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# GasTIPS®

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# Comments

## DOE-GTI Partnership Powers GasTIPS

For six years **GasTIPS** has publicized Gas Research Institute's (now Gas Technology Institute's) solutions to natural gas exploration, production and processing challenges faced by U.S. producers. Although the level of FERC-managed funding for GTI research has been reduced over the past two years, GTI continues to do work of interest to **GasTIPS** subscribers. At the same time, the U.S. Department of Energy has sharpened its focus on natural gas research, both upstream and downstream, by forming the Strategic Center for Natural Gas at the National Energy Technology Laboratory. So it makes perfect sense that these two entities would find synergy in together utilizing **GasTIPS** to publicize their efforts, both independent and joint, to provide solutions to gas exploration, production and processing problems. The result is a journal that will be published four times a year instead of three, and that will highlight insights gleaned from both GTI and DOE natural gas technology research projects. As the publisher, Hart Publications will provide additional articles on gas technology topics, when deemed appropriate.

This issue includes six articles, three of which focus on gas processing topics. Improving the efficiency and cost effectiveness of gas-sweetening processes has been a focus of GTI's for years and has become increasingly important as poorer quality gas

resources are beginning to be tapped. In addition, the increased need to remove sulfur from refined petroleum fuels is opening up additional applications for removing sulfur from refinery gas streams. The first of these articles looks at CrystaSulf™, a GTI-developed liquid redox process that is particularly well-suited for middle-range sulfur removal problems. The second article describes a new process proposed for sweetening natural gas streams that carries out the Claus reaction in an organic solvent. This process, developed at the University of California, appears to have substantial capital and operating cost advantages over conventional systems. A third article describes the benefits of using a computer simulator, ProTreat™, to accurately model amine gas treating systems in situations where both carbon dioxide and sulfur dioxide contaminants are present.

Two other articles look at leading edge technologies for improving the drillstring. Researchers at The Pennsylvania State University are investigating ways to use microwave technology to improve the performance of the tungsten carbide inserts in hard rock bits. And a manufacturer of composite material equipment is developing a cost-effective composite drill pipe that promises to combine high strength and light weight. Such pipe may eventually be used to drill high angle, deepwater boreholes more cost effectively. While these technologies

are not natural gas-specific, hard rocks and deep water are two of the more important challenges facing companies exploring for new gas reserves.

The sixth article takes a moment to familiarize **GasTIPS** readers with the Strategic Center for Natural Gas and highlights the structure of the Department of Energy's natural gas research program.

The Editors would like to thank all the readers who returned the postcard asking for an e-mail address included with the last issue. These e-mails will only be used to inform subscribers when a new issue of **GasTIPS** is available online.

We trust you'll find this issue of **GasTIPS** informative. Please contact the individuals listed at the end of each article to obtain more information on specific topics. If you have any questions or comments, please contact Karl Lang at [klang@chemweek.com/](mailto:klang@chemweek.com/).

*The Editors*

# Strategic Center for Natural Gas Integrates DOE's Natural Gas Program

by Brad Tomer  
SCNG Product Manager  
for Exploration, Production  
and Storage

*Recognizing the critical role of natural gas in meeting the need for clean energy, the U.S. Department of Energy has organized a group of individuals focused on improving our nation's natural gas system performance, from "borehole to burner tip."*

**W**hether the topic is reducing air pollution, increasing energy independence, improving the efficiency of electric power production or distribution, or progressing toward the day we can tank up with hydrogen on the way to work in the morning, a common thread is one particular form of hydrocarbon fuel ... natural gas. Gas burns cleanly, the U.S. has a lot in the ground, and more and better ways of using it efficiently are being developed at a rapid rate. Whatever your perspective, natural gas is an important part of the solution to our nation's energy puzzle.

This was one of the reasons that two years ago the U.S. Department of Energy (DOE) created the Strategic Center for Natural Gas (SCNG) at the National Energy Technology Laboratory (NETL). The goal was to focus and integrate federal efforts related to all aspects of natural gas as an energy source, from "borehole to burner tip". Better integration means better technology development results.

This article provides a quick overview of SCNG research efforts and in particular those related to exploration, production and processing, areas of particular interest to many *GasTIPS* readers.

## NETL Approach Emphasizes Partnership

NETL is somewhat of a unique entity within DOE—both its mission and approach to achieving that mission differ from those of other national laboratories. NETL is federally operated—a mode of operation that is common in other agencies, but not the norm within DOE. Also, while NETL performs research within its facilities, it places an emphasis on partnering with industrial and academic organizations to create commercially viable technical solutions to problems. Other labs do this as well, but it is the primary emphasis at NETL.

While part of a single entity, NETL engineers and geoscientists carry out their research and development activities from three physical locations: Morgantown, WV, Pittsburgh, PA, and Tulsa, OK. As the federal entity responsible for implementing coal, oil, and gas programs for DOE, NETL management reports to the DOE Assistant Secretary for Fossil Energy as its Secretarial Officer.

NETL partners with industry, universities, other national laboratories, private research organizations, and federal and state agencies. Research and development activities (currently nearly 900 projects with a total value of

\$7 billion) are conducted through partnerships, cooperative research and development agreements (CRADAs), grants, and contractual arrangements.

The organization of NETL follows a matrix structure. Five Product Management "offices" (SCNG, Coal and Environmental Systems, Fuels and Energy Efficiency, National Petroleum Technology Office, and Environmental Management and Defense Programs) deal with external stakeholders and define "what" NETL does. The remaining parts of the organization, (functional components like Project Management, Business and Logistics, etc.), focus on "how" to accomplish the work.

Within NETL, the mission of the Strategic Center for Natural Gas is to conduct comprehensive science and technology development in the areas of natural gas supply, infrastructure, and utilization, as directed by national policy. From a funding standpoint, the overall DOE natural gas budget in 2001 was \$238 MM of which roughly \$125 MM was managed by SCNG. Of that \$125 MM, about \$26 MM was used for upstream programs, \$8 MM for midstream, and \$90 MM downstream. About \$24.3 MM was directed towards E&P and methane hydrate projects, a number that jumped to \$30.5 MM in

the 2002 budget signed by the President a few months ago.

## SCNG's Central Role for Natural Gas

SCNG categorizes the federal natural gas research and development activities it is responsible for into three areas: Exploration and Production, Transmission, Distribution and Storage, and Processing and End Use. Each of these areas has a well-defined goal.

The overriding goal in Exploration and Production is to develop technologies that will assist industry in ensuring adequate supplies of reasonably-priced gas are available to meet expected high future demands. Near-term efforts focus on fully exploiting existing fields. Mid-term projects target the nation's vast marginal and unconventional resource. Long-term work is designed to encourage the exploration of the nation's frontier resources, including deep gas and methane hydrates.

The goal in Transmission, Distribution, and Storage is to foster the development and deployment of technologies that guarantee the reliability, flexibility, and safety of the nation's gas delivery and storage infrastructure as it adapts to future needs. The sub-areas are: gas storage, pipelines, system integration, protection/security, and environment and land issues.

The goal in Processing and End Use is to develop competitive, energy efficient, cost-effective means of using natural gas in new and existing markets. The main sub-areas here are: fuel cells and turbines, fuel cells for dispersed power generation, simple-cycle industrial gas turbines for distributed power generation, industrial, and cogeneration, and high efficiency engines and turbines.

SCNG also supports national natural

### Table 1: Topics of Drilling and Completion-Related SCNG E&P Projects Currently Underway

#### Improving Stimulation

- Real-time monitoring of stimulation jobs via electronic telemetry.
- Carbon dioxide/sand fracturing treatments.

#### Improving Cementing

- Ceramic borehole sealants for non-vertical wellbores.
- Lightweight cement.
- Improved zone isolation during cementing.

#### Developing New or Better Drilling Systems or Components

- Mud actuated hammer drilling system.
- High pressure coiled tubing drilling system.
- Laser drilling system.
- Hydraulic pulse drilling system.
- Advanced composite material drill pipe.
- Improved tungsten carbide drill bit cutter components.
- Improved diamond compact drill bit cutter components.

#### Developing Better Ways of Collecting or Transmitting Downhole Data

- Prediction of pore pressure ahead of the bit using a seismic signal.
- Formation logging in micro-boreholes.
- Micro-drilling with realtime downhole monitoring via coiled tubing.
- High temperature MWD system.
- High speed drillstring electronic data transmission.

gas strategic planning and policymaking, working with industry, other DOE offices, and other government agencies to craft strategic plans for natural gas that reflect a comprehensive, balanced portfolio of R&D activities.

## Exploration and Production Projects Within SCNG

Currently, SCNG is managing more than 70 projects in the Exploration and Production area. In addition, a substantial number of projects have reached their conclusion during the past eight months. Many of these have produced final results that will be reported on in the near future within the pages of *GasTIPS* or through other DOE publication channels.

The topics of investigation for the

projects currently underway cover a wide range that reflects the breadth of industry's technology needs. Many are related to advanced drilling technologies (Table 1) and two of these are the discussed in articles found in this issue of *GasTIPS* (see pages 29 and 34).

Approximately 11 percent of the SCNG E&P Budget targets technologies to ensure the maximum recovery from the nation's existing fields. These efforts include support to the Petroleum Technology Transfer Council to accelerate the utilization of existing exploration technologies in development settings. Another focus is the development of improved practices for secondary gas recovery. In addition, the Stripper Well program, and the recently-established Stripper Well

Consortium, are allowing independent operators to leverage federal funding to pursue solutions to a wide variety of technical issues that lead to the premature abandonment of gas wells.

Roughly 42 percent of the E&P Budget looks to mid-range efforts designed to greatly expand the nation's production from unconventional and marginal reservoirs. These efforts include a wide variety of novel advanced diagnostic, imaging, drilling, completion, and stimulation technologies with the promise to significantly reduce the costs and risks of exploring and producing gas at the economic margins of the resource base.

