CHEMICAL December 2010 CHEMICAL December 2010



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Facts at Your Fingertips: Project Design Decisions

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> CPI Job Market

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> Pilot Plant versus Simulation

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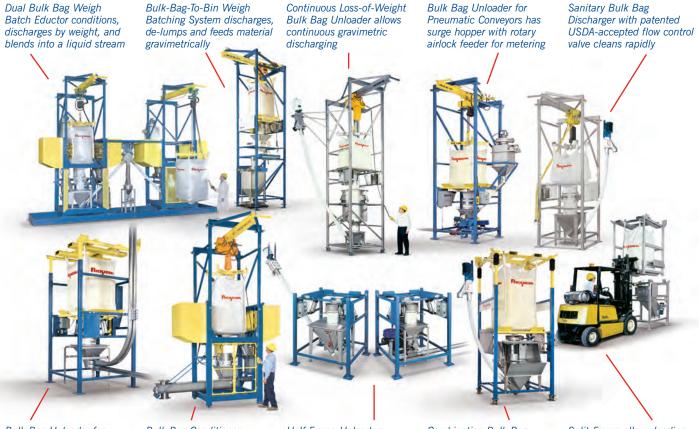


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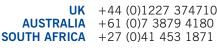
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plant? The author cautions too heavy a reliance on computer simulations in development projects, and salutes pilot plant engineers who corroborate modeling work in the tangible world

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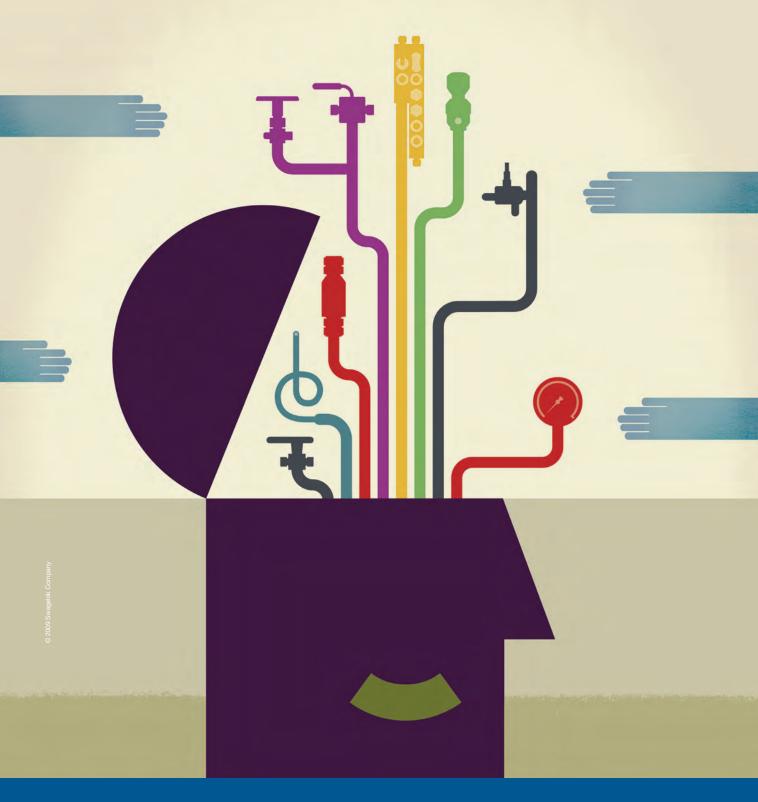
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Editor's Page

Blurring borders and the future

s the lines of demarcation separating scientific disciplines continue to blur, increasing clarity arises for the recognition that the scientific borderlands represent the fertile ground for future advancements. A recent illustration of that concept emerged at a symposium last month in New York, entitled "Research Frontiers in the Chemical Sciences." Sponsored by the Camille and Henry Dreyfus Foundation, the symposium brought together recipients of the Dreyfus Foundation's Teacher-Scholar awards in the chemical sciences for a networking event.

The impressive collection of young investigators at the symposium presented posters on topics such as nanomaterials synthesis using microplasma processes, microrheology of fluid interfaces, nanostructures as heterogeneous catalysts, control of chemistry on nanoscale-pattered surfaces and many more. While the projects presented at the symposium were diverse enough to defy attempts to identify themes, topics reflect an emphasis on the chemistry of biological systems, and a focus on the behavior of nanoscale materials. The inventive research projects reinforce the idea that modern research is increasingly and irreversibly interdisciplinary.

The scientists at the symposium will shape the future of chemistry and chemical engineering not only through the impact of their accomplishments on forthcoming scientific and technological advancements, but also by serving as mentors for young scientists, many of whom may join industrial R&D teams to seek solutions to both current challenges and those on the horizon.

One young chemical-engineering researcher I spoke with said the event was a positive experience because of the exposure he received to trends and thinking in areas outside his own research area. This idea of crosspollination of scientific ideas is in keeping with the multidisciplinary nature of cutting-edge chemical engineering and chemical R&D, but it also has particular relevance for those who will be working in the chemical process industries (CPI) for the coming decades. On page 15 of this issue, you will find a story about the current employment landscape for chemical engineers and other CPI professionals. Among the lessons that came through in preparing the article is that overcoming the challenges of the present will require contributions from those who can utilize insights and approaches from one industrial sector to tackle challenges in a different area or industry.

Doing so successfully necessitates the same kind of approach as that needed for carrying out category-defying research. Working well as part of a team, effective communication and an adaptive spirit are all predictors of success in many CPI jobs of the future, whether in industry or academia. A dynamic and fruitful future awaits chemical engineers who are tuned to the places where their specialties intersect with other disciplines.

Although the symposium had a decidedly academic feel, the links between research and industry seem to be on the minds of those steering the Drevfus Foundation. This mindset was made manifest by the topic

selected for another Dreyfus program: the biennial Dreyfus Prize in the Chemical Sciences. The second prize (Harvard University materials scientist George Whitesides won the inaugural Dreyfus prize in 2009) will be awarded in April 2011 to an individual who has advanced the field of catalysis in a significant way. It's a good bet that the accomplishments of the winner, whether working in industry or academia, were benefited by interdisciplinary thinking.

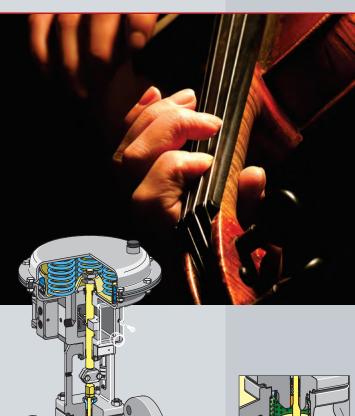


Scott Jenkins, associate editor

Director of Production & Manufacturing solson@accessintel.com

Not Mozart, Yet a **Classical Genius**





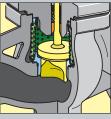
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Letters

The loss of a ChE icon

Longtime readers of this magazine will be saddened to learn that on October 11, Dr. James Fair passed away at the age of 90. At the time of his death, Dr. Fair held an appointment as the McKetta Centennial Energy Chair Emeritus in the chemical engineering department at The University of Texas (Austin,



James R. Fair October 14, 1920 -October 11, 2010

Tex.; www.utexas.edu), where he founded the long-lived Separations Research Program and served as its head from 1982 to 1996. After a distinguished career in industry, primarily with Monsanto Co. where I knew him, he moved to the University in July 1979 where he was awarded the first endowed chair in the college of engineering.

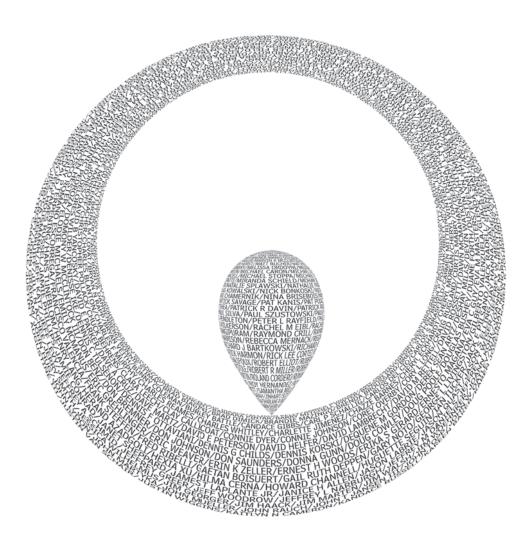
His industrial background included significant assignments in research, process design, manufacturing technical services and commercial development. During World War II, he was vitally involved with the government high-explosives and synthetic-rubber programs. His final position at Monsanto was director of corporate technology. In addition, he served as affiliate professor of chemical engineering at Washington University (St. Louis, Mo.) from 1964 to 1977.

Dr. Fair was a former director of the American Institute of Chemical Engineers (AIChE), a former president of James R. Fair, Inc., a former vice president of Fractionation Research, Inc., and was a registered professional engineer in Texas and Missouri. His many honors include Chemical Engineering's Personal Achievement Award; the Separations Science and Technology Award of the American Chemical Society; the Distinguished Engineering Graduate Award from The University of Texas at Austin; numerous AIChE awards and election to Fellow Grade of membership in that institution; and election to the National Academy of Engineering.

Dr. Fair authored or co-authored seven books and over 200 technical articles and book chapters. He supervised the master's and doctoral research of 42 graduate students. He lectured internationally on many occasions, and for 25 years taught a continuing education course under the sponsorship of AIChE, the course being the most popular of that organization and offered over 125 times. He held a B.S.Ch.E., M.S.Ch.E. and a Ph.D. in chemical engineering from Georgia Institute of Technology and the Universities of Michigan and Texas. Washington University and Clemson University awarded him honorary doctorates.

Jim Fair was a man of many dimensions, including tall height, long life and a wide smile. Throughout his long life he continually influenced those who worked with him as well as the lives of his many students. He had no peer. Jim, among his many personal attributes, also had a great sense of humor. For those of us who knew the man, he will remain in our hearts and dwell in our minds for our life time, but his work will live far into the future.

Bill Huitt and Jim's many friends and colleagues at Monsanto Co., St. Louis, Mo.



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Calendar

NORTH AMERICA

International Conference on Biomolecular Engineering. Society for Biological Engineering (New York). Phone: 1-646-495-1382; Web: www.aiche.org/sbe/events/ ICBE.aspx San Francisco, Calif. Jan. 16-19

Coatings Summit 2011. Vincentz Network (Hannover, Germany). Phone: +49-511-99-10-270; Web: european-coatings.com Washington, D.C. Jan. 17-19

2nd International Congress on Sustainability Science & Engineering. University of Arizona/College of Engineering (Tucson, Ariz.). Phone: 1-520-621-6596; Web: icosse11.org Tucson, Ariz. Jan. 9-13

35th International Conference & Exposition on Advanced Ceramics & Composites. The Ameri-

can Ceramic Society (Westerville, Ohio). Phone: 1-866-721-3322; Web: ceramics.org/icacc-11 Daytona Beach, Fla. Jan. 23-28

2011 ACI Annual Meeting and Industry

Convention. American Cleaning Institute (formerly the Soaps & Detergents Assn.; Washington, D.C.). Phone: 1-202-347-2900; Web: cleaninginstitute.org/about/ acimeetings.aspx Orlando, Fla.

Jan. 25-29

Lab Automation 2011. Assn. for Lab Automation (Geneva, Ill.). Phone: 1-888-733-1252; Web: labautomation.org Palm Springs, Calif. Jan. 29 - Feb. 2

Informex 2011. UBM International Media (Princeton, N.J.). Phone: 1-609-759-4700; Web: informex.com Charlotte, N.C. Feb. 7-10

AAAS Annual Meeting. American Assn. for the Advancement of Science (Washington, D.C.). Phone: 1-202-326-6400; Web: aaas.org Washington, D.C.

Feb. 7-10

TMS 2011: 140th Annual Meeting and Exhibition. The Minerals, Metals & Materials Soc. (Warrendale, Pa.). Phone: 1-724-776-9000; Web: tms.org San Diego, Calif. Feb. 27 - Mar. 3

Pharma & Biotech Licensing & Partnering. International Inst. for Business Information (Port Washington, N.Y.). Phone: 1-212-300-2520; Web: iibig.com/P1101 San Diego, Calif. Mar. 3-4

21st International Conference: Molding 2011: **Technologies for Business Success**. Executive Conference Management (Plymouth, Mich.). Phone: 1-734-737-0507; Web: executive-conference.org San Diego, Calif.

Mar. 27-31

5th Annual Energy Efficiency Finance Forum.

American Council for an Energy-Efficient Economy (Washington, D.C.); Phone: 1-704-341-2376; Web: aceee.org/conferences/2011/eeff Philadelphia, Pa.

Mav 3-4

SOUTH AMERICA

Expocomer 2011 Trade Fair. Chamber of Commerce, Industries and Agriculture of Panama (Panama City, Panama) and Messe Düsseldorf North America (Chicago); Phone: 1-312-781-5180; Web: mdna.com Mar. 23-26 Panama City, Panama

XIX International Solvent Extraction Confer-

ence. Gecamin Ltda. (Santiago, Chile). Phone: + 56 (2) 652-1575; Web: isec2011.com Santiago, Chile Oct. 3-7

EUROPE

10th International Electronics Recycling

Congress IERC 2011. ICM AG (Birrwil, Switzerland). Phone: +41 62 785 10 00; Web: icm.ch Salzburg, Austria Jan. 19-21

World CTL 2011. World CTL (Paris). Phone: +33-607-28- 5247; Email: management@world-ctl.com; Web: world-ctl.com Paris

Mar. 1-3

7th International Congress on Energy Efficiency & Renewable Energy Sources for South East Europe. Via Expo Ltd. (Sofia, Bulgaria). Phone: 0035-9329-454-59; Web: viaexpo.com Sofia, Bulgaria

Apr. 13-15

Techtextil 2011. Messe Frankfurt GmbH (Frankfurt, Germany). Phone: +49-69-75-75-0; Web: techtextil. messefrankfurt.com Frankfurt May 24–26

ASIA & ELSEWHERE

India Rubber Expo 2011. India Rubber Expo (Tamil Nadu, India). Phone: +91-44-42822207; Web: indiarubberexpo.in Chennai, India

Jan. 19-22

3rd International Conference on Drug

Discovery & Therapy. Eureka Science Ltd. (Sharjah, UAE). Phone: +971-6-5575783: Web: icddt. com Dubai, UAE

Feb. 7-10

10th International Nanotechnology Exhibition & Conference (Nanotech 2011).

ICS Convention Design, Inc. (Tokyo). Phone: +81-3-3219-3567; Web: nanotechexpo.jp/en/index.html Tokyo Feb. 16-18

Suzanne Shelley



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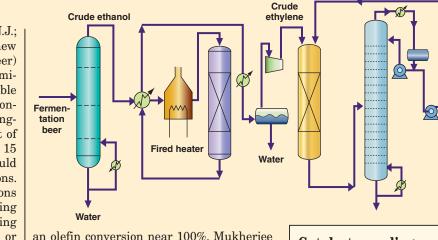
Edited by Gerald Ondrey

Scaleup for a new process to make Bio-Alkylate

Next spring, Exelus, Inc. (Livingston, N.J.; www.exelusinc.com) plans to pilot a new process that converts crude bioethanol (beer) into Bio-Alkylate — a fuel that is chemically identical to gasoline. New Renewable Fuel Standards (RFS) of the U.S. Environmental Protection Agency (EPA; Washington, D.C.) call for increasing the amount of ethanol that can be added to gasoline to 15 vol.% (so-called E15 gasoline), which would require significant engine modifications. "This technology eliminates the limitations of using bio-ethanol as a fuel by allowing gasoline blends up to E50 without requiring any changes to cars or fuelling stations or compromising mpg", says Exelus president Mitrajit Mukherjee.

In the new process (flowsheet), filtered beer is first vaporized in a stripping column, generating wet ethanol vapor. The vapor is heated further and dehydrated into ethylene over a solid-acid catalyst. Upon cooling, the water and ethylene are readily phase separated. Crude ethylene is then directly alkylated with excess isobutane over an engineered, zeolite catalyst producing Bio-Alkylate. Unreacted isobutane is distilled and recycled.

In laboratory trials, the in-house-developed catalyst showed exceptional activity for converting ethylene into high-octane, low-RVP (Reid vapor pressure) alkylate, with



Dehydration

reactor

Bio-alkylation

reactor

an olefin conversion near 100%. Mukherjee points out that the rates of reaction over this catalyst greatly exceed that reported in the literature for solid-acid catalysts, and avoids the hazards and costs associated with conventional isoparaffin-alkylation processes that are based on HF or H_2SO_4 liquid catalysts. Up to now, there are no commercial technologies capable of isoparaffin alkylation using ethylene, he says.

The new process is being developed with partial funding (\$1 million) from the U.S. Dept. of Energy's (DOE; Washington, D.C.) ARPA-E program, and a 1-gal/d pilot plant is being planned to start up next spring. Mukherjee estimates the cost of producing Bio-Alkylate at \$2/gal (without price credits for ethanol).

Catalyst recycling

A new process for selectively dissolving either palladium or gold from mixed-metal catalyst systems could offer a way to improve recycling of those materials. In traditional noble-metal recycling with aqua regia (mixture of nitric and hydrochloric acids), metals are often dissolved together, which introduces impurities into the recycled metals. Now scientists at the Georgia Institute of Technology (Atlanta, Ga.; www. gatech.edu) have developed

(Continues on p. 12)

A new catalyst reduces SO₂ emissions from H₂SO₄ plants

t last month's Sulfur 2010 Confer-Aence (November 1–4; Prague, Czech Republic), Haldor Topsøe A/S (Lyngby, Denmark; www.topsoe.com) introduced its latest sulfuric-acid catalyst, VK-701 Leap5, which promises to help operators of sulfuric acid plants meet morestringent SO₂-emission limits. When used in the final pass of single absorption H₂SO₄ plants, VK-701 Leap5 reduces SO₂ emissions by up to 40% compared to existing catalysts. The new catalyst also makes it possible to reach down to 50 ppm in existing 3+1 doubleabsorption plants or to design doubleabsorption plants with SO₂ emissions as low as 20–50 ppm SO₂, says Lene

Hansen, general manager — sulfuric acid, catalyst division.

Conventional sulfuric-acid catalysts are based on vanadium oxides promoted with alkali-metal sulfates on an inactive, porous silica support. In these so-called supported liquid phase (SLP) catalysts, the oxidation of SO₂ occurs as a homogeneous reaction in a liquid film covering the internal surface of the supported material. Although the detailed reaction mechanism is not entirely known, there is evidence that only the oxidation state V⁺⁵ is active. With Leap5 technology, Topsøe has substantially increased the amount of V⁺⁵ to about 70% of the total vanadium content (at 400–440°C) — about two to three times more than existing commercial catalysts, says Hansen. The activity of VK-701 operating at high conversion in a gas feed with 10 vol.% SO₂ and 10 vol.% O₂ is about two times higher than standard cesiumpromoted catalysts over the temperature range of $380-460^{\circ}$ C.

The first industrial installation of VK-701 was a single-absorption plant burning elemental sulfur and operating with a five-bed SO₂ converter. Replacing the Cs- and K-promoted catalysts in the final passes (beds four and five) led to a 35% reduction in SO₂ emissions at the same production rate (245 metric tons per day H_2SO_4).

December 2010

Makeup

isobutane

Distillation

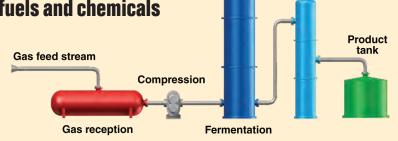
column

Microbes convert stack gases to fuels and chemicals

Ademonstration plant for a biological process that produces ethanol and 2,3 butanediol (2,3-BD) from the offgases of industrial plants will be started up in the third quarter of 2011 at a steel mill operated by Bao Steel (Shanghai). Developed by LanzaTech (Auckland, New Zealand; www. lanzatech.co.nz), the process will produce about 100,000 gal/yr of ethanol from a slipstream of stack gases, says Mike Schultz, the company's director of process development. He adds that negotiations are under way with Bao for a 50-million-gal/yr commercial plant that would start up toward the end of 2012.

