1.164
Melting point	°C	10.5	–4.29	–36.55	–36.98	42.2	–42		–41.6	–83	–40	–62	–114		–46.2
Dynamic viscosity (at 20°C)	cP	20.5		6.5			3.52							2.68[E]	2

Notes: [A] Liquid Density of phosphoric acid at 75% [B] Liquid density of sulfuric acid at 80% [C] Liquid density of sulfuric acid at 70% [D] Vapor density of nitric acid at 65% [E] Dynamic viscosity obtained after extrapolation [F] Boiling point at 38.2%

is manufactured by the combustion of sulfur with dry air to form sulfur dioxide (SO_2), then sulfur trioxide (SO_3) is produced through a catalytic conversion. Finally, sulfuric acid is obtained after absorption of SO_3 in water.

Sulfuric acid is a strong acid and a strong oxidizing agent; therefore it reacts violently with bases, combustible, reducing materials, water and organic compounds with the evolution of heat. It is highly corrosive to most common metals and forms a flammable/explosive gas.

Sulfuric acid is mostly used in the manufacturing of fertilizers, organic pigments, explosives and more. As a strong electrolyte it is used in electroplating baths for pickling, and for operations in the production of iron and steel. Moreover, it is extensively used as a solvent for ores and as a catalyst in the petroleum industry.

Nitric acid. HNO_3 is a solution of nitrogen dioxide (NO_2) in water; it is a colorless to light-brown fuming liquid with an acrid suffocating odor. Nitric acid is the second most important industrial acid; it is a highly oxidizing agent, used in the manufacture of chemicals, explosives, fertilizers, steel pickling and metal cleaning. However, the largest use for nitric acid is for the production of fertilizers.

Nitric acid is a strong acid that reacts violently in the presence of strong bases, reducing agents and combustible fluids, such as turpentine, charcoal and alcohol. It is corrosive to metals, forming flammable or explosive gas. Nitric acid also reacts violently with organic compounds.

Phosphoric acid. H_3PO_4 or orthophosphoric acid is a white solid with a melting point of 42°C, which is highly soluble in water, non-toxic and a relatively weak acid.

H_3PO_4 is the third most important acid in industry. It is used mostly in the production of phosphate fertilizers; but also in the manufacturing of agricultural feeds, soaps, detergents, waxes; and in the food industry as preservative, acidifier, clarifier or flavor enhancer; among other uses.

H_3PO_4 has two main methods of production: the wet process and electric furnace. It is commercially available at concentrations of 75, 80, 85 and 87 wt.% of PO_3. Higher concentrations, such as 105 wt.% (superphosphoric) and 115 to 118 wt.% (polyphosphoric) are also available. "Pure" or "technical grade" phosphoric acid is usually found at 85 wt.%.

Hydrogen chloride and hydrochloric acid. Hydrochloric acid is a solution of the gas hydrogen chloride; it is a poisonous, highly corrosive, hazardous liquid that reacts with most metals to form explosive hydrogen gas. Its appearance varies from pale yellow to colorless, according to purity.

Hydrochloric acid has many applications in the production of organic and inorganic compounds such as fertilizers, chlorides, dyes and more. HCl plays an important role in pickling of steel, acid treatment of oil wells, chemical cleaning and processing, and ore reduction among others.

When boiling all aqueous solutions, HCl forms an azeotropic constant-boiling mixture that contains 20.24% HCl and boils at 110°C (230°F).

Hydrogen fluoride and hydrofluoric acid. Anhydrous hydrogen fluoride (AHF) is a clear, colorless, corrosive fuming liquid with an extremely sharp odor. It easily dissolves in water to form hydrofluoric acid.

HF forms dense white vapor clouds if released. Both liquid and vapor can cause severe burns to all parts of the body. Specialized medical treatment is required for all exposures.

HF occurs naturally in volcanic gases and may result from industrial activities, such as coal-burning, and the manufacture or production of aluminum, phosphate fertilizer, steel and other chemical derivatives.

Commercially, HF is used to manufacture fluoropolymers, pharmaceuticals, aluminum, stainless steel,

TABLE 2. TOXICITY AND EMERGENCY RESPONSE DATA

	H_2SO_4		H_3PO_4	HNO_3	HF		HCl	
PEL (OSHA) [*6–8*]	1 mg/m^3		1 mg/m^3 TWA	2 ppm, 5 mg/m^3 TWA	3 ppm, 2 mg/m^3 TWA		5 ppm, 7 mg/m^3 ceiling	
REL (NIOSH) [*9*]	1 mg/m^3 TWA		1 mg/m^3 TWA; 3 mg/m^3 STEL	2 ppm, 5 mg/m^3 TWA; 4 ppm, 10 mg/m^3 STEL	3 ppm, 2.5 mg/m^3 TWA; 6 ppm, 5 mg/m^3 (15 min) ceiling		5 ppm, 7 mg/m^3 ceiling	
IDLH (NIOSH) [*9*]	15 mg/m^3		1,000 mg/m^3	25 ppm	30 ppm		50 ppm	
Incompatibilities & Reactivities	Organic materials, chlorates, carbides, fulminates, water, powdered metals. Reacts with water to produce heat. Corrosive to metals		Strong caustics, most metals, Reacts with metals to form H_2 gas. Do not mix with solutions containing bleach or ammonia	Combustible materials, metallic powders, hydrogen sulfide, carbides, alcohol. Reacts with water to produce heat. Corrosive to metals	Metals, water or steam. Corrosive to metals. Attacks glass and concrete		Hydroxides, amines, alkalis, copper, brass, zinc. Hydrochloric acid is highly corrosive to most metals	
UN Listing Number	1830: sulfuric acid; sulfuric acid, with more than 51% acid 1831: sulfuric acid, fuming; sulfuric acid, fuming, with less than 30% free sulfur trioxide; sulfuric acid, fuming, with not less than 30% free sulfur trioxide 1832: sulfuric acid, spent	2796: sulfuric acid, with not more than 51% acid	1805: phosphoric acid; phosphoric acid, liquid; phosphoric acid, solid; phosphoric acid, solution 3453: phosphoric acid, solid	2031: nitric acid, other than red fuming 2032: nitric acid, fuming; nitric acid, red fuming	1052: hydrogen fluoride, anhydrous	1790: hydrofluoric acid	1050: hydrogen chloride, anhydrous 2186: hydrogen chloride, refrigerated liquid	1789: hydrochloric acid; hydrochloric acid, solution
Emergency Response [*10*]	Guide 137	Guide 157	Guide 154	Guide 157	Guide 125	Guide 157	Guide 125	Guide 157

Notes: PEL: Permissible exposure limit REL: Recommended exposure limit IDLH: Immediately dangerous to life or health concentration TWA: Total weighted average STEL: Short time exposure limit ERG: Emergency Response Guidebook

high-octane gasoline, electronics (microchips and printed circuit board cleaning) and uranium isotopes. It is also used to etch glass or metal.

Safety and emergency response

Because acids are mostly hazardous chemicals, their toxicity levels and incompatibilities need to be taken into account when storing and transporting them, as well as how to respond in the event of a spillage.

Permissible exposure limits (PEL) for hazardous materials are given by the U.S. Occupational Safety and Health Administration (OSHA) regulations: 29 CFR 1910.1000, 29 CFR 1926.55 and 29 CFR 1915.1000 for the general, construction and maritime industries, respectively. Other toxicity levels, such as the Recommended Exposure Limit (REL) and Immediately Dangerous to Life and Health Concentrations (IDLH) are published in the U.S. National Institute of Occupational Safety and Health (NIOSH) Pocket Guide to Chemical Hazards. The chemical incompatibilities, health effects and other concerns when handling or storing hazardous chemicals are also given in the NIOSH Pocket Guide.

In the U.S., transportation of these acids or other hazardous materials is subject to the U.S. Department of Transportation Pipeline and Hazardous Materials Safety Transportation regulations. Transportation of hazardous materials in various forms (bulk, pipeline or tank cars) is subject to Title 49 of the Code of Federal Regulations (49 CFR).

In the event of spills of these acids or other hazardous materials, only properly trained personnel such as firemen and policemen (or properly trained plant personnel) should be involved in the emergency response and containment of the product.

The Emergency Response Guidebook 2008 (ERG2008) provides guidelines for managing emergencies when hazardous chemicals are involved. This guidebook is available in printed form, and can also be downloaded in convenient electronic form, including applications for smart phone that allow for quick searches of the chemicals and their associated guides. A new version of the Emergency Response Guidebook is scheduled for release this year (2012).

The chemical safety data for the acids covered in this article, including toxicity levels, incompatibilities and emergency response guides are summarized in Table 2.

Materials selection

The materials of construction, as well as any lining or internal coating requirements should be determined by a materials expert based on the acid, its concentration and storage conditions.

Aqueous acid solutions are very corrosive, and usually require special materials depending on the temperature or phase.

Some recommendations are given regarding the correct material selection depending on acid, such as in the following reference for H_2SO_4: NACE RP0391 — Materials for the Handling and Storage of Concentrated (90 to 100%) Sulfuric Acid at Ambient Temperatures; HF: NACE 5A171 — Materials for Storing and Handling Commercial Grades of Aqueous Hydrofluoric Acid and Anhydrous Hydrogen Fluoride.

Depending on the acid and storage, transport or process conditions, interior coatings or linings could also be considered. For instance, rail tank cars transporting concentrated sulfuric acid should be internally coated according to NACE SP0592 — Application of a Coating System to Interior Surfaces of New and Used Rail Tank Cars in Concentrated (90 to 98%) Sulfuric Acid Service.

TABLE 3. MATERIALS OF CONSTRUCTION, CLADDING & LINING [*12*]

Materials of construction	H_2SO_4
Aluminum	Aluminum alloys may be used to handle dilute (concentration below 10%) and concentrated acid (above 98%). It suffers corrosion for handling sulfuric acid in a range of concentration of 40–95 %
Carbon steel	It can be used to handle concentrated sulfuric acid at ambient temperatures under static and low-velocity condition. Corrosion resistance depends on temperature, acid concentration, iron content and flowrates
Cast Iron	Alloys with 14.5% content of silicon have shown best resistance to corrosion for sulfuric acid handling in all concentrations at temperatures up to the boiling point
Copper	Copper and copper alloys are not suitable for sulfuric acid handling
Lead	It has shown high resistance to corrosion in sulfuric acid handling up to 70% concentration. Although, this material is not recommended for pumps or valves
Nickel	Nickel 200 demonstrates good tolerance to sulfuric acid when it is handled at low or moderate temperatures
Niobium	It can be used for handling sulfuric acid at concentrations below 95% under oxidizing conditions
Gold	Exhibits excellent resistance to sulfuric acid up to 250°C (480°F) and is used when no corrosion can be tolerated
Platinum	Resists sulfuric acid in all concentrations and temperatures
Palladium	It is attacked by sulfuric acid in the presence of air
Rhodium	In wrought or cast form rhodium is not recommended for handling sulfuric acid
Stainless steel	Concentrated sulfuric acid turns extremely corrosive in presence of 316 and 304 stainless steels. The conventional austenitic grades show good resistance in dilute or highly concentrated acid at moderate temperatures
Zinc	It is slowly dissolved by dilute sulfuric acid; corrosion resistance depends on the concentration of the acid and the purity of the metal

TABLE 4. MATERIALS OF CONSTRUCTION, CLADDING & LINING [*12*]

Materials of construction	H_3PO_4
Aluminum	Aqueous solutions of phosphoric acid with concentration of 5 to 85% are highly corrosive for alloys 1100. Consequently this material is not recommended for phosphoric acid handling
Cast Iron	All cast irons can be considered to handle phosphoric acid; although the presence of contaminants must be previously evaluated since it can provoke severe cases of corrosion. High-silicon cast irons are ideal to manage phosphoric acid in all concentrations at any temperature, no presences of fluoride ions (F^-) are allowed
Copper	Copper and copper alloys can be used to manage pure phosphoric acid solutions in heat-exchanger tubes, pipes and fittings. System impurities can accelerate the rate of corrosion more than acid concentration
Lead	It is extensively used in the manufacture of phosphoric acid. It is highly resistant to corrosion
Nickel	Nickel alloys are appropriate for handling phosphoric acid. For dilute acid alloys 20Cb-3 and 825 are recommended; for concentrated acid at high temperatures alloy B-2 offers the highest corrosion resistance
Niobium	Resistant to corrosion for handling acid at temperatures below 100°C in all concentrations
Silver	Resistant to corrosion for handling acid at temperatures between 160 and 200°C in all concentrations
Tantalum	Resistant to corrosion for handling acid at temperatures up to the boiling point in all concentrations in absence of fluoride ions (F^-)
Stainless Steel	Conventional austenitic stainless steel has shown elevated corrosion resistance for all concentrations of phosphoric acid up to 65°C (150°F)

Tables 3–8 list some common metal alloys used in the CPI, along with their general acceptable use ranges (concentrations and temperatures) for each of the acids covered in this article.

Storage tanks

Usually aboveground storage tanks (ASTs) are used to store acid as they facilitate accessibility to tanks and ancillary equipment for inspection and maintenance. The storage tank should be sized for at least 50% more volume than required.

Tanks for acid storage are usually built of either metal (lined or non-lined), or fiber reinforced plastic (FRP). Metal tanks offer a higher durability, and can also resist higher stresses or impacts; whereas FRP tanks are economical, usually chemically inert, and can be a good alternative for low-volume, short storage times.

The mechanical design of tanks for acid storage usually follows either of the following codes:

- API STD 650 — Welded Steel Tanks for Oil Storage: for vertical tanks with flat bottoms and operating pressures less than 0.14 barg (2.5 psig)
- API STD 620 — Recommended Rules of Construction of Large, Welded, Low Pressure Storage Tanks: for vertical tanks with flat bottoms and operating pressures between 0.14 barg and 1.03 barg (2.5 psig and 15 psig)
- ASME BPV Code, Sect VIII, Div 1: for other operating pressures

Special design criteria, such as particular corrosion allowances or nozzle design, are also considered in acid storage tanks — either by special company or supplier criteria, or from professional associations. For instance, concentrated sulfuric acid tanks design should follow NACE SP0294 — Design, Fabrication, and Inspection of Storage Tank Systems for Concentrated Fresh and Process Sulfuric Acid and Oleum at Ambient Temperatures.

Tanks should allow access to the top nozzles and the vent system, and offer an appropriate facility for sampling. Periodically, it is necessary to homogenize the contents of the tank, because the acid that remains on the surface establishes a vapor-liquid equilibrium in which toxic and corrosive gases are released, so a recirculation system is recommended.

Special attention should be given to the acid physical properties in storage to prevent freezing, high corrosion rates or vaporization.

In general, corrosion rates increase at higher temperatures, so acids should be stored at the lowest possible temperature without freezing the acid. Higher corrosion rates could also result from heating of the metal surfaces due to sun radiation, so the tank exterior should be painted with a radiation reflecting color, such as white. Another regular measure to maintain acids at an appropriate temperature is coating the tank with an adequate material such as vinyl-based materials.

In places where the storage temperature could be below the acid freezing point, storage tanks and vessels should be provided with heating facilities, such as plate coils mounted on the outside of the tank wall, or external heat exchangers connected to

the tank. Internal heating coils are not recommended, because excess temperature in the coil walls accelerates corrosion and could cause leaks. Also, high-pressure steam is not recommended as a heating medium since heat exchange surfaces could exceed 100°C, causing severe corrosion.

Pressurized storage is required when the vapor pressure exceeds the atmospheric pressure at the storage temperature.

Common guidelines for acid storage tank design are summarized in *Chem. Eng.* May 2008, Facts at your Fingertips: Acid Storage.

When storing acids above ground, containment is also an issue. Tanks should be properly diked, or double walled, to contain spills. In general, containment should be at least for one tank volume (if not properly drained), or less provided there is adequate drainage to an acid neutralization pit, with blockage valves accessible to operators. Local code requirements should also be addressed when designing acid-tank containment; for instance, the U.S. State of Florida has specific requirements as given by Rule 62-762.891 — Mineral Acid Storage Tank Requirements.

Pumps

The design basis should be set before selecting a pump, that is, the operating conditions such as temperature, suction pressure, acid concentration, and so on.

A primary issue that must be taken into account while pumping acids is safety, so, the selected pump for the system cannot leave place for leakage; this is an advantage regularly offered by vertical submerged pumps over horizontal pumps. Also, material selection guidelines shall be followed to avoid casing, impeller or other internals damage.

Piping and fittings

Selecting pipe material and designing the pipe system is a very important issue in a plant, especially while handling acids. The system must ensure the acid is transported safely and efficiently. Piping should have as few flanges as possible, so the chance of having leaks becomes negligible.