The remaining 47 percent of the program's budget is dedicated to ensuring the long-range reliability of the nation's gas supply by encouraging the exploration of frontier resources. This effort includes R&D into drilling with lasers, and a new program entitled DeepTrek, that will pursue fundamental advances in materials and other technologies that may dramatically slash the costs of drilling below 20,000 feet. Also, the program is leading a national R&D effort into the area of natural methane hydrates, in cooperation with the United States Geological Survey, the Minerals Management Service, the National Oceanic and Atmospheric Administration, the Naval Research Lab, and, the National Science Foundation. DOE is investigating the connections between hydrates and drilling safety, sea-floor stability, the world's oceans, and global climate in order to allow the nation to safely tap the enormous potential of gas hydrates as a future source of energy.

### **Stakeholder Input Workshops Help Define Program**

NETL regularly hosts workshops to allow producers and others to identify

research needs that the federal government should pursue. For example, in March of last year, the SCNG hosted a workshop on deep drilling called DeepTrek. The workshop took place in Houston, TX and was designed to specifically identify ways to more economically exploit hydrocarbon resources at depths greater than 16,000 feet below the earth's surface.

At this workshop, speakers, currently drilling to these depths and beyond, outlined their experiences and problems. Attendees were then asked to identify ways to improve the drilling process for deep wells. The attendees offered suggestions on long- and short-term objectives for the SCNG to consider. The SCNG has now incorporated that input into the Deep Trek solicitation, which will be forthcoming in February 2002.

This is just one example of many workshops of this type that the SCNG holds. In preparation for these workshops, SCNG develops key issue areas around which the workshops are structured. The SCNG then asks workshop attendees to identify the key problems faced by their industry and for input to specific R&D areas that can overcome these problems. This sort of partnership provides SCNG with a perspective that is one important part of the development of a well-balanced R&D program.

### **SCNG Project Results in *GasTIPS***

During the coming months, look to find articles summarizing the results of SCNG projects within the covers of *GasTIPS*. In the meantime, visit the SCNG web site at [www.netl.doe.gov/scng](http://www.netl.doe.gov/scng) to find detailed descriptions of all of the current and past projects, online copies of the SCNG newsletter, and publications based on past research. There is also a special portion of the site devoted to the methane hydrate

program, found at [www.netl.doe.gov/scng/hydrate/](http://www.netl.doe.gov/scng/hydrate/).

Also, for those producers interested in participating in workshops like those described above or in responding to RFP solicitations for co-funded research or field demonstrations, notice of these activities can also be found on the NETL site. Once on the NETL site, click on "Business" to learn more of how to do business with NETL. ■

# CrystaSulf® Process Fills Mid-Size Niche for Sulfur Recovery in Multiple Applications

by Curtis Rueter  
CrystaTech, Inc.

*A new liquid redox sulfur removal process promises more reliability and less cost than existing methods for mid-sized treatment problems.*

As much as 25 percent of the natural gas in the United States may contain hydrogen sulfide ( $H_2S$ ) (Dalrymple, *et al.*, 1991), and worldwide the percentage could be as high as 30 percent, (Corrpt-Gandolphe, 1996). Because pipeline specifications limit  $H_2S$  to low levels, typically 4-16 ppmv, the need for cost-effective  $H_2S$  removal approaches is evident, particularly as new drilling moves into frontier areas that tend to contain resources with higher concentrations of sour gas.

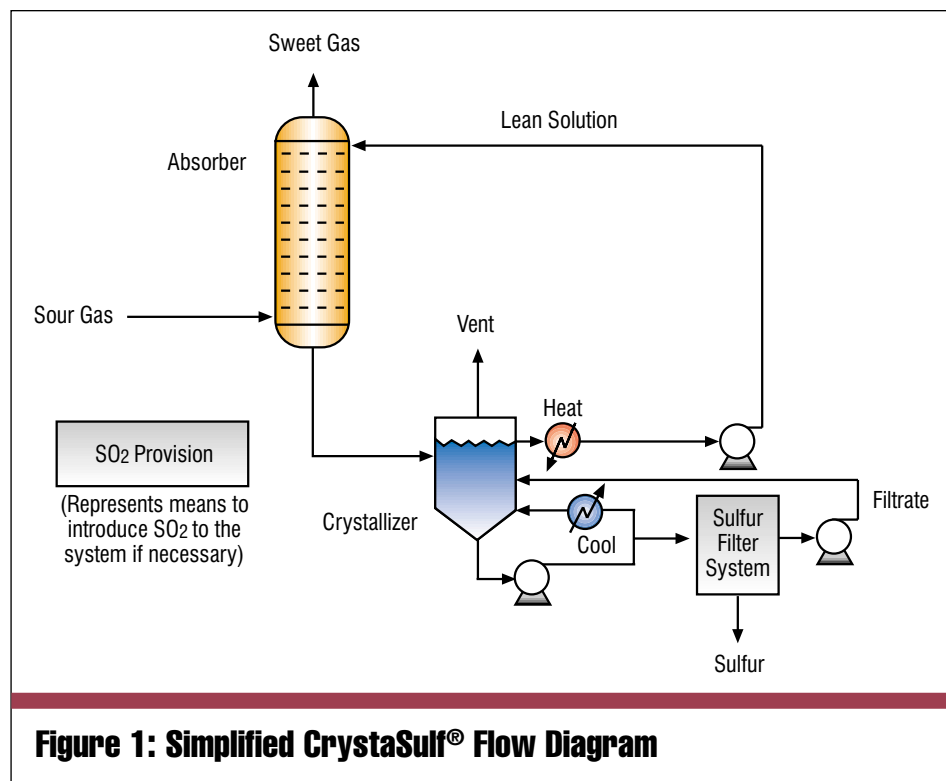
For small amounts of sulfur (< 0.1-0.2 long tons/day or LT/D), non-regenerable  $H_2S$  scavengers are frequently used, with good success, (Fisher, *et al.*, 1999). Amine/Claus/tail gas treating (TGT) combinations are well-established for larger scale treating (>25-30 LT/D). However, for the mid-size niche, the high operating costs associated with scavenging and the high capital costs associated with amine/Claus/TGT have made cost-effective treating challenging.

The CrystaSulf® process, developed in conjunction with the Gas Technology Institute (GTI) and exclusively licensed to CrystaTech, is a patented non-aqueous chemical process that removes  $H_2S$  from gas streams and is targeted at this mid-size sulfur recovery niche. CrystaSulf converts  $H_2S$  into elemental sulfur using nonaqueous chemistry and

solvents. While originally designed for high pressure applications, the same features that make CrystaSulf reliable at high pressure also make CrystaSulf a very good choice for numerous other applications as well. Its relatively low circulation rates result in reduced vessel sizes and lower capital costs in many applications, and its stable, high boiling point solution has minimal chemical losses (typically on the order of \$100 to \$200 per long ton). The first commercial unit is in the design stage and projected for a mid-2002 startup.

## Process Description

In the CrystaSulf process,  $H_2S$  is removed from the sour gas in a conventional tray absorber, which is typically comparable in size to an amine absorber (Figure 1). The  $H_2S$  reacts with dissolved sulfur dioxide ( $SO_2$ ) to produce dissolved elemental sulfur. Rich solution from the absorber passes to a flash tank (present in high pressure applications, but not in low pressure applications). Depending on the processing conditions and other options available at the site, it may at



**Figure 1: Simplified CrystaSulf® Flow Diagram**

times be economical to recompress the flash gas.

After the flash step, the solution flows to a crystallizer, where the temperature is lowered and solid elemental sulfur crystals form. The crystallizer/filter area is the only area where sulfur solids exist within the process, and they are removed by a filter system. The sulfur may be blended with Claus sulfur and sold, used in agriculture, or disposed of as non-hazardous waste. The crystallizer overflows to a surge tank, where a heater raises the solution temperature back to the circulating temperature and ensures that all elemental sulfur is dissolved in the solution. A conventional positive displacement pump transfers the solution back to the absorber.

Depending on the application,  $\text{SO}_2$  may be added to the solution in one of three ways. For smaller applications, liquid  $\text{SO}_2$  may be metered into the lean solution line. For larger applications, a portion of the product sulfur may be burned and the resulting  $\text{SO}_2$  absorbed into the CrystaSulf solution in a small separate contactor. Or, for applications where the  $\text{H}_2\text{S}$  feed gas can be combusted (e.g., amine acid gas), 1/3 of the inlet stream can be burned and the  $\text{SO}_2$  absorbed in a separate contactor.

In this process,  $\text{SO}_2$  binds chemically with species in the non-aqueous solution to form a non-volatile intermediate. A large quantity of  $\text{SO}_2$  can exist within the solution, and the solution has a very high capacity for holding more  $\text{SO}_2$ . This background concentration and excess capacity result in a buffering effect for the  $\text{H}_2\text{S}$  and  $\text{SO}_2$ , meaning that the process can be operated off the 2:1  $\text{H}_2\text{S}:\text{SO}_2$  stoichiometry in either direction for a significant period of time without losing removal efficiency.



**Figure 2: CrystaSulf® Pilot Unit Installed at West Texas Site**

The CrystaSulf process was tested thoroughly during approximately 5000 hours of pilot testing at a site in west Texas (Figure 2) that was treating  $\text{CO}_2$  for use in enhanced oil recovery. The pilot unit treated a gas stream containing 85 percent  $\text{CO}_2$  and 2000 ppmv  $\text{H}_2\text{S}$  at 300 psig. Although the site treatment requirement was only 100 ppmv  $\text{H}_2\text{S}$ , the CrystaSulf pilot unit consistently achieved 25-50 ppmv in the outlet gas with only 10 trays in the absorber, (McIntush, *et al.*, 2000). Achieving lower treatment specifications (e.g., 4 ppmv) is simply a matter of adding more trays to the contactor.