LanzaTech's process (flowsheet) converts carbon monoxide to ethanol by means of a specially cultivated strain of the *Clostridium* bacterium. The steel mill's waste gases mainly consist of CO, plus some H₂, CO₂, CH₄ and O₂. Particulate matter is filtered from the gases, then the O₂ is removed catalytically, since the bacterium is anaerobic.

The rest of the gas mixture is sparged into the aqueous solution of bacteria and converted to ethanol in a continuous process, at 35-40 °C. Hydrogen (needed for the process) is generated via a biological, water-gas-shift reaction, although if H₂ is present in the gas stream this reaction is mitigated and can



lead to additional ethanol production, says Schultz. The CO_2 is inert and simply passes through the reactor. A hybrid recovery system separates the ethanol and the coproduct 2,3-BD from the fermentation broth, which is recycled to the process.

Schultz says the process is competitive with processes that convert biomass to ethanol, with the advantage that it uses waste gases rather than crops. Such gases are often flared (as in the case of the steel mill) or burned for process heat. Schultz notes that 2.3-BD is an intermediate for such chemicals as butylenes, butadiene and methyl ethyl ketone. He adds that Lanza-Tech is working in the laboratory on a microbial process that converts CO directly to butanol or fatty acids. Last month, the company also announced plans to work with Pacific Northwest National Laboratory (Richland, Wash.; www.pnl.gov) to develop 2,3-BD as a drop-in jet fuel.

(Continued from p. 11)

Distillation

a scheme that combines thionyl chloride with a variety of organic reagents, such as pyridine, N, N-dimethylformamide (DMF), pyrimidine and imidazole, to selectively dissolve either gold or palladium, depending on the solvents chosen and their concentrations. No combination of solvents dissolves platinum, giving the solvent system the ability to separate Au or Pd from Pt in mixed metal catalysts. So far, the researchers have used the solvent system to separate Au and Pd from Pt, and to remove Au from a mixture of Au and Pd.

New catalyst approach for methane-to-ethylene conversion

Ananotechnology-based, bottom-up Approach to synthesizing catalysts for converting natural gas to ethylene could enable an alternative to the steam-cracking of crude oil, an energyintensive process conventionally used to produce ethylene, the world's most valuable commodity chemical. Scientists at the startup Siluria Technologies Inc. (San Francisco, Calif.; www.siluria. com) have developed a synthetic method that allows them to manipulate catalyst surface morphology such that the oxidative coupling of methane (OCM) reaction proceeds with high performance at low temperatures.

An industrially viable OCM method has been sought for decades, but past efforts have been unsuccessful because the high temperatures needed for activating methane reduce the reaction's selectivity. With many previously studied catalysts, methyl free radicals leave the catalyst surface before conversion to the desired ethylene product. Nonselective oxidation to CO_2 is common. A methane-based route to ethylene could introduce feedstock flexibility and uncouple the price of crude oil from that of commodity chemicals.

Marrying molecular biology with industrial chemistry, Siluria's catalyst synthesis process uses proteins on the surface of a genetically modified bacteriophage (bacteria-infecting virus) as nucleation sites for growing nanoscale wires of catalyst material. By growing the catalyst nanowires on an engineered biological template, Siluria is able to access crystal structures and surface morphologies not formed through conventional crystallization of the material. The novel crystal structures, in turn, give rise to catalyst active sites with unique properties that are critical to achieve the selectivity and yield required for an economically viable OCM process.

"Conventional top-down catalyst synthesis generates the most thermodynamically favorable crystal structures, but unfortunately, those are not the most effective in catalyzing OCM," explains Siluria president Alex Tkachenko. "We've developed a synthetic method for producing catalyst material with specialized surface properties, which turns out to be critical for an effective OCM process and delivering on the promise of methane activation to produce chemicals."

The catalyst materials are proprietary, doped metal oxides of early transition metals that are designed for compatibility with existing petrochemical industry infrastructure. Siluria has developed a library of compounds with a range of crystal structures, and has tested their behavior in catalyzing the OCM reaction. The company aims to achieve the catalyst performance required for commercialization in 2011, Tkachenko says.

Commercialization may be in the works for a 'cleaner' gold-recovery process

A"green" hydrometallurgical process for recovering gold from ore, developed by Haber, Inc. (Arlington, Mass.; www.habercorp.com), may make its commercial debut in Suriname. Albert Conti, Haber's CEO, says the Republic of Suriname has made a commitment to develop a strategic partnership with the company for the use of the process. The elimination of mercury pollution is a goal of the government in Suriname, where small-scale gold miners use the metal to absorb gold from gold concentrates, recovering the Au as a gold-mercury amalgam. Excess Hg is driven off as a vapor by heating. In contrast, Haber's process doesn't use Hg and avoids the use of cyanide, commonly used in large-scale mining to leach gold from ore.

The commitment follows recent government-sponsored tests of Haber's process in Paramaribo, Suriname's capital. A number of sulfide ores were processed and gold recovery ranged from 96.1–99.7%, says Conti. This compares with recoveries of about 35% for current small-scale commercial operations in Suriname, he says, noting that Suriname has more than 25,000 small-scale miners and about 100 larger gold-mining entities.

Conti declines to give specific details on the process, except to say that it involves a sequence of proprietary steps. Sulfide ore is crushed to 20–120 mesh and pretreated to liberate gold from sulfur-bearing compounds. The Au is leached from the ore by a lixiviant in an aqueous solution and recovered as a fine powder by adding a proprietary formulation that converts the dissolved Au from the ionic to the metallic form. Conti says the process is faster and cheaper than roasting or treating the ore with bacteria, the current, popular liberation methods.

Nanoclusters that selectively catalyze oxidation reactions

Professor Shu Kobayashi, University of Tokyo (Tokyo, Japan; www.s.u-tokyo. ac.jp), has discovered that bimetallic nanoclusters can catalyze the oxidation of alcohols. Kobayashi's group has demonstrated the ability to control the reaction pathways of alcohol oxidation to aldehydes, carboxylic acids or esters using nanoclusters composed of a few to several-tens of atoms of gold, platinum and palladium. These carbon-stabilized, polymer-incarcerated, bimetallic nanocluster catalysts exhibit selective oxidation of alcohols. The reactivity and selectivity are strongly dependent on the metal pairs in the cluster and the solvent system used. For example,

1-octylalcohol reacting over a Au/Pt cluster (room temperature, 1 atm of O_2 and a benzotrifluoride-water solvent) yields the corresponding aldehyde (C_7H_{15} -CHO) with 92% selectivity after 9 h, whereas a Au/Pd cluster (methanol-H₂O solvent) yields the corresponding ester (C_7H_{15} -COOCH₃) with 78% selectivity after 24 h.

Electron microscopy shows that the average diameter of the two clusters are about the same (2 nm), but the ratio of the metal atoms are different (1:1 for Au/Pt and 4:1 to 3:1 for Au/Pd). The research is said to be the first example of controlling the reaction pathway of gold-catalyzed reactions by introducing a second metal.

Miniature ESR spectrometer facilitates study of free radicals

Aminiaturized electron spin resonance (ESR) spectrometer developed by Active Spectrum Inc. (Foster City, Calif.; www.activespectrum.com) can facilitate studies of chemical species with unpaired electrons, such as organic free radicals and transition metal complexes. Available in both online and benchtop models, the Micro-ESR spectrometer greatly reduces the size, cost and complexity of ESR measurements, allowing the technique to be widely accessible to nonspecialist users in chemical processing and industrial research.

ESR spectroscopy is valuable for studying

the composition and concentration of shortlived free radicals in oils, food products and biological materials. A key advantage of ESR over optical spectroscopy techniques is its ability to scan dirty, mixed or opaque samples without any sample preparation. It is also highly specific — most substrates have no unpaired electrons and therefore do not interfere with ESR.

Traditional ESR equipment uses large and expensive 1970s-era microwave technology and magnets adapted from nuclear magnetic resonance (NMR) spectroscopy.

 $(Continues \ on \ p. \ 14)$

Fructose from fruit

Nutritis (Toulouse: www.nutritis.com), in cooperation with Novasep Process (Pompey, both France; www.novasep. com), plans to scale up a new process for producing fructose from apple juice. Details about the process were not disclosed, but the companies say the patented technology enables production of fructose that can be labeled as 100% fruit, making it suitable for fruitbased preparations such as dairy products and jams. Conventional industrial processes make fructose by the hydrolysis of sucrose from sugar cane, sugar beets and corn starch. Nutritis plans to produce fructose from substandard fruit that cannot otherwise be sold.

Nanoscrolls split water

Kazuhiko Maeda, assistant professor at The University of Tokyo (Tokyo; www.domen.t.utokyo.ac.jp) and professor Thomas E. Mallouk at Penn State University, have developed a highly efficient photocatalyst for splitting water with visible light. The catalyst — a nano-scale "scroll" of niobate has a guantum yield of 25% with 450-nm radiation. which is about 20 times more active than layered niobate systems, and ten times more than titanate-based photocatalysts, says Maeda.

To make the catalyst, an exfoliation process removes single layers from a niobate compound to give cylindrical scrolls. By combining the scrolls with a ruthenium-based dye, the catalyst becomes active with visible light. The H₂ production rate (3.5 mol/h) is also about 20 times faster than alternative catalysts. These improvements are believed to be due to the high specific area of the scrolls, which is about 200 times more than lavered niobate.

Fluorinated graphene

This year's physics Nobel Prize laureates, professors Andre Geim and Kostya Novoselov, and colleagues at The University of Manchester (U.K.; www.manchester.ac.uk), have successfully fluorinated each carbon atom of graphene to (*Continues on p. 14*)

ESR SPECTROMETER

(Continued from p. 13) The Micro-ESR sensor takes advantage of recent advances in wireless communications technology, and uses a samarium-cobalt permanent magnet to generate the magnetic field necessary to observe a signal. "We're essentially updating an old technology with modern microwave components borrowed from the wireless industry," says Active Spectrum president James White.

The result is a device that is 100 times smaller than conventional ESR equipment, but that maintains sensitivity in measuring free radicals. Also, "the dramatically lower cost of the device is a major advantage," White says.

Micro-ESR can be used to evaluate the degradation of engine oils and hydraulic fluids, and — with the help of spin-trapping compounds that convert transient free radicals into stable species - oxidative breakdown of food and concentrations of spin-labeled proteins. Micro-ESR can also be used for realtime monitoring of crude oil properties, such as asphaltene and vanadium content.

A more efficient use of renewable thermal energy

team of engineers from the Center for En-Aergy Technology, University of Adelaide (South Australia; www.adelaide.edu.au), the University of Nantes (France) and Mie University (Japan), have proposed a more efficient way to generate power from low to medium temperature (90-260°C) solar and geothermal resources. In the new system, called Renewable Assisted Power Generation (RAPG), the renewable energy is used to heat the boiler feed water (BFW), so the efficiency is no longer limited by the renewable resource's temperature, says team member Eric Hu. Instead, the efficiency is limited by the maximum temperature of the steam cycle.

In contrast to other solar boosting or combined power systems, in RAPG renewable-energy-generated heat or steam does not enter the turbine directly. Instead, the energy is used in place of steam normally extracted from turbine stages for BFW preheating in regenerative Rankine cycles. The steam that would be otherwise extracted is

therefore available to generate additional power in the turbine.

For three typical temperatures of geo- and solar-thermal resources (90, 215 and 260°C), the team compared the performance for the cases in which the resources were used to generate power in a standalone capacity or with RAPG in a typical 200-MW subcritical power plant and in a 600-MW supercritical steam plant. The team found that in the RAPG system the thermal efficiency of renewable energy generation exceeded the Carnot efficiency, which shows that the thermal efficiency was no longer limited by the temperature of the renewable source, but rather by the maximum temperature of the Rankine cycle, that is, the maximum steam temperature at the exit of the boiler.

In the case of a geothermal fluid at 215°C, for example, it was found that using the geothermal energy to heat BFW doubled the efficiency of power generation over the stand-alone case, and its contribution to total power was about 16%.

Biocatalysts for biodiesel production

esearchers from the Department of Environmental and Applied Chemical En-Gangneung-Wonju National gineering, University (Gangneung, South Korea; www. gwnu.ac.kr) are developing a two-step enzymatic route to biodiesel fuel that promises to reduce production costs. The process employs a lipase-producing bacterium and, sequentially, a commercial enzyme. The combination has the double advantage of reducing the use of commercial enzyme, thus lowering costs, and of reducing enzyme deactivation by methanol — a problem that has prohibited the commercialization of enzymatic biodiesel fuel production.

The university group, led by Prof Sung Ho

Yeom, uses a lipase-producing bacterium, Serratia marcescens, isolated from greasecontaminated soil and chemically mutated to increase its lipase production. The bacteria with highest lipase activity were mutated again to further enhance their lipase activity. These twice mutated bacteria exhibited 2.5-times higher intracellular lipase activity than the wild type. The enzyme combination was used for transesterification of soybean oil using methanol as an acyl donor. Although the biocatalyst was less inhibited

by methanol than the commercial enzyme,

it exhibits much-lower biodiesel-conversion

activity per mass than commercial enzyme,

thus the need for both.

(Continued from p. 13)

create a two-dimensional version of Teflon. Unlike its plastic counterpart, the so-called fluorographene is a crystalline material, one-molecule thick, which exhibits the high strength of graphene and does not react with other chemicals. The material also can withstand high temperatures, even in air. Potential applications of the new material - a wide-gap semiconductor that is optically transparent to visible light - include ultra-thin tunnel barriers for light-emitting devices and diodes.

Porous plastic powder

Ticona (Florence, Ky.; www. ticona.com), the engineering polymers business of Celanese Corp., has commercialized a new porous plastic powder that can be sintered into products with good strength and porosity for use in filtration applications for gases and liquids that require higher flowrates and lower pressure drops. The powder, tradenamed GUR X 192 VHMW (very high molecular weight) polyethylene, has a molecular weight of 600,000 g/mol; a particle size distribution (d₅₀) of 451 µm; a bulk density of 0.35 g/mL; and a 47% porosity with pore sizes of 250 µm. The patentpending material can be shaped and sintered at 180-220°C into porous objects with improved strength and porosity.

Hydrogen separation

Inorganic membranes for separating hydrogen in steammethane reformers (SMRs) have taken a step closer to commercialization following successful testing at a 20-Nm3/h experimental facility of Tecnimont KT (formerly Technip KTI S.p.A.: Rome, Italy; www.tecnimontkt. it). The facility, located at Chieti, Italy, incorporated a 0.4-m² Hysep membrane module developed by ECN (Petten, the Netherlands; www.ecn. nl) — for separating H₂ during the reforming reaction. Integrating reforming with separation enables the process to operate at "well below" 650°C instead of the 850–900°C required in traditional SMRs, says ECN. The membranes — thin-film Pd on ceramic supports - demonstrated a high flux and durability after 500 h of operation and more than 50 thermal cycles.

Newsfront

THANING CPI JOB MARKET SPURS HIRES

Cautious optimism drives modest hiring activity in the CPI, but headwinds remain

ngineers and others working in the chemical process industries (CPI) generally are experiencing an employment situation that is significantly improved over 12 to 18 months ago, with a tentative optimism among CPI companies driving significant hiring activity. Demand for chemical engineering skills remains strong, reflecting the fact that the chemical sciences represent key enablers in solving many broad challenges, including energy production and use, environmental protection and sustainability. However, the rosier outlook in the CPI job market is tempered by considerable uncertainty surrounding a host of issues, including the strength of the overall economic recovery and the possibility of shifting governmental policy. The uncertainty is fostering a sense of caution surrounding workforce decisions.

Blending optimism, caution

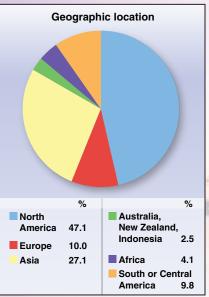
At the broadest level, the job market for chemical engineers and other engineers and technical workers in the CPI has stabilized and strengthened across all industry segments as a wider economic recovery has taken hold. Larry Jacobson, executive director of the National Society of Professional Engineers (NSPE; Alexandria, Va.; www. nspe.org), says "So many of today's challenges are looking for engineering solutions, so the demand for talented and versatile engineers is high."

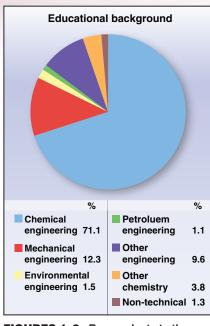
Kevin Swift, chief economist with

the American Chemistry Council (ACC; Washington, D.C.; www.americanchemistry.com), describes the prevailing attitude among chemicalrelated business leaders as one of "guarded optimism," with all industrial segments experiencing some recovery. Still, business leaders are facing a lot of uncertainty. Swift explains, with many still wondering about how changes to environmental regulations will come about and the possibility that countries will take legislative action on climate change in coming years, such as a cap-and-trade emissions policy for greenhouse gases.

Swift's views are consistent with those of Lawrence Sloan, president and CEO of the Society of Chemical Manufacturers & Affiliates (SOCMA; Washington, D.C.; www.socma.com), who echoes the "cautiously optimistic" sentiment based on the positions of SOCMA member companies. Sloan points to a wealth of anecdotal evidence that many companies are projecting positive growth and are planning to hire. Sloan cites more than a dozen examples of chemical manufacturing companies serving both industrial and pharmaceutical markets that are planning to hire personnel in the coming months, or that have recently done so.

According to Sloan's information, the hiring trend seems to apply to multiple job functions. "Companies are hiring in multiple areas — R&D positions, manufacturing positions, as





FIGURES 1–2. Respondents to the November *CE* salary survey predominantly lived in North America and Asia. Chemical engineering was by far the most common educational background, followed by mechanical engineering

well as sales, technical support and others," Sloan says.

The SOCMA companies, which tend to be small, U.S.-based specialty chemical firms that serve specific industrial and pharmaceutical markets, generally said the salaries they would offer new hires would match levels in the recent past, and that their open positions would represent a mix of experienced and entry-level jobs. The SOCMA membership also has concerns — for example, over efforts to reform the Toxic Substances Control Act (TSCA) — but those concerns do not seem to have dampened positive sales projections and hiring moves.

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Recruiting is more active

Recruiting firms working in the CPI space have observed a markedly increased number of personnel search projects for chemical engineers in 2010. For example, Patrick Ropella, CEO of the Ropella Group (Milton, Fla.; www.ropella.com), a search firm specializing in the chemical and allied industries, says his firm has seen an increase in search projects across all industry sectors. "There's no question that there's a more positive attitude than a year ago," he stated, "but at the same time, there's also a lot of trepidation, which makes for a slower hiring process." Ropella notes that his firm has seen a significant number of personnel searches aimed at helping a company enter a new market. Another positive indicator is the small number of chemical engineering searches to find staff for expansion projects. "Last year, there were virtually none of those," Ropella said.

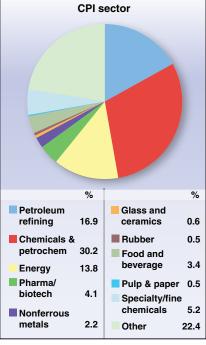
Although hiring freezes have largely been lifted, fear concerning possible new environmental regulations remains, and people don't hire when they are fearful, Ropella says. "We are slowly thawing out, but we still haven't broken free altogether," he adds.

Salary increases for job-changers are smaller than what has been seen in the past, he says. "Normally, a 10% increase in salary was the minimum to get the right person into a new position, but right now we're seeing a lot of single-digit percentage increases."

Rich Brandeis, a senior partner who manages the chemical engineering recruitment division at personnel search firm CPS (Westchester, Ill.; www.cps4jobs.com), has had a similar experience. "The hiring climate for chemical engineers is good right now, and it's gotten stronger throughout the year," he says, expecting the upward trend to continue.

Aside from the wider economic recovery, part of the reason for the activity could be that a lot of companies went a long time without hiring anyone, Brandeis suggests, so now, opportunities are good for mid-level chemical engineering positions across a wide range of industries.