TABLE 5. MATERIALS OF CONSTRUCTION, CLADDING & LINING [*12*]

Materials of construction	HNO_3
Aluminum	Aluminum alloys commonly used for nitric acid services are 1100 and 3003. Corrosion depends on temperature and concentration of the acid. Aluminum alloys are compatible with nitric acid at temperatures up to at least 71°C (160°F) when it is inhibited by hydrofluoric acid
Cast iron	Cast iron can be used for handling concentrated nitric acid under control conditions such as low temperature and low velocity. Corrosion attacks when handling dilute nitric acid
Stainless steel	For concentrations of 0 to 65%, most AISI 300-Series stainless steel has shown great corrosion resistance for temperatures up to the boiling point
Copper	Not suitable for use in nitric acid
Lead	It can be used for concentration between 52 and 70%
Molybdenum	Not suitable for use in nitric acid
Nickel	Nickel alloys are widely used in the production of nitric acid. Alloy 617 offers an excellent performance and corrosion resistance for handling nitric acid at high temperatures in the catalyst-support grids in high pressure plants
Niobium	It is completely resistant to nitric acid in all concentration at temperatures below 100°C
Gold	It is resistant to nitric acid in concentrations up to 50% above that it is attack by corrosion
Palladium	It is vulnerable to attack from corrosion when nitric acid is in presence of air
Rhodium	In wrought or cast form rhodium is resistant to corrosion produced by concentrated nitric acid at 100°C
Silver	Not suitable for use in nitric acid
Tin	Not suitable for use in nitric acid. Complex reaction occurs
Titanium	Appropriate for handling nitric acid at any concentration in temperatures below the boiling point. As temperatures exceed 80°C (175°F), corrosion becomes stronger depending on nitric acid purity. Titanium alloys can't be used for red fuming nitric acid due to a violent reaction that can take place in the system

TABLE 6. MATERIALS OF CONSTRUCTION, CLADDING & LINING [*12*]

Materials of construction	HF
Aluminum	Unsuitable for handling hydrofluoric acid
Stainless steel	Stainless-steel type 304 has a good performance for handling anhydrous hydrogen fluoride up to 200°C (390°F), it has poor resistance to dilute or concentrated hydrofluoric acid. On the other hand stainless-steel type 316 can be used for handling dilute acid at low temperatures
Copper	The use of copper alloys is affected by aeration and velocity, its corrosion resistance depends on the concentration and temperature
Lead	Fair corrosion resistance in a wide range of concentration and temperatures for handling hydrofluoric acid. Not recommended for handling dilute acid
Molybdenum	It offers great corrosion resistance to aqueous and anhydrous hydrofluoric acid with concentrations up to 50%, below 100°C (212°F)
Nickel	Nickel 200 is ideal for handling hot anhydrous hydrogen fluoride vapor, but it is not recommended for handling hydrofluoric acid in aqueous solutions
Niobium	Unsuitable for handling hydrofluoric acid
Tin	Unsuitable for handling hydrofluoric acid
Titanium	Unsuitable for handling hydrofluoric acid
Zirconium	Unsuitable for handling hydrofluoric acid

In order to select the piping material, the following aspects have to be defined: acid concentration, transport temperature, phase, fluid velocity, type of flow, impurities in the acid and solids presence.

Corrosion is often related to an acid's velocity. In order to maintain a low velocity of the fluid, a bigger pipe diameter is suggested.

Valves

Valves are used for various functions, including the following:

For blocking, gate valves or plug valves are regularly used. However, plug valves are preferred for this service, to ensure proper valve operation.

For control, globe or butterfly valves are suitable; they can be manually operated or be fitted with actuators.

Materials for different parts of the valves (disk, stem and seat) should be selected according to the acid concentration and operating conditions, by consulting the valve manufacturer.

Some common materials according to the acid to be handled are presented in Tables 9–13.

Acid handling

Sulfuric acid. Sulfuric acid must be stored separately from combustible and reducing substances in a well-ventilated environment at temperatures below 23°C (73.4°F). Concentrated acid needs to be isolated from water, as it may react violently, releas-

TABLE 7. MATERIALS OF CONSTRUCTION, CLADDING & LINING

Materials of construction	HCl
Aluminum	It is not appropriate for handling HCl; it has no resistance to corrosion
Cast Iron	Unalloyed cast iron systems are unsuitable for handling HCl, especially if high velocities are involve. A high-silicon iron alloyed with small amounts of molybdenum, chromium and copper can be used to handle hydrochloric acid up to 95° C (200°F) at all concentrations
Stainless steel	Corrosion attacks stainless steel (316) and stainless steel (304) when handling HCl at any concentration or temperature
Copper	Copper can be used to handle dilute hydrochloric acid only, due to its sensitivity to velocity, aeration and oxidizing impurities
Lead	It exhibits tolerance to corrosion at 24°C (75°F) and concentrations up to 15%. It is unsuitable for concentrated acid at higher temperatures
Nickel	Pure nickel and nickel-copper alloys can be used for handling hydrochloric acid below 10% concentration, without air presence, at low temperatures. The lower the concentration the higher can be the temperature of the system; for example, HCl at 0.5% can stand temperatures up to 200°C before corrosion attacks the alloy
Niobium	It has shown excellent corrosion resistance to handle HCl at any concentrations and temperatures up to 100°C (212°F)
Gold	It can be used for handling hydrochloric acid at any concentrations and atmospheric pressure up to the boiling point
Palladium	Unsuitable for handling hydrochloric acid
Rhodium	In cast or wrought form, rhodium has excellent corrosion resistance to handle concentrated hydrochloric acid in temperatures up to 100°C (212°F)
Silver	It is very susceptible to aeration when concentration and temperature are high
Tantalum	It has shown excellent corrosion resistance to handle HCl at any concentrations under atmospheric pressure and temperatures up to 90°C (195°F). It can be used to handle acid with concentrations below 25% up 190°C (375°F)
Titanium	Unsuitable for handling hydrochloric acid

TABLE 8. MATERIALS OF CONSTRUCTION, CLADDING & LINING

ACIDS	COMMON ALLOYS
H_2SO_4	For dilute and intermediate sulfuric acid (between 40 and 80% concentration) Incoloy alloys 25-6MO, 825, 020 and Inconel alloy G-3 have shown excellent corrosion resistance for temperatures up to 50 °C (120°F). When handling aggressive acid, Inconel alloys 625, 622, C-276 and 686 are suitable. For reducing conditions, Monel alloy 400 is appropriate in the absence of air for temperatures up to boiling point for concentrations below 15%. For storage of H_2SO_4, Monel alloy 400 can be used at room temperatures up to 80% concentration. Hastelloy B3, C-2000 and G-30 are also suitable for handling sulfuric acid
HNO_3	Chromium enhances corrosion resistance in alloys while handling nitric acid, due to this fact, Incoloy alloy 800 and 825 are adequate for nitric acid at all concentrations for temperatures up to the boiling point. Inconel alloy 600 and C-276 also offer good corrosion resistance to nitric acid for concentration over 20% at room temperature; alloy 690 has shown better corrosion resistant because its chromium content is higher. Hastelloy G-30 alloy and G-35 offer excellent corrosion resistance for this same reason
H_3PO_4	When handling phosphoric acid, Incoloy alloys 825, 020 and 25-6MO, as well as Inconel alloy G-3 are suitable and regularly used. For extreme conditions such as high temperature and high amount of impurities or halides contaminants, Inconel alloys 625, 622, C-276 and 686 are recommended. Hastelloy alloys B-3 and G-30 stand phosphoric acid in all concentrations and temperatures. Hastelloy alloy G-35 was especially designed for phosphoric acid wet processing in fertilizers manufacture
HCl	Incoloy alloys 25-6MO, 825 and 020, and Inconel alloy G-3 are used for dilute hydrochloric acid handling. Another alloy that offers good corrosion resistant in concentrations below 10% with aerated conditions at room temperature is Monel alloy 400. Nickel alloy 200 can be used at room temperature for concentrations up to 30% as well. For environments that contemplate the presence of oxidizing contaminants and hot hydrochloric acid, Inconel alloys 625, 622, C-276 and 686 are recommended. Hastelloy alloys B-3, C-2000 and G-30 are also suitable for handling hydrochloric acid, at all concentrations and temperatures
HF	The formation of fluoride films is key on engineering materials in order to offer good corrosion-resistance rates while handling hydrofluoric acid. Monel alloy 400 is widely used for this purpose, due to the fact that it has shown excellent corrosion resistance for all hydrofluoric acid services in all concentrations and temperatures up to (and even above) the boiling point. For anhydrous hydrogen fluoride up to 82°C (180°F), Nickel alloy 200 is commonly used. For dilute HF and temperatures up to 70°C (158°F) Inconel alloy 600 can also be used. Other alloys like Hastelloy C-2000 and Hastelloy G-30 are also recommended for handling hydrofluoric acid

ing heat. If sulfuric acid needs to be diluted or combined with water, then it has to be added to water carefully.

To manipulate sulfuric acid, proper personal protective equipment, such as gloves, a vapor respirator when ventilation is inadequate, face shield and full suit shall be used.

Nitric acid. Nitric acid must be stored separately in a corrosion resis-

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tant location, avoiding contact with powders, carbides, hydrogen, sulfide, turpentine and strong bases. Along the same lines it is important to mention that nitric acid's storage requires special conditions, such as adequate ventilation and especially low temperatures to ensure a cool environment for the solution, because heat may cause containers to burst and result in escape of poisonous gases; so it should not be stored above 23°C (73.4°F), and the container must remain dry and locked up.

Nitric acid and its vapors can cause severe damage during its handling to persons who have contact with it; the severity of the damage is related to the time of contact or exposure and the acid concentration.

Every process that involves nitric acid handling or storage must contemplate an adequate ventilation system that ensures airborne levels below the safety exposure limits allowed, not only this measure needs to be taken into account but also workers should be aware of the risks arising from management of nitric acid.

Phosphoric acid. Phosphoric acid can be described as a stable chemical, because it is not subject to thermal decomposition. However, the design criteria for its handling should be based on the acid concentrations and operating temperatures. The most important issue about this acid is the variation of its freezing point according to its concentration; the freezing point of standard concentrations are –17.5°C (0.5°F) at 75%, 4.6°C (40.2°F) at 80% and 21.1°C (70.01°F) at 85%, therefore it becomes necessary to heat phosphoric acid at high concentrations in order to maintain the acid as a liquid solution.

Unlike other acids, phosphoric acid does not react violently with metals; reaction occurs slowly and progressively with hydrogen as a product, so, caution should be exercised because the vapors formed are flammable.

Hydrofluoric acid. HF acid is a very hazardous material, both in liquid and vapor phase. It can cause severe burns, which may not be immediately painful or visible. HF is a strong irritant to the skin, eyes and respiratory tract. The fluoride ion easily penetrates the skin and generates destruction of tissue and severe bone damage.

Package sizes range from 500–1,000 mL for analytical products, to 10,000-L ISO containers. HF is delivered commercially in concentrations of 98 wt.%, 48–51 wt.% and 40 wt.%.

Due to HF's nature, strict measures shall be taken when handling the acid in industrial facilities. Such measures include administrative controls (for example, work permits); engineering controls (instrumentation: detectors, relief valves, emergency dump systems); and personal protection equipment (appropriate clothing).

When boiling all aqueous solutions, HF forms an azeotropic constant boiling mixture that contains 35.6% (by weight) HF and boils at 111.35°C (231.8 °F).

Hydrochloric acid. HCl must be stored in a corrosion resistant location. Even though the acid is nonflammable, when it is heated hydrochloric acid fumes are released,

TABLE 9. SPECIFIC EQUIPMENT, PIPING, VALVES AND PROTECTIVE CLOTHING GUIDELINES

Equipment	H_2SO_4
Tanks	Iron sulfate is produced in storage tanks of sulfuric acid; it is a consequence of interaction between the tank's surface and the acid. Usually iron sulfate precipitates, therefore the pump suction pipe should be placed above the tank bottom to avoid pumping solid residues that can compromise pump well-functioning. Under the same line, storage tanks must provide a facility to clean the tank bottom. The tank's maintenance should be performed periodically according to the laws of the state and the company policies
Pumps	According to the plant requirements, pumps used for sulfuric acid handling are usually horizontal centrifugal pumps or heavy duty vertical, submerged type For handling sulfuric acid at 93.19% (66 °Bè) usually horizontal centrifugal pumps with mechanical seals are used Common materials of construction are: cast iron or Alloy 20 wetted ends, Alloy 20 plunger, tetrafluoroethylene plastic chevron packings
Piping and fittings	For sulfuric acid service, welded pipe lines with schedule 80 are commonly used, these pipes should be kept full of acid to minimize corrosion attacks. Sulfuric acid also promotes hydrogen gas formation; for this reason it is necessary to avoid pressure buildup by venting the line In case of draining the pipe, the use of air is not recommended, because it can accelerate corrosion. Nitrogen can be used for such purposes
Valves	Butterfly Valves: Lead is an adequate stem and disk material for sulfuric acid at all concentrations; for concentrations lower than 75% at low temperatures Alloy 20 and Hastelloy have also been used. The seat should be made of PVF, with Viton and Hypalon also been used
Protective clothing	Properly fitted chemical safety goggles, face shield (8-in. high minimum) and protective clothing should be worn. Acid-proof clothing should be fitted snugly at neck and wrists, in a manner preventing drainage of acid to gloves or boots. Impervious rubber or polyvinyl chloride gloves with gauntlets covering forearms should be used. Boots made of the same material should be worn, with tops being covered by the trousers. Head protection via hard hat or full cover acid hood should be worn, as well as a respirator for protection against fumes

TABLE 10. SPECIFIC EQUIPMENT, PIPING, VALVES AND PROTECTIVE CLOTHING GUIDELINES

Equipment	H_3PO_4
Tanks	Heating coils should be provided in order to maintain the phosphoric acid above its freezing point, depending on ambient conditions and acid concentration. For instance, 85% H_3PO_4 freezes at 21.1°C
Pumps	All fittings should have wetted parts of 316 L stainless steel, with mechanical seals rather than packing. Centrifugal pumps are also used for phosphoric acid handling
Piping and Fittings	Stainless steel 316 is regularly used for piping because it has shown excellent results in corrosion resistance for all concentrations of phosphoric acid, even though the piping material can be the same used for storage When using stainless steel, the fittings and valves should be welded or flanged; screwed fittings are not recommended because they may allow leakage
Valves	Butterfly valves: 316 SS, Alloy 20 and Hastelloy C are good stem and disk materials for phosphoric acid at various concentrations, with Monel also showing fair results. Common seat materials include: PVF, Neoprene, Hypalon, Viton or EPT
Protective Clothing	Properly fitted chemical goggles and protective clothing should be worn. Impervious gloves and aprons are recommended. No special respiratory protection is required under ordinary conditions of use, provided that adequate ventilation is maintained. When vapor or mist concentrations exceed applicable standards, approved respiratory protective equipment must be used

TABLE 11. SPECIFIC EQUIPMENT, PIPING, VALVES AND PROTECTIVE CLOTHING GUIDELINES

Equipment	HNO_3
Tanks	For acid grades lower than 95 wt.%, tanks should be designed for slight pressure and vacuum, with fumes collected at a disposal system and sent to a scrubber. Vent piping should be designed taking into consideration possible corrosion from contact with moisture
Pumps	Wetted parts should be made of 304L stainless steel for concentrations lower than 95 wt.%; for higher concentrations, they should be made of titanium (with a water content higher than 1.34% to prevent spontaneous combustion), silicon iron or 3003 aluminum alloy [*11*]
Piping and Fittings	Piping made of 304L stainless steel is frequently used for HNO_3 up to 95 wt.%, and of aluminum for higher concentrations. Carbon steel (CS) piping with TFE, FEP or glass linings (up to certain temperatures) can also be used for all grades. [*11*]
Valves	Butterfly valves: 316 SS, Alloy 20 and Hastelloy C are good stem and disk materials for nitric acid at various concentrations. Seats made of Viton can handle various concentrations up to 70%; for low concentrations at low temperatures, Neoprene, Hypalon and EPT have also been used
Protective Clothing	Neoprene or natural rubber latex gloves are acceptable for handling nitric acid.

TABLE 12. SPECIFIC EQUIPMENT, PIPING, VALVES AND PROTECTIVE CLOTHING GUIDELINES

Equipment	HF
Tanks	Anhydrous and 70 wt.% HF up to 66°C, or HF between 60 to 70 wt.% up to 38°C, can be stored in carbon steel (CS) tanks, since the metal is passivated with an iron fluoride film when the fluid is in contact with the metal. Hydrogen corrosion may occur in steel tanks. Other grades of HF can be stored in tanks made of CS with natural rubber lining, polyethylene or unplasticized PVC [*11*]
Pumps	Diaphragm pumps with TFE or polychlorotrifluoroethylene (CTFE) diaphragms can handle anhydrous, 70 wt.% and electronic-grade HF. Centrifugal pump materials depend on grade: Ni-Cu alloy of Alloy 20 is used for anhydrous HF, Vinylidene chloride (VC)-lined steel for 70 wt.% and electronic-grade HF, and Penton-lined steel or solid Penton for electronic grade HF [*11*]
Piping and fittings	Anhydrous and 70% wt HF can be transported in seamless CS piping. The rating and schedule should be selected according to the operating pressure and corrosion allowance, with Sch 80 and Sch 160 commonly used for both grades, respectively. CS with VC, TFE and FEP lining is also used, depending on fluid temperature. Electronic-grade HF can be transported in unplasticized PVC pipe
Valves	Butterfly valves: Hastelloy C is the best material for the stem and disk, with Alloy 20 also exhibiting fair results. Common seat materials include: PVF, Hypalon and Viton; for pure (100%) HF, only PVF or Viton should be considered
Protective Clothing	Neoprene and natural rubber gloves are excellent for handling hydrofluoric acid in all concentrations, glove change is necessary before 8 hours

TABLE 13. SPECIFIC EQUIPMENT, PIPING, VALVES AND PROTECTIVE CLOTHING GUIDELINES

Equipment	HCl
Tanks	Outdoor tanks are preferred for storing hydrochloric acid; some common measures of protection when tank is placed indoors are coating the floor with asphalt or another corrosion resistant material to prevent several damages in case of leaks or spills. The tank must be provided with a vent so acid fumes do not accumulate in the tank and a drainage system so maintenance can be performed periodically. Vents should be routed to a scrubber
Pumps	Pumps similar to those used for H_2SO_4 and H_3PO_4 can be used. Centrifugal pumps lined with, or constructed of TFE, PVDF of Derakane are commonly used. Mechanical seals of carbon and ceramic faces with TFE or fluoroelastomer secondary seals, and Hastelloy C metal parts, are also recommended [*11*]
Piping and fittings	CS piping with TFE, PVDF, Derakane or polypropylene lining is frequently used for HCl. PVC or FRP piping have also been used, depending on fluid pressure
Valves	Butterfly Valves: common stem and disc materials include: lead or Hastelloy C. Common seat materials include: PVF, Neoprene, Hypalon and Viton
Protective clothing	For concentrations up to 40% neoprene and fluoroelastomer gloves are recommended for handling hydrochloric acid. For concentrated acid, butyl gloves are suitable

which can compromise the safety and toxicity levels allowed, therefore storage tanks need proper venting that shall be directed to a safe location and treatment facility.