### **Different Types of Applications**

Although originally developed for use in the direct treatment of high-pressure natural gas containing moderate (0.2-25 LT/D) amounts of sulfur, CrystaSulf can be applied to other high-pressure applications (e.g., removing  $\text{H}_2\text{S}$  from a hydrogen recycle stream on refinery hydrotreaters). In addition, it is applicable to a number of low-pressure direct treat situations, such as refinery

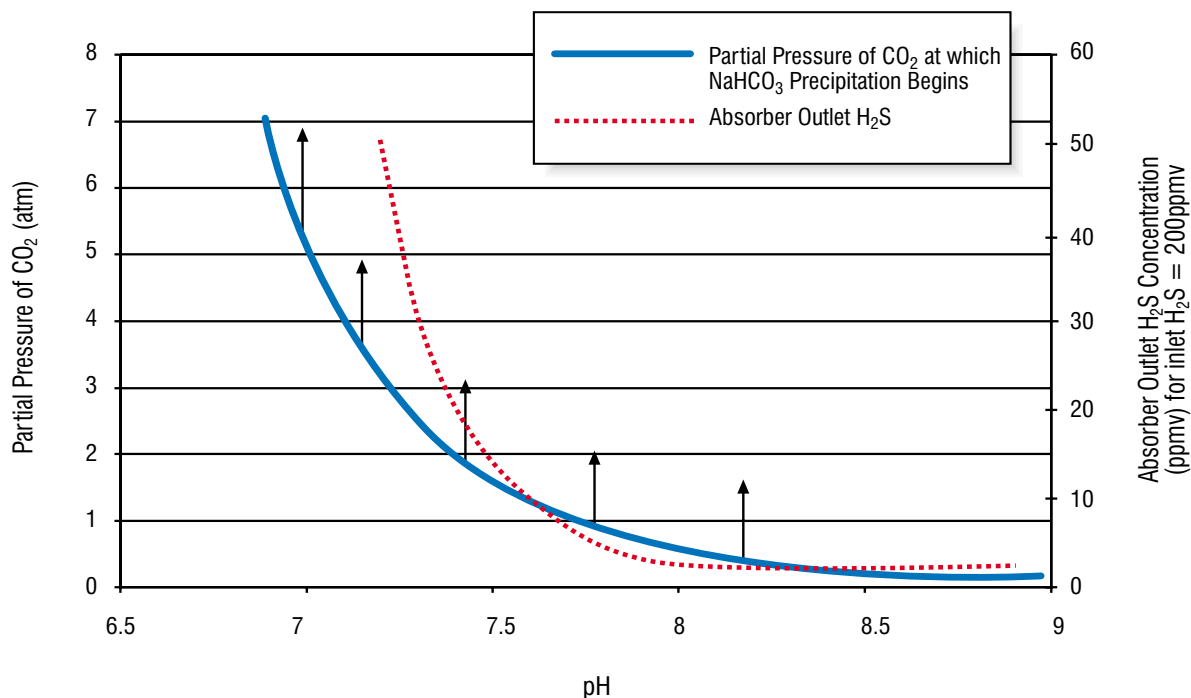
fuel gas, gasifier syngas, and gas streams from  $\text{CO}_2$  floods. Also, the process may be applied to Claus tail gas streams in the natural gas processing and refining industries. Each of these applications is described below.

### **High Pressure Natural Gas Applications**

Aqueous sulfur recovery approaches for directly treating natural gas in this size range (0.2-25 LT/D) have typically had several types of problems, (McIntush, *et al.*, 1995; Holloway, 1996), including plugging and pump wear related to circulating solids in the solution. In contrast, CrystaSulf's unique non-aqueous chemistry avoids these problems by keeping the sulfur dissolved in solution (no circulating solids).

A second issue with many aqueous sulfur recovery systems is the foaming that can result from chemical additives and hydrocarbon slugs entering the system. First, by forming large sulfur crystals, typically 50-500 microns in size, CrystaSulf avoids the need for additives and surfactants, eliminating





**Figure 3: Relationship of CO<sub>2</sub> Partial Pressure and Outlet H<sub>2</sub>S Concentration to System pH**

one source of foaming. Second, hydrocarbon slugs dissolve in the non-aqueous solution and will often be stripped back into the gas over time, but will not lead to foaming. Finally, because the sulfur is dissolved in the solution and there are no circulating solids in the pressurized portion of the system, particle-stabilized foams will not occur with the CrystaSulf process. Another issue with aqueous sulfur recovery systems directly treating high pressure natural gas has been the effect of CO<sub>2</sub>. CO<sub>2</sub> absorbs readily in aqueous systems, reducing the pH of the circulating solution. Aqueous systems must maintain a certain pH level to get the desired amount of H<sub>2</sub>S removal, and the primary absorption mechanism in these systems is negatively affected by lower pH (Figure 3). As a result, even modest partial pressures of CO<sub>2</sub> may result in increased caustic consumption to maintain pH and will make it more difficult to achieve the H<sub>2</sub>S removal specification required.

The other complicating factor is the potential for sodium bicarbonate precipitation in sodium containing aqueous sulfur recovery systems under significant CO<sub>2</sub> partial pressures. As shown on the left hand axis of the figure, a partial pressure of 0.5 atm CO<sub>2</sub> means that sodium bicarbonate precipitation may occur at approximately pH 8. However, as shown on the right hand axis, the system must operate at about this pH to achieve a 4 ppmv H<sub>2</sub>S specification. Bicarbonate precipitation may occur at high pH and H<sub>2</sub>S removal may suffer at low pH.

In general, the inability to run at high pH without precipitation problems and the poor removal at lower pH greatly limit the applicability of caustic scrubbing systems for high-pressure natural gas treating when CO<sub>2</sub> partial pressures are high. Dilute aqueous-iron systems may also see sodium bicarbonate precipitation if caustic (NaOH) is used for pH control. In one example, a dilute chelated iron

system applied to a gas stream with roughly 0.5 atm of CO<sub>2</sub> partial pressure was experiencing poor H<sub>2</sub>S removal due to operation at low pH; increasing the pH by caustic (NaOH) addition was not feasible because of bicarbonate precipitation, (Reicher, *et al.*, 2000). Switching to potassium hydroxide (KOH) resolved the problem, but at a much greater cost than NaOH on a per mole basis.

In contrast, CrystaSulf absorbs virtually no CO<sub>2</sub>, and as a non-aqueous system, has no pH to be affected by CO<sub>2</sub>. As a result, neither H<sub>2</sub>S removal capability nor chemical consumption rate is affected by the presence of CO<sub>2</sub>. In addition, CrystaSulf has a low circulation rate compared to other treatment options (Table 1). A low circulation rate helps to minimize the energy costs required to boost a liquid from atmospheric to high pressure. Lower circulation rates also enable designs that incorporate smaller vessels and reduced capital costs.

**Table 1: H<sub>2</sub>S Absorber Circulation Rates for Various Treatment Options (0.5 vol% H<sub>2</sub>S, 2.0 vol% CO<sub>2</sub>)**

System	gpm/LTPD
CrystaSulf®	20 - 50
Alkanolamine (MDEA)	30 - 60
Aqueous-iron	200 - 1,200
Caustic with bacterial regeneration	300 - 3,000

### HDS Recycle Streams

CrystaSulf's non-aqueous chemistry and ability to remove H<sub>2</sub>S at high pressure also create some unique advantages for treating hydrodesulfurization (HDS) recycle streams. Refineries are presently reviewing their gasoline and diesel hydrotreaters and determining how to comply with EPA's Tier II regulations, which will require that low-sulfur gasoline and ultra-low sulfur diesel be produced. Hydrotreating is likely to be the primary means of meeting these regulations.

In a conventional hydrotreater, sulfur-bearing hydrocarbon liquids (e.g., diesel) and an excess of hydrogen gas are fed to a reactor system containing a special catalyst. Hydrogen

reacts with the organic sulfur compounds in the liquids at elevated temperatures and pressures to produce H<sub>2</sub>S within the reactors. The (now) sweet hydrocarbon liquids are separated from the (now) sour hydrogen stream. The H<sub>2</sub>S is then removed from the hydrogen stream, typically by an alkanolamine system, and the excess hydrogen is recycled to the front of the process. Fresh hydrogen, typically made by reforming methane and containing around 5 percent methane as a contaminant, is added to the hydrogen recycle stream to replace the hydrogen consumed by the reaction or otherwise lost in the system. A portion of the recycled hydrogen is often purged or blown down from the system to avoid the accumulation of methane and other

light hydrocarbons formed by cracking across the catalyst.

CrystaSulf has two primary advantages in treating the hydrogen recycle stream off a hydrotreater. First, it can remove the H<sub>2</sub>S and provide point source sulfur recovery, which means that no additional load is added to the refinery's sulfur recovery unit (this is particularly important if the SRU is fully loaded). Second, the CrystaSulf circulation rate can be increased sufficiently so that the non-aqueous solution can effectively scrub the hydrocarbons from the HDS recycle stream, eliminating the need for the hydrogen purge and resulting in substantial hydrogen savings. Table 2 shows these benefits for a diesel hydrotreater at a 215 M barrel/day (BPD) refinery. If a conventional amine system is used along with the hydrogen purge, then 46.3 lb-moles hydrogen/hr are purged, along with approximately 8.2 lb-moles/hr of hydrocarbons. However, if CrystaSulf were used in this system, the light hydrocarbons could be scrubbed from the hydrogen recycle stream and vented off the flash gas vessel. The net

**Table 2: CrystaSulf Results in Less Hydrogen Loss, Better Removal of Light Ends**

Component	Hydrogen Purge Components (conventional amine), lbmol/hr	Flash Gas Components (CrystaSulf) lbmol/hr	Hydrogen Savings with nonaqueous approach lbmol/hr
CO <sub>2</sub>	—	0.003	32
H <sub>2</sub>	46.3	14.3	
H <sub>2</sub> S	0.03	—	
C <sub>1</sub>	6.1	3.83	
C <sub>2</sub>	1.0	4.63	
C <sub>3</sub>	0.45	1.99	
i-C <sub>4</sub>	0.3	1.34	
n-C <sub>4</sub>	0.3	1.29	
Total Light Hydrocarbons Removed (C <sub>1</sub> thru C <sub>4</sub> )	8.15	13.08	Additional 4.93 lb-mole/hr HC is removed

benefits are both a reduction in hydrogen consumption of 32 lb-moles/hr (equivalent to a savings exceeding \$1 MM/year at this refinery) as well as greater removal of hydrocarbons (an additional 4.93 lb-moles/hr), resulting in higher purity hydrogen and a more efficient HDS process.

### Other Direct Treatment Streams

In addition to the two examples described, CrystaSulf can be applied to a variety of low pressure streams as well. Examples of these include refinery fuel gas streams, gasification or syngas streams, and CO<sub>2</sub> for enhanced oil recovery, (McIntush, Reicher, *et al.*, 2001). The first commercial CrystaSulf plant will actually be installed on a refinery fuel gas stream, while the pre-commercial pilot plant operated on a 300 psig CO<sub>2</sub> stream.