Noticeably fewer out-of-work engineers are contacting Brandeis, and



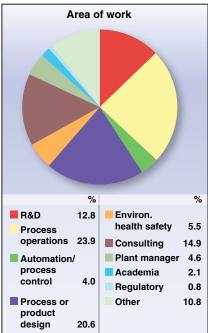


FIGURE 3–4. The largest number of survey respondents worked in the chemicals and petrochemicals industry. Process operations was the most common job area, although the distribution was more equitable

people seem to be more confident about making a career move. Also, we have seen more counteroffers by companies trying to retain workers, he says, which can be viewed as evidence for a stronger labor market. Brandeis agrees with others in pointing out that the hiring activity is broad-based — "pretty much across the board," he says. "Not many sectors are lagging, and the trend of improvement seems to be a longer-term one, rather than a short-term spike in activity."

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CE SALARY SURVEY

C lose to 1,500 readers of *Chemical Engineering* magazine from around the world responded to a nonscientific survey that attempted to gauge average salaries according to geographic regions of the world, level of experience, and industry sector. The results largely confirm that chemical engineering salaries are above average for the wider labor force, and improve with experience level. A profile of the respondents can be found throughout the article, and Tables 1–3 include some of the calculated salary averages. Thank you to all those who provided information for the survey. \Box

TABLE 1. AVERAGE ANNUAL SALARY OF SURVEY RESPONDENTS BY GEOGRAPHIC AREA (U.S. CURRENCY)

These averages include respondents from all educational back- grounds, industry sectors, experience levels and employer types	Average salary
All responding CPI workers	\$77,800
CPI workers living in North America	\$97,300
CPI workers living in Europe	\$69,500
CPI workers living in Africa	\$39,000
CPI workers living in Asia	\$37,000
CPI workers living in South and Central America	\$45,200
CPI workers living in Australia, New Zealand and Indonesia	\$107,000

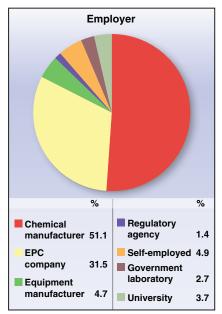


FIGURE 5. Over 82% of respondents work for chemical makers or engineering, procurement, construction (EPC) firms

University action

University recruitment represents another vantage point from which to gauge the hiring climate for engineers. Deborah Liverman, associate director of global education and career development at the Massachusetts Institute of Technology (MIT; Cambridge, Mass.; www.mit.edu) remarks that campus recruiting has been extremely busy at MIT this autumn. "I think it indicates the confidence felt by many companies," she says, especially since many autumn job offers have been made to students who will not begin work until summer 2011. "The confidence is enough for companies to be willing to make a hire this far out," she says.

In 2009, 47% of MIT chemical engineering graduates went directly to work,

as opposed to graduate school or other pursuits. The percentage increased to 61% going directly to work in 2010, a possible indicator of a greater number of work opportunities, especially for top students. In addition, a higher number of startup companies are recruiting graduates, as well as companies that are not the "traditional" employers of engineers, Liverman says.

In a reflection of the strong growth in emerging markets, Dow Chemical Co. (Midland, Mich.; www.dow.com) recently announced a robust campus recruiting effort in China. Dow says that in 2011, the company aims to recruit 80 qualified graduates from Chinese engineering schools for its Dow China operations. The total is 60% more than the number in 2010, Dow noted.

Nuanced picture

Within the current employment environment for chemical engineers and CPI professionals, a number of forces are at work that paint a more nuanced picture. One of the forces is industry's growing ability to utilize improved technology to maintain productivity with less personnel. "A lot of companies are doing more with less," says ACC's Swift, so in some cases, there is reduced pressure to hire. On the other hand, workforces at such lean levels can mean a robust market for contract workers and consultants.

In a recent virtual career fair, hosted by the American Chemical Society (ACS; Washington, D.C.; portal.acs. org), speakers mentioned other factors likely to affect the CPI employment situation. For example, an important trend to watch will continue to be the retirement of engineers in the "Baby BL Renewable Resources

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TABLE 2. SALARY OF SURVEY RESPONDENTS **BY EMPLOYER TYPE**

Averages here are based on respon- dents living in North America, with chemical engineering education at all experience levels and in all in- dustry sectors for the following types of employers:	Average annual salary
Manufacturer of equipment used in the CPI	\$103,000
Manufacturer of chemicals	\$117,720
Engineering, procurement and construction (EPC) firm	\$123,000
Consultant	\$170,000
University, government laboratory or regulatory agency	\$75,000

TABLE 3. SALARY OF SURVEY RESPONDENTS BY EXPERIENCE LEVEL

Averages here are based on re- spondents living in North America, with chemical engineering educa- tion, in all industry sectors, with the following levels of experience	Average annual salary
0-3 years experience	\$62,600
4-6 years experience	\$80,600
7-10 years experience	\$105,000
11-20 years experience	\$110,000
21-30 years experience	\$113,000
30+ years experience	\$126,000

Boom" generation. Many have delayed retirement because of the depressed economic conditions, she explained, but as they leave the workforce amid an improving economy, gaps will be created. ACC's Swift acknowledges that avoiding the loss of the practical knowledge base held by older workers as they retire will remain a key issue going forward.

While all industry sectors seem to be experiencing an improved job market, some areas have particularly high activity. Judging by the number of job search projects, Ropella points to energy technology (both traditional fossil-fuel-based energy and alternative sources) as "hot" areas of job growth activity. "There's also a lot of activity in mining and in jobs related to extractive technologies," he says. NSPE's Jacobson sees a robust future for engineers in the nuclear energy arena. Brandeis adds that there is particular demand for chemical engineers who are strong in capturing new process technology.

In the future, the global job market will see an extra premium on engineers whose skills go beyond technical expertise. "Employers love international

experience," says MIT's Liverman, as well as leadership and communications skills. Jacobson, of NSPE, says the largest demand is for engineers "who don't live in silos" — those with disparate experiences and diverse skill combinations from different areas of industry. "Those kinds of people have the tools to look at problems through a wide spectrum of glasses," Jacobson says.

The case of startup company Genomatica (San Diego, Calif.; www.genomatica.com), which is commercializing renewably sourced 1.4-butanediol, illustrates the point. The company has several openings, including ones for chemical engineers, which make up one-third of its R&D staff. In hiring decisions, it strongly considers a candidate's adeptness at interfacing with personnel

who have training backgrounds that differ from their own, as part of an integrated team. "We're looking for people who are skilled at speaking many different scientific languages," Genomatica CEO Christophe Schilling says.

Salary data

Available recent salary data for chemical engineers and others working in the CPI indicate that pay levels at the end of 2010 are similar to those observed in recent years. For example, salary data from NSPE indicate that chemical engineering salaries have increased from a median of \$107,000 in 2009 to \$112,750 in 2010. The results change slightly when the calculation includes engineers of all specialties who work in chemical processing, pharmaceuticals and related industries. In that case, the totals are \$104,858 median salary for 2009 and \$101.000 for 2010.

In November 2010, Chemical Engineering conducted a nonscientific survey of its readers to collect CPI salary information. (For more, see box, p. 17, Figures 1–5 and Tables 1–3).

Scott Jenkins

Newsfront

Endress+Hauser

ON THE LEVEL Level measurement technology was lacking in the

past, but new technologies are increasing reliability and overcoming traditional measurement obstacles

s level is a critical parameter for those in the chemical process industries (CPI), level measurement instrumentation has been around for some time. But as processors scramble to become more efficient, reduce waste, control inventory and abide by environmental regulations. level measurement has become even more important. As a result, device manufacturers are improving existing technologies with value-added characteristics, such as increased accuracy, better electronics, reduced maintenance, data-management capabilities and other advancements that allow processors to apply reliable level measurement to their toughest and most critical applications with confidence.

Level measurement plays a variety of roles in the CPI from measuring inventory levels to ensuring consistency during the production process to preventing pumps from running dry and tanks from overflowing. And there are as many different devices and technologies available as there are applications. But if you look closely at level measurement through the years, it's easy to see there was an opportunity to improve just about every type of technology or to switch to a newer technology to better solve existing problems, says Will Bailey, strategic business manager with Omhart Vega (Cincinnati, Ohio). "Some of the more antiquated technologies out there really needed improvement regarding the amount of maintenance they required. By switching to a newer technology or improved device that is more reliable and requires less maintenance, it provides a host of opportunities for processors to improve their processes, products and safety records," he says.

Historically it was not uncommon to use differential pressure technologies or displacers, such as mechanical floats, in level measurement applications. However, these technologies often had issues with changes to the specific gravity of a product, so there would be inaccuracies. To offset this, technicians had to recalibrate the devices for each new material or product, but with some of the newer, improved technologies, this step is not necessary. This reduces maintenance activity and improves the process.

Many older types of level measurement devices, such as displacers, required frequent mechanical checks and, just as often, removal from the process for cleaning. "Many of the available technologies no longer require that type of maintenance, so we're seeing a big push in the industry to move toward lower maintenance, higher accuracy technologies," says Bailey. "It simply and easily removes the challenges people historically dealt with regarding level measurement."

Radar to the rescue

Free-wave radar and guided-wave radar seem to be taking the industry by storm in recent years, and with good reason. Radar solves the problems mentioned above in a host of processes that have been traditionally difficult to deal with.

Radar devices can accurately handle specific gravity change, changes in capacitance or conductivity and there are few or no moving parts. The technology is also impervious to humidity and foams.

As a general rule of thumb — though it varies from application to application — guided-wave radar, which features

an antenna line or cable that goes into the vessel containing the product to be measured and a radar signal that travels up and down that line, is used on the process control side. Free wave, which is a non-contacting form of microwave level measurement, is more likely to be used in finished products for inventory control in both liquids and solids.

Not only is the ability to measure both liquids and solids a bonus, but radar technologies also offer improvements in signal reliability, which helps with traditionally difficult interface applications (where there is an emulsion layer between two materials that have separated in the storage or process vessel), or where vapors or dust are an issue.

Newsfront

Pepperl+Fuchs

In applications where there is foam or an emulsion layer, ultrasonic techniques were traditionally used. However, the technology was limited in that it would read the top of the emulsion layer or the foam. But improved signal reliability allows radar devices to differentiate and give an accurate reading.

When it comes to solids, most devices in the past provided good, accurate level measurement when the tank was static, but when there was product movement, the resulting dust in the vessel would cause the device to lose the signal. Improving signal reliability has allowed the signal to be maintained through fill and release of product.

Radar technologies are also able to work despite vapor conditions and high pressures or temperatures.

Because of the myriad of benefits and opportunities to use radar devices in a variety of applications, many level-measurement-device suppliers have launched new radar products, each with its own spin. "Before radar was a prevalent technology, there were other technologies you could use, but none of them really tackled these more difficult issues," says Jerry Boisvert, strategic marketing specialist with Siemens (New York, N.Y.). "It was like forcing your foot into a too small shoe, so we are working on introducing technologies that aren't a forced fit on difficult applications."

One such technology is Siemens' Sitrans LR400, a four-wire, 24 GHz FMCW (frequency modulated continuous wave technology) radar level transmitter for continuous monitoring of liquids and slurries in storage and process vessels including high-temperature and high-pressure applications, to a range of 164 ft. It is suitable for use on low dielectric media. The combination of 24 GHz and high signal-to-noise ratio contribute to improve signal reflection, smaller and more compact designs, better accuracy and the ability to solve problems that may have been difficult in the past, says Boisvert.

Honeywell (Morristown, N.J.) also offers SmartLine non-contact and guided-radar level-meters for traditionally difficult applications. The FIGURE 2. The Pulscon LTC transmitter for continuous level measurement in powdery to granular bulk solids and liquids provides measurement independent of density, temperature and dust and can also be used with foam on the surface

non-contact version employs FMCW technology for level measurement of solids and liquids and can be used to calculate volume. It provides a more stable measurement than pulse radar and is suitable for use in agitated process conditions in demanding applications. The guided version operates on time delay reflectometry to measure level, distance, interface, level and interface, volume and mass. It is suited for measuring liquids as well as granular solids.

Endress+Hauser (E+H; Greenwood, Ind.) offers an interesting twist in radar devices via its Levelflex FMP55, which is a multiparameter technology that provides interface and level measurement in liquids. The FMP55 combines

LEVEL MEASUREMENT SERVICE PROVIDERS

Automation Products Group www.apgsensors.com BinMaster www.binmaster.com Endress+Hauser us.endress.com Honeywell www.honeywell.com K-Tek www.ktekcorp.com Ohmart/VEGA www.ohmartvega.com www.pepperl-fuchs.com Pepperl+Fuchs Siemens usa.siemens.com

guided radar and capacitance technologies in the same unit on the same rod or cable. "The advantage of this is that by using two technologies, it can measure interface well, especially those that have an emulsion or rag layer," says Gene Henry, product business manager with E+H. "The capacitance technology measures interfaces that have rag layers well but can't measure overall level. Guided radar measures overall [level] well, but is not always reliable with interfaces. By combining the two into one instrument, you can have the best of both worlds."

Pepperl+Fuchs (P+F; Twinsburg, Ohio) also offers a radar device with something extra. The Pulscon LTC transmitter for continuous level measurement in powdery to granular bulk solids and liquids provides measure-

AVOIDING OVERSPILL

While not as technologically advanced as many of the continuous level products, point level measurement is taking on an increasingly important role for chemical processors since environmental safety concerns have moved to the forefront. "Most chemical processors are looking closely at their level measurement devices for overspill protection due to the environmental and safety impact an accidental overfill can create," says E+H's Henry. "If you have a hazardous or flammable material that gets out of the tank, vapors may spread away from the site and a fire or explosion can result either on your site or in another area, so overspill protection is key for chemical and petrochemical processors who want to keep their safety record intact."

The increasing importance of overspill protection has led to two trends. The first being the use of SIL-rated instruments (SIL = safety integrity level; for more on SIL, see Tolerable Risk, *Chem. Eng.*, September 2007, pp. 69–74). With SIL-rated instruments, it is not just the instrument, but the fact that to maintain an SIL rating, there must be a total solutiondriven process that includes testing the instruments and the system to make sure they are working. "To maintain a SIL rating, there have to be frequent system and instrument checks and tests, which all plays into having a program for overspill protection," says Henry.

The other occurrence resulting from increased overspill protection is the replacement of mechanical high-level switches to more advanced technologies. "Historically floats or mechanical switches were used in these applications, but now we are see more vibrating level switches, many of which offer an SIL rating, because they are more reliable," says Omhart Vega's Bailey.

Vibrating fork technology is growing in popularity because it is immune to a lot of influences such as viscosity, solids forming within a liquid, foams, temperature and humidity. "It functions equally well in a lot of environments, which isn't the case with many traditional, point level switches," says P+F's product manager, Mike Mendicino.

Bailey adds that as environmental regulations continue to tighten, many processors are going the extra mile and adding some sort of continuous technology to at least one point level device. "This provides redundancy and reliability and shows a tremendous commitment to trying to prevent overspills," says Bailey.

are also growing in popularity. "These devices can be set so that an alarm is sent to the control system and at the same time an emergency text can be sent to the field tech or plant manager to notify them of a problem with level," he says. The technology is also advancing to the point where it can perform self diagnostics via a sensor check or complete check within the electronics or sensor itself and send alerts or alarms, allowing someone to make changes via the control system rather than sending out a technician to determine the problem, Boisvert adds.

Joy LePree

FIGURE 3. This ultrasonic technology calculates tank level or volume while the network or Internet communications capability provides access to data from virtually anywhere

ment independent of density, temperature and dust and can also be used with foam on the surface. The Pulscon TLC is usable as a single measuring cell or can be integrated into a Profibus PA system.

Using the data

Similar to P+F, other device providers are making advances in their technologies that help deal with data, but there's more than one way to skin the data management cat. "The idea of improving level measurement devices around the information side of things versus making the sensors themselves more proficient at measuring level is gaining in popularity," says Jon Cox, applications engineer with Automation Products Group (Logan, Utah). "As a result we are seeing more products that provide information through cell networks or the Internet or the dispersion of information directly through the sensor."

His company, for example, recently launched the LOE network/ Web enabled ultrasonic sensor. The ultrasonic technology calculates tank level or volume while the network- or Internet-communications capability provides access to data from virtually anywhere. There are two programmable solid-state relays for pump and alarm point control and email, and text message alerts are sent whenever user-defined values are reached. Adjustments can be made via built-in web pages so that no programming software is required.

Siemens' Boisvert adds that wireless continuous-measurement devices



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Fractionation Column

Did somebody say pilot plant?

uring the first three days of last month's AIChE Annual Meeting (November 7–12; Salt Lake City, Utah), like many attendees, I hopped from session to session listening to those papers that were of the most interest to me. Generally, I attended the energy, environmental and separation sessions. I saw many graphs, tables, process flow diagrams and conclusions. I am guessing that almost half of the projects that the speakers described were computer simulations. Of those computer simulation projects, very few described how their models were corroborated.

Regarding separations, for example, I do not remember any presenter stating that their thermodynamic models were checked against laboratory data, and, many of the systems being studied were non-ideal and highly nonideal. Meanwhile, I saw biofuels computer studies that failed to use the "f" word, filtering.

That made Don Glatz's presentation on the fourth day all the more refreshing. I do not recall whether he performed computer simulations of the processes that he studied, but, if he did, he had real pilot-plant data to corroborate the models employed by his computer software. Don works for Koch Modular Process Systems (KMPS; Paramus, N.J.; www.modularprocess.com). Don's slide presentation included drawings and photographs of a Houston pilot plant. KMPS had performed liquid-liquid extraction testing on two completely different feed streams, one using a Karr reciprocating plate extractor, and one using a Scheibel rotating disc contractor. The columns, the pumps, the valves, the





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

feeds and the products were all *real*.

That said. I am worried about the next generation of chemical engineers - the Facebook/Youtube/Twitter gang of today's High Schools, the 100-textmessages-per-day teenagers, "hardware and software geniuses" according to their helicoptering parents. Eventually, a significant fraction of them will be asked to actually pump, catalyze and boil some real fluids. Valves will need to be opened and closed. Columns will need to be pressured up. Hands will need to get dirty. The aptitudes of the next generation might reside elsewhere. Compounding my worries is the appearance that the U.S. governmental agencies that approve grants seem to devote just as much money to computer simulation work as they do pilot planting. Computer simulations certainly save money, but corroborations of that work with pilot-plant and industrial data will certainly sometimes be required.

It would be foolish of me to be computer software averse. I grew up performing thermodynamic and process simulation work - carrying 8-in. thick decks of input cards back and forth to the computer window associated with my company's mainframe computer. I loved it, but eventually I had to go into real plants to check the willingness of Mother Nature to abide by my simulations' wishes. She was never completely compliant.

I salute the pilot plant engineers and technicians of our industry. I also salute the simulation engineers. Clearly, we need them both. Someday, I would like to introduce them to each other. Mike Resetarits

resetarits@fri.org

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Department Editor: Scott Jenkins

Project design decision-making: Option lists

Most of the more than \$100 billion in capital spending and \$1.2 trillion in prodution costs spent annually by the chemical process industries (CPI) is committed by decisions made in the early phases of process development and plant design. A more disciplined approach to decision-making that fosters early attention to costs and more thorough consideration of available options has the potential to lower costs for most projects and better balance capital and production costs.

The Economic Design Model is a method to formalize this disciplined approach to cost considerations [1]. The model has three phases: setting project objectives; creating a thorough and comprehensive list of options; and conducting an economic analysis of the technically workable options. The model is helpful for early-stage decision-making (product and process development, feasibility evaluation and conceptual phases). Large amounts of money are at stake in these early phases because the decisions made there set the future economic framework.

CREATING OPTIONS LISTS

The heart of the Economic Design Model in a process- or plant-design project involves creating a full list of options for completing the project. To identify the best of the available options, engineers must diverge early in a project to consider multiple options before converging on a decision. Doing so requires carefully and deliberately constructing an option list that is more comprehensive than a typical one. Experience indicates that more than one workable technical option usually exists, so the decision comes down to economics. Economics decisions, in turn, are best made on the basis of balancing capital and production costs - evaluating whether it makes sense to spend additional capital to reduce production costs, or if it is better to reduce capital outlay but be left with higher production costs.