Operators handling hydrochloric acid must wear protective equipment and it is advisable for them to take a shower and gargle with sodium bicarbonate after manipulating the acid in order to avoid teeth corrosion in other activities performed by the operator.

Undesirable reactions can take place between hydrochloric acid and the following compounds: chromate, permanganate and sulfate. Such reactions generate chlorine gas as a result. A subsequent reaction occurs with metal peroxide forming its corresponding chloride.

When storing hydrochloric acid, proper ventilation has to be ensured in order to maintain the acid concentration in air below the permitted limit of exposure. ■

Edited by Gerald Ondrey

Authors

Alberto Baumeister is the CEO and co-owner of the Ecotek group of companies, which is located in the City of Knowledge, Panama (Calle 60, PH Obarrio 60, Piso 15, Ofc. 15-A, Obarrio, Panamá, Rep. De. Panamá; Phone: +507-203-8490; Fax: +507-203-8491; Email: abaumeister@ecotekgrp.com). He has experience as coordinator and senior process consultant in engineering projects for the chemical, petrochemical, refining, oil and gas industries. He has a Master's Diploma in water treatment management from Universidad de León (Spain, 2011), a specialization in management for engineers at Instituto de Estudios Superiores de Administración (Venezuela, 1990), and a degree in chemical engineering from Universidad Metropolitana (1987), graduating first of his class. He has been a professor of the Chemical Engineering School at Universidad Metropolitana between 1995 and 2007 and has written several technical publications for international associations.

Sebastiano Giardinella is the vice president and co-owner of the Ecotek group of companies (same address as above; Email: sgiardeinella@ecotekgrp.com). He has experience as process leader, and in project planning and control, in engineering projects for the chemical, petrochemical, refining, oil and gas industries. He is a certified project management professional (PMP), has Master's Degree in project management from Universidad Latina de Panamá (Panama, 2009), and a degree in chemical engineering from Universidad Simón Bolívar (Venezuela, 2006), graduating *summa cum laude*; and is currently a student of the M.Sc. in renewable energy development at Heriot-Watt University (Scotland). He is also professor of project management at Universidad Latina de Panamá, and has written technical publications for *Chemical Engineering* magazine, international associations and academic institutions.

Mayhell Coronado is a process engineer of the Ecotek group of companies (same address as above; email: mcoronado@ecotekgrp.com). She has experience in the development of conceptual and basic engineering projects for the oil and gas, and chemical industries, as well as in support to company management. She has a degree in chemical engineering from Universidad Metropolitana (Venezuela), where she taught thermodynamics, transport phenomena and general chemistry as professor assistant, and has published a work on the effects of microbial contamination in diesel fuel used for thermal power generation.

From Batch to Continuous Processing

Continuous flow reactors can provide many benefits over batch processes. This article answers why and how

Robert Ashe
AM Technology

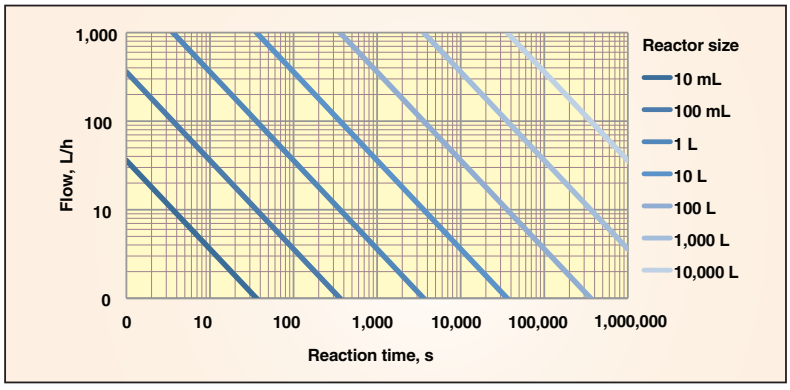

FIGURE 1. Reactor sizes for a range of throughputs and reaction times are shown

Small chemical reactors offer a number of benefits compared to large reactors, such as better heat transfer and mixing. While small batch vessels are impractical at the industrial scale, continuous flow reactors can provide the benefits of small physical size without the practical difficulties of multiple vessels. This article considers the four basic needs of flow reactors (volumetric capacity, heat transfer, plug flow and mixing) and how they influence choice of equipment.

Flow reactors

A small chemical reactor has a higher ratio of heat transfer area to working volume than a large reactor. It can also deliver more mixing energy per unit volume without bending the agitator shaft. Since the outcome of most chemical reactions is linked in some way to mixing or heat transfer (or both), small reactors have inherent advantages over large ones. Depending on the reaction type, these advantages can contribute toward substantially lower capital and operating costs. Small batch vessels, however, are impractical at the industrial scale since hundreds or even thousands of process cycles would be required for commercial throughputs. The solution to this problem is to use a flow reactor. This is a channel or series of mixed stages in which process materials react as they flow through it. It is analogous to a series of small, stirred batch vessels on a conveyor belt. A flow reactor provides the benefits of small physical size without the practical difficulties of charging and emptying multiple small vessels.

Historically, flow reactors have been more common in bulk chemical processes where the problems of vessel size are most acute. With growing pressure on chemical manufacturers to meet ever-tougher cost and regulatory targets however, the range of applications for flow reactors has started to expand rapidly.

Flow reactors are not new technology, but many of the applications they are now being developed for are new, and as such progress is confronted by the usual adoption problems of market resistance, availability of the right equipment and lack of user knowhow.

User knowhow is arguably the biggest hurdle to progress since most engineers and scientists were trained in batch methods and have had little experience with flow technology. While good knowhow does exist, this is still concentrated within a relatively small number of individuals or groups. For the wider industrial community, many basic questions relating to the business case and implementation methods for adopting flow processes fail to get answered properly. For example, the question of energy saving is often dismissed on the grounds that the temperature cycle in a flow reactor is broadly similar to that of a batch reactor and therefore, there is no net energy saving. This, however, disregards the fact that a flow reactor may have many tons less of hardware to heat and cool, and does not require a five-fold oversizing of the utilities to cope with uneven heating and cooling loads. Implementation of flow reactors also remains far too reliant on trial-and-error rather than systematic methods. All too often good applications for flow technology get abandoned during trials for lack of the right equipment or operating methods.

There are difficult challenges for flow reactors, such as fouling and large solids. Apart from these however, flow reactors are easier to scale up than batch systems, since the size differences between laboratory and production scale are greatly reduced. Success, however, is contingent on a

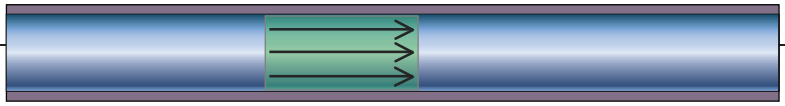

FIGURE 2. In plug flow, the fluid velocity across the face of a channel is uniform

good understanding of the underlying process characteristics and a broad knowledge of reactor types to address the specific needs.

Reasons for continuous

The benefits of flow reactors vary according to the nature of the process, but they typically include the following:

- Improved yield and quality — For competitive and consecutive reactions, flow reactors can deliver significant improvements to yield and purity
- Reduced solvent use — For heat-transfer limited reactions, flow reactors can operate with leaner reaction mixtures and therefore with less solvent. Similarly, continuous countercurrent processes for operations, such as extraction, yield better separation with less solvent
- Capital cost — For mixing or heat-transfer limited processes, flow reactors are smaller and often significantly smaller than batch reactors. Smaller physical size contributes to lower equipment costs as well as smaller utilities and buildings. Capital expenditure reductions on building costs alone for a continuous plant can amount to 50% or more [*1*]
- Utility costs — Over 50% of the energy used in a batch reactor is wasted on the hardware, cross mixing between the heating and cooling fluids and uneven utility loads. Flow reactors can reduce this waste by 90% or more by virtue of reduced physical size and steady state operation
- Plant flexibility — Batch reactors have good flexibility in terms of the unit operations they can perform, but very limited flexibility in terms of working capacity. A flow reactor is more specialized in terms of unit operations it can perform, but the capacity per cycle can be varied by orders of magnitude by changing the cycle time. This can contribute to greater plant flexibility and therefore less process equipment
- Safety costs — For hazardous processes, the cost of managing safety in small flow reactors is inherently cheaper than large batch reactors. Small pressure vessels are cheaper to fabricate and require smaller emergency-relief systems

The extent to which these benefits apply depends on factors such as process type, product value and throughput. It also depends on whether the application is for an existing facility or a new one.

Manufacturers from the chemical process industries (CPI) often view the batch versus continuous question as an either/or option, and reject flow reactors on the grounds that the batch vessels are still required for work-up operations. While this may be true in many cases, the commercial advantages of using flow reactors within existing batch processes should not be underestimated. Flow reactors generally occupy small footprints and can be integrated within existing batch plants. Where flow reactors can make a material difference to yield or reduction in batch failures, the commercial case for using them within existing batch processes can be self-evident. There are also other reasons for using flow reactors within existing batch processes, such as debottlenecking, reducing energy waste, more efficient use of solvents, improved safety, reduced problems of bursting disc failure (which can disable an entire production facility) and increasing plant flexibility.

Understanding the process

While microreactors can be used successfully with very little process information, the same does not apply to larger flow reactors. Scaling up flow reactors by trial-and-error is difficult and potentially dangerous. A good understanding of the process is required before trials are undertaken.

A variety of analytical devices can be used to study flow processes, but one of the most useful tools for generating design data is a reaction calorimeter, since this provides information about both kinetics and heat of reaction. It is also worth noting that the differences between batch and flow reactors relate to scale (there is no flow effect) and a small reaction calorimeter with similar heat transfer and mixing characteristics (to the proposed flow reactor) can provide reliable design data for scaleup.

Even where processes are relatively well understood however, there is no substitute for testing under flow conditions. The equipment used for scale-up development should be as similar as possible to the full sized reactor. This generally favors the use of large flow reactors for scaleup work rather than microreactors (other than where microreactors can be used at the production scale).

Reactor capacity

The volumetric capacity of a flow reactor should be as small as possible. Apart from the obvious benefits of small footprint and higher performance, small flow reactors have proportionately lower startup and shutdown losses. The physical size is calculated from the relationship:

Reactor size (L) = *Throughput* (L/s) × *Reaction time* (s)

Reactor sizes for a range of throughputs and reaction times are shown in Figure 1.

A flow reactor can be scaled up by a process of numbering up (or scaling out). For this, the channel size is kept constant and capacity is increased by using multiple parallel channels of similar size. The size of the scaled up system can be predicted accurately when numbering up, since the functional capabilities of the channels remain unchanged.

The reactor can also be scaled up by increasing the channel length, although scope for this is limited by pressure drop. In some cases, longer channels will give better performance (due to higher velocities) and therefore require smaller working capacities than predicted.

Increasing the tube diameter is often the only practical way of scaling up, especially where high reactor volumes are required. Increasing the diameter, however, often reduces performance, and therefore the scaled system may have to be larger than predicted to compensate for the difference.

When scaling up, it is preferable to use the maximum tube diameter possible, subject to satisfactory performance. Short, large-diameter tubes have lower fabrication costs, a reduced tendency to block and a lower pressure drop. They are also easier to clean.

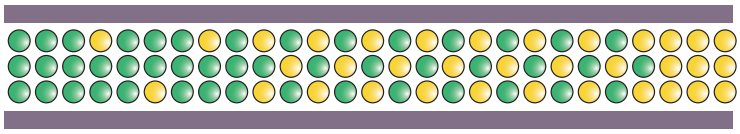

FIGURE 3a. In a plug flow reactor, material is only retained in the reactor while it is reacting, and it cannot be discharged until the required residence time has elapsed

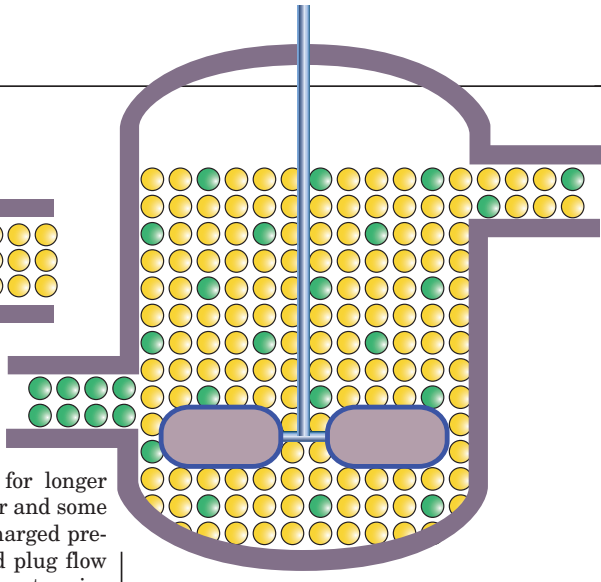

FIGURE 3b. An example of a system that is not plug flow is the continuously stirred tank reactor (CSTR), which is fully back-mixed. In the absence of plug flow, reacted material is retained for longer than required, and unreacted material is discharged prematurely. It is therefore inherently less efficient than either a plug reactor or an ideal batch reactor

Plug flow

Plug flow describes a flow pattern where the fluid velocity across the face of a channel is uniform (Figure 2). It also implies no back mixing and all fluid elements having the same residence time. While plug flow is required for most applications, ideal plug flow cannot be achieved in practice due to the effects of molecular diffusion, surface drag, bends and obstructions in the channel.

Microreactors typically have channel diameters of less than 0.5 mm. In these systems, the plug flow characteristics benefit from low fluid velocities [*2*]. This is a useful characteristic as the reaction time can be varied from less than a second to many minutes in relatively short channels.

In larger diameter channels, good plug-flow characteristics benefit from high fluid velocities [*2*], and the channel length has to be varied to suit the reaction time. Good radial mixing is also desirable to prevent the fluid from separating into zones of fast — and slow — moving fluid. Radial mixing can be achieved by means of baffles, static mixing elements, dynamic mixing and turbulent flow. A good approximation to plug flow can also be achieved by using multiple stirred tanks in series, and the higher the number of tanks used the better. Above ten stages, however, the cost of additional stages starts to outweigh the incremental benefits.

Plug flow is determined by experimental methods such as dye injection tests. These data need to be treated with care, because the quality of plug flow required will change with the reaction rate. The observed quality of plug flow can also change as the physical properties of the fluid changes.

Plug flow provides the means for separating reacted and unreacted material (Figure 3a). Without plug flow, some product is retained for longer than required in the reactor and some unreacted material is discharged prematurely (Figure 3b). Good plug flow is the key to minimizing reactor size and optimizing yield and purity. A common misconception is that plug flow is not required for zero order reactions (where the reaction rate is independent of reactant concentration). This is not correct. Plug flow is important for virtually all processes, even for zero order reactions. The exception to this is fast zero-order reactions (typically a few seconds or less) where reactor size is not a significant cost issue and there are no consecutive reactions.

For many applications, the benefits of good plug flow are not limited to residence time control. Where the reaction rate is dependent on reactant concentration (nth order reactions), low back mixing reduces dilution of unreacted material giving faster reaction rates and higher yields per unit volume. Plug flow also provides the means for preventing material from over reacting, and reducing reactions between reactants and product.

Where the outcome of a reaction is particularly sensitive to plug flow, high fluid velocities and long channels are desirable. From the perspective of pressure drop and reactor cost, however, there are practical advantages to minimizing channel length. A good compromise is to use long small-diameter tubes where the reaction rate is high (and therefore more sensitive to back mixing) and larger diameter tubes where the reaction rate is slow.

Mixing

Mixing can be characterized in many ways, such as applied mixing energy, uniformity of mixing, time to achieve a uniform blend, Reynolds number or mass transfer characteristics for two phase mixtures. There are also different mechanisms for promoting mixing, such as molecular diffusion, turbulent flow or splitting and folding of fluid streams. In a flow reactor, the optimum mixing condition is good radial mixing (mixing across the tube) with low axial mixing (mixing along the tube).

Scale is important for mixing. For example, a small flow reactor can blend a fluid in a fraction of a second, whereas a 1,600-L batch reactor can take 10 to 20 seconds [*3*].

Microreactors operate under laminar flow conditions with Reynolds numbers of typically less than one hundred [*4*]. The flow streams are parallel, and these systems rely principally on molecular diffusion for radial mixing. Microreactors have a reputation for good mixing that is not always deserved. For demanding mixing applications, such as with two phase fluids, it is often necessary to employ static mixing elements within the channel to promote radial mixing.

The need for good radial mixing becomes increasingly important as the diameter of a flow reactor is increased. Without radial mixing, large tubes bring problems of poor blending, poor plug flow and reduced heat-transfer performance.