In addition to the operating advantages described previously (e.g., absence of circulating solids), CrystaSulf's stable solution and chemistry is a key advantage in applications that have various contaminants (CO<sub>2</sub>, CO, H<sub>2</sub>, O<sub>2</sub>, HCN, NH<sub>3</sub>, etc.) that often negatively influence other processes. For example, while the effect of CO<sub>2</sub> on aqueous sulfur recovery processes has already been discussed, the presence of even small amounts of oxygen (a few hundred ppm) can greatly increase amine degradation rates. Hydrogen cyanide (HCN) and carbon monoxide are not expected to be handled well by biologically-based processes, and HCN has been known to cause problems in coke oven gas treatment by at least one aqueous sulfur recovery system.

### Claus Tail Gas

To achieve high levels of sulfur recovery (> 99.8 percent), H<sub>2</sub>S-recycle processes are typically used in Claus tail gas applications. Because Claus unit tail gas is typically at high

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temperature (around 300°F) and contains a significant amount of SO<sub>2</sub>, it must first be reacted with hydrogen to convert the SO<sub>2</sub> to H<sub>2</sub>S, and then cooled (typically in a water quench system) down to 100-120°F before being sent to an amine system operating at near-atmospheric pressure. If SO<sub>2</sub> breaks through the hydrogenation reactor to the amine system, the amine is often seriously damaged and must be replaced. The acid gas from the amine regenerator is then recycled to the front end of the Claus unit.

In contrast, CrystaSulf has a number of advantages for this application:

- The quench system is eliminated. The high-boiling point solution (> 250°C) treats the gas directly without separate, dedicated cooling equipment.
- The hydrogenation reactor is eliminated. H<sub>2</sub>S and SO<sub>2</sub> are reacted together in the CrystaSulf solution.
- No recycle of H<sub>2</sub>S or CO<sub>2</sub> to the front end of the plant is required. Recycling these species may reduce the Claus reaction furnace capacity or temperature. Eliminating the

recycle simplifies the Claus unit controls and increases capacity.

- Solution buffering capacity is high. The solution has a high capacity for absorbing SO<sub>2</sub>, and the process normally operates with a background level of SO<sub>2</sub> absorbed in the solution (in a stable intermediate form). Thus, even if the tail gas goes off-ratio for a period of time, CrystaSulf will continue to achieve high levels of overall control efficiency.
- Solution is resistant to contaminants. CrystaSulf solution is not harmed by SO<sub>2</sub> or any other species commonly found in Claus tail gas. If an excursion occurs that sends high levels of SO<sub>2</sub> to the CrystaSulf unit, there is absolutely no harm done to the solution.
- Ease of sulfur disposal. Formation of large sulfur crystals that are 99 percent sulfur and leave the filter with only 2 percent water. As a result, the sulfur product is relatively dry and is of sufficient purity that it can be blended into the Claus sulfur.

for sale. Liquid SO<sub>2</sub> will be purchased from a readily available local source. The installation of the unit will reduce the refinery's overall emissions to a level below existing air quality thresholds, meaning the refinery will be able to increase throughput while decreasing emissions. This alone will save the refinery an estimated \$100,000/year in regulatory compliance costs. ■

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*For more information on CrystaSulf technology solutions for your gas processing applications, contact Curtis Rueter at CrystaTech, at 303-466-5056 or via e-mail at [curtis.rueter@crystatech.com/](mailto:curtis.rueter@crystatech.com/).*

## **First Plant to be Constructed**

After the successful pilot unit operation, the process was deemed ready for commercial applications. The contract for the first CrystaSulf unit, currently in the design stage, was signed in August 2001, and this unit is presently slated for a mid-2002 startup. Located at a refinery in Corpus Christ, Texas, the unit will clean a fuel gas stream at approximately 100 psig and 120°F. The inlet gas stream contains 13 percent H<sub>2</sub>S and high concentrations of CO<sub>2</sub>, CO, and O<sub>2</sub>, while the treated gas will have less than 100 ppmv. The flash tank will be operated at atmospheric pressure and recovered vapors recompressed into the fuel gas system. The overall process will recover 2.7 tonnes/day of sulfur, which will likely be blended into a local sulfur terminal

# New Approach To Sulfur Removal Could Reduce Costs

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*A new process proposed for sweetening natural gas streams carries out the Claus reaction in an organic solvent. It appears to have substantial capital and operating cost advantages over conventional systems.*

**A**s sweeter natural gas and crude oil supplies are exhausted worldwide, refiners and gas processors are faced with need to process less desirable (higher sulfur content) feedstocks. At the same time, governments are requiring that fuels processed from these feedstocks contain less residual sulfur, as a regulatory control on exhaust emissions. The sulfur species found in natural gas or obtained during the desulfurization of crude oil is generally hydrogen sulfide ( $H_2S$ ), with perhaps small amounts of mercaptans, and the conventional means of recovering the sulfur is the modified Claus process. In the past, the gas leaving a Claus plant (tail gas) was burned to convert the unreacted (and lethal)  $H_2S$  to sulfur dioxide ( $SO_2$ ) which, when vented to the atmosphere, contributed to acid rain. To avoid this result, tail gas clean-up units (TGCUs) have been added that often have capital and operating costs as great as the parent Claus plant. The Shell Claus Off-gas Treatment (SCOT) process is a common example.

Both the refining and natural gas industries are interested in sulfur recovery processes that promise lower cost, higher overall sulfur recoveries or both. A solvent-based sulfur recovery process (UC Sulfur Recovery Process or UCSRP) being developed at the University of California at Berkeley

appears to be significantly lower in both capital and operating costs than a Claus/SCOT unit of equivalent sulfur capacity, based on an engineering design comparison.

In the UCSRP, a gas stream containing  $SO_2$  and excess  $H_2S$  is fed to a reactor with a circulating organic solvent above the melting point of sulfur. Bright yellow liquid sulfur, free of  $H_2S$ ,  $SO_2$  and solvent, is produced. Unreacted  $H_2S$  flows to a furnace where it burns to form a small amount of elemental sulfur together with  $SO_2$ , which are recovered in an absorber/stripper operation and sent back to the reactor. Overall sulfur recovery is 99.9+ percent. For a 77.3 tonnes/d (76 LT/d) plant, estimated capital costs were 60 percent and estimated operating costs 68 percent of those for a conventional three-stage Claus/SCOT plant. This article compares overall sulfur recovery, capital and operating costs, and operational features of the two processes.

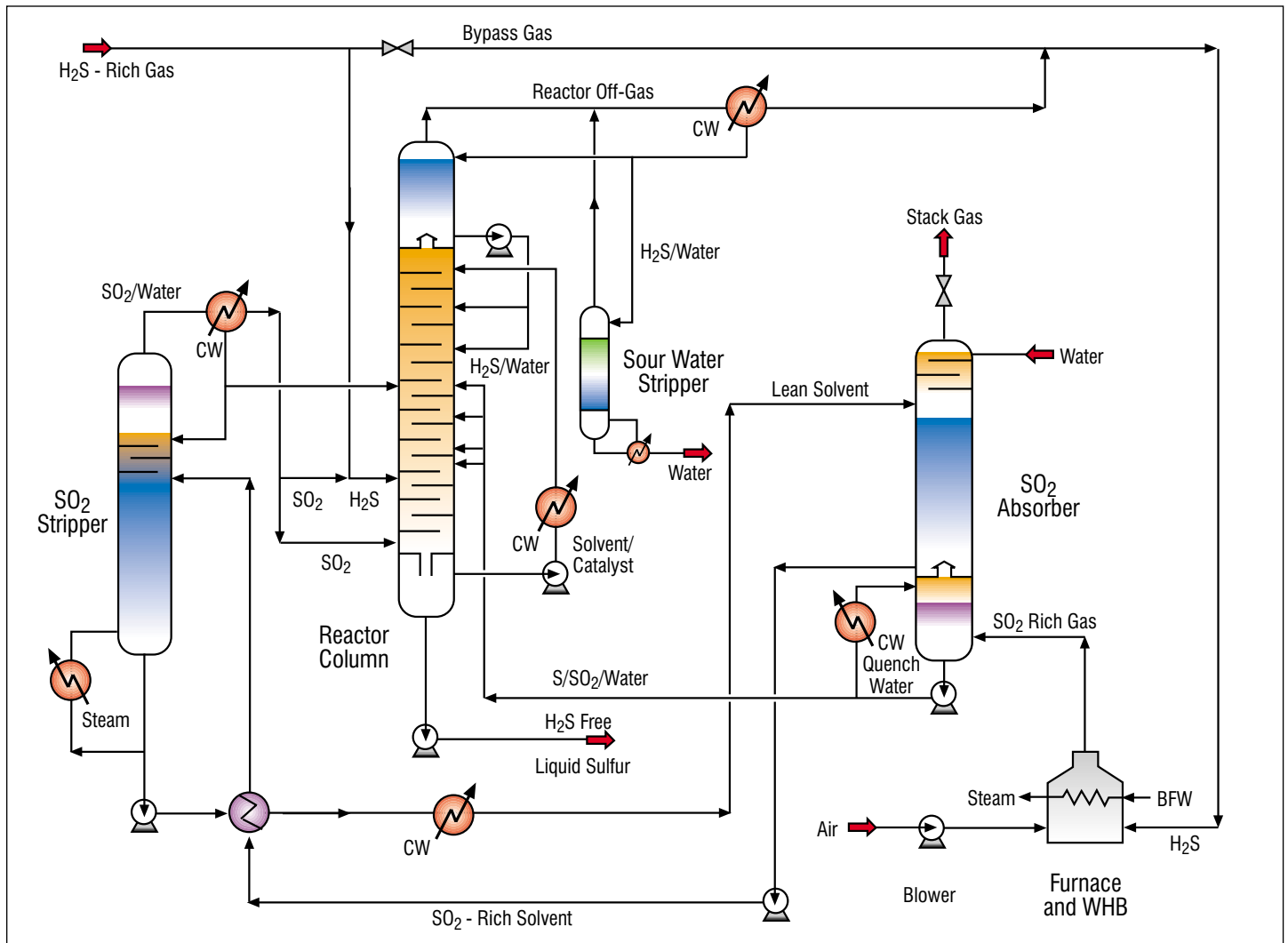
## Claus/SCOT Process Overview

The Claus process produces elemental sulfur by burning part of the  $H_2S$  to form  $SO_2$  and reacting uncombusted  $H_2S$  with  $SO_2$  (Changela, et al., 1990; Paskall, 1989). Most modern sulfur recovery plants use the modified Claus process, where the reaction is carried out in steps. In the acid-gas burner, 1/3 of the

$H_2S$  in the feed gas is oxidized to  $SO_2$ . Simultaneously, an uncatalyzed reaction occurs in the furnace between the unburned  $H_2S$  and the  $SO_2$ , which are in stoichiometric ratio, converting about 60 percent of each to sulfur vapor. The gas leaving the furnace is then cooled to condense sulfur, reheated, and passed through a catalytic converter.