Reasons why option creation falls short Three main reasons exist for why the consideration of options in a design project often falls short. One involves the heavy schedule demands placed on engineers. These demands can create the perception among engineers that there is not sufficient time for anything other than to find a workable solution to the design problem. Another can be viewed as a consequence of standardized plant design, which generates a reluctance to make changes that would alter the standardized designs (even when improved technology is available or economic conditions have changed). The third reason for why option lists often fall short of the mark is that creating effective ones can simply be hard to do.

Option creation tools

The need for increased rigor in creating options lists may be more difficult, but there are several tools that can help greatly. While brainstorming and its variations can be used for generating options, there are additional tools that bring discipline to the option creation process and help overcome the factors that could cause

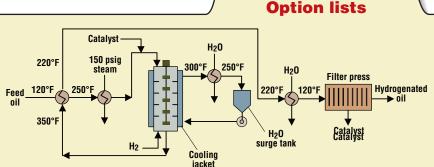


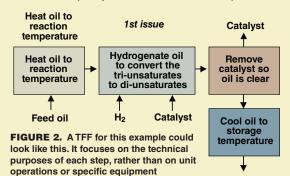
FIGURE 1. An engineer might develop a flowsheet like this one to meet business and technical objectives for a hypothetical oil hydrogenation example

the option lists to fall short. The tools promote divergence before design answers were selected, promote higher levels of understanding of the design and help spark creativity.

Flowsheet development. The first tool is an alternate methodology for development of the design flowsheet. The concept is to focus on the technical purpose of each step in the process, rather than specific

unit operations or equipment types. The resulting block diagrams, called technical function flowsheets (TFFs), help to slow convergence to design specifics until all possible options have been thoroughly considered. Technical function descriptions are verb-object combinations, rather than nouns usually used to describe unit operations. For example, in the case of a hypothetical oil hydrogentation process, technical function phrases could be: 'separate solids from the oil," "heat oil" or "react oil with hydrogen." These descriptions are intentionally less specific and more generalized than the descriptions of unit operations that could fulfill those technical functions, such as pressure filter, shell-and-tube heat exchanger, and multistage stirred-column reactor (Figures 1 and 2). TFFs are backed up by material and energy balances (often based on pilot plant data) and a detailed table that explains the purpose for each function, lists important operating conditions (temperature, pressure, flow, and so on) and describes the basis for selecting operating conditions. Unit operations guides. Use of the TFF methodology sets the stage for the option list creation. When it comes time to select unit operations for the different technical functions, the Unit Operations Guides, as published in [2], will help engineers define most of the potential options. Eight guides are available, covering the following topics: blending and mixing; drying (water removal only); heat transfer (inlcuding evaporation); mass transfer (including crystallization); material transport; mechanical separation; reactions; and size modification. Question lists. Intended to increase the depth of understanding about a process, the question list is a series of process-related questions that will lead to more design options and higher quality designs. It is often the case that general

Technical Function Flowsheet, Oil Hydrogenation (with permission from CRC Press)



questions that lay the foundation for a project are not addressed well, either because the answer appears at first glance to be obvious, or because of the perception that there is not enough time to answer them sufficiently, or because no one thought to ask the question in the first place.

The general process and process-interaction questions address broad project issues, such as the following examples: Should a product intermediate be bought or synthesized? Should the process be a batch or continuous one? What grade of a raw material should be purchased? Should recycle or purge streams be used, and do those streams need to be treated before reuse or disposal? How does this unit operation affect the downstream process?

USING THE MODEL TO FINALIZE

Once a comprehensive list of options exists, project engineers can move on to the next stage of the Economic Design Model, in which each option is analyzed to determine whether the option can meet the technical needs of the project, as well as to determine which of the technically feasible is the most economical option.

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Note: This edition of "Facts at Your Fingertips" was adapted from Capital and Production Costs: Improving the Bottom Line, an article authored by Thane Brown that appears in the January 2010 issue of *Chemical Engineering*.

People

WHO'S WHO



Rahie

Hansen

Oseco (Broken Arrow, Okla.), a manufacturer of rupture disks, names Darren Doyle vice president of sales.

Koch Membrane Systems (Wilmington, Mass.), a supplier of membrane filtration technology and systems support, promotes Hamid Rabie to chief operating officer.

Abresist Corp. (Urbana, Ind.), a provider of abrasion-resistant protective linings, promotes Mark McCombs to sales representative for parts of Indiana, Ohio, Kentucky and Michigan.

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Robert D. Hansen becomes president of Dow Corning Corp. (Midland, Mich.). Stephanie A. Burns remains chairman and CEO.

Design engineer Wei Chen joins **Applied Seals North America** (Newark, Calif.), a supplier of perfluoroelastomer sealing products for ultraclean manufacturing environments for nanoelectronics and biomedical products.

Awesco (Albany, N.Y.), a distributor of gas and welding equipment and





McCoole

Stephanou

Y

services, promotes Dale McCoole to director of sales.

Bayer Technology Services (BTS; Leverkusen, Germany), which provides engineering, construction and process optimization, names Jim Stephanou head of BTS Americas, located in Baytown, Tex.

Christel Fenge joins Sartorius Stedim Biotech (Goettingen, Germany), as vice president of marketing for fermentation technologies. Suzanne Shelley

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Cover Story

Heat Transfer Fluid Leaks: Break the Fire Triangle

Extreme processing temperatures present the greatest risk. Know where leaks are most likely to occur and how to prevent them



Conrad E. Gamble and **Matthias Schopf** Solutia Inc.

anagement of process plants requires the understanding and application of the fundamental tenets of safe design, construction, and operation of each facility. In that context, many years of practical experience with systems that use very high-temperature organic heat-transfer media has led to much knowledge regarding design features to adopt and those that should not be repeated.

For the media itself, the severe stresses of extreme operating temperatures involved can sometimes narrow the number of candidate fluids to one: the eutectic mixture of diphenyl and diphenyl oxide (DP:DPO; Figure 1). This article shares best practices that have been developed over the years to ensure the safest possible use of this heat transfer fluid (HTF). Effective methods to identify and determine the order of magnitude of leaks are discussed. Meanwhile, a review of the concepts necessary for effectively maintaining a leak-tight system is presented. And, for occasions in which unexpected leaks do occur, a thorough

explanation of the fire risk is offered and approaches that can minimize ignition potential and property damage are addressed.

INTRODUCTION Basics of system requirements

Process plants require heating and usually cooling capabilities. A "typical" HTF system is composed of an energy source, such as fired heaters or waste heat recovery systems; pumps to force the fluid flow; an expansion tank to absorb the volume expansion of the fluid; and last but not least, a heat consumer. High-temperature heattransfer systems are usually closed systems, and hence, a release of fluid can typically only occur in case of accidents or malfunctions. Heat transfer media used in such systems are usually water-and-steam, water based fluids, mineral oils or synthetic heattransfer fluids specially designed for this purpose. Based on their physical and chemical properties, such fluids imply certain handling risks in addition to the general risk of operating an HTF system.

Besides classical process plants in the chemical, petrochemical and plastic industries, two other applications have recently come into the focus by utilizing large volumes of DP:DPO HTFs (see box entitled Large volume applications): electrical energy production by concentrating solar power (CSP) and converting natural gas into synthetic oils in a process called gas to liquids (GTL).

As with all HTF systems, the design must accommodate the volume expansion of the heating fluid, which is typically provided by an expansion or surge tank of sufficient volume and headspace. For plants using DP:DPO near its maximum temperature of 400°C (750°F), the volume expansion from ambient temperature is greater than 30% [1]. The expansion vessel is typically pressurized to keep the HTF in liquid phase and is equipped with a vent system to permit the exit of nitrogen, degradation products, and a portion of DP:DPO into the ullage vent-collection system. The extensive network of HTF handling piping, instruments, and vessels, combined with



FIGURE 1. Extreme operating temperatures sometimes narrow the number of candidate fluids to one: the eutectic mixture of diphenyl and diphenyl oxide (DP:DPO)

elevated vapor pressures, can increase the potential for leaks from systems if adequate design and maintenance measures are not also incorporated.

Incident history of leaks, fires

Examination of the leak history from DP:DPO handling systems indicates that the primary sources are flanged connections, flexible connectors or rotary joints, and pump seals. Fireresistant gaskets used in DP:DPO service can require substantial compressive force on the gasket face. In cases where insufficient flexibility is provided in piping networks (Figure 2), the resulting force applied to the flange pair reduces compression on a portion of the gasket, leading to leakage. Such leaks can sometimes be addressed by tightening sufficiently, but only as a temporary repair. Permanent repair will require gasket replacement and improved piping flexibility.

Stainless-steel flexible hoses were first installed on the early solar energy generating systems (SEGS) in the California Mojave Desert in the 1980s. Some of the original flex hose remain in service today, while most has been replaced over time. Experience in the mode of failure finds that the hose develops small cracks through which the DP:DPO fluid seeps slowly at first. The leakage can be visually observed as a darkened area on the outside of the hose assembly. Prompt maintenance can permit the piping section to be shut down and isolated for hose replacement prior to a rupture. When proper experience and care is applied during hose installation, the hose can be kept free of the torsion and misalignment that reduce its service life. It is important to follow hose manufacturer recommendations on inspection and replacement frequency, as the hose has smaller wall thickness than rigid pipe.

Rotary joints have the advantage of using greater wall thickness for a more robust joint, yet have the flexibility to support the daily cycling demands of the mirrored rows of heat collection elements (HCEs). Designs can incorporate high-temperature, metal-to-metal seals and can include provision for injectable, graphite-based packing for

LARGE VOLUME APPLICATIONS

CSP plants

Concentrating solar power (CSP) plants require a reliable heat-transfer fluid to efficiently absorb the sun's energy and transfer it to the steam generator. The HTF is the single component of the system, which traverses the solarenergy collector field (mirror arrays) and the power generation unit (power block). The uninterrupted circulating flow of the fluid and thermal energy is essential for system operation. Any resistance to flow reduces demonstrated efficiency from theoretical levels, and interruptions in flow can take the entire system offline.

[']The designs of existing and planned CSP plants that utilize DP:DPO heat transfer fluid have similar features. Large circulation pumps capable of delivering flowrates up to 10,000 gal/min or more provide the motive force for circulating the fluid through field supply headers of decreasing diameters extending to the farthest mirror rows. HTF flowrates through the individual heat-collection element (HCE) rows are regulated to maintain the desired HTF temperature in the return headers. The heated fluid is used in large shell-and-tube heat exchangers to provide superheated steam, which is then utilized in traditional power turbines. The HTF continually circulates throughout the operational hours to support the movement of solar energy from direct solar and thermal storage systems, if provided.

GTL-processes

In the gas-to-liquids (GTL) process, natural gas is chemically converted into fuel liquids. The base is the Fischer-Tropsch (F-T) process of converting mixtures of hydrogen and carbon monoxide into liquid hydrocarbons. In general, GTL technologies are three-step processes. The first step is conversion of the natural gas into a hydrogen and carbon monoxide mixture usually called synthesis gas or syngas. The second step is a catalytic reaction of this syngas into a mixture of hydrocarbons (F-T). Finally, the last step is a cracking and conversion of the hydrocarbons into the desired final products [2]. Due to high temperature requirements of some of the process steps, DP:DPO heat transfer fluid is utilized for process heat control.

> maintaining adequate sealing. Some users, however, have reported that some of the rotary joints failed more, and without warning.

> Whether from flexible hose or rotary joints, the close proximity of these devices to nearby ignition sources can lead to the ignition, or even autoignition of a released cloud of HTF mist if surface temperatures exceed 1,100°F (593°C). For safety of personnel, response typically involves remote isolation of the piping circuit to allow the fire to burn itself out in remote or noncongested areas. Alternate measures may be appropriate to protect against peripheral damage in more congested areas. Extinguishing the fire prior to stopping the HTF release may result in later, repeated ignition. Operating practices must incorporate the frequent monitoring of the integrity of field piping systems through regular inspections and/or remote video surveillance, or both.

> Pumps in DP:DPO service can have single or double mechanical seals. Some of the pumped liquid may be required to adequately lubricate seal-

Cover Story

ing surfaces during operation. Excessive temperatures at the seal faces can also vaporize the HTF resulting in no lubrication and ultimately cause mechanical damage to seal face materials. This mechanism can create particulate matter that erodes the seal face and create a separation of seal faces, thereby resulting in leakage. A seal flush can be effective in minimizing excessive seal wear by removing harmful corrosive or fouling deposits with filtered fluid [3].

It should be noted that while infrequent insulation fires are experienced very-high-temperature organic in HTF systems, it is extremely rare in DP:DPO systems. DP:DPO has a lower relative-normal boiling point than other high temperature HTFs, permitting its more rapid evaporation from soaked insulation before it can be heated to its autoignition temperature (AIT) by the oxidation exotherm within porous insulation. Cellular glass insulation has proven most effective in interrupting the mechanism of HTF insulation fires among most organic HTF chemistries.

Consequences of leaks, fires

There are no positive consequences of leaks and fires, except perhaps the heightened awareness of design or construction flaws, which leads to improvement. The immediate negative consequences include risks to human life and health from released fluid ignition and fire fighting response, or inhalation of partially combusted hydrocarbons. Personnel should be trained and equipped to respond by remotely isolating systems, and staying out of the path of smoke, vapors and liquid runoff. In CSP plants, the amount of fluid contained in a 100-m mirrored row with 70-mm O.D. heatcollection elements will be limited to nearly 0.33 m^3 (87 gal), plus the volume contained in piping up to the isolation valve(s). Much of the HTF released from piping containment will flash into the vapor phase, and then quickly condense into a mist cloud. The balance will rapidly cool in contact with equipment and earth, remaining in liquid phase. Contaminated soils will require disposal or treatment per regulatory require-

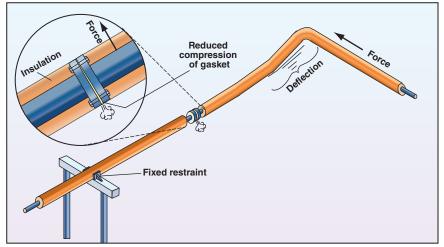


FIGURE 2. Poor piping flexibility can cause leaks

TABLE 1. DP:DPO KEY FIRE-RELATED PROPERTIES					
Characteristic	Method	°C	°F	Vol. % in air	
Flash point (open cup)	ASTM D-92	124	255	-	
Fire point	ASTM D-92	127	260	-	
Flash point (closed cup)	ASTM D-93	110	230	-	
Autoignition temperature	ASTM E-659	601	1,114	-	
Lower combustion limit	-	149 260	300 500	0.8 0.5	
Upper combustion limit	-	149 260	300 500	3.3 6.2	

ments, possibly including incineration or bioremediation. Hot fluid released and collected should not be reused, as it will have become oxidized and perhaps contaminated. Equipment wetted with HTF condensate should be cleaned for proper housekeeping, and to remove residue that could potentially fuel a future fire.

In areas where leakage is of highest risk (such as flex hose or rotary joints, pump seals, valves or instrument manifolds, and so on) efforts should be made to limit the amount of instrumentation and wiring potentially exposed to fire. Doing so will minimize the downtime required to return the equipment into operation. Any equipment, piping, instrumentation, insulation or controls that have been exposed to fire or DP:DPO spray should be inspected by qualified engineers and technicians for reliable operating integrity, prior to returning it into service.

FLUID CHEMISTRY AND INHERENT PROPERTIES Formulation

DP:DPO heat transfer fluids are a eutectic mixture of diphenyl and diphenyl oxide. These two components exhibit the highest thermal stability available among organic heat-transfer media. Their eutectic ratio ensures the lowest freeze point possible, thereby creating the widest working range of temperature. As diphenyl and diphenyl oxide exhibit tremendous thermal stability, the weakness of the specific fluid formulation would then lie in its impurities. Therefore, it is most desirable to have very high assay of the two primary constituents, and limit the percentage of impurities possible. Doing so protects thermal stability of the fluid and also ensures that the physical properties of the HTF will have minimal deviation from its key components.

Low chlorides content (< 10 ppm) ensures long life of stainless-steel



FIGURE 3. Pipe flanges are potential sources of leaks. Design considerations are important here

system components without excessive risk of stress-corrosion cracking. There are SEGS systems, for example, that have been utilizing low-chloridecontent heat-transfer fluid since the 1980s with no reported cracking issues. Low chlorides content should therefore be a requirement of DP:DPO fluids used in plants to help maintain longterm mechanical integrity.

Tendency toward leakage

Operators of high temperature, organic heat-transfer fluids should be aware of the potential for leakage of DP:DPO from components that may be leak-tight when pressure tested with water. It has been noted that, "the physical and solvent properties of organic fluids allow the fluids to penetrate ordinary valve and pump packing and bleed through porous materials including some cast irons" [4]. For piping flanges, fire-resistant gaskets are preferred, which can require higher compression to adequately seal as compared to softer gasket materials. To help minimize leak potential and keep the DP:DPO confined within the system, engineers and safety professionals have developed key practices as outlined later in the section on preventing leakage.

When DP:DPO eutectics freeze, the material contracts in volume by over 6%. If the product then melts between frozen plugs of product or other mechanical boundaries, tremendous pressure can result, possibly leading to release of the product through the weakest constraint, such as flanged connections, valve stems, pump seals, and so on. When thawing a frozen section of piping or equipment, it is very important to accommodate the expansion in volume into unobstructed piping or equipment.

Key fire-related properties

Key properties of interest in gauging the potential for fire with organic heat transfer fluid are provided for DP:DPO in Table 1.

Minimum ignition energy (MIE) is a measure of an amount of energy below which an explosive mixture in air will not be affected, and above which the mixture can be affected. Measured values for heat transfer media are not readily found in published literature. As T. H. Pratt explains, the MIE concept applies only to capacitive spark discharge and that, based upon numerous factors, "one must recognize that MIEs vary with most everything" [5]. Taking this into account, NFPA 77-2007 states that the MIE for most saturated hvdrocarbon vapors is near 0.25 mJ, and that mists can have MIE values one or two orders of magnitude higher [6]. This value can have considerable deviation from the MIE experienced in actual field environments.

PREVENTING LEAKAGE, COMPONENT SELECTION Reliable system design

Complete guidance on system reliability cannot be adequately covered within the constraints of this article; however, many important concepts are provided herein in support of designers and operators. Economics of projects often require use of the lowest cost materials that are fully compatible for the service to be utilized. Since the mid-1970s and the development of the first low-chloride (< 10 ppm) formulation of heat transfer fluids, stainless-steel metallurgy for instrument tubing and specialized equipment could be used without fear of chlorides-related stress cracking potential. It is important to note that any significant extension of operating temperatures above those currently used in DP:DPO HTF systems would require more exotic and costly alloys for piping and equipment, as well as the development of satisfactory, new heat-transfer fluids. Useful reference standards and codes are included in the References section [7-14].

With the HTF being significantly above its normal boiling point during operation, adequate overpressure protection in the form of pressure relief valves (PRVs) and rupture disks must be provided with adequate capacity for relief between selected points of isolation. With proper design considerations, the PRVs can be installed in series with rupture disks to minimize fugitive emissions from the system. General good practices for HTF system design are typically available from the HTF manufacturer, including information on system components, filtration and expansion tank design.

Piping, flanges and gaskets

Seamless carbon steel has been demonstrated as an appropriate piping material for use with organic HTFs in plants up to their maximum bulk operating temperatures. Threaded fittings in piping are to be avoided in preference to allwelded construction. Graphite-based, paste thread sealants have demonstrated marginal success with threaded connections that cannot be avoided, such as instrument connections, pumpcasing drain plugs, and so on.

Cover Story

With any metallurgy employed, the linear expansion and contraction of the piping must be accommodated by use of expansion loops or flexible connection members, and pipe supports should be generously spaced to prevent sagging. When thin-walled expansion joints are selected, manufacturers' recommendations for inspection and replacement frequency should be strictly followed. Piping should be provided with "shoes", which can slide unrestrained axially on the pipe supports with thermal cycles. Where flanges are necessary, Class 300 and Class 600 ring-joint flanges or raised-face flanges are commonly used. While the number of flanged pairs should be minimized to reduce the number of potential leak points and fugitive emissions, designers should consider maintainability and install such connections to all major pieces of equipment and in other key locations for ease of servicing equipment (Figure 3).