There are two general methods for promoting radial mixing: dynamic

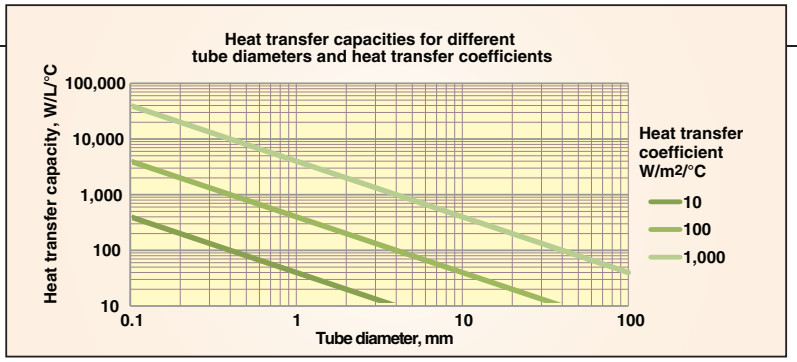

FIGURE 4. Cooling capacities for different tube sizes and heat-transfer coefficients are shown here

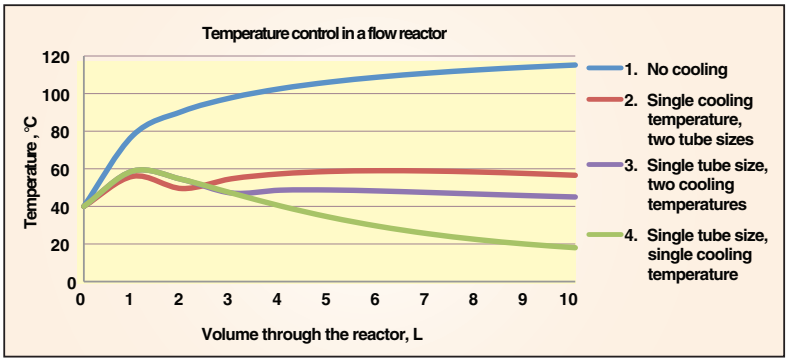

FIGURE 5. This example illustrates different approaches for temperature control

mixing and static mixing. In dynamic mixing, mechanical stirrers generate radial mixing. In static mixing, radial mixing is promoted by either turbulent flow or elements that break up the flow pattern (baffles or static mixers).

Mixing is a difficult parameter to characterize, and the mixing conditions can also vary within the channel as the physical properties of the process material changes. For this reason, there is no substitute for testing. Some general considerations for mixing in larger systems include the following:

- Good radial mixing is required throughout large flow reactors even when the process fluid is fully blended in order to maintain good plug flow and where applicable, efficient heat transfer
- For some processes, such as competitive reactions, mixing at the point where reactant streams combine can be essential for optimum performance. For these, different mixing strategies may be required at the inlet and early stages of the reactor
- Processes with more than one phase (solid, liquid, gas) usually have mass transfer limitations. To overcome the limitations, these processes require high mixing efficiencies throughout the reactor to maximize interfacial area- and mass-transfer coefficients
- Where solids are present in the process fluid, efficient mixing is required to keep them uniformly dispersed and free flowing. Dynamically mixed flow reactors have the advantage of keeping solids suspended if the flow is interrupted. In a statically mixed system, an interruption to flow can lead to a serious blockage
- Where the process fluid contains fragile materials, such as crystals or live cells, mixing is constrained by the need to prevent product damage

Heat transfer

In a tubular flow reactor, heat is added or removed by a cooling jacket. The amount of heat that can be transferred per unit volume of product in a tube (Q_v) is determined by the relationship:

$$Q_v = (4 \times U \times \Delta T) / D$$

The heat transfer coefficient (U) is typically in the range of 50–1,500 W/m^2/°C. There is little scope for varying this since the parameters that determine it are fixed by other needs.

The heat transfer rate can be controlled by varying the temperature difference between the process fluid and the heat transfer fluid (ΔT). This is typically in the range of 0 to 50°C, but it can be higher where the process fluid can tolerate extreme surface temperatures.

The amount of heat that can be transferred per unit volume of product (Q) is inversely proportional to tube diameter (D). This means that the tube diameter needs to be small enough to cope with the process heat load. Cooling capacities for different tube sizes and heat transfer coefficients are shown in Figure 4.

Where the product can tolerate extreme temperatures, heat transfer is not important to design and short large-diameter tubes are preferable for reasons of cost, low pressure drop and ease of cleaning. In such cases, the feed and discharge temperatures can be controlled by separate feed and discharge heat exchangers, respectively. If the process temperature can rise above the normal boiling point, elevated operating pressures are required to suppress boiling. Boiling in a flow reactor is undesirable as it reduces the working volume.

Temperature control. For many processes, good temperature control is necessary and reactor design has to take account of variations in process heat load along the channel. In microreactors, high levels of heat can be added or removed with low temperature differences between the jacket and the process fluid. This means that a single cooling strategy can often be applied without causing serious overcooling where the process heat load is low. In larger flow reactors, higher temperature differences between the jacket and process fluid are necessary to compensate for the lower surface-to-volume ratios. If a single cooling strategy is employed under these conditions, problems of overcooling can arise in areas where the process heat load is low (for example at the end of an nth order reaction). While it is feasible to manage this with co-current flow (between the heat transfer fluid and process fluid), a more practical approach is to treat the channel as a series of stages with different channel sizes or jacket temperatures (to cope with different heat loads). The example shown in

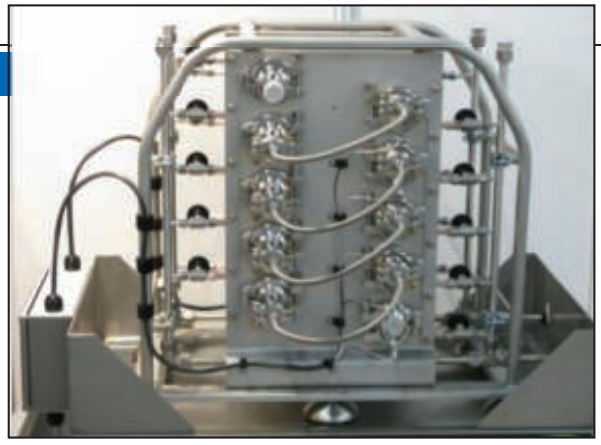

FIGURE 6. This dynamically mixed plug-flow reactor uses transverse mixing in tubes

Figure 5 illustrates different cooling strategies for an exothermic reaction in a 10-L reactor:

- In the first case, no cooling is applied, and the tube diameter is 38 mm. The overall reactor length is 9 m, and the product temperature rises to over 100°C
- In the second case, a single coolant temperature of 0°C is used. The first cooling stage (2 L) uses a 5-mm-dia. tube, and the second stage (8 L) uses a 38-mm-dia. tube. The overall reactor length is 109 m and the process temperature is held at 40–60°C
- In the third case, a single tube of 5-mm dia. is used. The first cooling stage (3 L) uses a cooling jacket at 10°C, and the second stage (7 L) uses a cooling jacket at 40°C. The overall reactor length is 509 m and the temperature is held at 40–60°C
- In the fourth case, a single tube of 5-mm dia. is used, and the cooling jacket is at 10°C throughout. The overall reactor length is 509 m, and the process material is progressively cooled to below 20°C

The first case is the simplest and cheapest solution since the tube length is short and no cooling system is required. In the second and third cases, the process temperature is controlled within a range of 20°C but the second case has the advantage of shorter tube length. In the fourth case, the product is subject to severe overcooling, which will affect the reaction rate (and hence loss of yield).

Common flow reactors

Most flow reactors fall into two broad categories according to whether they use static or dynamic mixing.

- Static flow reactors rely on fluid movement through the reactor to generate radial mixing and the higher the axial velocity, the better the mixing. Having no moving parts, they can be fabricated with very small channel diameters. They are generally smaller reactors suited to fast reactions with homogenous fluids (although multiphase mixtures can be handled in some cases)
- Dynamic flow reactors use mechanical stirring to mix the product. The mixing performance is independent of fluid velocity through the reactor. These are generally larger systems suited to slower reactions and multiphase mixtures

Flow reactor types

This section summarizes common flow reactor types. It has to be accepted that the comments are generalizations and there will be considerable overlap in terms of capabilities between different reactor types.

Microreactors (statically mixed): These typically have channel diameters of less than half a millimeter, although channels of up to 1 mm or more are often described as microreactors. The flow channel may be a simple tube or may include static mixing elements to promote mixing. They operate under laminar flow conditions with a Reynolds number of less than 100 [*4*].

Advantages: Very good heat transfer; Good plug flow at low velocities; Can handle reaction times from less than one second to many minutes within short channels; Low startup and shutdown losses; Can be used with very little process knowhow

Disadvantages: Generally poor mixing with two phase fluids, although this can be addressed with static mixing elements; Very high cost per unit volume; Poor solids handling capabilities and easily blocked; Difficult to clean other than by flushing; High pressure drop

Typical applications: Ideal for R&D where limited quantities of reagents are available; Can be used for low throughput product for reaction times of a few seconds or less

Tubular flow reactors (statically mixed): These are simple tubes ranging from a few millimeters in diameter to over 50 mm. They rely on turbulent flow for effective mixing and plug flow.

Advantages: Moderate to good heat-transfer capacity depending on tube diameter; Simple with low fabrication cost; Low pressure drop in larger diameter systems; Larger-diameter tubular reactors are easy to clean

Disadvantages: High tube lengths other than for short reaction times; Poor to moderate mixing, which limits performance with multiphase mixtures; Tube lengths have to be varied with reaction time; Poor to moderate solids handling and easily blocked if flow is interrupted; Startup and shutdown losses can be high

Typical applications: Generally suitable for small or large scale production with low viscosity fluids and limited mixing requirements; Generally better for dedicated applications since performance is sensitive to fluid velocity (and therefore length has to suit the reaction time)

Tubular flow reactors with baffles or static mixing elements (statically mixed): Baffles or static mixers in tubes give better mixing and plug flow than simple tubes, and this improves with increased density of mixing elements.

Advantages: Moderate to good heat transfer depending on tube diameter; Moderate to good mixing depending on density of mixing elements and fluid velocity; More flexible than simple tubular reactors in terms of reaction time for a given length; Can operate effectively under laminar as well as turbulent flow conditions subject to adequate density of mixing elements

Disadvantages: Higher fabrication cost than simple tubes; While performance is improved with higher density mixing elements this is achieved at the cost of higher pressure drop and increased difficulty of cleaning; Generally poor solids handling capabilities and easily blocked with high density mixing elements; On large diameter systems with high density mixing ele-

ments, hot spots can occur at the center of the tube

Typical applications: Suitable for a broad range of manufacturing operations, but preferably with clean fluids; For reasons of cost and pressure drop, generally better suited to reaction times of a few minutes or less; Given the practical difficulties of cleaning, generally better for dedicated applications

Oscillatory flow reactors (statically mixed): In static flow reactors, mixing relies on axial movement of fluid through the channel. The penalty for this is high tube length and high pressure drop for processes with long reaction times. The oscillatory flow reactor addresses this problem using bidirectional flow. This gives higher average velocities in short, larger diameter tubes even with long reaction times.

Advantages: Significant reduction in overall length compared to other static flow reactors; Better solids handling characteristics than other static flow reactors; Moderate to good mixing; Generally more flexible than other static flow reactors in terms of residence time

Disadvantages: The additional variable of oscillatory flow makes characterization more difficult; Accumulation of gas or vapor in the reactor can severely impair mixing performance; Performance is a trade off between good mixing (which favors short tubes) and good plug flow (which favors long tubes); An additional pump is required for the oscillatory flow; This has to generate relatively high pressures and can present material selection problems with some process fluids

Typical applications: Suited to a broad range of slower reactions and can handle slurries

Single, continuously stirred-tank reactor (dynamically mixed): This is a conventional stirred tank with continuous feed and discharge. This is not a plug flow reactor, which severely limits its suitability for most applications.

Advantages: Good mixing that is independent of fluid velocity through the reactor; Good solids handling; Very low pressure drop (gravity transfer can often be used)

Disadvantages: Poor performance for most applications due to the lack of plug flow; Significant oversizing is required to compensate for the high level of reacted material retained in the reactor; Moderate to poor heat transfer capacity depending on size

Typical applications: Only a small number of applications suit this type of reactor. These are generally zero order processes where reactor size is not a significant cost factor and there are no consecutive or competitive reactions

Multiple, continuously stirred-tank reactors in series (dynamically mixed): The lack of plug flow in a single-stage stirred tank can be overcome by using multiple stirred tanks in series. While as few as three tanks in series have been employed by some users, a substantial improvement in plug flow (in terms of residence time distribution) is observed by using ten stages [*5*]. Above ten stages, the trade-off between cost and performance becomes increasingly difficult to justify. For very small systems, agitated cell reactors that employ transverse mixing can be used. For larger systems, conventional tanks with rotational stirrers can be used.

Advantages: Good mixing that is independent of fluid velocity through the reactor; Good solids handling; Very low pressure drop (gravity transfer can often be used)

Disadvantages: Small systems are expensive due to the high number of stirred stages required for optimum performance; Large systems suffer from the same heat-transfer and mixing limitations of conventional batch reactors

Typical applications: Process development with multi-phase mixtures; At the industrial scale, they are more common where volumetric capacities of thousands of liters are required

Dynamically mixed plug-flow reactors: Plug flow reactors need to be long and tubular to achieve good plug flow. While rotating stirrers can be designed for use in tubes, they generally present significant cost and fabrication problems. An alternative design for agitated tube reactors uses transverse mixing with free-moving agitator elements (Figure 6). These generate strong radial mixing in long tubes without the cost and technical problems associated with mechanical seals, baffles and shaft stability.

Advantages: Good mixing that is independent of fluid velocity through the reactor; Good plug flow; High flexibility in terms of reaction time range for a given tube length; Short, large diameter tubes that are easy to clean; Good handling characteristics for multi-phase mixtures. Very low pressure drop (gravity transfer can often be used)

Disadvantages: Subject to minimum diameter constraints, which limits the scope for strong exotherms where good temperature control is required

Typical applications: Processes with long reaction times, slurries and gas-liquid mixtures

Conclusion

The batch to continuous debate encompasses a broad spectrum of views. At one extreme are those who remain fully committed to batch methods. For some applications batch reactors are clearly a better solution or the only option. Processes with sticky materials for example are inherently unsuited to flow systems as are some slurries with large solids. In the absence of heat transfer or mixing constraints, batch reactors are also cheaper than flow reactors (although significantly less energy efficient). The resistance to change from batch to continuous is often justified on the grounds that existing batch processes are well optimized and offer little scope for improvement. This is true for some processes and particularly so when the cost of switching from batch to continuous is factored in.

Where there are mixing or heat transfer constraints however, the commercial benefits of flow reactors are often clear cut and compelling. It has been suggested that as much as 60% of commercially important non-polymer reactions could be run more efficiently in flow reactors [*6*] (and higher if peripheral benefits are included). To quantify these benefits, however, account needs to be taken of many factors that contribute to lower cost of goods. It also has to be recognized that most processes in use today were developed for batch reactors and would

not necessarily be the first choice of method given the option of more-capable flow reactors.

At the other extreme of the batch to continuous debate are advocates of fully continuous processes where multiple steps are carried out within a single process train. The difficulty here is that good residence-time control is critical to many flow processes. Problems in one stage of a continuous process will impact the entire process train, and the system will only be as good as the least reliable step. As process equipment and knowhow develop in this field, the scope for integrating multiple steps will improve, but as things stand, multi-step continuous processes will remain the exception rather than the rule.

The near term future for chemical process technology must lie between the extremes of fully batch and fully continuous [7]. There are many common processes where incumbent methods are severely constrained by heat transfer and mixing limitations. Integrating flow reactors within existing batch processes for the right applications will contribute to: smaller equipment, greater plant flexibility, fewer batch failures, improved yield/quality, improved safety, lower energy costs and reduced solvent use. Progress in this area, however, will depend on a broader understanding of the subject in terms of methods for identifying suitable applications for flow reactors, a better knowledge of the available equipment and systematic methods for process development. ■

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Author

Robert Ashe (AM Technology, The Heath Business & Technical Park, Runcorn, Cheshire, WA7 4QX, U.K.; Phone: +44 (0) 1928.515454; Email: robert.ashe@amtechuk.com) is a chemical engineer with over 35 years of experience in fine chemicals, pharmaceuticals and food manufacturing. Much of his career has been devoted to chemical plant design in high-value chemical manufacturing. In 2000, he co-founded AM Technology with David Morris. This company specializes in the manufacture of flow reactors and holds a broad portfolio of patents for dynamic mixing in tubes.

Blending, Sampling and Segregation

These aspects of dry solids mixing represent three sides of the same coin. Do not discount them

Thomas G. Troxel
Jenike & Johanson, Inc.

FIGURE 1. This pattern shows how the faster velocity in the center displaces material relative to the slower material at the walls

Blending and segregation are two opposite and competing processes in solids handling that ideally fit the expression "two sides of the same coin." But, if you think about it, coins have three sides and sampling completes the idiomatic expression perfectly. Blending is a necessary and essential process operation widely employed in many industries and materials, from nanoscale powders to run-of-mine ores. Segregation is almost always an unwanted consequence of handling operations that reverses the blending process or creates a need for blending where it might not otherwise be necessary if it were not for the effects of segregation. Sampling is the vitally important tool for measuring and quantifying a blend, understanding the sources and effects of segregation and for troubleshooting problems.

Blending

Blending is the process of combining two or more materials to achieve a combined product. The mixture may be a combination of dissimilar materials such as cement, sand and aggregate to make concrete; or cereal flakes, raisins, nuts and marshmallows to make breakfast cereal. A blend may also be a combination of chemically similar particles blended to create a uniform mixture of particle sizes, or another property such as color, flavor, melt index and so on. Blending bulk solids can be achieved in many ways including batch and continuous methods and with a wide variety of equipment, covering the range from extremely low intensity blending with gravity recirculation or layering components onto a belt, to high intensity blending with high-speed, high specific-horsepower blenders.

All of the various equipment and processes for blending solids rely on three principal mechanisms for achieving a blend: convection, diffusion and shear.

Convection. Convection is the transfer of a collection of particles from one location to another. This can occur as a result of material cascading in a tumble blender, material moving against the blade of a ribbon or paddle blender or as a result of gas pressure pulses in a pneumatic blender.

Diffusion. Diffusion is the random redistribution of particles that occurs as a result of increased particle mobility. Increased mobility typically occurs when the bulk density of the material is decreased sufficiently to allow individual particles to move relative to one another. Fluidization in fluidized bed reactors or granulators results in diffusion. Mechanical blenders move collections of particles by convection, but when the speed of the agitator is sufficient to locally fluidize material, diffusion occurs. Tumble blenders, continuous screw blenders and agitated blenders also provide opportunities for diffusion where material is cascading or in free-fall.