Most modified Claus plants have two or three catalytic converters, operating at successively lower temperatures in the range 220° – 180°C, each followed by its own condenser and all but the last condenser followed by a reheater. The two-converter system may achieve a sulfur yield of up to 94 percent whereas the three-converter system may achieve a sulfur yield up to 98 percent. The gas leaving the final catalytic condenser is typically sent to a TGCU. Several variations of the Claus process have been developed to handle a wide range of feed-gas compositions.

The SCOT process consists of two sections: a hydrogenation/hydrolysis section, followed by water quench, and a selective-amine gas-treating section. In the first section, the tail gas from the Claus unit is heated to the range of 250° – 300°C, then reacted with hydrogen (or a reducing gas) over a cobalt molybdate catalyst. All sulfur compounds ( $SO_2$ , S, COS,  $CS_2$ , etc.) are converted to  $H_2S$ . The off-gas from the reactor is cooled to



**Figure 1: Process Flow Diagram for the UC Sulfur Recovery Process**

approximately 180°C in a waste-heat boiler followed by a water quench. Finally, H<sub>2</sub>S is selectively absorbed by an alkanolamine solution such as MDEA or DIPA. The rich amine solution is stripped and the H<sub>2</sub>S-rich stream is recycled to the front end of the Claus plant (Naber, et al., 1973; Goar, and Sames, 1983; Goar, 1975). The treated gas from the SCOT absorber is normally incinerated if the H<sub>2</sub>S content exceeds 100 ppmv. The incinerated gases are discharged from a high stack.

### UCSRP Overview

The UCSRP is a solvent-based process for reacting hydrogen sulfide (H<sub>2</sub>S) and

sulfur dioxide (SO<sub>2</sub>) to form elemental sulfur. The reaction medium is an organic liquid that catalyzes the reaction. H<sub>2</sub>S-rich gas enters the UCSRP unit at about 50°C and 1.7 bar, typical of a Claus-plant feed. The reactor resembles a bubble-cap distillation or gas-absorption column. A small fraction of the SO<sub>2</sub>-rich gas stream enters the reactor column below the bottom bubble-cap tray (Figure 1). The remainder of the SO<sub>2</sub>-rich gas stream together with about 75 percent of the H<sub>2</sub>S-rich stream enters a tray or two higher and H<sub>2</sub>S is in substantial excess at all points above. The remainder of the inlet H<sub>2</sub>S bypasses the

reactor to reduce the gas flow through it. Hydrocarbons, mercaptans, carbon dioxide, and many other potential impurities in the H<sub>2</sub>S are inert to the reaction and pass through the reactor unchanged. Although some ammonia can be tolerated within the reactor, it is generally preferable for most of the ammonia-containing H<sub>2</sub>S entering the UCSRP to be part of the bypass stream that flows directly to the furnace. The gases are absorbed into the hot solvent where an exothermic reaction takes place. The reactor is kept nearly isothermal by injecting water into the solvent flowing between trays to absorb the heat of reaction by vaporizing. The

sulfur forms a second liquid phase. The solvent, which is recycled in a circulation loop from the bottom to the upper part of the reactor column, is saturated at all times with elemental sulfur.

Both liquid phases flow down the column, being separated by decantation at the bottom of the column where the solvent stream is collected for recycle. On the bottom one or two trays H<sub>2</sub>S is stripped and/or reacted from the solvent by the entering SO<sub>2</sub>. The decanted solvent contains negligible H<sub>2</sub>S and a very low concentration of SO<sub>2</sub>. In addition, H<sub>2</sub>S is also stripped (or reacted away) from the liquid sulfur flowing through this section. The liquid sulfur from the reactor then flows to a sulfur pit. The overhead vapor from the reactor column is scrubbed with a water reflux to remove solvent vapor and unreacted SO<sub>2</sub> (which reacts very rapidly with H<sub>2</sub>S in water), then passes through condenser. Part of the water separated from the reactor off-gas is pumped back to the reactor as reflux. The net condensed water is sent to a sour-water stripper where dissolved H<sub>2</sub>S and CO<sub>2</sub> are stripped overhead and pure water is produced.

Unreacted H<sub>2</sub>S leaves the condenser, mixes with the bypassed acid-gas feed and flows to the furnace where it is burned with slightly substoichiometric air to form SO<sub>2</sub> and a small amount of S<sub>2</sub> (see Reaction Furnace section below).

The hot combustion gas, containing S, SO<sub>2</sub>, H<sub>2</sub>O, CO<sub>2</sub>, and N<sub>2</sub>, is cooled by producing 4-bar (or higher pressure) steam in the waste-heat boiler and is then sent to the SO<sub>2</sub> absorber. At the bottom of the absorber a water quench cools the combustion gas to near-ambient temperature, condensing a large fraction of the water vapor as well as the sulfur vapor. The net dilute slurry (about 2 to 4 weight percent) of colloidal sulfur, slightly acidic because of dissolved SO<sub>2</sub> and small amounts of

**Table 1: Assumptions for Comparison of UCSRP and Claus/SCOT**

- Modular construction was used for both plants.
- Plant capacity of 77.3 tonnes (76 long tons) per day of sulfur
- Location in a remote section of western Texas. No import or export of electricity. Plants self-contained except for raw materials and chemicals.
- Project contingency 20% of base plant cost.
- Electric power generated by a gas fueled generator (1000 BTU/Scf fuel gas) with a heat rate of 9,000 BTU/hp-hr (28% thermal eff.).
- Stream factor 96% (350 days/year).
- UCSRP and Claus/SCOT plants have 4 operators working eight hours per day, three shifts per day, seven days per week, 52 weeks per year at \$14.00 per hour.

higher sulfoxy acids, is sent to the reactor column to provide part of the cooling mentioned above.

The cooled combustion gas then flows counter-current to the stream of lean solvent that absorbs the SO<sub>2</sub>. The off-gas from the top of the SO<sub>2</sub> absorber is sent to the atmosphere via a tall stack. The stack gas, which does not require heating or incineration, may have an SO<sub>2</sub> content of 100 ppmv or less. The SO<sub>2</sub>-rich solvent is pumped to the stripper where the SO<sub>2</sub> is stripped from the solvent and sent to the reactor. A condenser for the stripper vapor separates water for the stripper reflux, while the net water from the stripper vapor is also used for cooling in the reactor column. After being cross-exchanged by the rich solvent, the lean solvent is further cooled and returned to the absorber.

### Assumptions and Design Criteria

The evaluation of the UCSRP and its comparison to the conventional Claus/Scott process was carried out using a consistent set of assumptions and design criteria. The assumptions are listed in Table 1. Two different H<sub>2</sub>S/CO<sub>2</sub> feed gas ratios were evaluated: 67/33 and 30/70 (Table 2).

Bulk costs for process units were determined using Kellogg, Brown and Root's (KBR) estimating program, ICARUS Process Evaluator (IPE). Claus costs were updated from an earlier GRI study (Changela, et al., Topical Report Task 3), and SCOT unit costs were developed as part of a separate GRI study (Strickland, et al., Topical Report Task 49). The costs for the UCSRP process were developed from sized equipment lists. Two sets of cost estimates for each case were requested from KBR's estimating group. One set assumed the metallurgy of the plant to be stainless steel and the other set assumed the metallurgy to be carbon steel. Vendor quotations were obtained for the more expensive or specialized equipment items.

Standard sizing techniques were used for the design of both process configurations, based on experience, residence time, and acceptable space or gas separation velocities. Sulfur pits were sized for a seven-day storage capacity and the sulfur load-out pump was rated for less than eight hours pump-out. Proprietary information from UC Berkeley was used to size the UCSRP equipment.

**Table 2: Feed Gas Composition**

	67/32 H <sub>2</sub> S/CO <sub>2</sub>	30/70 H <sub>2</sub> S/CO <sub>2</sub>
CH <sub>4</sub> (Mole %)	0.2	0.2
H <sub>2</sub> S (Mole %)	62.6	27.8
CO <sub>2</sub> (Mole %)	30.2	64.9
H <sub>2</sub> O (Mole %)	7	7
Feed (kmol/hr)	151.8	350.7
Pressure (bara)	1.7	1.7
Temperature (°C)	50	50
Cost (\$/MMBtu)	1.50	1.50
Claus Plant Configuration	Straight through flow	Split flow

**Table 3: Equipment Counts for Both Options**

Feed	UCSRP	Claus/SCOT
67/33 H <sub>2</sub> S/CO <sub>2</sub>	42	64
30/70 H <sub>2</sub> S/CO <sub>2</sub>	42	66

## Design Comparison

### ► Sulfur Recovery.

The expected overall sulfur recovery for the UCSRP process is 99.9+%. Sulfur loss consists primarily of SO<sub>2</sub> vented from the SO<sub>2</sub> absorber overhead. The design of the UCSRP absorber is conventional, with the design parameters being: gas/solvent flow ratio, number of equivalent stages in the absorber, solubility of SO<sub>2</sub> in the solvent (a function of composition, temperature and pressure) and concentration of SO<sub>2</sub> in the lean solvent feed to the absorber (which in turn depends upon the operation of the stripper). Many glycol ethers are quite good physical solvents for SO<sub>2</sub> and solubility data have been obtained for several of them.