When used with raised-face flanges. gaskets should have a metal ring for blow-out resistance and graphite-filler within 316 stainless-steel, spiral windings for fire resistance. A number of manufacturers offer such gaskets, and some designs provide more-exotic-alloy spiral windings for improved memory and longer use-life for cycling temperature services. A key necessity for flanged connections is to ensure that adequate and uniform sealing compression (seating stress) is provided on the gasket faces. Data on compression force of gasket surfaces are available from gasket manufacturers, which will then determine proper bolt/stud/nut selection and torque requirements.

For non-circular gaskets, such as in some pump casings and valve bonnets, foil-inserted graphite has been successfully used. Where larger gaskets are required for heat exchanger heads with partitions, and so on, foilinserted graphite, tang or corrugated metal-inserted graphite can be used. With any gasket type selected, proper installation techniques prescribed by the manufacturer should be followed.

Pumps and pump seals

Pumps in HTF service are generally of cast steel for withstanding the stresses of thermal shock. Pumps in

high temperature service can have double mechanical seals, or can be of a sealless design. The pumped fluid is used to lubricate and cool bearings in either type. Excessive temperatures at mechanical seal faces (typically tungsten carbide, stellite, or silicon carbide) can vaporize the HTF resulting in no lubrication and mechanical damage to seal face materials. This mechanism can create particles that can erode the seal face and create a separation of seal faces resulting in leakage. Therefore, stuffing box and seal gland cooling is important to maintain lower temperatures and also improved lubricity of the HTF. A seal flush can be effective in minimizing excessive seal face wear and particle accumulation on flex metal bellows by removing harmful deposits with filtered fluid [15].

Valves

Valves in DP:DPO service may include forged- or cast-steel, or stainless-steel bodies, balls, plugs and disks. 13-Chrome (Cr) stems and 13-Cr or stellited seats should be considered. Fire-resistant graphite packing should be specified in select configurations available. Bellows seal designs can provide physical barriers for reduced emissions and leaks. With frequent updates of valve feature availability, the designer should always consult with the valve manufacturer to consider the latest innovations available. Soft-seat materials should be avoided since they can burn out in case of fire, potentially adding to the complexity of the HTF release. Small valves with welded end-connections should be considered to reduce potential leak points, and larger valves should be considered with flanged end-connections. Connecting piping should have adequate flexibility to minimize torque induced from thermal expansion and contraction of piping applied to flanges, which may develop leaks.

Maintenance practices

Simply put, three keys for adequate maintenance practice are as follows:

- 1. Respond to identified leaks promptly with repairs
- 2. Repairs should address the cause of

the failure, and not just the consequences of the failure

3. Learn, document, and practice the recommended preventive maintenance of the equipment

LEAK DETECTION

Human senses

DP:DPO fluid can be operated in liquid or vapor phases. When there is leakage, even above its normal boiling point, the vapor emitted can quickly condense to form a visible, near-white mist cloud. The cloud quickly increases its transparency as it dissipates. For small leaks it may be possible to observe liquid droplets present at the source of the leak, such as valve stems, pump seals, flanges, and so on, as well as on the ground. As mentioned earlier, a small leak from a flex hose can appear as a darkened, wetted area. When cool ambient conditions permit, frozen DP:DPO may be observed as white crystalline solid. Visual detection capability for leaks can be greatly enhanced by use of remote video-surveillance monitoring.

The DP:DPO eutectic mixture has been measured to have an odor threshold of 9 parts per billion (ppb) in air, making detection by odor possible without exceeding established airborne exposure limits for either component. In indoor areas, the airborne HTF may not dissipate readily, preventing easy tracing back to the leak point. In outdoor environments, the odor is more closely localized around the leak point due to the more rapid dissipation of the components in air. While odor may be a sensitive detection technique, the use of instrumentation is a preferred detection-and-measurement method with superior quantifying capability for leak tracing.

Specialized instrumentation

Instruments using the principal of photoionization are well suited for use in CSP and process plants. Typically, these instruments can readily detect DP:DPO concentrations in air as low as the ppb range. For realtime personnel exposure monitoring, the ppb sensitivity is appropriate to quantify exposure at levels below established occupational exposure limits for the fluid's components. For maintenance

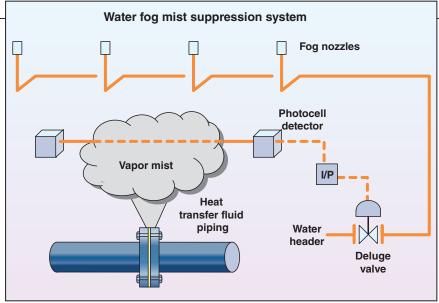


FIGURE 4. Vapor or mist detection systems automatically detect the presence of clouds of DP:DPO and can automatically trigger water-fog deluge systems and alarms, initiate forced ventilation, and de-energize area equipment

needs, parts-per-million (ppm) sensitivity is sufficient to determine orders of magnitude of identified leak sources so that repairs can be scheduled on a prioritized basis. The instruments can be purchased as handheld units for lightweight mobility, or as fixedmount, continuous area monitoring stations. The devices pull air samples through the instrument for measurements. Drawbacks include inability to distinguish DP:DPO vapor from other measureable organics and combustibles (although, this is not a problem in most CSP plant areas), and tendency to condense vapors within the sample inlet when sampling high concentration leak points. The internal condensation can require the instrument to be offline until disassembled for thorough cleaning. Experience in handling the unit can quickly overcome this issue. Cost of these instruments currently varies from approximately \$3,000 for small, ppm handheld models, to \$5,000 and higher for the ppb and fixed-mounted systems. They can be purchased with intrinsically safe certifications for use in hazardous areas.

To prevent the potential ignition of vapor- and mist-cloud leaks within indoor areas with reduced ventilation, custom-developed instrumentation has been installed to automatically detect the presence of clouds of DP:DPO with the capability of automatically triggering water-fog deluge systems and alarms, initiating forced ventilation, and de-energizing area equipment with a continuous monitoring capability. This design has incorporated the use of multiple-sensor actuation to avoid accidental activation from only one monitor (Figure 4). The use of water-fog systems has been found in testing to rapidly reduce airborne DP:DPO concentrations to below combustible concentrations in air [16].

It is important to follow the manufacturer recommendations with respect to the use and maintenance of these instruments.

Process level indications

Today's facilities will typically have installed instrumentation for the routine measurement of liquid level in process vessels, including bulk storage tanks, surge or expansion tanks and ullage vessels, condensate tanks, and others. Modern instrumentation often permits excellent level-trend capability so that response time to unexpected level changes is greatly enhanced by use of configured deviation alarms with a distributed control system (DCS). Common suitable instrumentation types can include displacer, differential pressure and radar within their respective design limitations of temperature. Externally mounted floats within stainless-steel chambers can provide 0 to 100% level indication and remote, continuous monitoring on DCS screens. Changes in liquid level in vessels can be a somewhat crude, but important component of detection material loss.

Potential fire scenarios

The prerequisite to a fire is a leak from point sources, such as described in the earlier section on incident history.

Insulation fires. Insulation fires are not uncommon with high-temperature, organic heat-transfer fluids. However, in one extensive study of such fires by Britton, he concludes, "Least prone should be DP:DPO types, which have very high AITs (above 500°C) and relatively low flash-points of around 130°C" [17]. DP:DPO-soaked insulation apparently avoids the insulation fire mechanism by its more rapid rate of evaporation out of the insulation than other higher-boiling heat-transfer-fluid chemistries, while avoiding the close approach of its autoignition temperature.

Spark- or flame-produced ignition. With DP:DPO fluid, a more expected cause of fire would be from spark- or flame-produced ignition. Releases of liquid DP:DPO above its fire point of 127°C (260°F) are susceptible to ignition from area sources, such as exposed electrical contacts, spark-producing tools, open flames and so on. However, hot HTF can rapidly cool once released into its environment, both by evaporative cooling and by conducting thermal energy into the heat sinks provided by the ground, equipment and so on. A good design practice is to provide sloping of underlying surfaces such that any liquid release can drain away from process equipment, thereby minimizing potential ignition. Also, once any release of HTF is observed, all sparkproducing work should be halted until the situation has stabilized.

Autoignition. Autoignition of a DP:DPO cloud has been reported in one CSP plant where the HTF leaked in close proximity to the heat collection element (HCE). When a vapor or mist cloud of DP:DPO within its combustible limits of concentration in air contacts a surface at temperatures above the autoignition point of the HTF, it can ignite. The cloud is consumed rapidly and can continue to burn at the source of ongoing leakage until its flow is stopped. Where possible, designers are encouraged to consider incorporating remote isolation capability for piping circuits, so that the fuel to the fire can be safely interrupted.

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FIRE PREVENTION & FIGHTING TECHNIQUES Breaking the fire triangle

Three necessary components make up the fire triangle (Figure 5) for an HTF system: oxygen, an ignition source and fuel (DP:DPO). How do we address each of these successfully to avoid their fiery combination?

Oxygen. Within process vessels, provide blanketing with dry, inert gas. Nitrogen is typically used, and should be a minimum of 99% purity.

Ignition sources. HCEs of CSP plants are the previously identified point of AIT concern, as the surfaces can reach temperatures above the DP:DPO AIT, particularly when leakage reduces the flowrate of the HTF through the HCE. To date there has been no known provision made to avert the potential drifting of a DP:DPO cloud across the HCE. Upon the first detection of leakage from piping components in the proximity of potential ignition sources, prompt maintenance should be scheduled for repair or replacement. Potential for open flames (such as smoking, welding and cutting torches) can be addressed by strict compliance with adequate administrative controls. Electrical area classification design should be properly addressed so that it conforms to applicable code requirements. This should establish a minimum radius around each potential DP:DPO leak point, and should be determined according to specified standards for the requirements of electrical enclosures, switches, motors and so on.

Fuel (DP:DPO). Use of HTFs at very high temperatures often requires an organic heat-transfer fluid of high-

est thermal stability, which is the DP:DPO eutectic fluid. The proper approach to eliminating the presence of DP:DPO outside of its containment is by careful design and installation of the system and its components, and incorporating a culture of effective preventive maintenance and inspections for the system's proper operational integrity. For the unexpected leaks, designs incorporating remote isolation capability can help minimize DP:DPO release and personnel exposure during these events.

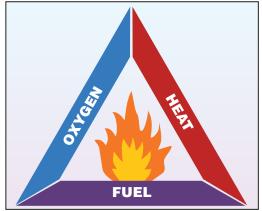


FIGURE 5. Oxygen, an ignition source and fuel (DP:DPO) are the three necessary components of the fire triangle for an HTF fire scenario

Fire fighting response

For small or incipient-stage fires with a limited and minimal source of fuel (DP:DPO), the use of a handheld fire extinguisher can be an effective and appropriate selection to extinguish a fire. Approach to the fire should be from upwind to avoid breathing partially combusted materials, with standard sweeping discharge of drychemical or carbon-dioxide media. Consider placement of 20-30 lb (9-14 kg) dry-chemical fire extinguishers in easily accessible locations at each level of structure where the fire potential exists in HTF handling areas [12]. Personnel must be properly trained in the operation of selected extinguishers and techniques for approaching and extinguishing HTF fires.

Larger dry-chemical units are available on trailers and can be quickly transported to distant locations within the expanse of a facility. It is recommended that personnel intended to operate such equipment receive specialized training and certification prior to being authorized to respond. Onsite fire brigades can address fires much more rapidly than offsite fire departments, and can help to minimize potential damage, system downtime and repairs.

As mentioned earlier, fires involving larger releases of HTF in non-congested areas of a facility that are beyond the capability of incipient-stage response techniques are perhaps best managed by remote isolation of the DP:DPO source, and allowing the fire to burn out without risk to employees. Runoff of liquid should be directed away from areas that might result in costly damage, extensive downtime and repairs. Water monitor stations equipped with water fog nozzles may provide needed protection and cooling of equipment in certain areas, which should be assessed in planning for a fire-water management scheme.

Indoor areas may require sprinkler protection when potential leak points

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from HTF piping and equipment exist. Consideration may be given to automatic leak-detection systems, coupled with the capability to de-energize area electrical equipment and activate zones of water fog deluge systems and alarms. If ventilation is activated, it may negate the desired effect of water fog sprays in scrubbing the HTF mist from the air. Pooled or contained DP:DPO liquid settles to form a bottom layer once it has cooled to a temperature at which its density is greater than the density of the water.

For any of the above approaches for fire response, consultation with insurance providers is recommended.

FINAL THOUGHTS

The primary intent of this paper is to stress the importance of good design, installation, and maintenance of equipment in DP:DPO handling facilities in preventing HTF release and fire events. By keeping the HTF within the piping as intended by robust design, the frequency, severity, and consequences of leaks and fires can be minimized or even prevented altogether. All components of a complex HTF system cannot be addressed within the context of this paper, therefore the designer and facil-

edgeable specialists representing suppliers of the heat transfer fluid, piping components and specialty fittings, and insurance providers to properly protect against leaks and their consequences. Edited by Rebekkah Marshall

ity operator should consult with knowl-

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Filtration Centrifuges: An Overview Peter Schmidt Andritz KMPT GmbH

The first step in selecting the right centrifuge is understanding what types are available

o solve the task of solid-liquid (S-L) separation in the chemical process industries (CPI) a wide variety of methods are invariably used. Many of these methods are not mutually exclusive and include a selection of technologies that can be expediently combined to provide an efficient operating system.

A good understanding of the options available and details of the individual system modules can enhance the efficiency of the overall concept. For example, mechanical separation using pusher centrifuges has proved to be a very expedient intermediate stage for the dewatering of sodium bicarbonate downstream of the vacuum filter and before calcination. By reducing the residual moisture between filter discharge and centrifuge discharge, some 38% of the required evaporation heat can be saved; and just a fraction of the saved energy is required to operate the centrifuge. This helps to protect the environment and also saves costs.

This article presents a basic overview of different types of centrifuges with descriptions of how they operate and where they are applied in the CPI.

Some definitions

To begin with, a distinction is made between thermal and mechanical separation of solid products from liquids. Whereas thermal S-L separators can

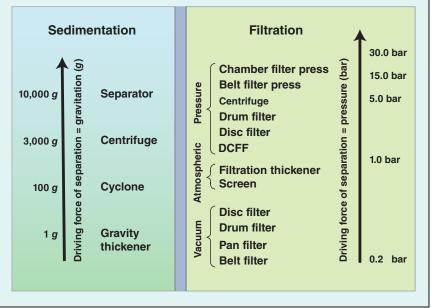


FIGURE 1. A classification scheme of the various types of equipment available for solid-liquid separation is shown here

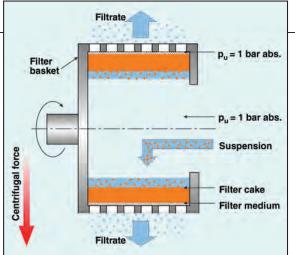
be grouped under the general term "dryer", it is not so easy to classify the equipment used in the first stage of mechanical S-L separation. For a finer distinction it is necessary to consider the flow directions of the solid and the liquid phases. If these are both in the same direction one talks of filtration; in the case of opposing directions the process is referred to as sedimentation (Figure 1). There is also the special case in which the flow directions of the solid and liquid phases are at right angles to each other, this is referred to as cross flow filtration.

In most sedimentation processes, the difference in density between the solid and liquid phase is utilized, but it is also possible to use electric or magnetic fields for separating purposes. To some extent, the natural sedimentation in the earth's gravitational field is used to this end, for example, by gravity thickeners. This natural sedimentation is enhanced by also superimposing a centrifugal field, as is done by cyclones and centrifuges.

Filtration, on the other hand, uses a filter medium that retains the solid phase while allowing the liquid phase to flow through.

Another way to distinguish filtration methods is to consider the driving potential that moves the liquid phase, or filtrate, through the filter medium. Separation can take place in the simplest of ways: using a screen in the earth's gravitational field or by imposing a pressure gradient. Such pressure gradients can be generated by applying a vacuum to the filtrate side. Here, however, natural limits are quickly revealed.

Far greater potentials are to be found when the pressure gradient is applied to the solids side using overpressure. This brings us to the focus of the article, filtration centrifuges, which are used when a cake-forming filtration is required.



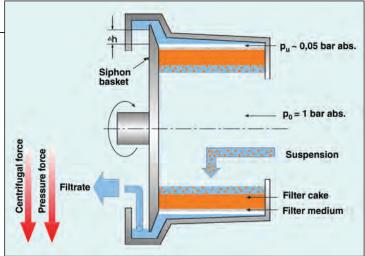


FIGURE 4. A horizontal peeler centrifuge with siphon basket back-

washes the heel while adding pressure for higher filtration efficiency

FIGURE 2. A cross section of a horizontal peeler centrifuge with screen basket is shown here

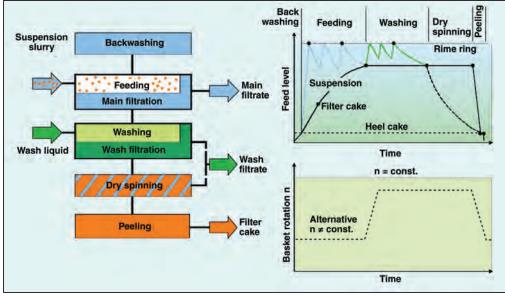


FIGURE 3. Batch process for a peeler centrifuge involves several steps

Filtration centrifuges

At the most basic level, nearly everyone is familiar with filtration centrifuges; this is the separation principle used in common appliances, such as washing machines and salad spinners.

The design of a centrifuge is usually always the same: a basket holds the filter medium, and the mixture to be separated rotates about an axis. Filtrate is spun outwards through the filter medium and the solids remain in the basket for easy discharge. In the simplest of centrifuges this operation is performed manually. On an industrial scale, however, the discharge is nearly always performed automatically.

Filtration centrifuges can also be further distinguished. On the one hand there are discontinuously operating centrifuges where the processes are carried out in batchwise steps, and then there are the continuous operating centrifuges where all process steps are carried out simultaneously.

In addition to this, there are many other features and variations in centrifuge design, for instance, the horizontal or vertical alignment of the rotation axis. In the various filtration centrifuge types, different combinations of these additional features have proven their worth in numerous applications. The following sections describe the evolution of such features.

Batch centrifuges

Peeler. Chronologically, the discontinuous or batch-operating peeler centrifuge represents the first generation, with the first horizontal peeler centrifuge launched in 1928 by Krauss-Maffei. As shown in Figure 2, these machines feature a cantilever-mounted basket that rotates in a closed process housing. The door on the front side

of liquid (washing); spinning of the liquid up to the desired residual-moisture level (dry spinning); and finally, the discharge of the cake from the basket (peeling) using a peeler blade.

Attention is drawn to the fact that during the peeling off of the cake this taking place at full speed in high quality machines — the peeler blade cannot be swung in flush with the filter medium (usually a mesh of plastic fibers or fine metal wires), as otherwise there is a risk of the mesh being damaged or peeled off. As a result, a residual heel is left in the basket that can be used again several times with many products. However, after a certain period of time, the heel becomes clogged with fine particles. Because of the increased filtration resistance. continued use becomes uneconomical and the residual heel has to be removed. This can be done pneumati-

tains, among other things, the motor for driving the basket shaft. In a typical batch run, the S-L mixture (feed) is first fed to the basket. Centrifugation then pro-

houses all the fittings re-

quired for operating a pro-

cess run. The drive part

mounted at the rear con-

is first fed to the basket. Centrifugation then proceeds through the following sequence of steps (Figure 3): cake formation on the filter medium in the supersaturated state (sedimentation); dewatering of the cake up to saturation (intermediate spinning); washing of the cake with a new supply

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FIGURE 5. This photo shows the basket of a baffle ring centrifuge

cally with pressure blows or hydraulically by dissolving and breaking up the cake through swirling action.