Shear. The definition of shear as it applies to solids blending is sometimes misunderstood. Shear occurs in a flowing granular solid as a result of a velocity gradient, and can develop as either a discontinuous shear (for instance, a shear or slip plane) or as a continuous gradient of velocity. In either case there can be some overlap in what could be called shear and what might be characterized as convection. The important difference, at least as it applies to solids blending, is in the intensity. Shear planes that develop in gravity blending or in a tumble blender or screw blender will achieve blending predominantly by a

convective process. Shear in a high-speed mixer will have a different end result in that it will be much more effective in breaking up agglomerates of fine powders and distributing small-particle-size material with high surface activity.

Figure 1 is a cross section view through a bin showing the displacement of material as it flows. The pattern shows how the faster velocity in the center displaces material relative to the slower material at the walls. This displacement pattern can be described as shear deformation, but the blending that occurs is caused by convection of collections of material from the central part of the bin that reach the outlet sooner than material near the wall. If material withdrawn from the bin is circulated back to the top, the discreet fill pattern that was placed into the bin will be smeared and homogenized, and some degree of blending will be achieved after several recirculations [*1*].

In a high-speed agitated blender, shear occurs with significantly higher intensity near the agitator but affects a relatively small volume of material. The high-intensity shear can break agglomerates, plate fine particles onto the surface of larger particles and redistribute particles with much higher intensity, causing better diffusion of small particles that may adhere to themselves or other particles. Ideally, the convective component of blending in this type of blender brings all of the material in the blender into the high shear zones.

Blend quality

In casual conversation, terms such as "uniform blend", "homogenous mixture", and "well blended" are often used without quantifying what they mean. In the heavily scrutinized pharmaceutical industry, regulations and guidelines dictate specific and often rigorous methodology for quantifying the uniformity of a mixture. Food and other consumer products must also meet trade regulations for delivering the stated quantity of product and composition of ingredients. Quantifying the structure of a blend requires defining not only the composition (for instance, the proportion of each component and allowable variation) but also the quantity of material that is to be measured. The compositional requirement of a blend is usually well known because it is either the entire objective of the process to produce a particular mixture, such as a pharmaceutical tablet or box of cereal, or it is an intermediate mixture that has well defined requirements. Determining the amount of material to measure is sometimes less clear, and may be different depending on who is answering the question. In pharmaceutical applications the answer is clear in that it is almost always based on the unit dose of the blend that makes up the final tablet.

FIGURE 2. Sifting segregation occurs when a mixture containing a range of particle sizes is allowed to form a pile. Smaller particles sift between larger particles as each layer of material slides along the pile surface

In other applications the sample size subjected to scrutiny is not as clear cut. Take, for example, a box of cereal. In one specific example a manufacturer produced a cereal with a cereal particle and a marshmallow candy particle. The packaging line filled boxes from a series of conveyors that fed a mixture of the two components into a gravimetric packaging machine that dispensed the required weight of the blend into each package. The two components of the mixture were of similar size and density and the only blending that occurred was in the handling system along several conveyors and in a surge hopper. Each component was metered onto the conveyor system at the correct ratio, but there was not a discreet blending process. The manufacturing plants fine tuned their systems to get an acceptable mixture into each box.

In this case, each box was always within weight tolerance for the stated package quantity, but there was no direct control of the ratio of the two components in any box. In an effort to improve quality, a new packaging line was installed that handled each component individually up to the packaging head where each component was weighed in a series of weigh hoppers, and then each box was filled from several hoppers of each component, selected by the packaging system controller, to control both the proportion of each component and the total weight in a box. This would seem an ideal solution since it would guarantee both accurate package weight and composition. From the producers point of view, they had improved quality by insuring that every package met their quality standard.

However, to everyone's surprise, consumer complaints from packages produced at this plant went up. What the plant had gained in compositional accuracy they had sacrificed in uniformity. Consumers found that individual bowls of cereal poured from the new packages had much wider variations of the two components than boxes filled from the old filling line. The new filling line had given up control of blending, even though the blending in the old system was very "low tech". The new system allowed only about one second from the time the ingredients were separate until they were combined in the package. There was very little opportunity to do any blending, and the result was that some boxes were well blended while others were segregated. From the manufacturers point of view, they had "improved quality" and were taking responsibility for what they could control to the highest degree of precision, but they were not controlling the sample size of interest to consumers.

Random blend. The simplified case of blending two different ingredients that do not have significant bonding or other interactions is often referred to as a random blend. In a random blend, all individual particles are free to move relative to each other, and

FIGURE 3. In the example described in the box on p. 44, cylindrical pellets with a nominal size of about 3 mm were being blended with nearly spherical beads with a size range from about 1 mm down to 150 microns

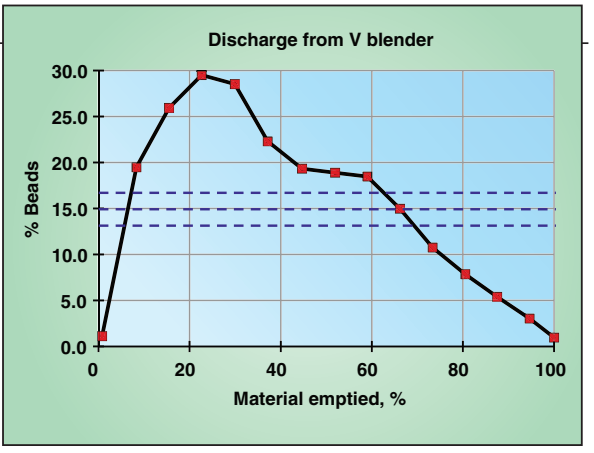


FIGURE 4. Implementation of a more-rigorous sampling method in the example on p. 44, revealed inadequate blending. Area between dotted lines represents desired blend ratio

particle bonding is very weak or does not occur. Particles that form random blends can be easy to blend, because the particles move easily relative to one another, but that also means that they 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. This process is referred to as segregation. Random blends are never perfect, and even in the best-blended state there will be random variations in concentrations of components.

Ideal random blends are uncommon in most industrial applications, although the cereal example described above comes close. In most dry blending applications, particles have some tendency to interact with one another by way of chemical, molecular, physical, or other means such that individual particles can agglomerate, coat or bond to one another. When particle interaction occurs, the blend is referred to as an ordered, structured, or interactive blend. In an ideal world, this type of blending process could create new particles, each consisting of the combined individual ingredients. A perfect particle created in this blender would consist of the desired ratio of each ingredient. This concept of combining ingredients to create new, chemically homogeneous particles is one of the primary objectives of wet and dry granulation processes. In most industrial blending processes, the reality is somewhere between a random blend and an ordered blend. Some particles of the blend may have very little tendency to interact while other blend components may have significant interaction.

Segregation

Segregation is the separation of particles that have identifiable differences. By definition, identical particles cannot segregate. Segregation can be driven by many forces including gravity, electrostatic forces, fluid drag forces and elastic forces.

Sifting. The most prevalent type of segregation is sifting, which results in separation by particle size. Sifting segregation occurs to some degree in virtually every industry that handles bulk solids. Sifting will occur in mixtures of different size particles when particles are sufficiently large so that surface forces are weak relative to gravity (usually larger than 100 microns), when particles have mobility relative to one another (in other words, they do not adhere to each other) and when there is some mechanism to allow particles to move relative to one another. Collections of different size particles will not spontaneously segregate when at rest, but will often readily segregate when allowed to move in many common, industrial handling processes.

Figure 2 shows an example of sifting segregation that occurs when a mixture containing a range of particle sizes is allowed to form a pile. Smaller particles sift between larger particles as each layer of material slides along the pile surface. As the pile builds up, the finer fraction deposits preferentially near the center and coarser fraction near the edges.

A blend of perfect structured particles of identical size will not segregate after discharge from the blender. However, if there are size differences between the particles, then segregation by size may occur. Even if all the particles are chemically homogeneous, this segregation may cause problems because it may affect other blend properties, such as solubility or bulk density. There are many examples where this type of segregation is important, such as with spray-dried laundry detergent and food products, and other natural materials such as clay cat litter, coal and other mineral materials. In these examples the particles vary in size, but are virtually identical from a chemical standpoint.

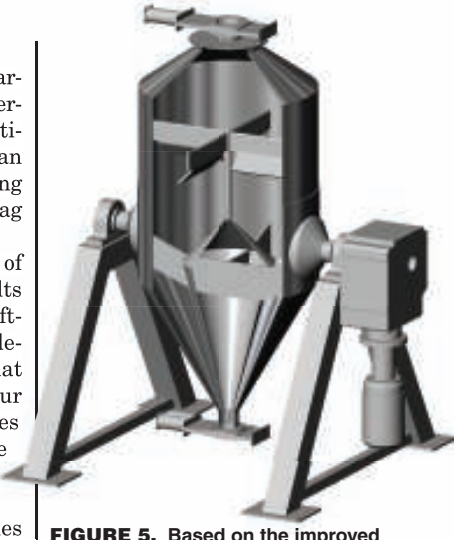


FIGURE 5. Based on the improved sampling results, the example blender was modified to improve the blending performance and minimize segregation mechanisms

In many other industrial-blending applications, particles not only differ in size, but also in chemical composition. And in most cases, particles of different size also have different chemical composition, so sifting segregation not only results in physical

AN EXAMPLE

In an example involving plastic compounding, engineers for the project had experience using a V-type rotating blender for similar materials at other plants. Batch ingredients would be fed into a scale hopper and then discharged into the blender. The blended batch would then be discharged into a surge bin feeding an extruder. While this process had worked successfully at other compounding plants, this plant would blend ingredients with a wider size range. The most extreme case involved blending the two materials shown in Figure 3, which consisted of cylindrical pellets with a nominal size of about 3 mm and nearly spherical beads with a size range from about 1mm down to 150 microns.

Engineers for the project ran tests in a small-scale V-blender and evaluated blend uniformity by taking samples from the blender. The overall blend results showed a lot of scatter. Some samples were acceptable, while others were significantly outside the desired blend ratio. Engineers also noticed that when the batch was discharged after a test, the material in the receiving container was severely segregated. This prompted development of a more rigorous sampling method that included collecting samples from the discharge of the blender from beginning to end. When this was done the results, shown in Figure 4, clearly showed inadequate blending. Based on this realization, further testing was done to determine if a tumbling type of blender could be used. After a series of trials, a cylindrical blend vessel with an conical hopper and insert was found to produce a blend within specification, and discharge a blended stream to the downstream vessel.

This example illustrates several key points about blending, segregation and sampling. First, the blender must provide the correct blending action for the solids. These two materials are very free flowing and hence blend easily. Blend time in the tumbling type blender is very short, on the order of tens of revolutions. However, the other side of the coin is equally important because the material also segregates readily, so maintaining the material in a blended state is difficult. This required two modifications to the geometry of the blend container. First, the dead zone near the center of the blender had to be reduced because the blender geometry did not allow enough diffusion to mix material in the center of the blender. Convection dominated the action in the blender, and diffusion occurred on the cascading surfaces, but some material remained poorly blended in the center. In addition to the blending performance, the flow pattern that developed during discharge had to be changed from funnel flow, where some of the blend remained stagnant during discharge, to mass flow, where all of the blend flowed as it discharged. In funnel flow, the highly segregating material could de-mix along shear surfaces during discharge. A mass flow pattern was achieved by designing a sufficiently steep hopper-shaped blend container with an internal bullet to displace material in the center of the container. The final configuration of the blender, shown in Figure 5, produced the results shown in Figure 6.

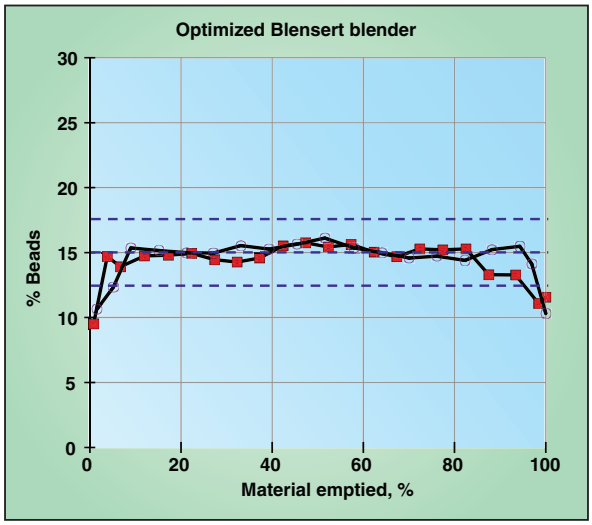
The other important point illustrated in this example is sampling. Without sampling the blender discharge stream from start to finish, it would be impossible to know if the blend delivered to the extruder would be uniform and within specification.

A blending process shouldn't be considered as an independent step in a block diagram, but really must be treated as a synergy of the mixing activity, segregation and sampling to produce the desired end result. ❑

property differences associated with size distribution, such as bulk density and fluidization behavior, but also differences in chemical composition.

Fluidization segregation and particle entrainment. Other common mechanisms of segregation that act primarily on particle size are fluidization segregation and particle entrainment or dusting segregation. Fluidization segregation occurs where gas movement through a bed of solids causes finer, lighter particles to rise to the top surface of a fluidized blend of powder, while the larger, heavier particles concentrate at the bottom of the bed. Particle entrainment or dusting segregation occurs when fine particles in a blend are carried by air currents (such as during transfer of a blend into a container) and then settle preferentially at the container walls.

FIGURE 6. The sampling results for the new blender design show the improved performance. Area between dotted lines represents desired blend ratio



Sampling

Sampling is essential in determining the state of the blend in a blender, in downstream equipment or in the final delivered product. Samples are analyzed to measure the variables of interest to the application, such as particle size, chemical composition, pH, dissolution rate, color and so on. The overall average of the sample results represents the average composition of the blend. Variations between samples provide a measure of blend uniformity. Variability can be expressed by statistical measures, such as standard deviation, coefficient of variation, or relative standard deviation, as well as other measures [2].

The single most important rule of sampling is to collect a sample that accurately represents the state of the blend at the point where it is sampled. In many industrial processes this is a significant challenge due to either the difficulty of getting a sample at the desired location or because of the potential for sampling error that is introduced by the sampling method. The two "golden rules" of sampling are to always collect a full stream sample, and always collect a sample while it is moving [3].

In the case of the cereal example given above, sampling is easy because the sample is either a whole box of cereal or individual servings poured from the box. In either case the sample is easy to get and it is possible to analyze the entire sample. For other processes, significant challenges can prevent the use of good sampling techniques. In an example involving limestone

at a circulating fluidized-bed boiler, the size distribution of the limestone used for fluegas desulfurization came into question. This became a critically important question because the plant could not meet emission regulations above about 50% output, and the blame was falling on the size distribution of the limestone produced by the onsite preparation plant. The plant produced about 50 ton/h of limestone, and to get a meaningful sample was not a trivial task. To satisfy all parties involved, the plant had to modify its chute systems to put in large diverter gates and position several operators to coordinate the collection of simultaneous full-stream samples in 55-gal drum quantities.

Samples of this size pose another challenge in analysis. Usually it is not practical or convenient to analyze such large sample quantities, so samples must be reduced or split. Simply grabbing a small sample from a large sample container can introduce significant sampling error, especially if sifting segregation can occur. The preferred method of splitting samples is to use a spin riffler, which can divide a sample into a number of smaller splits, usually from 6 to 16 divisions. Spin rifflers are capable of providing a high degree of uniformity between the individual sub-samples.

Another challenging task is sampling from a static bed in a blender. Clearly these samples cannot be collected while the material is moving, so some means of getting a meaningful sample must be used. Simply scooping material from the top surface is insufficient for many reasons, including particle segregation, which could make the top surface significantly different from material deeper in the bed. A sampling thief is often used to collect samples from a stationary bed.

A sampling thief is a probe that can be inserted into a bed of material to collect a sample from below the surface. Many designs exist, but in their basic form, they consist of a rod, frequently with a pointed tip. The rod has cavities cut into it along its length which are covered by a tube that fits closely over the rod and has openings that correspond to the cavities in the rod. The tube can be rotated to cover the cavities or open them. When the rod is inserted into the bed of material, and the tube rotated to exposed the cavities, blended material will fill the cavities, which can then be covered again and withdrawn. Thief sampling has been used extensively and studied thoroughly. While it remains the only practical way to collect samples from all positions in a static bed, it has been shown to have biases and limitations that can cause sampling error. These can come from properties of the blend and from operator technique. Some typical sources of error include local segregation as material fills the cavities, smearing as the thief is inserted into the bed, and preferential adhesion or repulsion of some components of the blend.

Sampling will always introduce

some degree of error, which can affect the sampling results in three ways. Perhaps the most common effect is that sampling increases the variability of samples, which produces data showing that the uniformity of the blend is worse than it actually is. In some cases, this presumed "fact" is used to suggest that a blend is actually better than what the sampling data shows.

Sampling error can also produce bias, or an overall shift in the results, indicating that the measured average from the collected samples is higher or lower than the known blend composition. This type of error can be confounding because people often jump to the conclusion that there is "missing" or "extra" material, somehow. It is more likely that one component adheres to, or is repelled by, the thief, or preferential flow of one component into the thief shifts the sample composition. Another possibility is that a sample is collected from an inappropriate location, such as a known dead zone in the blender that may be holding the "missing" material. When this type of error is observed it usually requires additional detective work to determine the cause of the bias.

Sampling devices can also produce samples that show lower variability than are actually present due to smearing of the sample. This type of error is often referred to as "counterfeiting." Counterfeiting is hard to detect because it may produce results that show blending is good.