It is expected that the liquid sulfur will be free of dissolved H<sub>2</sub>S because of its contact with SO<sub>2</sub>-containing solvent at the bottom of the reactor column. The UCSRP configuration makes it very unlikely that H<sub>2</sub>S vapor will reach the atmosphere without having been burned to SO<sub>2</sub>.

### ► Temperature Management.

The reactor is operated within a range of 125° – 135°C. If the feed gas is lean in H<sub>2</sub>S, then the relative magnitude of the heat of reaction is reduced. The solvent flow must be sufficient to make its sensible heat exceed that of the gas stream. The water content of the solvent will then adjust, through the control process, to prevent excessive cooling by evaporation at the bottom of the reactor.

### ► Equipment Counts.

A single reactor in the UCSRP Process replaces the Claus unit, which has 3 stages. A two-stage Claus plant would save investment capital for the Claus option, but require a higher operating cost for the SCOT section. The furnace section of the UCSRP has equipment similar to the thermal section of a Claus unit (air blower, reaction furnace, waste heat boiler, and thermal condenser).

The Absorber and Stripping sections in UCSRP take the place of the SCOT unit in terms of clean-up function, but the number of equipment items is far fewer than are found in the Hydrogena-

tion/ Hydrolysis, Quench, Stripping and Regeneration sections of a SCOT unit (Table 3). In addition to a lower equipment count, the size of equipment in UCSRP is likely to be smaller than for Claus/SCOT. Some parts of the Claus and SCOT equipment are relatively large because a high-temperature, low-pressure gas is being processed.

### ► Reaction Furnace.

The UCSRP furnace is expected to behave similarly to a Claus plant furnace, but will operate at a higher temperature for a given feed-gas composition because there is much less dilution by excess H<sub>2</sub>S. For instance, with a process feed containing about 33% CO<sub>2</sub>, the adiabatic flame temperature for the furnace burner is about 2570°F (1410°C). The slightly H<sub>2</sub>S-rich combustion requires only a single stage, with about 1 to 2 percent of the H<sub>2</sub>S being converted to S<sub>2</sub>. The S<sub>2</sub> ensures complete reaction of the O<sub>2</sub> and precludes formation of both SO<sub>3</sub> and NO<sub>x</sub> because the temperature is high enough for chemical equilibrium to be established. Similarly, the high concentration of SO<sub>2</sub> forms an atmosphere that is much more oxidizing than that in a Claus furnace and will prevent the formation of soot, CO, COS or H<sub>2</sub>. Since the absorption of SO<sub>2</sub> is favored by low temperature, the gases from the reaction furnace are quenched to near-ambient temperature with a water wash at the base of the SO<sub>2</sub> absorber. Most of the water of combustion, as well as the sulfur vapor, is condensed in this step. The net water collected in this step contains 2 to 4 weight percent sulfur and is sent to the reactor column where the water serves as coolant and the sulfur is collected as product. The rest of the water is absorbed with the SO<sub>2</sub> from the combustion gas by the solvent in the stripper. Because the furnace is downstream of the reactor, and the SO<sub>2</sub> is recycled to the reactor, control of the



overall process stoichiometry is made automatic by controlling the furnace stoichiometry as described.

► **Utilities.**

The power requirements in UCSRP may be larger than for the Claus/SCOT option primarily because of the air blower and cooling water system. For both processes, the major power-consuming units are the air blowers. The air blower in UCSRP has to overcome the pressure drop through the reaction furnace, waste-heat boiler and SO<sub>2</sub> absorber. In Claus/SCOT, the air blower has to overcome the pressure drop of the Claus furnace and three Claus stages, plus the drop through the Hydrogenation/Hydrolysis, Quench and Amine Absorber sections of the SCOT unit. UCSRP does not require fuel gas except for power generation. The reducing-gas generator of the SCOT unit requires a small quantity of fuel gas. More importantly, Claus/SCOT must include an incinerator, which is a significant fuel gas user, if the treated gas contains more than 10 ppmv H<sub>2</sub>S.

Some external cooling is required in both UCSRP and Claus/SCOT. Furthermore, the SCOT unit generally requires additional trim cooling for the quench water pump-around and the lean amine. In this analysis, it was assumed that air cooling may be used for the UCSRP process since the lowest temperature required is about 40°C (condensers, quench water and solvent to SO<sub>2</sub> absorber).

The UCSRP process requires about the same amount of steam as that of the Claus/SCOT Unit. For UCSRP, the reboiler for the SO<sub>2</sub> stripper is a large steam user (about 36% of the steam produced in the waste-heat boiler) primarily because of the heat load required to strip out the SO<sub>2</sub> and the remaining water of combustion after heating the solution to 140°C. Although it has a higher power requirement than

Claus/SCOT, overall the UCSRP process has lower utilities costs than Claus/SCOT.

► **Catalyst and Chemicals.**

Claus catalyst life can be 3 years or longer, and SCOT catalyst life can be 5 years or longer. The UCSRP solvent operates at temperatures where deterioration of the solvent is unlikely to be significant unless degradation is found to result from contaminants in the acid gas feed. At present, one can expect that the UCSRP will have lower costs for replacement of solvent and other chemicals than Claus/SCOT.

► **Emissions and Safety.**

The emission sources within UCSRP and Claus/SCOT are identical, but the quantities of emissions are expected to be less for UCSRP. It is possible that surge or storage tanks that vent to atmosphere may require some device to prevent release of volatile organic compounds (VOCs) and H<sub>2</sub>S in the vent gases. In the Claus and SCOT units, the major emissions are from the incinerated gas. These emission sources compare to emissions from the SO<sub>2</sub> absorber off-gas in the UCSRP case. Neither process should emit more than 250 ppmv SO<sub>2</sub>. NO<sub>x</sub>-suppression equipment is not required for the reaction furnace of either process since both operate under reducing conditions. The quantity of sour water discharged or requiring treatment in Claus/SCOT will be greater than for UCSRP. There appears to be no significant difference in safety considerations between the processes.

► **Metallurgy.**

Corrosion data obtained in the laboratory indicate that the UCSRP plant could be all carbon steel. The experiments used diethylene glycol methyl ether (DGM) containing 5 weight percent water, saturated with sulfur, as the solvent. The solvent contained either 1.0 mole SO<sub>2</sub> or 0.9

mole H<sub>2</sub>S per liter. The maximum temperature was 140°C for SO<sub>2</sub> and 120°C for H<sub>2</sub>S. The metals tested were carbon steel, 304 stainless and 316 stainless. With SO<sub>2</sub> at the highest temperature, after a test of 24 days, the corrosion rates for all three metals were essentially the same at about 8 μm (3 mil) per year. With H<sub>2</sub>S at the highest temperature, after a test of 24 days the corrosion rates for all three metals were also essentially the same at about 0.1 μm (0.05 mils) per year. In the cost estimates, KBR conservatively specified stainless steel for the absorber, stripper, reactor and lean/rich exchanger and used carbon steel in the rest of the equipment.

► **Turndown and the Effect of Pressure.**

Turndown does not represent any significant problems for UCSRP and, if anything, will be easier with UCSRP than with Claus/SCOT. At lower operating rates, solvent residence time on the reactor trays will increase relative to the gas flow, which will improve the driving force for gas absorption. The effect of pressure on UCSRP is minimal since the system already runs at low pressure.

## **Economic Comparison**

► **Capital Costs.**

Relative investment costs for UCSRP are lower than for Claus/SCOT. The Direct Fixed Capital (DFC) for UCSRP is estimated to be 61% of the DFC for a conventional straight-through Claus/SCOT unit treating an H<sub>2</sub>S/CO<sub>2</sub> ratio of 67/32, and 60% of that for a split-flow system treating an H<sub>2</sub>S/CO<sub>2</sub> ratio of 30/70. If carbon steel is used throughout, the relative capital costs for UCSRP are 59% and 58%, respectively, of those for conventional Claus/SCOT units for the high and low H<sub>2</sub>S feed cases.

It is well to note that the estimate of the capital cost for a given piece of equipment has an uncertainty of about 25%. However, every effort was made to do all estimates for both processes using the same methodology and making the same assumptions. As a result, random errors tend to offset each other and the uncertainty of the comparison should be better than 25%.

#### ► **Operating Costs.**

The relative operating and maintenance costs for UCSRP are estimated to be 68% of those for the straight-through Claus/SCOT unit for an H<sub>2</sub>S/CO<sub>2</sub> ratio of 67/32, and 67% for the case of the split-flow system treating an H<sub>2</sub>S/CO<sub>2</sub> ratio of 30/70. If carbon steel is used throughout, the relative operating costs for UCSRP are 67% and 65%, respectively, of those for a conventional Claus/SCOT unit for the high- and low-H<sub>2</sub>S feed cases.

### **Operational Considerations**

#### ► **Sulfur Separation.**

In the UCSRP the reactor column also serves as a 3-phase separator (V/L/L). The reactor operates at temperatures above the melting point of sulfur, and hence should avoid sulfur-plugging problems. Because sulfur is produced as a liquid, there is no need for flotation and melting. Instead, sulfur is coalesced and decanted. The high density of liquid sulfur (1.8 g/mL) should allow it to separate rapidly from the solvent. Since the solvent in the reactor will contain dissolved (and possibly entrained) sulfur, this might cause sulfur blockage if the temperature of the solvent drops too much after leaving the reactor. The pump and piping for the solvent pump-arounds will require steam tracing, just as will the reactor column itself.

#### ► **Vapor Recovery.**

The H<sub>2</sub>S-rich gas exiting the solvent section of the reactor column will be

saturated with sulfur and solvent vapors and may contain some SO<sub>2</sub>. To prevent loss of these components a knock-down section of trays at the top of the column carries water at 95° – 105°C where sulfur is condensed as a very dilute slurry, solvent vapor is absorbed and the SO<sub>2</sub> is reacted away in the aqueous phase to form a very dilute mixture of sulfoxy acids and colloidal sulfur. The net flow of water through this section is only a few percent of the solvent flow through the lower section of the reactor column. This stream of water mixes homogeneously with the solvent when injected as coolant in the down-comers of the upper section of the column. Similar water-wash sections prevent loss of solvent vapors at the tops of the SO<sub>2</sub> absorber and stripper columns.