Siphon peeler. The siphon-peeler centrifuge (Figure 4) is a design variant of the peeler centrifuge that enhances the performance. This design uses a rotary siphon that enables the residual heel to be backwashed from the filtrate side, which frees the capillaries of fine particles. In this way, the residual heel can be used much longer without necessitating its time-consuming removal.

A further advantage of the siphon variant is that, besides the applied filtration pressure generated by the mass of the liquid, there is an additional vacuum created behind the filter medium (through the low level in the siphon chamber) that increases the filtration rate.

Because it is possible to extend the length of the siphon-peeler pipe, feeding into a liquid pool is also possible with this type of machine.

Horizontal peeler centrifuges both standard and with siphon basket — are very flexible in use. They can be used for starch, herbicides or fine chemicals, to name just a few application examples.

Vertical-basket peeler. Another variant of the peeler centrifuge is the vertical-basket peeler centrifuge. The operating principle is practically the same as the horizontal peeler, except that the product is not discharged after peeling via a chute or conveyor screw, but rather drops through the openings in the bottom of the basket during the peeling process. To do this, the centrifuge is decelerated to enable

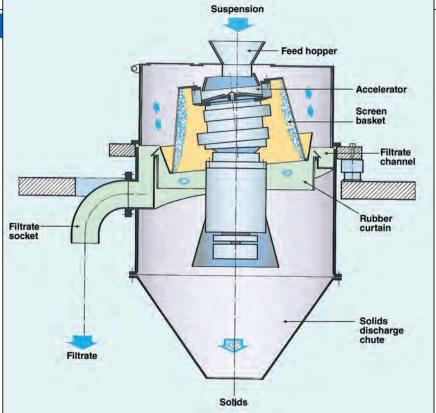


FIGURE 6. The basket of a tumbler centrifuge is mounted on a U-joint, which results in a tumbling motion that enables continuous product discharge

a controlled product discharge. This, however, extends the batch processing time and consequently reduces the throughput rate.

Nevertheless, this vertical variant reduces capital costs due to its compact design. A typical application of vertical-basket peeler centrifuges is for the dewatering of gypsum produced in fluegas desulfurization units.

In general, the throughput of peeler centrifuges is somewhat restricted in comparison with the other types of filtration centrifuges. This is where the continuously operating centrifuges, in which all process steps are carried out simultaneously, enables a huge increase in the throughput rate.

Continuous centrifuges

In continuous centrifuges a further distinction is made with regard to the conveying mechanism used for discharging product.

Sliding discharge. The sliding discharge centrifuge uses the simplest possible conveying mechanism. Instead of a cylindrical shell common to peeler centrifuges, the horizontally arranged basket has a conical shell that opens out towards the discharge of the centrifuge. The solids in the basket have a product-specific friction angle that has

to be taken into account when selecting the opening angle of the basket. Through the slope resistance of the centrifugal force and the pressure of the following solids, the product slides through the basket without requiring any mechanical tool. To keep the friction angle of the product and the basket angle small, a fine-hole sheets or slotted screens rather than a mesh, are used in continuous centrifuges. The slotted screens are made of profiled wires between which the screen slots point in the transport direction.

Baffle ring. A special variant of the sliding discharge centrifuge is the baffle ring centrifuge (Figure 5). Here it is not the cake transport as a closed product layer that is intended, but rather a dewetting of individual particles. To this end, steps are arranged on the shell surface so that the particles are in a free fall for a short period of time. When the individual particles hit the next step, the surface moisture is spun off the particle. If, in the closed cake layer, there is still interstitial water between the particles that are in direct contact, this impact effect can attain cake residual-moisture levels of less than 0.1 wt.%. This application example, however, is almost solely restricted to coarse synthetic granules.

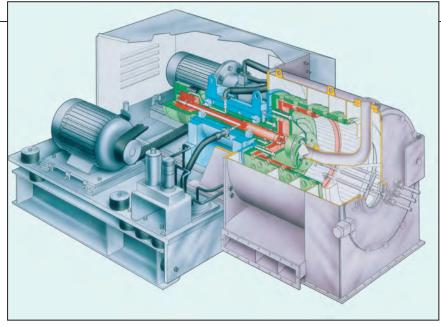


FIGURE 7. This inside view of a two-stage pusher centrifuge shows the rotating parts in green and the rotating and oscillating parts in red. The bearing housing is blue

Vibrating basket. The next modification of the sliding discharge centrifuge is the vibrating basket centrifuge. Here, the opening angle of the conical basket is first selected in such a way that no sliding action or product transport will take place. However, by superimposing an axial vibrating movement to the rotation, the friction can be overcome for a short while through the impulse that is transferred to the product by the vibration of the shaft and the basket. This results in a stepwise transport through the basket.

The vibrating basket centrifuges stand out with their substantial solids throughput rates of up to 350 ton/h. The centrifugal force (C-value), however, is limited to approximately 150 times the earth's gravitational acceleration (150g), as otherwise the transport impulse can no longer be generated by means of vibration.

The main field of application of vibrating basket centrifuges is in the treatment of fine coal.

Tumblers. The most complex way to generate a transport impulse is found in the tumbler centrifuge (Figure 6). Here again basically the same principle is applied as in a sliding centrifuge, using a conical basket. In this case, however, the basket is mounted via a U-joint and an articulated hollow shaft at a slight angle to the actual axis of rotation. Through a different speed of the inner drive shaft to that of the outer hollow shaft, the angular offset wanders independent of the rotation. The movement of the basket can be seen to wobble like a child's spinning top.

In doing so, every point on the basket periodically passes through the area of the slightest inclination and the area of the greatest inclination, relative to the surface of the screen. In the area of the greatest inclination, the product starts to slide; in the area of the slightest inclination, the product is slowed down again until it stops. There is therefore no cake that is moved as an intact surface, as in the vibration basket centrifuge, or as a cake ring, as in the pusher centrifuge. Instead, there is a cake that is constantly subjected to a rotating wave movement.

Since the intensity of the transport impulse increases to the same degree as the centrifugal acceleration, the tumbler centrifuge is not subjected to the constraints of a vibrating basket centrifuge. C-values of up to 8,000g are possible, and throughput rates of up to 300 ton/h can be achieved.

The tumbler centrifuge is used today for dewatering carnallite, for example. **Worm/screen.** The conical-shaped basket used in the above centrifuges are also used in the worm/screen centrifuge. In the conical basket, there is also a conical screw that rotates in the same direction as the basket, but at a slighter differential speed, resulting in a forced transport of the product in the centrifugal field.

In this case, it is not necessary to exactly coordinate the basket angle to the product friction angle because the screw takes on a regulating function. Where the screw retains the still liquid slurry at the smaller diameter, it pro-actively conveys the product at the large diameter, the friction angle of which has increased due to the reduced residual moisture.

As a result of this effect, and through the forced product transport, the worm/screen centrifuge is relatively insensitive to feed fluctuations and interruptions in the product supply.

Typical applications are the filtration of iron sulfate and polystyrene. **Pusher:** The pusher centrifuge also has a forced product transport. This centrifuge has no conical basket. Its cylindrical basket is fixed to a hollow shaft and has a plate on the basket bottom that is mounted on a pusher rod, which runs inside the hollow shaft. The pusher rod oscillates axially so that the plate, known as the pusher plate, performs a pushing motion at the bottom of the basket.

The product that is fed centrally through a rotating feed system, dewaters in the feed zone on the screen. The cake ring that then forms is pushed towards the discharge by the pusher plate. The vacant space that appears when the pusher bottom is driven back is again filled with new product, allowing a new cake ring to develop that, during the forward motion, pushes the previous one further forward.

For a continuous product transport, the pusher centrifuge requires a constant topping up with product. The pusher centrifuge therefore relies on the feed conditions being kept as constant as possible. Through the cake ring that is moved over the screen as a compact block, the throughput of particles is very low for a continuous operating filtration centrifuge, as most of the fine particles are retained in the cake. In a modern pusher centrifuge throughput rates of up to 150 ton/h can be reached.

After the single-stage pusher centrifuge was introduced, there soon followed two-stage pusher centrifuges (Figure 7). Here, a second, shorter inner basket with smaller diameter is installed between the pusher plate and the basket. In the two-stage pusher centrifuge the pusher movement that in the single-stage machine only takes place between the pusher plate

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and the basket — also takes place between the inner basket and the outer basket. With two shorter baskets instead of the one long basket, the twostage centrifuges have the advantages that the height of the cake is reduced. the filtration resistance is lower and the pushing force required is less than in single-stage machines. Even if the single-stage pusher centrifuges provide a higher throughput rate due to the higher cake volume in the basket, the two-stage machines have become established as the more popular version today. Machines are also available with three and more stages for special applications (Figure 8).

The main fields of application are crystalline products with average particle sizes of around 80 μ m up to 10 mm, and solids feed concentrations from around 25 wt.%. Some examples of applications are the dewatering of ammonium sulfate, sodium chloride and adipic acid.



FIGURE 8. Closeups of the interior of two-stage (left) and three-stage (right) pusher centrifuges

Final remarks

To select the right type of centrifuge it is most important to maintain close contact with the equipment manufacturer. For the coordination of the full system, it is also expedient for the manufacturer to not only be able to supply the complete range of centrifuges, but also the equipment integrated in the process line upstream or downstream of the centrifuge.

Edited by Gerald Ondrey



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Optimizing Reciprocating Compressors for CPI Plants

Follow this guidance to improve the design, performance and reliability of these widely used machines

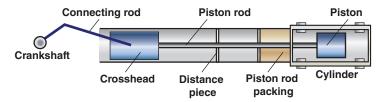


FIGURE 1. Reciprocating compressors compress air or other gases using a piston that is driven by a crankshaft. Shown here are the main parts of a reciprocating compressor

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Reciprocating compressors the most commonly used type of compressor throughout the chemical process industries (CPI) — are flexible and efficient, and they can generate high head (from several bar to several thousand bar) independent of gas density. Worldwide, the installed reciprocating compressor horsepower is approximately two times that of centrifugal compressors.

However, the maintenance costs associated with reciprocating compressors are approximately three times greater than those for centrifugal compressors (due to valve, unloader and packing-maintenance requirements). This article provides practical recommendations for users to consider in an effort to improve the selection, operation and maintenance of reciprocating compressors in CPI applications.

Compressor design

Figure 1 shows the basic design of a reciprocating compressor. Close attention to the selection of the piston rod packing can improve performance, because this is a common source of reliability problems associated with reciprocating compressors, and is a common path for the leakage of potentially hazardous process gases. Experience shows that packing life can be extended by as much as a fac-

tor of three by adding the proper coating (tungsten carbide is a widely used coating material for piston rods).

Interstage cooling is required when the machine or gas being compressed has a temperature limit. In this case, as the gas cools, any liquid that may form is separated in interstage facilities and then the gas is returned to next compressor stage for further compression. Each compressor stage may consist one or more cylinders. Vendors usually offer a range of interstage pressures. The ability to optimize interstage pressures can help to minimize the total cost of ownership for the compressor and interstage facilities. This optimization can be done by evaluating the initial cost and operating costs of compressors and interstage facilities for various interstage pressures.

During operation, interstage pressures will increase during part-load operation (that is, operation at lower flow that results when an unloader device is used; this is discussed below) combined with variation in pressure at the suction inlet.

In a typical reciprocating-compressor design, the first stage may contain one or more cylinders and a clearance pocket. An additional bottle may be added to the cylinder with an actuated on/off valve. To avoid unwanted interstage pressure increases, users may consider installing additional clearance pocket(s) on the first-stage cylinder(s) and using part-load operation via the compressor control logic.

By selecting the right interstage design pressure, users can ensure proper operation in the face of part-load operation and variation in suction pressure. In general, the interstage design pressures should be around 15%higher than the interstage basic design values for applications that are working with common part-load steps (such as 25%, 50%, 75% and 100% capacity) and are expected to experience a variation in suction pressure of around +/- 7% during operation.

In some applications, reciprocating compressors must be designed to operate reliably in the face of considerable suction pressure variations while still providing full design flow at the desired discharge pressure. These operating requirements will have a direct impact on compressor sizing, especially the frame rating and motor power required for the unit.

Figure 2 shows load curves for the connecting rod of a reciprocating compressor in petroleum refining service. Variation in suction pressure (in this case, a roughly 7% reduction in suction pressure) results in a higher load on the rod. As a general rule, the compressor should be designed so that the maximum-anticipated rod load

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does not exceed 80% of the allowable rod load.

As shown on the *y*-axis in Figure 2, the rod load shall change sign from negative to positive and then negative again during one revolution of the crankshaft in order to provide proper lubrication for the mechanism (especially for the cross-head pin). The duration of rod sign reversal (the period during which load has the opposite sign) should not be less than 15 degrees of crank angle. The rod-load reversal peak (maximum amount of load in the reversed direction) should not be less than 3% of the actual combined load in the opposite direction. These minimum requirements should be satisfied under all possible operating conditions (especially in the face of suction-pressure variation and partload operation, such as when an unloader device is used to decrease flow through the compressor).

In many cases, higher values of rod-reversal duration and peak are considered during compressor design to increase reliability. In Figure 2, minimum load-reversal duration corresponds to 50% capacity, and the reversal duration is more than 70 deg.

In general, the optimum speed for the reliable operation of reciprocating compressors is around 350 rpm. For compressors operating below 400 kW, speed on the order of 450 rpm is suitable. However, for those operating below 100 kW, higher speeds (even as high as 700 rpm) are acceptable.

Lubricated cylinders and packing may be preferred to extend service life. However, non-lubricated cylinder compressors should be used when the possibility of oil contamination cannot be tolerated in downstream operations (for instance, when trace amounts of lubricating oils could cause catalyst problems in downstream reactors).

For the optimum operation of reciprocating compressors, sufficient inertia — provided by a properly sized flywheel — is mandatory to regulate the variable reciprocating torque. Figure 3 shows brake torque versus crank angle for one revolution of the crankshaft for a reciprocating compressor used in a petroleum refinery setting. The red and blue curves rep-

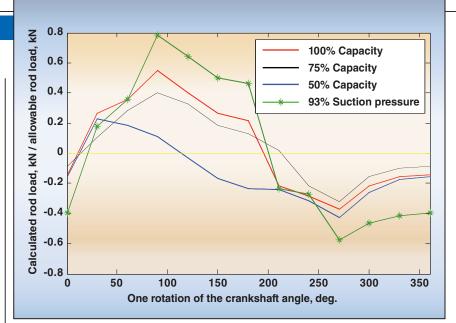


FIGURE 2. These rod-load curves are for a reciprocating compressor in refinery service. This figure shows variation in the ratio of rod load to allowable rod load in one revolution of the compressor crankshaft (from 0 to 360 deg.). Individual curves show the rod load for various operating conditions, including 50%, 75% and 100% compressor capacity, as well as full flow with 7% suction reduction (that is, operation at 93% of rated suction pressure)

resent compressor torque for normal full-load capacity and half-load (50%) capacity, respectively.

A step-less capacity-control system uses a hydraulically actuated, fingertype unloader. This device unloads the suction valve for only a portion of compression cycle to achieve the desired adjusted capacity.

A finger-type unloader has fingershaped parts that act on the cylinder valve elements and keep them open for a defined duration during the compression cycle. Users should note that these finger-type unloaders have the potential to damage the valve-sealing elements and thus may have greater maintenance requirements.

A step-less capacity control system is recommended for larger machines (units rated above 2 MW, when large operation variation is expected). In these cases, step-less capacity control (working in the range of 20-100% capacity) is extensively used due to process requirements.

In general, valves and unloaders are responsible for nearly half (roughly 45%) of unscheduled reciprocatingcompressor shutdowns, so valve and unloader selection can have a strong impact on reliability. And many consider the automatic cylinder valves to be the most critical components of such machines, as they are responsible for many unscheduled maintenance events. For large compressors (that is, those that operate at relatively low speeds with high pressure ratios), relatively large-bore ringtype valves (above 100 mm, or 4 in.) combined with plug-type unloaders should be considered first, to avoid reliability issues associated with fingertype unloaders. Since ring-type valves and plug unloaders are not available for smaller-sized compressors (those that operate at relatively higher speeds), such units typically use platetype valves.

During operation, the rotating parts of the compressor, power transmission and driver will act like springs connected in series. This torsional dynamic system may create resonance (where one natural frequency of system coincides with one of excitation torque). In reciprocating compressor trains, there is always a risk of torsional resonance and torsional fatigue failure (that is, damage to component resulting from excessive cyclic loads).

Couplings that connect the driver to the compressor can be modified to tune the system to avoid torsional resonance. Several coupling options are available as follows:

- 1. Direct, forged-flange rigid connection (no coupling) between driver and compressor
- 2. High-torsional-stiffness coupling is allowed by torsional analysis. Since

coupling options are limited, it may not be possible to find an acceptable coupling with the required torsional characteristics and service factor, especially for large compressors (above 3 MW)

3. Flexible coupling (which provides more elasticity and damping, but may require greater maintenance since elastic elements in such a coupling may need frequent replacement)

The most common reasons for problems caused by torsional vibration are lack of comprehensive torsionalvibration analysis, improper application and maintenance of couplings (especially flexible ones) and lack of appropriate monitoring. As a general rule-of-thumb, the shaft diameter of the electric motor should be equal to or greater than the reciprocating crankshaft diameter (because the crankcase is generally forged from a stronger steel grade compared to the motor rotor).

Condition monitoring

Condition monitoring, when done properly, can pay for itself by helping operators identify potential systems malfunctions at an early stage. A rigorous program should include monitoring of these important conditions:

Vibration (including continuous vibration monitoring of the compressor and motor casing, providing both alarm and shutdown capabilities):

• In general, velocity transducers are preferred over accelerometers (because interested frequencies for monitoring better match with velocity-measurement sensors). The optimum configuration for using a velocity transducer is to install one on each end of the crankcase, about halfway up from the base plate in line with a main bearing, both for compressor and motor

• Crosshead accelerometer (alarm)

Temperature:

- High gas-discharge temperature for each cylinder (with both alarm and shutdown capabilities)
- Pressure packing piston-rod temperature (alarm)
- High crosshead pin temperature (alarm), only for relatively large

compressors (around or above 3 $\ensuremath{MW}\xspace$)

- High compressor main, and motor bearing, temperatures (alarm)
- Valve temperature (monitoring)
- Oil temperature, out of compressor frame (alarm)
- High jacket-water temperature of each cylinder (alarm)

In addition, proximity probes, typically located under the piston rods, provide alarm capabilities but are not used for shutdown. These are used to measure the rod position and determine wear or malfunctions. Such probes can quickly identify problems such as piston or rider band malfunctions, cracks in the piston rod attachment, a broken crosshead shoe or even a liquid carryover to a cylinder.

Improving maintenance

To support regular maintenance, the installation of any reciprocating compressor must ensure proper access to the entire compressor system, especially the non-drive end. In particular, adequate space and work areas must be provided to enable the complete withdrawal of the piston, removal of the cooler bundles or piping spool and laydown area (to carry out maintenance, dismantling of parts and repairs).

Similarly, three crane capacities must be properly identified: The total capacity of the overhead crane (to lift components for routine maintenance), the maximum maintenance weight (to ensure that the heaviest parts, usually the motor, can be lifted during overhauls), and the maximum installation weight (maximum skid weight, usually the compressor skid).

For a typical 7-MW API 618 compressor train for petroleum-refinery service, these crane capacities would be roughly 11 tons, 55 tons and 100 tons, respectively, and the required crane height would be roughly 12 m (around 40 ft)

Any time a given compressor must be stopped for an extended time, it should be turned a quarter-turn every week, using a barring device (this is a device that slowly turns the compressor to avoid locking and other problems that often arise during long stoppages of reciprocating compressors). A manual barring device can be used for relatively small compressors. A pneumatic barring device must be used for compressors rated above 750 kW (provided there is no area classification or power-availability problem).