The purpose of sampling is to accurately quantify the state of a blend or uncover blend quality issues, not to mask or overlook suspected problems. Samples should be intentionally collected from suspected regions where the blend may be less uniform, not just from the middle of the blender, or during the "best" part of a production run. Fringe regions may include the surface of the material, regions where the material may have been stagnant during blending and at the beginning and end of discharge from a batch bin. ■

Edited by Rebekkah Marshall

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Author

Thomas G. Troxel is a vice president at Jenike & Johanson, Inc. (3485 Empresa Dr., San Luis Obispo, CA 93401; Phone: 805-541-0901; Email: tgtroxel@jenike.com). He holds a B.S. in Engineering Science (ES) from California Polytechnic State University (Cal Poly) in San Luis Obispo. After graduating as the Outstanding Senior in the ES program in 1981, he worked at General Dynamics Co. He then helped to open Jenike & Johanson's San Luis Obispo, California facility in 1982. Troxel has been intimately involved in many aspects of the firm's consulting and research activities on a wide range of projects including flow properties testing, modeling, blending, pneumatic conveying and fluidization. He has been a major force behind the firm's expansion in mechanical design engineering and custom-built equipment.

Reduce Hazards in Process Vacuum Systems

Stanley S. Grossel
Process Safety and Design Consultant

Reduce explosion risks, and chemical and physical hazards, to ensure safer operation of vacuum pumps and related systems

Vacuum conditions for many unit operations — including distillation, evaporation, drying, crystallization, filtration and more — are typically achieved using the following three approaches:

- Steam ejector systems
- Mechanical vacuum pumps
- Integrated vacuum systems (which combine steam ejectors and mechanical vacuum pumps)

This article is the second part of a two-part series,[1] and provides practical guidance for addressing — during design and operation — some of the most commonly encountered hazards and maximizing the overall safety of these systems.

This section reviews the potential hazards that can occur with process vacuum pumps and systems. These include chemical sources of hazards, explosion-related hazards and physical sources of hazards. They are discussed below, and are based on material in Reference 1.

Chemical sources of hazards

Chemical reactions and explosions. All possible chemical reactions that a vacuum system might encounter at any point in mechanical vacuum pump systems should be considered — particularly those that could result in a fire or explosion (deflagration). And such an analysis should consider these scenarios during normal use, potential misuse and failure conditions.

For instance, experience has shown that explosions have occurred in situations involving materials that were not originally considered by the system designer, and in which the failure mode of that particular scenario had not been taken into account. During this analysis, the reactions that should be considered include homogeneous, heterogeneous and abnormal reactions. Two other sources of chemical hazards are toxic and corrosive materials. Each of these chemical sources of hazards is also discussed below.

Homogeneous reactions. Homogeneous reactions occur in the gas phase between two or more gas molecules. If such reactions are possible in a given manufacturing process, the process pressure and reactant concentrations must be carefully controlled to prevent excessive reaction rates.

Heterogeneous reactions. Heterogeneous reactions require a solid surface (a catalyst) to occur. Most heterogeneous reactions become homogeneous at higher pressures, normally at pressures well below atmospheric pressure. This means that the way in which the gases react inside the process equipment will not necessarily relate to the way that they react when compressed by a mechanical vacuum pump.

Abnormal reactions. Abnormal reactions can occur when chemicals come into contact with gases or materials that the designer did not anticipate. This can occur, for example, when there is a leak that either allows atmospheric gases (air) to leak into the system, or toxic, flammable, or explosive gases to leak into the atmosphere.

To prevent the occurrence of abnormal reactions, high-vacuum applications should typically maintain a leak tightness of 1×10^{-5} mbar-L/s (1×10^{-3} Pa-L/s), or lower. It must be ensured that all valves in the system are leak-tight across their seats.

In estimating an acceptable air-leakage rate, do not overlook the implications of process contamination by air leakage. If leakage into the vacuum system could in any way adversely affect the process, or create a safety hazard, the acceptable air leakage may be considerably less than that presented in Ref. *1*, pp. 83–86, for medium- and high-vacuum systems.

Consideration should be given to the fact that gases that do not normally come into contact with each other during the process cycle may be mixed in the pumping system and exhaust piping. It is possible that water vapor or cleaning solutions may be present in the process equipment after routine maintenance procedures due to the equipment having been washed and cleaned. Water vapor may also enter the system from exhaust ducts and exhaust scrubbers.Where solvents are used to flush process deposits from the vacuum system, it is important to ensure that the selected solvent is compatible with all the process materials in the vacuum system.

Explosion-related hazards

Explosions can occur in vacuum pumps. The most common causes of violent rupture of a vacuum system due to an explosion results from the ignition of flammable materials, or the blockage or restriction of the pump exhaust. In general, the source of explosion hazards in vacuum systems fall into one of the these categories:

- Oxidants
- Flammable materials
- Pyrophoric materials
- Unstable materials

Oxidants. Oxidants, such as oxygen, ozone, fluorine and others are often pumped using vacuum pumps. Oxidants readily react with a variety of substances and materials. The reaction often produces heat and an increased gas volume with a potential for fire and overpressure in the pump or exhaust system. The practices discussed below are recommended to minimize the potential for an explosion in pumps handling oxidants:

1. Editor's note: Part 1 of this two-part article appeared in the September 2012 issue (pp. 59–66).

1. Always use perfluoropolyether (PFPE) lubricant in pumps that are used to pump oxygen in concentrations above 25 vol.% in an inert gas. Although PFPE lubricants are preferred, hydrocarbon-based lubricants may be used if a suitable inert purge is used to guarantee that the oil is not exposed to unsafe levels of oxidant.
2. If it is not desired to use PFPE lubricants in oil-sealed rotary vane or piston vacuum pumps, dilute the oxidant to a safe concentration using an inert gas (dry nitrogen). However, this approach is only feasible for relatively low flowrates of oxidant gases.
3. One must install safety features in a mechanical pump system to ensure that the minimum flow of the dilution gas required to reduce the oxidant concentration to a safe level is always available, and to ensure that the flow of oxidant does not exceed the maximum allowed flowrate. The safety system must be designed so that the flow of oxidant stops immediately if these conditions are not met.

Flammable materials. Many gases are flammable and can explode (deflagrate) if they are within the flammability limits, and there is an ignition source of sufficient energy available (for instance, from a localized heat source). It may be possible to reduce the explosion hazard if the concentration of flammable gases is lowered by introducing an inert gas at several points within the pump or system.

Another way to reduce the probability of an explosion is to eliminate potential ignition sources. It may be difficult to eliminate all ignition sources because there is often a possibility of static discharge or friction hot spots where rotating machinery is used.

Where it is not possible under abnormal conditions to avoid the flammable zone, the equipment must be designed to contain any resulting explosion without rupturing or transmitting a flame to the environment. This is typically achieved by installing a flame arrester on the exhaust piping.

Pyrophoric materials. A number of gases (such as silane, phosphine and others) are pyrophoric — that is, they are spontaneously flammable in air at atmospheric pressure, so combustion could occur if the gases were to come in contact with air anywhere in the vacuum system. This can happen if air leaks into the system, or if the system exhaust comes in contact with the atmosphere. In a confined space (such as an exhaust duct, a dust filter, or an oil box of a mechanical pump) combustion can cause an explosion.

If, in addition to pyrophoric materials, an oxidant is also present in the process, the probability of an explosion occurring at both atmospheric and process conditions increases greatly. Similarly, if oxidants from other processes are vented through a common extraction (treatment) system, combustion or an explosion could result. It is necessary, therefore, that an independent extraction system be used when pyrophoric materials are pumped.

While the use of PFPE lubricants in the pump will not prevent the ignition and explosion of pyrophoric materials, it will help to minimize the risk of a subsequent oil fire. However, PFPE lubricants can absorb process gases which, in the case of pyrophoric materials, can lead to local ignition when the lubricant is exposed to air. The probability of this occurring can be reduced by using a dry vacuum pump, which contains no lubricant in the swept volume.

Unstable materials. Sodium azide is occasionally used in the preparation of products for freeze drying and other manufacturing processes. Sodium azide can produce hydrozoic acid, the vapors of which can react with heavy metals to form unstable metal azides, which may then explode spontaneously.

Brass, copper, cadmium, tin and zinc are commonly used in many components in vacuum pumps, accessories and piping. If one's process system uses or produces sodium azide, it must be ensured that the gas path in the process system does not contain heavy metals.

Toxic materials. Toxic materials are not necessarily explosive in nature, but they are hazardous to one's health, and thus require special care. The following methods for handling them in vacuum pump systems should be considered:

- *Gas dilution:* Systems can be installed to allow the dilution of toxic process gases as they pass through the vacuum pump and into the exhaust piping. Dilution may be used to reduce the concentration of the gas to below its toxic limit
- *Leak detection*: Many vacuum pumps are generally designed to be leak tight to a level of $< 1 \times 10^{-3}$ mbar-L/S ($< 1 \times 10^{-1}$ Pa-L/s). However, the leak-tightness of the adjoining process system cannot be ensured and thus should be checked with a reliable leak-detection method (such as helium mass-spectrometry leak detection) to confirm the integrity of the vacuum and exhaust systems
- *Shaft sealing (dry pumps):* Many vacuum pumps use a gas purge to ensure that process gases do not enter the gearbox, and thereby, potentially escape into the atmosphere. The integrity of the purge gas supply must be ensured when handling toxic gases. Non-venting regulators must be used in combination with a non-return check valve
- *Shaft sealing (other pumps):* Oil-flooded shaft-seal designs (for instance, for mechanical booster pumps and rotary vane pumps) minimize the risk of process gas leakage (or, of the in-leakage of air), and can provide a visual warning (oil leakage or oil level reduction) before a hazard arises. Other seal designs may not give adequate warning of failure
- *Magnetic drives:* Where total hermetic sealing is required, dry vacuum pumps can be fitted with a magnetic drive employing a ceramic containment vessel (housing); this eliminates the need for shaft sealing on the motor input shaft

If pressure-relief valves or rupture disks are used to relieve excess pressure, ensure that they are safely vented into a suitable exhaust system to prevent the toxic gas from escaping into the atmosphere and possibly coming in contact with people.

Corrosive materials. Corrosive gases generally attack the material they are in contact with by the mechanism of ion exchange, a process that can only happen in the presence of a suitable liquid solvent (such as water). This mechanism cannot occur when the material is in the vapor phase, even if a suitable solvent is present.

The possibility of corrosion occurring in mechanical vacuum pumps should be considered when carrying out a hazard analysis.

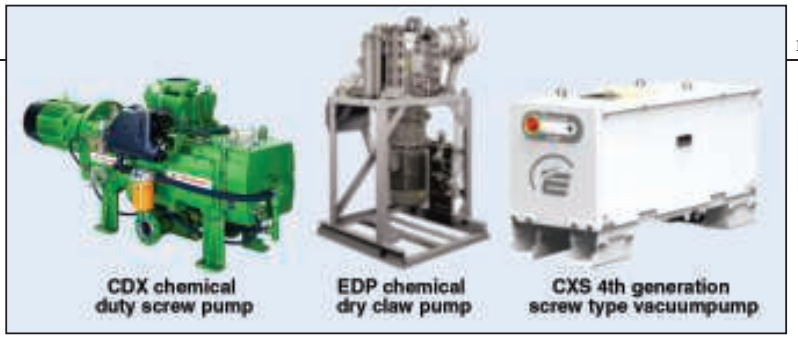

FIGURE 1. Prudent design, operating and maintenance strategies are needed to minimze the potential hazards associated with all types of vacuum systems

Many vacuum pumps are not designed to resist wet corrosive materials. Therefore, the following points should be considered:

- *Anhydrous materials:* The removal of a liquid solvent (water) will prevent corrosion. However, special care must be taken to prevent the ingress of wet air from the exhaust of the vacuum pump, especially when the pump is stopped and air is sucked back into the pump. An inert purge should be used as part of the shutdown procedure in order to flush corrosive vapors out of the system prior to shutdown
- *Dilution:* Use a suitable dilution gas to prevent condensation of corrosive vapors and thus prevent the possible resulting corrosion
- *Temperature:* Increase the pump- and exhaust-line temperature to prevent condensation and, therefore, corrosion. Where the low temperature of the incoming gas flow could lead to condensation and subsequent corrosion, an inert gas purge should be used to avoid condensation
- *Corrosion of safety equipment:* Where safety critical equipment (such as flame arrester elements, temperature sensors, safety relief valves and so on) could be damaged by corrosive products in the process gas flow, their materials of construction must be selected to avoid this hazard
- *Phase changes:* Unplanned phase changes can result in condensation and possible corrosion. Consideration of changes in temperature and pressure is required to avoid this hazard
- *Unplanned reactions:* These can lead to the generation of corrosive products. Careful consideration should be given to the possibility of cross contamination when equipment is used for more than one purpose

To minimize hazards from chemical sources and to minimize explosion hazards, attention should be paid to the items listed below to ensure safe operation of vacuum pumps and systems:

- Consider all possible chemical reactions within your system
- Make allowance for abnormal chemical reactions, including those that could occur under fault conditions
- Refer to material safety data sheets when you assess the potential hazards associated with your process materials
- Use dilution techniques to minimize reactions with oxidants and flammable materials
- Use dry vacuum pumps if flammable materials could enter the flammable zone under abnormal conditions
- Use the correct type of lubricant in your pump when you pump oxidants, and consider the use of a dry pump
- Do not use heavy metals in the gas path of your system if your process uses or produces sodium azide
- Take specific care when handling toxic, corrosive, or unstable materials
- Ensure that the concentration of flammable materials in the vacuum system does not permit these materials to enter the flammable zone. During operation, there should be a suitable supply of inert gas (nitrogen) to achieve dilution to a safe level under all foreseeable operating conditions and failure modes
- Ensure that exhaust blockages cannot occur during operation, either because of mechanical components (such as valves or blinds) or because of process materials or byproducts depositing in pipelines, filters and other exhaust components
- Use only PFPE (perfluoropolyether) oils to lubricate pump mechanisms that are exposed to high concentrations of oxygen or other oxidants. Other types of oils sold as "non-flammable" may only be suitable for use with concentrations of oxidants up to 30% v/v
- Ensure that the accidental overpressure of a deliberately closed and isolated vacuum system cannot occur, for instance, as a result of a fault in a pressure regulator or purge control system
- Where the pumped product can react violently with water, it is recommended that a cooling fluid other than water (for example, a heat transfer fluid) be used in the cooling circuit

Physical sources of hazards

Exhaust system over-pressure. The most common physical hazard is an over-pressurized exhaust due to a blockage or restriction in the exhaust system. All high-vacuum pumps are specifically designed to operate with high outlet-to-inlet compression ratios. They are designed to exhaust to atmospheric pressure, or to a pressure only slightly higher. If the exhaust is restricted or blocked, many typical vacuum pumps can generate an exhaust pressure in excess of 7 barg (101.6 psig). This can lead to failure of the pump or other components in the system.

In addition to the potential over-pressure caused by the operation of the pump, the introduction of a compressed gas (such as a purge or dilution gas) can also over-pressure the system if the exhaust system is restricted or blocked.

Where a vacuum pump is fitted with a flame arrester on the exhaust side, it is essential that the exhaust back pressure does not exceed the maximum limit stated in the vacuum system's instruction manual. A regularly scheduled maintenance program should be instituted to ensure that process deposits do not block the exhaust system and flame arrester. In addition, it may be desirable to install a pressure sensor located between the pump and the flame arrester to detect if a blockage is occurring and to sound an alarm.

Phase change. Another cause of overpressure is sublimation or phase change, which can lead to blockage of process piping. The instruction manual supplied with a vacuum pump should be read for information on the maximum and recommended continuous backpressure, and the exhaust system should be designed so that these limitations can be met.

Some recommendations for oil-

sealed rotary vane and piston pumps, dry pumps, and exhaust system design are summarized below:

- For oil-sealed rotary vane and piston pumps, design the exhaust system so that the pump is never subjected to a back-pressure greater than 1 bar gauge (14.5 psig). Under normal operating conditions, the pump should not run continuously with a back-pressure of more than 0.35 bar gauge (5.1 psig)
- Many dry pumps also generate high exhaust pressures when the exhaust system is blocked or restricted. One well-known dry-pump manufacturer recommends that its dry pumps should not be operated with an exhaust pressure higher than 0.3 bar gauge (4.4 psig). If there is a risk of high exhaust pressures occurring, this vendor recommends that the system incorporate an exhaust pressure switch to shut down the pump and prevent the recommended pressure from being exceeded
- It is recommended that the exhaust system be designed to withstand an internal pressure of at least 1.3 bar gauge (18.9 psig) for oil-sealed pumps and at least 10 bar gauge (14.5 psig) for dry pumps, unless over-pressure relief is provided.

If a vacuum pump exhaust system contains accessory components that might cause restrictions or blockages the protection methods shown in the Table (p. 53) should be provided.

Inlet overpressure. The most common cause of over-pressure in vacuum-pump inlet piping is the introduction of compressed gas (such as purge gases) when the pump is not operating. If components in the inlet piping are not suitable for the pressures that result, the piping will rupture and process gases will leak from the system.

Care must be taken before connecting compressed gas supplies to the vacuum system through pressure regulators, which are designed to provide a low-pressure flow so that the regulated pressure is within the pressure rating of the vacuum system. The non-venting regulators most commonly used will cause the pressure within the vacuum system to rise to the pressure of the gas supply to the regulator, if operated under conditions where there is no process gas flow through the system. These two methods can help to prevent over-pressurization:

- Reduce the pressure and allow the gases to bypass the pump into a freely vented exhaust volume
- Monitor the pressure of the vacuum system and use a positive-closure valve to shut off the supply of compressed gas at a preset pressure level

Inlet overpressure can also result from incorrect vacuum pump operation, and special precautions must be taken until it has been established that the pump is operating correctly.

If the direction of rotation of the pump is incorrect and the pump is operated with the inlet blocked or restricted, the pump will generate high pressure in the inlet piping. This could result in rupture of the pump, the piping and components in the piping.