#### ► **Start-up and Shut-down.**

The initial start-up period of a Claus plant is long, primarily because of the time required to bring the various units up to their operating temperatures, and then to achieve the desired tail-gas ratio. It is anticipated that less initial start-up time will be required for UCSRP since operating temperatures are lower (except for the furnace) and the process is not as dependent as the Claus process on precise air control. During start-up the furnace can be operated with a fuel gas to generate steam and the solvent flows through the reactor column and stripper can be circulated to bring the system up to temperature before the feed of H<sub>2</sub>S is begun. Planned shut-downs should also be easier with UCSRP. There will be no need to strip the sulfur from the catalyst beds nor to switch gradually from acid gas to fuel gas, as must be done in Claus plants. For unplanned shut-downs, care will be needed in UCSRP to prevent sulfur solidification. The sulfur decanter section at the bottom of the reactor should be fitted with internal steam coils (much like a sulfur

pit or tank in a Claus plant) to prevent sulfur solidification. Level-control valves that release the produced sulfur from the reactor to storage will require steam jacketing. An auxiliary boiler is provided for cases when steam is needed to prevent sulfur from solidifying.

#### ► **Scale-Up and Scale-Down.**

In a separate economic study performed by a different organization for another purpose, UCSRP was compared to Claus/SCOT for a sulfur plant with a capacity of 950 tonnes/d. The relative capital cost for the UCSRP was estimated to be 62 percent of that for a Claus/SCOT system, a result remarkably close to the estimates above despite the vastly larger scale of the project and the difference in authors.

Claus/SCOT units have a minimum size of about 20 tonnes/d, because of their relatively high operating temperature. The minimum practical size for the UCSRP is estimated to be 1 to 10 tonnes sulfur per day. UCSRP plants of this size will be competitive with aqueous redox systems (such as Stretford, Sulferox and Lo-Cat), due to its substantially lower chemical costs and ability to produce much purer sulfur.

### **Additional Process Research Requirements**

While the conceptual design and cost/performance comparison has been completed, a number of areas require additional research to refine and confirm UCSRP performance assumptions. In addition, a demonstration pilot plant is being planned as a next step in proving the process.

#### ► **Reaction Kinetics.**

The only experimental data (Neumann and Lynn, 1986) on the kinetics of the reaction between H<sub>2</sub>S and SO<sub>2</sub> in the solvent medium were obtained at room

temperature, where the second-order rate constant is of the order of 50 to 100 liters/mol-sec. At 120 – 140°C, the kinetics should be 10 to 100 times faster than at room temperature. Since data at the higher temperature were lacking, the quantities of H<sub>2</sub>S and SO<sub>2</sub> reacting on each theoretical stage were calculated by assuming a rate constant of 50 liters/mol-sec, a depth of liquid of 3 inches and physical equilibrium between the liquid and gas leaving that stage. Eleven theoretical stages were required to obtain 99% reaction of the SO<sub>2</sub>. This assumption is probably conservative, with the result that some combination of reduced catalyst concentration, reduced solvent inventory per stage and/or reduced number of stages can be realized in practice.

#### ► **Sulfur Purity.**

It is assumed that the sulfur purity for UCSRP will be equal or superior to Claus, with ash and carbon content in the sulfur being less than that made in a typical Claus unit. The investigators for this study believe that little or no hydrocarbons will dissolve in the liquid sulfur at reactor conditions since their gas-phase concentrations will be low. If there are mercaptans in the feed gas, some of them (as well as SO<sub>2</sub>) could be absorbed by the sulfur. A steam strip or water wash will be used if needed to remove dissolved solvent and SO<sub>2</sub>, as well as any other impurities, from the liquid sulfur.

#### ► **Solvent Selection.**

The first choice of solvent is diethylene glycol methyl ether, DGM, sold as *Dowanol DM* by *Dow Chemical* or as *Methyl Carbitol* by *Union Carbide*. DGM has the advantages of being relatively low in cost, being a good solvent for SO<sub>2</sub> absorption and having a very low solubility in liquid sulfur (Sciamanna and Lynn, 1988a, 1988b). According to *Dow Chemical*, DGM is

stable at temperatures up to 140°C in the presence of air and up to 200°C in an atmosphere free of O<sub>2</sub>. Triglyme, tetraglyme, and Selexol are, respectively, the dimethyl ethers of triethylene glycol, of tetra-ethylene glycol and of polyethylene glycols of still higher molecular weight. They are low in toxicity and are better solvents for SO<sub>2</sub> than DGM, but have a solubility of about 0.5 wt% in liquid sulfur (Sciamanna and Lynn, 1988a, 1988b).

A steam strip (for triglyme) or a water-wash step could be used to recover the solvent from the sulfur. N-methyl pyrrolidone is a very good solvent for SO<sub>2</sub> (better than the glymes), and has a very low toxicity, so it can be used in the SO<sub>2</sub> absorber/stripper loop if its cost is not prohibitive. However, its solubility in sulfur makes it problematic for the reactor.

The solubilities of sulfur, H<sub>2</sub>S, SO<sub>2</sub>, CO<sub>2</sub>, propane and n-butane have been determined in several of the above solvents as a function of temperature, pressure, and the water content of the solvent (Sciamanna and Lynn, 1988a). This information is of considerable value in guiding solvent selection. Additional solubility data for different solutes or solvents are relatively easy to obtain.

#### ► **Solvent Degradation.**

No solvent degradation has been detected in the experiments done to date; however, these experiments have not included runs of hundreds of hours duration at temperatures well above the melting point of sulfur. Such experiments will be run at GTI with each of the solvents that show promise based on their costs and properties. In a typical (automated) experiment a sample of solvent will be put in a stirred, thermostated reactor flask at a temperature of 130°C. Small, metered streams of pure H<sub>2</sub>S and SO<sub>2</sub> will be sparged in. The stream flow of SO<sub>2</sub> will

be adjusted so that bubbles of that gas just reached the surface. The volumetric flow of H<sub>2</sub>S will be set at about 2.2 times that of the SO<sub>2</sub>. Liquid sulfur will be removed from the flask as needed and analyzed for solvent content. Small samples of solvent would be removed periodically and analyzed to detect formation of degradation products, whose composition is at present unknown. Such experiments should be run continuously for 500 to 1000 hours, depending on the nature of the results. In addition to any indication of solvent degradation, one will obtain a qualitative indication of the reaction kinetics from observing the behavior of the gas bubbles as they rise through the solvent. In addition, samples of carbon and stainless steel will be inserted near the stirrer in the reaction flask to check on the rate of corrosion.

#### ► **Reactor Design.**

Before constructing a multi-tray reactor column it would be advisable to construct and operate a single-tray column to determine tray efficiency and to observe tray hydraulics, in a device similar to that used by Hix and Lynn. (Hix and Lynn, 1991). The tray would be of the bubble-cap type. The gas stream would consist of nitrogen as well as the reactants and reaction products, and would be recirculated with a blower. Provision would be made for monitoring the concentrations of H<sub>2</sub>S and SO<sub>2</sub>, for injecting metered flows of them as needed, and for removing liquid sulfur and water vapor as they are formed. The column would be perhaps six inches in diameter, with glass sections above and below the tray. A circulating flow of solvent, which could be either heated or cooled, would also be provided. Provision would be made to heat the tray as well as the rest of the system either by steam jacketing or electrically-heated tracing. These

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studies would determine: the minimum acceptable L/D in the reactor column, and whether sulfur will settle adequately in a reactor containing appropriate jacketing and heating coils. The data obtained from the operation of this system should facilitate the design of the reactor for the first pilot or demonstration plant. Future studies should focus on kinetics, mass transfer and physical properties.

### ► **Demonstration Plant.**

A demonstration plant in the size range of 1 to 10 tonnes per day is thought to be sufficiently large to prove the technology. Planning for such a plant is currently underway.

## Conclusion

Based on a conceptual engineering design analysis, UCSRP appears to be significantly lower in both capital and operating costs than a Claus/SCOT unit of equivalent sulfur capacity. The process appears technically feasible, although some issues require further study. It should be noted that the reactor is the only step in the UCSRP that is not a standard operation in the chemical industry. Since the reactor operates at relatively low pressure, mild temperatures and under non-corrosive conditions, the development effort required to realize the potentials of the process should be modest. ■

*This article is summarized from a paper presented by the authors at the 2001 International Gas Research Conference, held November, 5-8, 2001, in Amsterdam, The Netherlands. For more information on the status of this research please contact Prof. Scott Lynn, Department of Chemical Engineering, University of California, Berkeley, at (510) 642-1634; E-mail: lynn@cchemberkeley.edu or Dennis Leppin, Gas Technology Institute, (847) 768-0521; E-mail: dennis.leppin@gastechnology.org/.*

# Simulator Provides Guidance for Increasing CO<sub>2</sub> Slip in Gas Treating Applications

by Ralph H. Weiland and  
John C. Dingman,  
*Optimized Gas Treating, Inc.*

*Accurate computer simulation can help gas processing facility designers take full advantage of amine absorber column internals when dealing with natural gas streams that contain both hydrogen sulfide and carbon dioxide.*

**W**hen treating a gas stream containing both H<sub>2</sub>S and CO<sub>2</sub>, it can often be desirable to remove most of the H<sub>2</sub>S but not all of the CO<sub>2</sub>, only reducing the CO<sub>2</sub> content to a specific amount (typically 2 to 3 percent). Selective removal of H<sub>2</sub>S with CO<sub>2</sub> rejection (“slip”) results in lower solvent circulation rates, better quality sulfur plant feed, and concomitantly lower capital and operating costs. Producing a gas with specified levels of both CO<sub>2</sub> and H<sub>2</sub>S can be achieved using “formulated” solvents, most of which are based on mixtures of MDEA and another primary or secondary amine used as an activator to adjust the level of CO<sub>2</sub> absorption. However, there are times when the maximum possible rejection of CO<sub>2</sub> consistent with any maximum H<sub>2</sub>S specification is desirable. One example is tail-gas treating. Another is the treating of a gas stream with relatively low total acid gas content and a very high CO<sub>2</sub> to H<sub>2</sub>S ratio where a higher than normally allowable CO<sub>2</sub> content output stream can be blended with other gas streams.