For larger compressors (2 MW or larger), these special tools are often needed to carry out routine maintenance on reciprocating compressors. These tools cannot be easily purchased; they must be specially designed and fabricated based on the actual machine:

- Bearing extractor
- Piston extractor
- Valve extractor
- Piston fit-up tool
- Hydraulic tightening system
- Crosshead assembling tool
- Special lifting tools
- Partition plate-assembling tools
- Mandrels for wear bands

During maintenance of compressor mechanical components, the following criteria are important:

- Cylinder clearance for the outboard end should be around 4–6 mm (0.2–0.3 in.), and for the inboard end, clearance should be around 2–4 mm (0.1–0.2 in.)
- The allowable temperature of the machine bearings, piston rod, connecting rod bearing and crosshead should be maintained around 85°C, and for the crosshead pin, it should be maintained around 90°C
- The vibration level of the crankcase should not exceed 100 microns, and the expected vibration level of the cylinders should be around 150 microns (these vibration recommendations are peak-to-peak vibration readings for an installed, troublefree, middle-range machine around 1 MW)
- Bearings, piston rings and piston shoes should also be inspected regularly.

Auxiliaries and accessories

For auxiliaries and accessories, the optimum configuration is to install a local panel near the compressor skid (around 250 mm, or one foot away from the compressor skid), and on a standalone skid to minimize the potential for vibration damage.

The oil system should include two

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oil pumps, both sized for a capacity that is 20% greater than the maximum oil flow required for the compressor. At a minimum, two pumps should be used. Either a run-down tank (this is a stainless-steel tank that allows the supply oil to safely coast down the machine in the event that both pumps have failed), or a crank-shaft-driven main oil pump is required. Dual (two) removable bundle shell-and-tube oil coolers (TEMA C), double oil filters with a removable element and stainless-steel piping are also necessary.

Liquids should not be allowed to accumulate inside the compressor cylinder. For any application, a suction drum (sized appropriately with regard to application-specific retention time, flow velocity, and, if required, a mistcollection system to capture contaminants) with a drain provision should be provided. It may be part of pulsation control. To control pulsation, a vertical vessel is sometimes used as both suction drum and suction pulsation vessel, but this not recommended since vertical pulsation vessels cause relatively long piping to the compressor, which may lead to dynamic problems. Similarly, these are sometimes conflicting requirements for pulsation damping and suction separation, so this combined approach is not preferred. It may be used only for small compressors, let's assume below 250 kW, with relatively light gases, such as those lighter than nitrogen.

Cooling-water systems are generally used as a heat sink for reciprocating compressors, to avoid hot spots and improve machine stability and reliability. To design a cooling system, first, the generated heat should be calculated. Then, the anticipated temperature rise should be identified. The cooling-water inlet temperature should be selected between 6°C and 16°C above the inlet gas temperature. When selecting the pump to deliver cooling water to the compressor cylinder and packings, consider this:

• With regard to the slope of the operating curve (discussed in greater detail below), the selected operating point should not be in the flat or near-flat part of curve; rather, enough slope is needed for proper operation

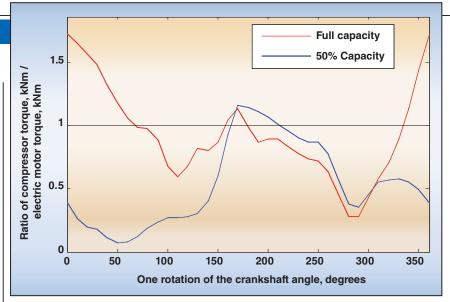


FIGURE 3. This plot of reciprocating compressor torque versus crank angle shows the variation in compressor torque represented by the ratio of compressor torque/ motor torque, in one revolution of compressor crankshaft (0 to 360 deg.). The curves show compressor torque in two operation conditions — 50% capacity and 100% (full) capacity

- There should be a continuous rise from a selected operating point to shut off
- There should be proper shut-off pressure compared to a selected operating point (preferably 10% but a minimum of 6%)

Note: usually a larger size pump shall be selected to meet these recommendations.

Any reciprocating compressor system should be designed with a margin of excess flow capacity for the cooling system, to enable it to respond to situations that deviate from normal operation, where the need may arise later for additional cooling flow to remove excess generated heat (for example, unloaded operation when the compressor is idle, overload conditions, or future expansion, if applicable). The recommended cooling pump capacity margin is 10-25% (that is, pump rated capacity is 10-25% more than required normal flow).

Users should consider suitably sized pulsation vessels and correct any potential pulsation resonance in piping rather than using damping devices, such as orifices, choke tube and so on to dampen pulsation. Acoustic reviews should be performed during compressor system design to guarantee all anticipated combinations of pressures, speeds and load steps (including the use of flow-reduction steps that rely on unloaders, which can vary the compressor flow).

Pulsation limits are recommended

around 85–95% of API 618 (Approach 3) limits to provide some margin (5–15% of API 618 limit values) to mitigate risk during construction and installation periods, and to cope with unanticipated deviations and problems. Similarly, pulsation vessels are generally fabricated before finalization of the piping-design-and-pulsation study, and enough margin should be provided to meet potential risks.

For nearly all applications, horizontal suction and horizontal discharge vessels are preferred. Long distances between vertical pulsation vessels and compressors increase the likelihood of pulsation problems.

Improving performance

The maximum predicted discharge temperature for any API 618 reciprocating compressor for CPI applications must not exceed 150°C, and must not exceed 135°C for hydrogen-rich service. In general, gas discharge temperatures below 118°C tend to lead to longer life for the wearing parts.

When it comes to optimum pressuredrop values for pulsation dampeners and suppression devices, the pressure drop maximum is 1% of absolute pressure. For the intercooler, pressure drop around 0.70 bar or 2% of absolute pressure is recommended.

Readers should note that the use of orifice plates to dampen pulsation, especially on high-speed, single-act compressors (that is, those that compress gas on only one head of cylinder), can contribute to significant pressure drops.

To gain a better understanding of reciprocating compressor performance and track ongoing operation, the following performance curves should be developed:

- Suction pressure versus load
- Suction pressure versus flow
- Discharge pressure versus load
- Discharge pressure versus flow
- Suction pressure versus discharge pressure, per load step (that is, for each flow-reduction step using unloaders, typically 50%, 75%, 100% flow; 25% is rarely used because of the potential for reliability and load-reversal issues)

Such flow curves typically plot the minimum achievable flowrate to the maximum achievable flowrate in specified increment steps (for instance, in 10% steps). (Flow-versus-dischargepressure plots of specific suction pressures may be an acceptable alternative when suction-pressure variations are limited). A review of the steepness of the proposed load curves can help the engineer to quickly identify which load curves (and where) are too steep. In these situations, small changes in pressure can have significant changes in load and flow. In general, compressors with steep load curves are hard to automate and tune. Thus, steep load curves usually indicate improper sizing of cylinders.

Optimum conditions

When scoping a reciprocating compressor system, it is absolutely necessary to have a minimum of two technically accepted proposals from qualified vendors. Small and medium compressors should be delivered fully fabricated as one skid-mounted package. Larger compressors are typically delivered as a prefabricated system (including the crankcase, distance pieces, and so on) with dismantled cylinders. Assembled cylinders are typically delivered to the site separately and installed later. It is common for the vendor to provide site-supervision work for cylinder installation at a negotiated lump-sum price.

Edited by Suzanne Shelley

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Chemical Lifecycle Management

Sustainability demands a higher level of oversight that seeks new ways to effectively and cost efficiently procure, handle and dispose of, or recycle materials

Victor Belenchia Clean Harbors

hemicals in production and laboratory environments alike have three distinct lifecycle phases: the first is procurement and inventory storage; the second is use in a manufacturing process or research program; and the third is post-use, including onsite handling, removal and beneficial reuse, recycle or disposal.

A number of considerations including regulations, safety, material utilization efficiency, costs and sustainability govern all of the steps in this lifecycle. The challenge for laboratory and production managers is to balance these sometimes competing forces to create an effective process that meets the organization's overall needs.

Proper chemical handling during processing is typically emphasized in production facilities, but how to handle chemicals post-use is often given less attention. While this article examines the full chemical lifecycle, it focuses on this post-use phase.

Sustainable lifecycles

Every laboratory and chemical processor must follow environmental, health and safety (EH&S) procedures and regulations. This is often a challenge, especially for smaller operations, since implementation frequently rests on individual managers,

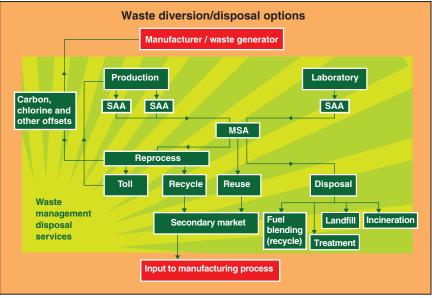


FIGURE 1. Managing post-use chemicals is a vital part of chemical lifecycle management. Recycling "waste" chemicals is an option that can contribute greatly to sustainability goals

local teams and staff who have other primary responsibilities. Regardless of size, laboratory and production managers must periodically review their policies, conduct training and audit their chemical inventories and waste materials. Requirements, processes and regulations change, and it is management's responsibility to remain in compliance, while meeting everyday production demands.

Environmental sustainability continues to increase in importance and is embraced as a corporate goal at many manufacturing organizations. This takes chemical management beyond the traditional regulatory and safety considerations. The chemical process industries (CPI) have become even greater stewards of the materials throughout their entire lifecycles as they seek ways to further reuse or recycle materials that were historically disposed of as waste (Figure 1).

Sustainability requires the minimization of consumption and waste in order to reduce environmental impact through process optimization and reuse or recycling whenever possible. This can require additional recordkeeping and administrative steps. The challenge, when it comes to chemical lifecycle management, is to find operationally efficient and cost-effective ways to reduce the overall waste stream through process optimization, efficient material use, tolling or selling recyclable materials to a secondary market.

Purchasing and storage

management Effective chemical starts before the chemicals arrive at the site. Laboratory and operations personnel need to work carefully with purchasing staff to order the correct amounts, minimize order errors and ensure that proper receiving and storage provisions are in place. For operations personnel, it is a balance between anticipating customer orders and manufacturing volume in order to eliminate production interruptions, minimize storage and other inventory costs, and to avoid having to dispose of outdated materials. Purchasing staff can consolidate orders to lower costs, but they must have an understanding of the chemical properties and intended uses in order to ensure that materials are consumed before they begin to degrade and negatively

affect chemical reactions or final product properties. Overall, the CPI have done a good job utilizing continuous improvement techniques to manage their supply chains.

Once received, the materials must be stored in accordance with local, state and provincial regulations, or the International Code Council's (ICC) International Building and International Fire Codes, and handled in accordance with company EH&S procedures. In the U.S., handling must follow the Occupational Health & Safety Admin.'s (OSHA) Code of Federal Regulations (Title 29 CFR 1910.1200), which covers Hazard Communication Standards (HCS), the federal Risk Management Program for certain highly toxic chemical compounds regulated under the Clean Air Act, and the Chemical Hvgiene Standard for laboratories (Title 29 CFR 1910.1450).

Production and laboratory use

As materials move into the second phase of the lifecycle — use in a production environment or laboratory the potential for handling errors increases. Although industrial users and researchers are concerned with, and trained in, safe handling procedures for hazardous materials, they are less likely to focus on storage and removal. This leads to some potential problems in chemical lifecycle management.

Chemicals are generally well managed in production environments since production procedures are welldefined and engineered. Failure most often results from human error or system breakdowns. Although the results can be catastrophic, they are largely preventable through adherence to established EH&S procedures, effective maintenance practices and frequent training.

Research environments are less systematized, which presents greater opportunity for errors. Researchers often work independently on projects and they use smaller quantities of a greater number of chemicals. This presents an exponentially higher number of possible chemical reactions and storage issues.

Working alone or on small teams, researchers often neglect to properly label chemicals in secondary containers at their stations. Unlabeled or improperly labeled chemicals violate hazardous communication (Hazcom) regulations. Informal practices also lead to the potential for abandoned chemicals as researchers move on to other jobs or projects. Unidentified chemicals may require testing, special handling and expensive disposal if they cannot be verified by the researcher.

Consistency and training, supported by accurate, up-to-date labeling and recordkeeping, are key ingredients to a successful chemical management program.

Post-use

Handling of solvents, catalysts and other chemicals can become much more problematic once they have been used in a production or laboratory process. They are no longer in their original containers and are often combined with other substances. In the U.S. for example, OSHA, the Environmental Protection Agency (EPA), Dept. of Transportation (DOT) and, in some cases, Drug Enforcement Admin. (DEA) regulations also come into play.

A few important fundamental facts must be kept in mind at all times: In the U.S., all chemical storage areas fall under Resource Conservation and Recovery Act (RCRA) regulations and are subject to inspection by regulators. To comply with that, a limited number of conveniently positioned and properly organized satellite accumulation areas (SAAs), under the control of the EH&S or chemical hygiene manager, should be set up near production or research areas.

There are a number of RCRA rules for SAAs. For instance, containers must be in good condition without rust, dents, or cracks; they cannot be stored near drains or other structures that could pose an environmental risk: hazardous waste containers cannot exceed 55 gal; acutely hazardous waste cannot exceed one quart; SAAs must be inspected weekly. There are additional guidelines including requirements for clear and visible labels stating "hazardous waste" and listing the container contents, as well as the "three-day rule" that requires that the containers are immediately marked with the current date when full, and removed within three days to the main storage area (MSA).

The MSA is governed by additional rules. One of the most important relates to allowed accumulation time once materials enter the MSA. Small quantity generators (SQGs) — organizations that generate more than 100 kg but less than 1,000 kg of hazardous waste per month — can accumulate materials for 180 days from the start date.¹

Large quantity generators (LQGs) — those that generate 1,000 kg or more of hazardous waste per month, or more than 1 kg per month of acutely hazardous waste — are subject to a 90-day accumulation rule.²

Regardless of size, the MSAs must be secured against unauthorized entry; hazardous waste and container contents labeling rules must be followed; containers must be inspected weekly with reports kept on file; and preparedness and prevention equipment (for example an emergency phone, alarm, fire suppression and spill prevention equipment) is required.

There are additional regulations and exceptions to the rules noted above. For instance, peroxide formers, such as ethers and dioxane, are generally managed under a peroxideformer program that tracks the material based on the manufacture and retention dates from the day it arrives onsite until the day it is consumed or disposed of. SAA storage times and 90/180-day MSA rules are all subordinate to the peroxide-former deadlines. So, if the earliest peroxide-former retention date is 45 days in the future, that is the container's deadline. Even the waste disposal company must track peroxide formers and follow expedited destruction schedules based on the retention date.

Managing chemical storage areas.

A minimum number of properly trained staff should be assigned to transport materials from the SAAs to the MSA and manage the materials in all of the storage areas. Some operations are large enough to have a dedi-

 http://www.epa.gov/solidwaste/hazard/ generation/lqg.htm

^{1.} http://www.epa.gov/waste/hazard/generation/ sqg/index.htm

TRI CHEMICALS

Environmental Manager

cated full-time staff, while for others it is a part-time function (that requires the same annual refresher training). Properly organizing the handling and in-house movement of chemicals removes the responsibility from researchers and substantially reduces training requirements.

Another benefit of using dedicated staff is that it puts a third party in position to regularly inspect warehouse, production and laboratory areas to ensure that chemicals are properly stored and handled in accordance with regulatory and company policies. Here are some best practices that we have seen in the field and that we practice at our own sites and for customers:

- Ensure that proper procedures (such as labeling and tracking) are being followed at each production and laboratory location, and that waste chemicals are stored only at approved sites
- Conduct periodic safety training for research and production personnel
- Establish collection schedules to efficiently move materials from SAAs to the MSA in a timely way
- Ensure that SAA containers are in good condition, properly closed and placed within secondary containers, and that incompatible materials are properly segregated
- Catalog, store and consolidate MSA materials in bulk containers and pack smaller quantities, elbow bottles, and so on, for transportation
- Identify unknowns before shipping
- Follow up on the disposal or recovery of chemicals for company and regulatory reporting

Software. Most large companies utilize chemical inventory management systems, while many laboratories still use spreadsheets to manage their inventories. Spreadsheets can be helpful, but they are not the answer for corporate-wide or comprehensive lifecycle-management programs. They are generally home-grown standalone applications without built-in reporting mechanisms. They do not extend beyond the department or integrate with corporate systems. They do not tie into industry resources and documentation, such as material safety data sheet (MSDS) databases, that are necessary to build chemical profiles. An added benefit of recycling and reusing chemicals can be reducing the volume of materials listed in Toxics Release Inventory (TRI) reports. This can contribute to reduced taxes and annual fees associated with RCRA regulations. TRI chemicals have included the following:

- Aluminum (fume or dust)
- Aluminum oxide (fibrous forms)
- Ammonia (anhydrous and aqueous) Asbestos (friable)
- Hydrochloric acid (acid aerosols)
- Nitrate compounds (water dissociable; aqueous solution)
- Phosphorus (yellow or white)
- Sulfuric acid (acid aerosols) Vanadium (except when contained in an

alloy)

Zinc (fume or dust)

The TRI listing for the following three chemicals is based on the chemical activity rather than the form of the chemical:

1. Dioxin and dioxin-like compounds (only if they are manufactured at the facility; or are processed or otherwise used when present as contaminants in a chemical but only if they were created during the manufacture of that chemical)

 Isopropyl alcohol (only if it is being manufactured by the strong acid process)
 Saccharin (only if it is being manufactured)

 $\begin{array}{l} {\rm SOURCE: U.S. Environmental Protection Agency: EPA 260-R-09-006 October 2009 "Toxic Chemical Release Inventory Reporting Forms and Instructions — Revised 2009 Version" (http://www.epa.gov/TRI/report/rfi/ry2009rfi121709.pdf). See Table II-3, p. 143 for the full alphabetical list of chemicals and qualifiers \\ \end{array}$

Perhaps, most importantly, they may not stand up to regulatory scrutiny if the need arises.

Specialized software and online chemical-waste-management systems handle all of these issues and also address many other important criteria, such as chemical movements, 90/180-day storage-time limits, chemical expiration dates and reporting. More sophisticated software and online tracking systems assign cost center allocations, and facilitate movement, inventorying and packing, as well as documenting the transfer of the material to a certified waste disposal company.

Waste disposal companies

Given the complexity of effective waste-chemical management, many companies find that it is more efficient and cost effective to turn to waste disposal companies to handle their chemical waste throughout the entire post-use lifecycle phase. Service companies manage the entire process including onsite movements, storage, packing and removal. In that way, generators do not have to invest as heavily in RCRA training and can rely on the contractor to apply its expertise, provide staff and backup staff as necessary, monitor regulatory federal, state and local developments, and implement best practices.

This allows plant and laboratory management to properly oversee the function without adding headcount. It also enables staff to focus on their core responsibilities and not be burdened with additional training or oversight of chemical management functions. Using the same outsourcer to handle materials in-house as well as for removal and disposal eliminates interorganizational hand-offs and can further reduce costs.

Communications between waste generators and chemical disposal companies. Before a waste disposal company can remove hazardous chemicals from a generator, all paperwork must be in order, the materials must be properly labeled and packaged and ready to go. It is important that chemical waste is properly characterized in order to avoid unnecessary expense or, in a worst case, additional transshipping to another waste disposal site. Confusion creates lost time and increased costs for chemical characterization tests and EPA and DOT documentation.

Communications with the waste disposal company are key. Direct links between the generator and the waste disposal company are efficient since any materials or paperwork that are not in compliance will be identified early, waste profiles and DOT manifests can be generated, and placards assigned before the disposal company arrives on site.

A direct link to the waste disposal company can also report containers that are ready for packing and/or pickup, enabling the removal company to cost-effectively schedule pickups. Automated procedures also help companies stay in compliance with RCRA 90/180-day rules or special handling requirements for peroxide formers and other acute hazards.

Regardless of the degree of automation and communications, under-

SOLVENT RECYCLE AND REUSE

nome waste disposal companies offer more sophisticated services, such as the ability to recycle solvents. Examples of solvents that are typically recycled for reuse include the following:

1-Ethyl-2-pyrrolidone Acetone Decalin (Decahydronaphthalene) Dimethyl sulfoxide Ethanol solutions (excluding specially denatured alcohols) Ethyl acetate Ethylene glycol Isoamyl alcohol Isopropyl alcohol Isopropyl alcohol (high water) Methanol

Methyl ethyl ketone Methylene chloride N,N-Dimethyl acetamide Naptha N-Methyl-2-pyrrolidone N-Propyl bromide Perchloroethylene Petroleum distillates Propylene glycol Tetrachloroethylene Tetrahydrofuran Toluene Xylenes (ortho-, meta-, para-)

standing the chemical waste disposal process minimizes costs up front by smoothing the transition from the MSA to disposal and reuse. Communication with the waste disposal company has become even more critical over the last few years due to increased opportunities for chemical reuse, recycling and tolling.