Always use a blanking plate (line blind) loosely secured by screws to the pump inlet until it has been established that the direction of rotation for the pump is correct.

Operation at high rotational speeds could result in pump break-up. Do not operate the pump at rotational speeds above the maximum designed speed of rotation; this is particularly important where frequency inverters are used for speed control.

General design practices

More details about the design practices described below can be found in Ref. *1*.

Pressure ratings in a system. Vacuum systems and piping are designed to operate with internal pressures below atmospheric pressure. In practice, however, it is necessary to design the system (pump and piping) for use with internal pressures above atmospheric pressure as well. It may be necessary to incorporate pressure relief devices to prevent over-pressurization.

Exhaust systems must always be designed to produce the smallest possible backpressure to the pump during operation. It is important, however, to design the exhaust system with an adequate pressure rating; it must be suitable for the pressures that can be generated by the pump and by the introduction into the system of a compressed gas, and be suitable for use with the overpressure-protection measures used.

Where the vacuum system is used to handle a vapor or gas mixture in the flammable range, and where there is a potential ignition source, the pump, piping and associated vessels should have a minimum design pressure of 10 bar gauge (145 psig).

Eliminate stagnant volumes. A stagnant volume is any closed volume in a vacuum pipe or component that is not subjected to a through flow of gas. Examples are the gear box of a mechanical booster pump or the gauge head of an instrument. Valved piping and nitrogen-gas inlet pipes can also become stagnant when they are isolated.

Stagnant volumes must be taken into account when one considers the mixture and reaction of process gases that are not normally present together in the process vessel. A stagnant volume is not purged and may be filled with process gases as the pressure in the system rises and falls. In this way, gases that pass through the system at one stage of the process can be retained. These may then react with gases from a subsequent phase of the process. Thorough evacuation of the process chamber between episodes involving the introduction of incompatible gases will lower the risk of an explosion.

Special care must be taken when considering cross-contamination in stagnant volumes, especially when the pressure is relatively high (close to atmospheric pressure) and the gases are potentially explosive. In particular, one should consider the hazard of buildup in filters and separators. Where appropriate, use high-integrity, continuous flows of inert purge gas to reduce the probability of cross-contamination.

When pumping flammable gases, it is possible for stagnant volumes to fill with potentially explosive gases or vapors that cannot be removed by normal purging. When an ignition source may also be present, specific purging of the stagnant volume should be considered.

Sources of potentially explosive gas or vapor mixtures. When a flammable gas or vapor is mixed with the correct concentration of oxygen or other suitable oxidant, it will form a potentially explosive mixture, which will ignite in the presence of an ignition source.

While it is generally apparent if a pumped gas or vapor is potentially

TABLE. OPTIONS FOR PROTECTING VACUUM PUMP EXHAUST SYSTEMS

Component	Protection method
Valve in exhaust	Interlock the valve so that its piping is always open when the pump is operating
	Incorporate a pressure relief bypass
Exhaust scrubber	Incorporate a pressure relief bypass
	Incorporate a pressure monitor or sensor and interlock this with the pump motor so that the pump is shut down when the exhaust pressure is too high
Flame arrester	Provide an exhaust pressure measurement or a differential-pressure measurement
Oil mist filter	Incorporate a pressure-relief device

explosive, there are some conditions where a potentially explosive mixture is produced due to conditions that were not considered when the process was designed. One must identify all possible sources of potentially explosive mixtures that could be generated by the process vacuum equipment. Examples are listed:

- *Cross-contamination.* Where a vacuum pump is being used for a number of duties, it is possible that its use with individual gases or vapors is safe, but if the pump is not purged before use with another gas or vapor, then cross-contamination could occur with unexpected reactions
- *Cleaning fluids.* An application may be viewed as benign, but the use of flammable cleaning fluids and the subsequent drying by evacuation through the vacuum pump can create a potentially explosive mixture
- *Unexpected material.* On "house vacuum" duties where the vacuum pump is used to provide a distributed vacuum system, it is possible to pump flammable gases or vapors that were not considered during system design. These gases or vapors may have autoignition temperatures that are lower than the internal temperatures of the vacuum pump
- *Dissolved vapors.* Low autoignition-temperature vapors generally have high vapor pressures. Pumping these vapors at pressures above the vapor pressure will generally result in internal dry-pump stage temperatures below the autoignition point. However, it is possible for these vapors to become absorbed in a liquid with a lower vapor pressure, and for them to be released as the pressure falls below the vapor pressure of the dissolved vapors. At this lower pressure, it is possible for the internal dry-pump stage temperature to exceed the autoignition point
- *Air leakage*. The accidental ingress of air or oxidant into a vacuum system may change the concentration of a flammable gas or vapor and create a potentially explosive mixture
- *Flammable sealing liquids*. Where a flammable liquid is used as the sealing liquid in a liquid-ring vacuum pump, air ingress will create a potentially explosive internal mixture

Avoiding the flammable zone. A flammable gas or vapor will only create a potentially explosive atmosphere if it is combined with sufficient air or oxygen (or another oxidant, such as chlorine) and the concentration of the vapor in the mixture lies between the lower flammability limit (LFL) and the upper flammability limit (UFL).

To be potentially explosive, it is also necessary for the concentration of oxygen to be above the limiting oxidant concentration (LOC). There are several strategies that can be used to avoid operating with gas mixtures in the flammable region. The choice of strategy will depend on the outcome of the hazard analysis for the process and the pumping system. Recommended strategies include the following:

- *Maintain the flammable gas concentration below the LFL.* This is accomplished by diluting the flammable mixture with an inert gas (usually nitrogen) that is introduced into the pump inlet and/or purge connections. Refer to NFPA 69 [2] for recommendations as to what percentage of the LFL the flammable concentration should be maintained. The required integrity of the dilution system and of any alarms or interlocks will depend on the nature of the hazards that would result if the dilution system were to fail
- *Maintain the oxidant concentration below the LOC.* This mode of operation requires the use of oxygen-concentration monitoring of the pumped gases to ensure safe operation. To minimize the risk of flammable gas accidentally entering the flammable zone, a safety margin below the LOC should be maintained. NFPA 69 [2] presents recommendations for these safety margins. The preferred method of maintaining the oxidant (usually oxygen) level below the LOC is to carry out both rigorous exclusion of air and oxygen from the process and pump system, and dilution of the pumped gas with an inert purge gas (usually nitrogen) that is introduced into the pump inlet or purge connections, if needed. The required integrity of the air/oxygen-exclusion measures (for example, purging) and of any alarms or interlocks will depend on the hazardous zone that would result, were the exclusion and dilution systems to fail

Precautions typically required to rigorously exclude air from the process and pump system include the following:

- *Eliminate air leaks.* Use a leak detector or conduct a pressure-rise test
- *Remove all air from the system before the start of the process.* Before any flammable gas is admitted into the process, the system should be fully evacuated and purged with an inert gas (usually nitrogen) to remove all air from the system. At the end of the process, repeat this procedure before the system is finally vented to the atmosphere
- *Maintain the integrity of shaft-seal gas.* For dry-vacuum pumps, ensure that any shaft-seal gas cannot be supplied with, or contaminated by, air under any circumstances (for dry-vacuum pumps). In addition, ensure that any gas ballast port is either sealed, or only used to introduce inert gas
- *Follow instructions and maintain proper lubrication.* For wet vacuum pumps (rotary piston or rotary vane pumps), maintain the shaft seals in accordance with the manufacturer's instructions, and use a pumped, pressurized oil-lubrication system with an alarm that indicates the loss of oil pressure. This system may comprise an external accessory to provide filtered and pressurized lubricating oil, with a pressure switch. Ensure that any gas ballast port is either sealed, or only used to introduce inert gas. Provide an adequate purge of inert gas to the oil box, to remove air before the start of the process
- *Maintain the drive-shaft seal for vacuum booster pumps (such as the Roots pump).* Maintain the primary drive shaft seal fully in accordance with the manufacturer's instruc-

tions, and ensure that any purge or breather port connections can only be used to introduce inert gas

- *Guard against reverse flow.* Ensure that the system operating procedures and protective equpment protect the system from any reverse air flow that might result from a pump failure. Ensure that any pumped flammable gases are safely disposed of at the final vent from the pump exhaust. Ensure that flammable gas mixtures cannot occur in the exhaust piping. This is done by using suitable inert-gas purging of the piping before the start of, and after the end of, the flammable gas process, and by the use of adequate inert-gas purging during operation, to prevent turbulent back-mixing of air down the exhaust
- *Maintain the flammable gas concentration above the UFL.* Where the flammable gas concentrations are very high, operation above the UFL can be used. Procedures for doing this are given in NFPA 69 [2]. To maintain the oxygen at a safe level, rigorous exclusion of air and oxygen from the process and pump system is required. This can be accomplished by dilution of the pumped gas with an inert gas (usually nitrogen) or by addition of enrichment flammable gas (NFPA 69 discusses the use of methane for this), introduced into the pump inlet and/or purge connections

Use of flame-arrester protection systems. When dry vacuum pumps are used for pumping gases or vapors that could enter the flammable zone under abnormal conditions, exhaust flame arresters should be installed to quench any resulting flame front in the event of an ignition. Flame arresters should be closely coupled to the exhaust connections.

Where there is the possibility of continuous burning within a dry vacuum pump, special precautions should be taken. Such conditions could occur due to a continuous ignition source resulting from the accidental pumping of a gas or vapor with an auto-ignition temperature lower than the internal pump temperatures. In this case, one must use a PT100 or equivalent temperature-sensing device on the pump side of each flame arrester to detect a burn condition, and adopt a suitable flame-suppression strategy to make the system safe in the event of a burn being detected. The suitable strategy depends on the application, but could include the following:

- *Stopping the supply of fuel.* Closing a valve located on the inlet of the dry vacuum pump will prevent the supply of fuel (process gas or vapor) into the vacuum pump
- *Stopping the source of ignition.* Stopping the pump by turning off power to the motor will bring the pump to a halt in approximately 10 seconds
- *Inerting the area of the burn*. The rapid addition of inert gas into the area of the burn (typically, but not always located in the exhaust manifold of the pump) will eliminate the flame. Note that it is possible for a flame to reignite if the source of ignition is not removed

Piping design

This section discusses design practices for piping components used with mechanical vacuum pumps.

Bellows. Bellows are short, thin-walled components with deep convolutions. They are used to reduce the transfer of vibration from a pump to a vacuum system. They are available in metal, rubber or plastic construction.

Always install bellows in a straight line with both ends rigidly constrained. When installed correctly, the bellows can withstand a small positive internal pressure (refer to the Instruction Manual supplied with the bellows for details). Note: Do not use bellows on dry vacuum-pumps' exhausts: use braided flexible bellows (see below).

Braided flexible bellows. Braided flexible bellows are bellows with an outer protective layer of woven stainless-steel braid. They are suitable for use as exhaust connections on dry pumps and other applications where there is significant gas pulsation or the possibility of high gas pressures.

Braided flexible bellows are intended for installation in static systems. They are not suitable for repeated flexing, as this could cause fatigue failure. When a braided flexible bellows is installed, one must comply with the minimum bend radius given in the manufacturers's instruction manual.

Flexible hoses. Flexible hoses have a thicker wall section and shallower convolutions than bellows. Flexible hoses provide a convenient method for the connection of vacuum system components and help to compensate for misalignment or small movements in rigid vacuum piping. Flexible hoses can be formed into relatively sharp bends that will hold their position.

Flexible hoses are intended for installation in static systems. They are not suitable for repeated flexing, as this could cause fatigue failure. When flexible hoses are used, use the shortest possible length and avoid unnecessary bends. Do not use flexible hoses on dry pump exhausts.

Anchor points. Piping and piping components must be anchored correctly. For example, if bellows are anchored incorrectly, they will not reduce the vibration generated by the pump, and this could lead to fatigue in the piping.

Seals. Where there is the possibility of positive pressure occurring in any part of the vacuum system (even under failure conditions), one must use trapped O-ring type seals, which are capable of withstanding the expected vacuum and positive pressures.

Physical over-pressure protection

Over-pressure can be caused by a restriction or blockage in the vacuum system or in one of its components. The over-pressure may also occur as a result of compressed-gas flow from the pump or from external compressed gas supplies (such as those for a dilution system). There are two main methods of over-pressure protection, as follows:

Pressure relief. Rupture disks or pressure-relief valves may be used to relieve an overpressure situation. These devices must be provided with discharge piping to convey the relief device discharge fluid to a safe location (where plant personnel are not usually located), and the vent line should have no restrictions. If the process produces solid byproducts, the pressure-relief devices must be inspected on a regularly scheduled basis to ensure that they are not blocked or restricted.

Over-pressure alarm or trip. This method of protection is used by several vacuum-pump system manufacturers.

This type of protection can be used for any vacuum-pump system, but it is particularly suitable for systems that produce solid byproducts.

Since the use of an alarm or trip system forms part of a primary safety system, it must incorporate a design of high integrity. One should not rely on a simple pressure switch or sensor installed in a flow line, as the condensation of solid particles or corrosion of the sensor can cause the device to fail without indication of the failure.

It is also recommended that a sensor be used that incorporates a nitrogen purge flow to the sensor. A purge flow keeps the sensor relatively free from blockage, and such a sensor is designed so that, if the sensor is blocked, the nitrogen supply pressure is high enough to operate the pressure alarm and trip. The normal settings for such devices are usually no higher than 0.3 bar gauge (4.35 psig) and no greater than 0.4 bar gauge (5.80 psig).

The control system should be designed so that both the pump and any gas inlets (which are capable of pressurizing the system above its maximum operating pressure) are shut down when the trip point is reached.

Purge systems. The correct use of a purge (usually nitrogen) can ensure that corrosive products are removed, preventing them from damaging the vacuum pump, and also from plugging or damaging protective systems such as flame arresters. In addition, the removal of process gases ensures that undesired and potentially dangerous chemical reactions do not occur between process gases and vapors used in different process cycles. ■

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1. Edwards Ltd., Safety Manual-Vacuum Pump and Vacuum (corporate manual).
2. NFPA 69, Standard on Explosion Prevention Systems, National Fire Protection Association, Quincy, Mass., 2008.

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Author

Stanley S. Grossel is president of Process Safety and Design, Inc. (4 Marble Court, Unit 9, Clifton, N.J. 07013-2212; Phone: 973-779-3668; Email: psadi28@aol.com). He provides consulting services related to process safety and loss prevention, storage and handling of bulk solids and powders, air pollution control, and process design of batch plants. Prior to his 1993 retirement, Grossel spent 25 years in the Engineering Dept. of Hoffman-La-Roche, in a variety of roles. He was honored as the only Senior Engineering Fellow ever to be designated by Hoffman-LaRoche. Grossel is a fellow of AIChE, and holds AIChE's Norton Walton/Russell Miller Award for outstanding contributions to loss prevention. He has lectured and been published widely, and has authored major standards and guidelines. Grossel is also a contributor on process safety to the 7th Ed. of "Perry's Chemical Engineers' Handbook." He holds a B.S.Ch.E. from City College of New York and an M.S.Ch.E. from Drexel Inst. of Technology, and has completed courses toward a doctoral degree.

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

According to my version of the story, Robert Kohler said, "We don't need visas to enter Hungary." According to Robert's version, he said, "It's easy to get visas to enter Hungary." Either way, he was wrong. Either way, he got us to Budapest.

Robert Kohler and I visited the OMV plant in Schwechat, Austria, on a Friday morning. At Robert's urging, we decided to go to Budapest for the weekend, without visas. We took a Vienna-to-Budapest train that stopped at the Hungarian border. The immigration agent was unimpressed by our visa-less American passports. He ordered us off the train — and our luggage too.

We found ourselves in a very small border station that some might call a cottage. A tall black-haired taxi driver with a brown leather beret approached us hurriedly and requested $20 each for emergency visa service. We had no other options that we knew about. Robert and I agreed to his price. The taxi driver ran to his cab. Robert and I power-walked behind him, lugging our luggage behind us. Before the car doors were closed, the taxi was speeding through a recently harvested corn field. After a 15-min ride, most of my teeth were loose. When the cab exited the cornfield, we were faced by a highway border station with buildings that looked governmental. The taxi driver stated, in reasonably good English, "Leave your bags in the cab. We. Must. Hurry."

Inside the building, we encountered a long line. Our taxi driver took us to the front of the line, by shouting something in Hungarian that did not completely satisfy everybody that we were cutting in front of. Nevertheless, we left the building with photographs and stamps in our passports.

We jumped back into the taxi and sped toward an automobile border crossing that had three closed lanes and three open lanes. Each open lane had 20 cars in each. Our driver took us to one of the closed lanes. Unfortunately, his friends weren't working the gates on that particular day. Our taxi driver slowed but did not stop. Two very young Hungarian soldiers were very unimpressed by the explanation that our driver shouted as we drove past them. They un-slung their weapons. They looked at each other, wondering whether to shoot or not. Robert and I looked at each other, wondering whether to slouch or not. They did not. We did. Within two minutes we were back at the train station and on the very same train that we were thrown off of, and headed toward Budapest.