Reuse and recycle. Some hazardous and non-hazardous waste and byproducts with residual value can be directly reused in other processes by another manufacturer. In the past, these materials may have been landfilled or incinerated because it was less expensive and there were few disposal alternatives. Today, both environmental and financial incentives encourage materials reuse.

Beneficial reuse and recycling provide additional revenue streams, or at least the potential for cost reductions, for the generator; however, they add complexity to the removal-anddisposal process. Rather than a oneway transaction where the disposal company removes and disposes of the material, reuse and recycling may now include waste removal discounts for materials sold on the secondary market. Tolling, where reprocessed materials are returned to the generator, also results in more complex price negotiations based on fluctuating rates for virgin materials.

Recycling, where the waste disposal company reprocesses chemicals for new uses, can also substantially advance generators' sustainability performance. The materials stav out of the waste stream and become feedstock for another company's process, as outlined in Figure 1.

There are several other benefits that accrue from the reuse and recycling of chemical materials. In some cases reuse and recycling operations reclassify wastes, removing them from the generator's hazardous waste manifest, which reduces the volume of materials listed in Toxics Release Inventory (TRI) reports and advances waste minimization goals. They contribute to reduced taxes and annual fees associated with RCRA regulations (see box titled TRI Chemicals) and other corporate responsibility and environmental reports.

Clearly, sustainable disposal, reuse and recycling require a more sophisticated service level from the waste disposal company. Only a few are able to analyze the waste with an eye toward potential uses and the secondary market. They also have knowledge of the underlying regulations and access to the separation and purification resources necessary to reprocess the materials (see box titled Solvent Recycle and Reuse). Companies that offer these services along with in-house processing, tolling and resale capabilities, are best suited to develop comprehensive waste management programs that fully leverage sustainable secondary uses for waste chemicals.

It makes financial and environmental sense to explore these opportunities with a waste processor.

Edited by Dorothy Lozowski

Author



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20 years in the chemical industry with American Cyanamid and Cytec Industries in various technical and commercial positions and has been with Clean Harbors for the last six years in his current role



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FOCUS ON Personal Protective Equipment

Software now available for bump testing gas detectors

As part of the Altair 4X Gas Detector (photo), a new Altair 4 QuickCheck Station software version is available and is currently installed in production units. This version allows the Altair 4 QuickCheck Station to bump test both Altair 4 and Altair 4X Gas Detectors. — MSA, Pittsburgh, Pa. www.msanet.com

Safety glasses that easily turn into goggle

Swap protective eyewear (photo) comes with temples for use as safety glasses as well as a head strap to make it a safety goggle. Ideal for workers who perform a variety of tasks throughout the day, Swap can easily be converted from spectacles to goggles by swapping the temples and head strap with a simple snap. The lenses meet ANSI Z87.1+ and CSA Z94.3 standards and offer 99.9% UVA, UVB and UVC protection. — Gateway Safety, Inc., Cleveland, Ohio **www.gatewaysafety.com**

Stable stairs ensure safety at the loading dock

Elite Series Safety Stairs (photo) deliver enhanced rigidity and longer lasting tread for safe access from a loading rack or dock to an elevated walkway. The stairs provide a stronger engineered connection point that eliminates shakiness and increases the stability of an attached fall-protection enclosure. Construction materials include carbon steel, aluminum and fiberglass, or a combination of materials. — Benko Products, Inc., Sheffield Village, Ohio www.green-mfg.com

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N95 (photo) feature V-shaped pleats

that expand to help provide a com-

fortable seal, and to flex with mouth

movements for easier talking. The in-

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also feature embossed front panels,

which helps them to retain their

shape, and have side tabs designed

for easy positioning on the face for a

comfortable fit. A nose clip provides a

custom fit and secure seal. — 3M, St.

www.3m.com/occsafety

Paul, Minn.

3M

Gatewa Safety

This hazmat suit is comfortable to wear

CSP 5900 (photo) is a new singleexposure, disposable, Level A gastight chemical protective suit for hazardous materials (hazmat) incidents. The fully encapsu-

Benko Products

lated suit is especially designed to provide complete protection against hazardous gases, liquids and particles. The CSP 5900 is approved to NFPA 1994 standards and also meets European requirements for protective clothing for industrial applications and fire brigades. The suit is made of Zytron 500, the softest laminate material on the market, says the firm. — Dräger Safety, Inc., Pittsburgh, Pa. www.draeger.com

This blanket soaks up oily spills even when it's windy

This firm has recently introduced the Pig Oil-Only Weighted Blanket (photo) to capture leaks and drips outdoors, even in windy conditions. The blanket features a hydrophobic polypropylene filler that absorbs petroleum-based liquids, but no water. Heavy-duty seams form self-contained pockets within the



New Pig



Dräger Safety

blanket to maximize coverage area and keep the filler evenly distributed. The blanket helps companies to comply with 40 CFR 112.7 and 40 CFR 122.26. — *New Pig Corp., Tipton, Pa.* **www.newpig.com**

Clothing for when safety depends on visibility

This firm has recently completed the launch of its line of high-visibility safety clothing. The new line is suited for workers in public works, road security, building construction and other occupations where high-visibility clothing will enhance their safety. The most recent additions to complete the line are a five-in-one waterproof, high-visibility coat that can be worn in four seasons: and two types of rain pants, one nylon for warmer weather, and the other featuring a quilted lining for colder weather. - Linde Canada, Mississauga, Ontario, Canada

www.lindecanada.com

Ear protection that's comfortable and effective

The Pilot push-in ear plug for occupational hearing protection features an innovative hybrid design that combines

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www.how ard leight.com

New liners keep workers warm in very cold weather

This firm has expanded its N-Ferno Extreme Warming product line with the introduction of two new threelayer FR Winter liners (photo), which are available in regular and shoulder lengths. The liners feature three layers of warmth protection: A Banox FR3 Cotton shell, a foam mid-layer, and a poly-fleece liner. These winter liners not only help to keep workers warm on the most frigid work days, but they also keep them protected with the Banox FR3 cotton shell. — *Ergodyne, St. Paul, Minn.*

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www.rstahl.com

Ergodyne

Lighter, more-effective suits for firefighters

Developed in cooperation with Akao Co., these firefighting suits are made with Triprotech arimid-fiber fabric. Weighing just 2.5 kg, the new suits are 10% lighter than existing suits, and are 60% more effective in preventing second- and third-degree burns compared to the lightest suits of Akao. Akao will begin marketing the suits next March. — Teijin Techno Products, Ltd., Osaka, Japan

www.teijin-technoproducts.co.jp

A new hazmat suit for Level B

This firm has launched its first Level B style hazmat suit. OneSuit Shield is developed from the firm's proprietary Coretech Barrier Membrane, ensuring the flexibility and comfort that emergency responders are used to in the firm's Level A portfolio. The new suit is currently in testing for NFPA 1992 and 1994 standards. Level B hazmat suits are used for interacting with controlled and known chemicals in liquid form. — Saint-Gobain Performance Plastics, Merrimack, N.H. **www.onesuittec.com**

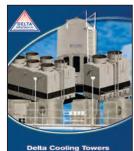
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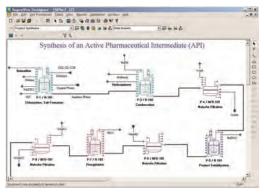


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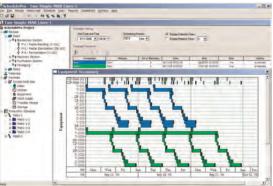
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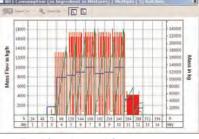
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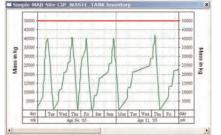
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Economic Indicators

PLANT WATCH

Total and CPI to study coal-to-olefins petrochemical project in China

November 5, 2010 — Total S.A. (Paris, France; www.total.com) and the Chinese energy group China Power Investment Corp. (CPI) intend to build a coal-based petrochemical plant in China. The two companies signed a letter of understanding to study the construction of a plant in Inner Mongolia. Both companies will now launch a feasibility study on a project for a 1-million ton/yr polyolefins production site, based on methanol produced from the gasification of coal. The investment for the plant, which is due to start production after 2015, is estimated at \notin 2–3 billion.

Yulin selects Unipol PE process for plant in China

November 3, 2010 — Univation Technologies LLC (Houston; www.univation.com) has announced that Yulin Energy and Chemical Ltd. of Yanchang Petroleum Group Co. (Yulin) has selected Univation's Unipol PE process for a 300,000 metric ton per year (m.t./yr) linear low-density polyethylene (PE) plant.The facility will be located in Shaanxi Province, People's Republic of China.The Unipol PE process facility will be fed by a unique combination of conventional feedstock and coal-to-olefins feedstock. Planned startup of the facility is 2013.

Stamicarbon's urea technology licensed in Argentina

October 30, 2010 — Stamicarbon B.V. (Sittard, the Netherlands; www.stamicarbon. com), the licensing and IP Center of Maire Tecnimont S.p.A. (www.mairetecnimont. it), has signed a license agreement with Tierra del Fuego Energia Y Quimica S.A., an Argentina-based company controlled by shareholders from China. The agreement concerns a urea synthesis plant and a urea granulation plant with a capacity of 2,700 m.t./d to be built in southern Argentina. Startup is planned in 2012.

Lanxess invests €9 million to expand EVM production

October 28, 2010 — Specialty chemicals company Lanxess AG (Leverkusen, Germany; www.lanxess.com) is to expand the production capacity at its Dormagen, Germany, site for its ethylene-vinyl acetate copolymer (EVM) line sold under the product brands Levapren and Levamelt by 30% to

BUSINESS NEWS

15,000 m.t./yr.The new capacities are due to be available from the second half of 2012.

Evonik to expand capacity of precipitated silicates

October 22, 2010 — Evonik Industries AG (Essen, Germany; www.evonik.com) is planning to increase its silica (precipitated silicic acids) production capacity by 25% over the next four years. Investments to ensure this increase in production capacity will be in the mid double-digit million Euro range. Total capacity expansion is expected to be in the six-digit metric ton region and will be implemented mainly at Evonik's existing silica sites in Asia and Europe.

Yara Praxair to build air separation unit in Sweden

October 22, 2010 — Yara Praxair (www. yarapraxair.com) — a 50-50 joint venture (JV) between Praxair, Inc. (Danbury, Conn.; www.praxair.com) and Yara International ASA — will invest in a new air separation unit to be located in Perstorp, Sweden.The plant, with a capacity of 200 ton/d, is scheduled to start up in 2012.

World's first onsite, large-scale specialty gas plant

October 19, 2010 — Air Products and Chemicals, Inc. (Lehigh Valley, Pa.; www.airproducts.com) has signed a contract with Anhui Sanan OptoElectronics Co., a subsidiary of Sanan OptoElectronics, to build two onsite ammonia plants at Sanan's new high-brightness light emitting diode (LED) manufacturing facility in the Wuhu Economic and Technological Development Area, Anhui Province, China. These new facilities are said to be the first and largest onsite specialty gas plants in the world. Each plant will be capable of supplying 2,000 m.t./yr of ammonia.

MERGERS AND ACQUISITIONS

Chevron to gain natural gas resource through Atlas Energy acquisition

November 9, 2010 — Chevron Corp. (San Ramon, Cal.; www.chevron.com) will acquire Atlas Energy, Inc. (Philadelphia, Pa.; www.atlasenergyresources.com) for cash of \$3.2 billion and assumed pro-forma net debt of approximately \$1.1 billion.The acquisition will provide Chevron with a natural gas resource position primarily located in southwestern Pennsylvania's Marcellus Shale.The acquisition is subject to certain Atlas Energy restructuring transactions, approval by Atlas Energy shareholders and regulatory clearance.

CB&I signs agreement with Sabic to license aromatics technology

November 9, 2010 — CB&I (The Woodlands, Tex.; www.cbi.com) has signed an agreement with Sabic Americas Inc. (Houston; www.sabic.com/americas/en) to jointly develop and commercialize toluene methylation technology.Toluene methylation is used to selectively produce *para-*xylene within an aromatics complex.The technology was developed around a proprietary Sabic catalyst and process.The terms of the agreement were not disclosed.

Lanxess strengthens portfolio with two businesses from Flexsys

November 8, 2010 — Lanxess AG has agreed to acquire two businesses from Flexsys, a division of Solutia Inc. (St. Louis, Mo.). Lanxess will acquire the primary accelerator business and the anti-reversion agent Perkalink 900. Both parties have decided not to disclose the acquisition prices. The acquisition of the primary accelerator business requires approval from the relevant antitrust authorities.

BASF enters agreement to market additional molecular sieve products

November 8, 2010 — BASF Catalysts LLC (Iselin, N.J.; www.basf.com) has entered into an original equipment manufacturing agreement with Chemiewerk Bad Köstritz (Thüringen, Germany; www.cwk-bk.de) to market molecular sieve products targeted at the natural gas, petroleum refining, petrochemical and air-drying markets. BASF will manage the business through its Adsorbents group, which is part of the BASF Process Catalysts and Technologies business.

Toyo and LG Group form `green' JV

October 12, 2010 — Toyo Engineering Corp. (Toyo; Chiba, Japan; www.toyo-eng.co.jp) has established a JV with LG Group (Seoul, South Korea; www.lgcorp.com) through Toyo Engineering Korea Ltd. and Serveone Co., both of which are 100% subsidiaries of their respective parent companies. The JV, named LG Toyo Engineering Co., is located in Seoul. Serveone controls 70% of the venture with the remaining 30% share controlled by Toyo-Korea.

Dorothy Lozowski

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

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Economic Indicators

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

CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	Sept.'10 Prelim.	Aug.'10 Final	Sept.'09 Final	Annual Index:
CEIndex	552.5	549.5	525.7	2002 = 395.6
Equipment	662.4	657.3	621.5	2003 = 402.0
Heat exchangers & tanks	611.8	605.8	563.4	2004 = 444.2
Process machinery	625.3	621.7	604.0	2004 = 444.2
Pipe, valves & fittings	— 834.1	827.1	768.3	2005 = 468.2
Process instruments	422.0	416.9	409.7	2006 = 499.6
Pumps & compressors	902.9	902.5	895.9	
Electrical equipment	482.5	482.7	464.7	2007 = 525.4
Structural supports & misc	680.9	675.6	632.5	2008 = 575.4
Construction labor	329.0	330.0	327.5	
Buildings	502.5	503.0	493.2	2009 = 521.9
Engineering & supervision	337.3	337.9	345.4	L



2009

2010

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

CURRENT BUSINESS INDICATORS

LATEST

89.1

91.3

157.6

120.9

Sep.'10 =

Sep.'10 =

Sep.'10 =

Sep.'10 =

Jul.'10 = 1,754.4

88.4

71.4

90.7

158.4

Aug.'10 =

Aug.'10 =

Aug.'10 =

Aug.'10 =

Aug.'10 = 260.3

Aug.'10 =

PREVIOUS

88.9

71.8

264.1

159.3

121.4

90.8

1,773.0

YEAR AGO Oct.'09 = 85.0 Sep.'09 = 1,639.5

67.9

243.2

86.0

150.4

120.0

Oct.'09 =

Oct.'09 =

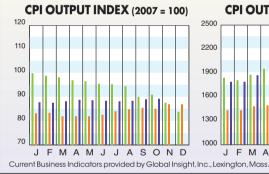
Oct.'09 =

Oct.'09 =

Oct.'09 =

2008

CPI output index (2007 = 100)	Oct.'10	=	
CPI value of output, \$ billions	Sep.'10	=	
CPI operating rate, %	Oct.'10	=	
Producer prices, industrial chemicals (1982 = 100)	Oct.'10	=	
Industrial Production in Manufacturing (2007=100)	Oct.'10	=	
Hourly earnings index, chemical & allied products (1992 = 100)	Oct.'10	=	
Productivity index, chemicals & allied products (1992 = 100)	Oct.'10	=	



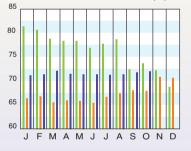
CPI OUTPUT VALUE (\$ BILLIONS) 2500 1900 1600

MAMJJASOND

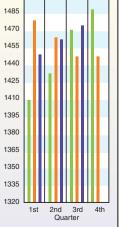
1500

JF

119.8 **CPI OPERATING RATE (%)**



(1926 = 100)	3rd Q 2010	2nd Q 2010	1st Q 2010	4th Q 2009	3rd Q 2009
1 & S INDEX	1,473.3	1,461.3	1,448.3	1,446.5	1,446.4
rocess industries, average	1,534.4	1,522.1	1,510.3	1,511.9	1,515.1
Cement	1,530.0	1,519.2	1,508.1	1,508.2	1,509.7
Chemicals	1,505.2	1,493.5	1,481.8	1,483.1	1,485.8
Clay products	1,518.3	1,505.6	1,496.0	1,494.3	1,495.8
Glass	1,428.5	1,416.4	1,403.0	1,400.1	1,400.4
Paint	1,542.1	1,527.6	1,515.1	1,514.1	1,515.1
Paper	1,444.5	1,430.1	1,416.4	1,415.8	1,416.3
Petroleum products	1,637.0	1,625.9	1,615.6	1,617.6	1,625.2
Rubber	1,579.3	1,564.2	1,551.0	1,560.5	1,560.7
Related industries					
Electrical power	1,419.2	1,414.0	1,389.6	1,377.3	1,370.8
Mining, milling	1,576.7	1,569.1	1,552.1	1,548.1	1,547.6
			3 3 3 9 9	17/05	1,767.3
Refrigeration	1,804.8	1,786.9	1,772.2	1,769.5	1,707.3



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2009 = 1.468.6

2007 = 1,373.3

CURRENT TRENDS

apital equipment prices (as Ureflected in the CE Plant Cost Index) increased from August to September, throwing yet another curve into an already atypical year. Typically, capital equipment prices escalate from January to around August or September and then decline through the end of the year. In 2010, however, equipment prices peaked in May, declined through August, and then switched directions again in September. Meanwhile, preliminary data suggests that the CEPCI increased again in October (official data will be released in the January issue).

Visit www.che.com/pci for more on capital cost trends and methodology.

Oct.'10 = Sep.'10 = 1,792.0 Aug.'10 = 72.0 Sep.'10 = Oct.'10 = Oct.'10 = 267.6 Sep.'10 = Oct.'10 =

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2005 = 1,244.5

2003 = 1,123.6

Patent applications for polymer nanocomposites have increased by 375% in the last seven years.

Process Economics Program Report: Polymer Nanocomposites

Polymer nanocomposites have garnered a great deal of interest from academia and industry in nanoscience and nanotechnology. In this new report, SRI Consulting's Process Economics Program (PEP) reviews the current state of polymer nanocomposites and focuses on nanoparticles and polymer nanocomposites that are already commercialized or have a commercial potential.

Continual progress has been made with commercialization in automotive, biological implants, sports equipment, packaging, and aircraft components. However, wide spread commercialization has moved at a slower pace than expected. Several critical technological challenges still need to be overcome, including reducing cost and significantly improving manufacturing technology. The field of polymer nanocomposites is still very much in the development phase. This report is a technology survey covering technology trends, the current state of literature covering manufacturing polymer nanocomposites, material properties and challenges. The status and potential applications, current markets and producers for polymer nanocomposites are reviewed. The report covers polymer nanocomposites containing clay-based and carbon-based nanoparticles, along with an overview of polymer nanocomposites containing other types of nanoparticles including metal oxide, polymeric, and cellulose nanoparticles.

For more information and to purchase this report, contact Angela Faterkowski, +1 281 203 6275, afaterkowski@sriconsulting.com or visit our website.

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