Some Americans feel that, when they travel abroad, their American passports and American dollars will pave their way to and from anywhere and will shield them from all troubles. Whether at the border to Hungary, or in a market square in Mongolia, the U.S. State Department does not really know you. You're on your own. Be careful. ■

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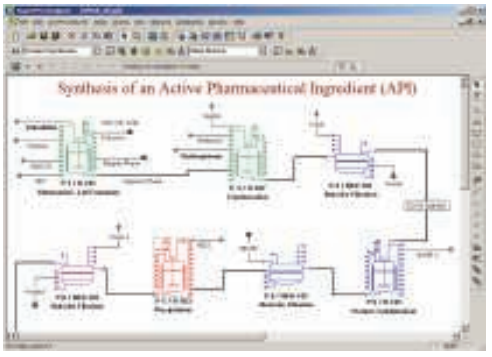

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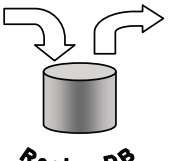

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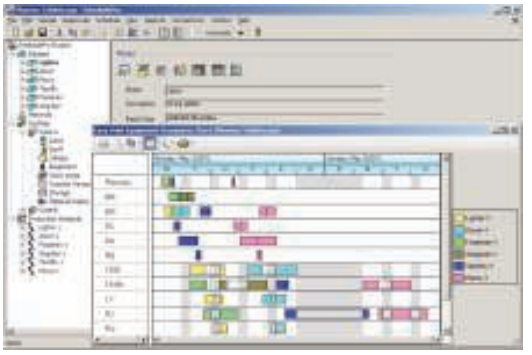

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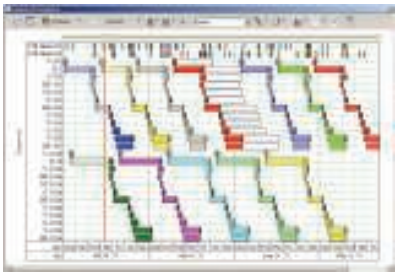

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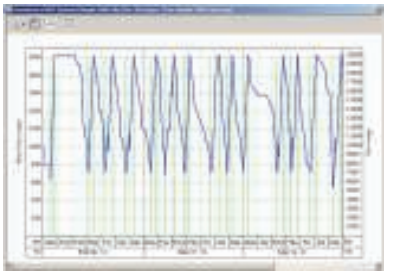

Managing inventories for input, intermediate, and output materials

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FREE PRODUCT INFO

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03 Inorganic Chemicals
04 Plastics, Synthetic Resins
05 Drugs & Cosmetics
06 Soaps & Detergents
07 Paints & Allied Products
08 Organic Chemicals
09 Agricultural Chemicals
10 Petroleum Refining, Coal Products
11 Rubber & Misc. Plastics
12 Stone, Clay, Glass, Ceramics
13 Metallurgical & Metal Products
14 Engineering, Design & Construction Firms
15 Engineering/Environmental Services
16 Equipment Manufacturer
17 Energy incl. Co-generation
18 Other_____

JOB FUNCTION

20 Corporate Management
21 Plant Operations incl. Maintenance
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

1	16	31	46	61	76	91	106	121	136	151	166	181	196	211	226	241	256	271	286	301	316	331	346	361	376	391	406	421	436	451	466	481	496	511	526	541	556	571	586
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12	27	42	57	72	87	102	117	132	147	162	177	192	207	222	237	252	267	282	297	312	327	342	357	372	387	402	417	432	447	462	477	492	507	522	537	552	567	582	597
13	28	43	58	73	88	103	118	133	148	163	178	193	208	223	238	253	268	283	298	313	328	343	358	373	388	403	418	433	448	463	478	493	508	523	538	553	568	583	598
14	29	44	59	74	89	104	119	134	149	164	179	194	209	224	239	254	269	284	299	314	329	344	359	374	389	404	419	434	449	464	479	494	509	524	539	554	569	584	599
15	30	45	60	75	90	105	120	135	150	165	180	195	210	225	240	255	270	285	300	315	330	345	360	375	390	405	420	435	450	465	480	495	510	525	540	555	570	585	600

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Jason Bullock,
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Chemical Engineering
8325 Broadway, Ste. 202/PMB 261
Pearland, TX 77581
Tel: 281-485-4077; Fax: 281-485-1285
E-mail: jbullock@che.com;
Arkansas, Arizona, California, Colorado, Georgia, Kansas, Louisiana, Missouri, New Mexico, Nevada, Oklahoma, Texas, Washington

Dan Gentile
District Sales Manager
Tel: 512-918-8075
E-mail: dgentile@che.com
Indiana, Illinois, Iowa, Minnesota, Wisconsin

Diane Hammes
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Chemical Engineering;
11000 Richmond Ave, Suite 690, Houston, TX 77042
Tel: 713-444-9939
E-mail: dhammes@che.com
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International

Petra Trautes
Chemical Engineering
Zeilweg 44
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Germany
Phone: +49-69-58604760
Fax: +49-69-5700-2484
Email: ptrautes@che.com
Austria, Czech Republic, Benelux, Eastern Europe, Germany, Scandinavia, Switzerland, United Kingdom

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

Katshuhiro Ishii
Chemical Engineering
Ace Media Service Inc., 12-6, 4-chome
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E-mail: amskatsu@dream.com
Japan

Ferruccio Silvera
Chemical Engineering
Silvera Pubblicita
Viale Monza, 24 Milano 20127, Italy
Tel: 39-02-284-6716;
Fax: 39-02-289-3849
E-mail: ferruccio@silvera.it/www.silvera.it
Andorra, France, Gibraltar, Greece, Israel, Italy, Portugal, Spain

Rudy Teng
Sales Represntative
Chemical Engineering;
Room 1102 #20 Aly 199 Baiyang Road
Pudong Shanghai 201204
China
Tel: +86 21 50592439
Fax: +86 21 50592442
MP: +86 13818181202
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*** International Edition**

See bottom of next page for advertising sales representatives' contact information

Classified Index October 2012

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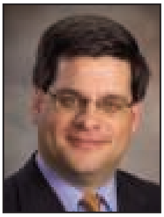
E-mail: dhammes@che.com Tel: 713-343-1885

OCTOBER WHO'S WHO


Thill


Peeters


Rosenberger


Pace


Lind

SNC-Lavalin (Montreal, Quebec), a global engineering and construction group that specializes in infrastructure and operations and maintenance services, names *Robert Card* president and CEO.

Tony Thill becomes president of **CST International** (Lexana, Kan.), a global manufacturer of factory-coated metal storage tanks, aluminum domes and related products.

Blachoh Fluid Control (Riverside, Calif.), a maker of industrial fluid control products, names *Bill Bendel* technical sales manager and *Terri Simmons* global customer service manager.

Dow Corning Electronics Solutions (Midland, Mich.) promotes *Eric Peeters* to vice president.

The Hallstar Co. (Chicago, Ill.), a producer of specialty ingredients for use in industrial and personal care applications, promotes *David Rosenberger* to vice president of sales and *Louis Pace* to chief operating officer.

Conrad Winters becomes director of drug product development for **Hovione** (Loures, Portugal), a global producer of active pharmaceutical ingredients and drug product intermediates.

Abresist Kalenborn Corp. (Urbana, Ind.), a manufacturer of impact- and wear-resistant linings, appoints *Jennifer Lind* as a territory manager for Mississippi, Alabama, western Tennessee, eastern Arkansas and the panhandle of Florida. ■

Suzanne Shelley

BUSINESS NEWS

PLANT WATCH

Solvay increases fluorinated-derivatives production capacity in France
September 6, 2012 — Solvay S.A. (Brussels, Belgium; www.solvay.com) is investing over €10 million to double the capacity of the fluorinated aliphatic-derivatives production line at its plant in Salindres, France. The project's first stage is currently underway. The Group decided to double production capacity for triflic acid and triflic anhydride, as well as for lithium bis(trifluoromethanesulfonyl)imide to meet the rapidly expanding requirements of electronic applications and the battery and pharmaceuticals markets.

BASF to build production plant for specialty zeolites in Ludwigshafen
September 6, 2012 — BASF SE (Ludwigshafen, Germany; www.basf.com) is investing in the construction of a production plant for specialty zeolites at its Ludwigshafen, Germany site. The plant is expected to start up operations in the 1st Q of 2014. BASF currently produces specialty zeolites at its operating site in Seneca, S.C., where production capacity is also being expanded.

NatureWorks broadens Ingeo product portfolio with Sulzer equipment
September 6, 2012 — Sulzer Chemtech Ltd. (Winterthur, Switzerland; www.sulzerchemtech.com) is supplying proprietary production equipment to the NatureWorks LLC (Minnetonka, Minn.; www.natureworksllc.com) Blair, Nebr., facility to enable NatureWorks to increase production of Ingeo biopolymer and produce high-performance resins and lactides. Ingeo production capacity at Blair will rise from 140,000 to 150,000 metric tons per year (m.t./yr). Commissioning of the new equipment is expected in the 1st Q of 2013 with capacity increases and new products becoming available in the 2nd Q.

Shell to construct the world's first oil-sands carbon capture and storage project
September 5, 2012 — Shell (The Hague, the Netherlands; www.shell.com) plans to go ahead with the first carbon capture and storage (CCS) project for an oil sands operation in Canada. The so-called Quest project will be built on behalf of the Athabasca Oil Sands Project joint venture owners (Shell, Chevron and Marathon Oil). From late 2015, Quest will capture and store, deep underground, more than 1-million m.t./yr of CO_2 produced in bitumen processing. The Alberta government will invest $745 million in Quest from a $2-billion fund to support CCS, while the Government of Canada will invest $120 million through its Clean Energy Fund. Quest is said to be the world's first commercial-scale CCS project to tackle carbon emissions in the oil sands. Shell has received the necessary federal and provincial regulatory approvals for Quest.

Lanxess to build the world's largest EPDM plant in China
September 4, 2012 — Lanxess AG (Leverkusen, Germany; www.lanxess.com) is building what is said to be the world's largest plant for ethylene propylene diene monomer (EPDM) synthetic rubber in China. The company is investing €235 million in the plant in Changzhou, Jiangsu Province. The plant will have a capacity of 160,000 m.t./yr and is expected to start up in 2015. All necessary permits have been obtained from the local authorities.

Technology from Veolia Water Solutions is selected for potash and salt recovery
August 30, 2012 — Veolia Water Solutions & Technologies (Saint Maurice, France; www.veoliawaterst.com) has been awarded the supply of a large-scale evaporation and crystallization plant by Iberpotash, S.A., a business unit of ICL Fertilizers. Iberpotash produces potash fertilizers from sylvinite mining from deposits located near Suria, Spain. The plant, to be completed in 2014, will recover and purify high-quality sodium chloride and potassium chloride from byproduct salts resulting from the production of fertilizer. Utilizing technology from Veolia, Iberpotash will expand production capabilities to include 750,000 tons/yr of chemical and food-grade salts, as well as recover up to 50,000 tons/yr of white potash from the process.

Linde to build Vietnam's largest air separation unit
August 29, 2012 — The Linde Group (Munich, Germany; www.linde.com) has been awarded a long-term contract to supply Vietnamese steel-company Posco SS-Vina (PSSV) with industrial gases. The deal will see Linde construct the country's largest air separation unit (ASU) at the Phu My industrial park in Ba Ria. The Group will be investing a total of around €40 million in the project. The ASU will be able to produce 35,000 Nm^3/h of gases, and is set to go onstream in 2014.

Lummus propane-dehydrogenation technology selected for unit in China
August 28, 2012 — CB&I (The Woodlands, Tex.; www.cbi.com) has been awarded a contract by the Hebei Haiwei Group for the license and basic engineering design of a grassroots propane dehydrogenation unit to be located in Jingxian, Hebei Province, China. The unit will use the Catofin propane dehydrogenation process from Lummus Technology that employs Süd-Chemie's latest Catofin catalyst to produce 500,000 m.t./yr of propylene. The unit is expected to start up in 2015.

Foster Wheeler awarded contract for gas-to-chemicals complex in Brazil
August 23, 2012 — Foster Wheeler AG (Zug, Switzerland; www.fwc.com) says that a subsidiary of its Global Engineering and Construction Group has been awarded a contract by Petróleo Brasileiro S.A. (Petrobras) for a gas-to-chemicals complex in Linhares, Espirito Santo State, Brazil. Foster Wheeler will provide basic and front-end engineering design, as well as additional assistance. The design phase is scheduled for completion at the end of 2013. The complex is expected to produce more than 1-million m.t./yr of ammonia and urea fertilizers, methanol, acetic acid, plus formic acid and melamine.

MERGERS AND ACQUISITIONS

BP agrees to sell Carson refinery and Arco Retail Network to Tesoro
August 13, 2012 — BP (London, U.K.; www.bp.com) has reached an agreement to sell its Carson, Calif. petroleum refinery and related logistics and marketing assets in the region to Tesoro Corp. for $2.5 billion in cash (including the estimated value of hydrocarbon inventories and subject to post-closing adjustments) as part of a previously announced plan to reshape BP's U.S. fuels business. Subject to regulatory and other approvals, Tesoro will acquire the 266,000-bbl/d refinery near Los Angeles as well as the associated logistics network of pipelines and storage terminals and the Arco-branded retail marketing network in Southern California, Arizona and Nevada. The sale also includes BP's interests in associated cogeneration and coke calcining operations. The closing is expected to happen before mid-2013. This is part of the previously announced program to divest $38 billion of assets by the end of 2013. ■

Dorothy Lozowski

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October 2012; VOL. 119; NO. 10

FOR MORE ECONOMIC INDICATORS, SEE NEXT PAGE

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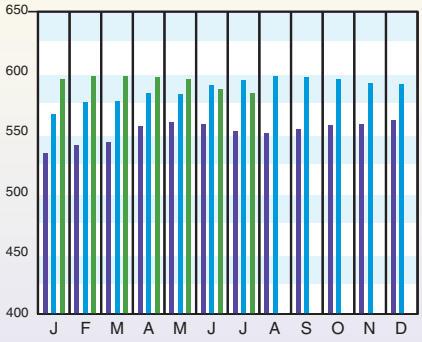
CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	July '12 Prelim.	June '12 Final	July '11 Final
***CE* Index**	582.2	585.6	593.2
Equipment	708.5	713.9	724.1
Heat exchangers & tanks	652.0	661.4	681.8
Process machinery	664.7	666.5	675.8
Pipe, valves & fittings	911.3	917.7	915.3
Process instruments	424.2	425.1	446.9
Pumps & compressors	928.9	927.0	909.5
Electrical equipment	509.7	513.7	512.5
Structural supports & misc	757.8	759.9	764.7
Construction labor	323.9	322.6	326.9
Buildings	525.6	527.1	520.5
Engineering & supervision	328.0	327.9	332.1

Annual Index:

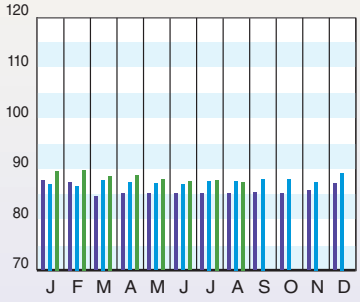
2004 = 444.2
2005 = 468.2
2006 = 499.6
2007 = 525.4
2008 = 575.4
2009 = 521.9
2010 = 550.8
2011 = 585.7



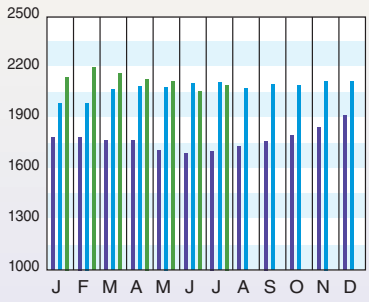
CURRENT BUSINESS INDICATORS

	LATEST		PREVIOUS		YEAR AGO	
CPI output index (2007 = 100)	Aug.'12 =	87.4	Jul.'12 =	87.8	Jun.'12 =	87.5	Aug.'11 =	87.6
CPI value of output, $ billions	Jul.'12 =	2,097.5	Jun.'12 =	2,059.0	May.'12 =	2,116.1	Jul.'11 =	2,113.3
CPI operating rate, %	Aug.'12 =	75.4	Jul.'12 =	75.7	Jun.'12 =	75.5	Aug.'11 =	75.6
Producer prices, industrial chemicals (1982 = 100)	Aug.'12 =	292.9	Jul.'12 =	295.4	Jun.'12 =	312.7	Aug.'11 =	337.0
Industrial Production in Manufacturing (2007=100)	Aug.'12 =	94.1	Jul.'12 =	94.8	Jun.'12 =	94.5	Aug.'11 =	90.7
Hourly earnings index, chemical & allied products (1992 = 100)	Aug.'12 =	156.9	Jul.'12 =	158.9	Jun.'12 =	157.2	Aug.'11 =	156.6
Productivity index, chemicals & allied products (1992 = 100)	Aug.'12 =	102.5	Jul.'12 =	103.2	Jun.'12 =	103.7	Aug.'11 =	107.2

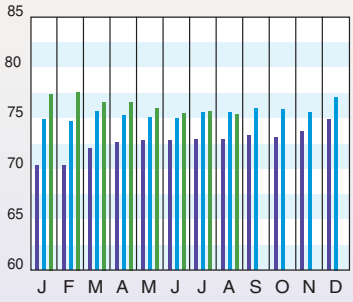
CPI OUTPUT INDEX (2007 = 100)



CPI OUTPUT VALUE ($ BILLIONS)



CPI OPERATING RATE (%)



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

CURRENT TRENDS

Capital equipment prices, as reflected in the *CE* Plant Cost Index (CEPCI; top), dropped 1.9% from June to July (the most recent data). Meanwhile, the Current Business Indicators from IHS Global Insight (middle), including the operating rate, dropped slightly from July to August.

According to the American Chemistry Council's (ACC; Washington, D.C.; www.americanchemistry.com) latest weekly economic report at *CE* press time, production by the U.S. chemical industry fell by 0.3% in August. The drop left production at 85.7% of its average level from 2007, ACC noted. The lower production in August follows a similar decline in July and flat growth in June. The data indicate that production of organic and inorganic basic chemicals was up slightly, as was that of synthetic rubber, but those gains were offset by declines in plastic resins and artificial fibers, according to the ACC report.

Meanwhile, wholesale sales of chemicals rose by 0.5% in July, to $10.2 billion. This follows a 1.3% gain in June, ACC noted. Chemical inventories were also up. Compared to a year ago, chemical sales at the wholesale level were up 4.8%, outpacing the 3.0% rise in inventories, ACC said.

For more on capital cost trends and methodology, visit: www.che.com/pci ■

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