In high CO<sub>2</sub>-rejection applications there is an optimal number of absorber column trays or an optimal depth of absorber packing that maximizes CO<sub>2</sub> rejection while still meeting the H<sub>2</sub>S treating goal. The processing facility

designer must determine the number of trays or depth of packing (of a specific type) and decide on how to provide enough flexibility to be able to handle the inevitable changes in the volumes and acid gas composition experienced over the life of most plants.

The reasons for selectivity differences between trays and random packings concern the effects of tray and packing hydraulics on mass transfer fundamentals, an important but often overlooked factor in gas treating. A computer simulator incorporating true mass transfer rates based on a detailed depiction of real trays and packing material, both depth and type, can provide far more rigorous predictive modeling of a process and its columns. As a result, such a simulator can be an extremely valuable tool for predicting sensitivity to variations in both design and operating parameters. A simpler, equilibrium model cannot achieve the same accuracy.

The ProTreat™ simulator, developed by Optimized Gas Treating, Inc. with financial support from the Gas Research Institute (now Gas Technology Institute), can be used to uncover ways to maximize CO<sub>2</sub> slip and relate the findings to column internals details and actual column structure. This article provides several examples of its application.

## Understanding Selectivity

All alkaline solvents are thermodynamically selective towards CO<sub>2</sub> but kinetically selective towards H<sub>2</sub>S. However, if we are to use this understanding to figure out how to increase CO<sub>2</sub> slip (i.e., to improve selectivity) then we must also recognize two other important facts about acid gas-amine systems: (1) CO<sub>2</sub> and H<sub>2</sub>S react quite differently in alkaline solution, and (2) their physical absorption rates are controlled by resistances in entirely different phases.

How do these facts relate to selectivity? When CO<sub>2</sub> dissolves into the solvent, it binds chemically to the amine at finite rates of reaction, forming reaction products. At low temperatures, these reaction products are stable and require heat and stripping vapor to decompose them and reverse the reactions. On the other hand, when H<sub>2</sub>S dissolves into an amine, it converts immediately to sulfide and bisulfide ions via instantaneous protonation reactions with hydrogen ions, without directly involving the amine at all. These protonation reactions are immediately reversible and the extent of reversibility depends on solvent alkalinity, not reaction kinetics. While CO<sub>2</sub> reacts relatively slowly and H<sub>2</sub>S rapidly, the CO<sub>2</sub> forms stable reaction products, whereas, H<sub>2</sub>S forms readily-

decomposed products in a reaction that depends only on alkalinity.

What this means is that if the gas mixture and the solvent are exposed to each other for only a short time,  $H_2S$  absorbs more rapidly than  $CO_2$  because the instantaneous  $H_2S$  reaction keeps the  $H_2S$  concentration in the unreacted form low in the liquid and this maintains the driving force. However, the  $CO_2$  reaction isn't fast enough to prevent the  $CO_2$  concentration from building up and slowing down its absorption rate. The reaction kinetics have made the process selective towards  $H_2S$ . If, on the other hand, we allow the phases to remain in contact for a long time, both gases continue to absorb, but as the  $CO_2$  absorbs it consumes amine and reduces the solvent's alkalinity. At some point, this reduction in alkalinity becomes too low to keep all the  $H_2S$  in a protonated form. Consequently  $HS^-$  and  $S^{=}$  deprotonate and the  $H_2S$  starts to desorb. Meanwhile, the  $CO_2$ , still driven by the (almost) irreversible reaction, continues to absorb and react. Reaction equilibrium favors keeping  $CO_2$  in solution even to the extent of releasing already-absorbed  $H_2S$  if necessary. Part of the trick to controlling selectivity is a combination of manipulating the factors affecting the chemistry (because changing the reaction kinetics profoundly affects the  $CO_2$  absorption rate) and controlling contact times.

But the absorption of both gases, although greatly influenced by  $CO_2$  reaction kinetics, is also controlled by diffusion. For  $CO_2$ , the diffusional resistance is predominantly in the liquid phase, while for  $H_2S$  it is in the gas phase. The other part of the trick then is that by carefully selecting the tower internals to favor mass transfer of an acid gas in one phase over the other, it should be possible to alter the relative

absorption rates and hence the selectivity. This gives one an additional means to enhance  $CO_2$  slip.

So, the secret to controlling selectivity lies in choosing an amine with the right alkalinity and the right reactivity towards  $CO_2$ , allowing gas-liquid contact for the right length of time, and using the right kind of equipment with the right internals. A mass transfer rate model is perfectly suited to dealing with such issues of all-important detail. Equilibrium stage models simply cannot capture these effects.

### The Need for a Mass Transfer Rate Model

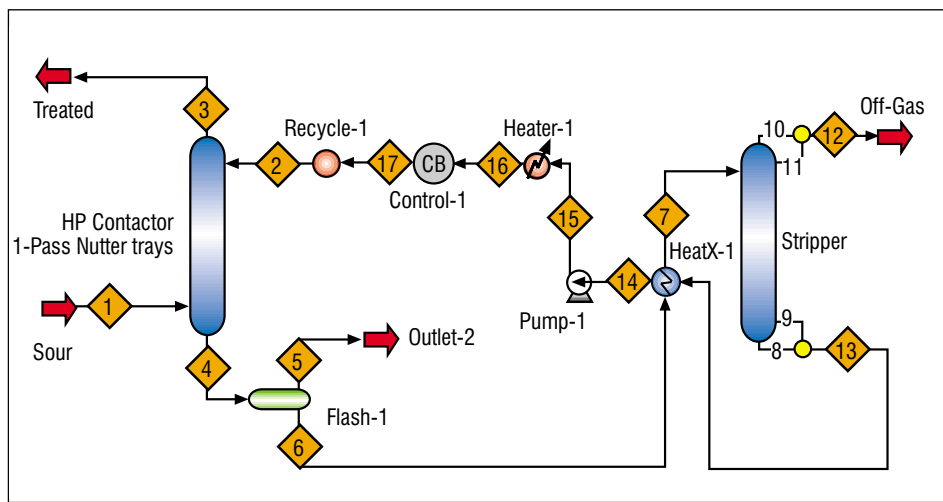
The rigorous, mass transfer rate approach used for all column calculations by ProTreat modeling is completely predictive, because it is not dependent on the availability or the need for empirical adjustments to simulate new applications correctly. In fact, there are *no* empirical adjustments in the ProTreat model. True mass transfer rate modeling is built on five key elements:

- Mass and energy balances around individual phases on a tray or in a packed segment

- Conventional thermodynamic phase equilibrium
- Equilibrium across vapor-liquid interfaces
- Effect of chemical kinetics on mass transfer rates, particularly in the liquid phase
- Mass and heat transfer rate models for transport across vapor-liquid interfaces.

There are intricate interrelated effects between these five key elements. They are affected by a variety of chemical and physical, phase and component transport properties such as chemical kinetics, diffusion coefficients, solvent viscosity, and other heat and mass transport properties, salting-out effects on acid-gas solubilities, and the mass transfer characteristics of the actual hardware being used.

The tower is modeled in full detail as a piece of real equipment, not as an idealization. From a separations standpoint, multi-pass trays perform differently from single-pass trays. Metal packing gives different results from plastics and ceramics. In addition, all solvent properties, including the changes in these properties caused by acid gas loading, affect mass transfer coefficients and thereby influence the



**Figure 1: Process Flow Diagram**

**Table 1: Stream Conditions for Case Study 1**

	Sour Gas (Inlet 1)	Lean Solvent (Stream 2)
Temperature (°F)	90	110
Pressure (psig)	900	900
Flow (MMscfd or US gpm)	20	Various
H <sub>2</sub> S (vol% or loading)	1.0	Various
CO <sub>2</sub> (vol% or loading)	10.0	Various
Methane (vol% or loading)	85.0	
Ethane (vol% or loading)	4.0	
MDEA (wt%)		45

actual separation achievable with a specific column under a given set of operating conditions. A true mass transfer rate simulation uses a distributed parameter model: mechanistic, rich in detail, and fully predictive. This is in contrast to the nonpredictive equilibrium stage approach based on a lumped parameter model that assigns all the physical and chemical complexities to one or two parameters such as stage efficiencies for the acid gases and liquid residence times.

### Case Study 1: Selective H<sub>2</sub>S Removal from High-CO<sub>2</sub> Gas

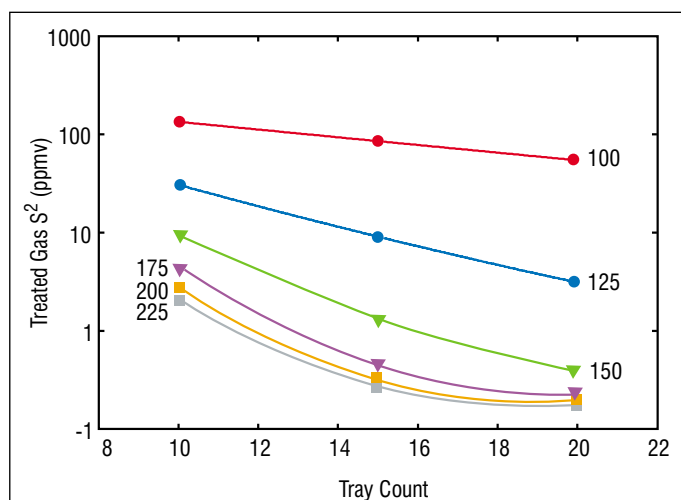
This simulator case study describes removal of H<sub>2</sub>S from a high pressure gas stream using a conventional flow scheme (Figure 1) with a 3-ft diameter column contactor containing Nutter trays or Raschig rings. The example demonstrates how trays compare with equivalent depths of a random packing, and the effect of tray count and packed depth on H<sub>2</sub>S removal and CO<sub>2</sub> rejection. The feed streams to the contactor are detailed in Table 1. The effect of tray count on the H<sub>2</sub>S content

of the treated gas, the percentage CO<sub>2</sub> slipped, and the quality of the off-gas from the regenerator (a potential Claus plant feed) are determined for several solvent rates.

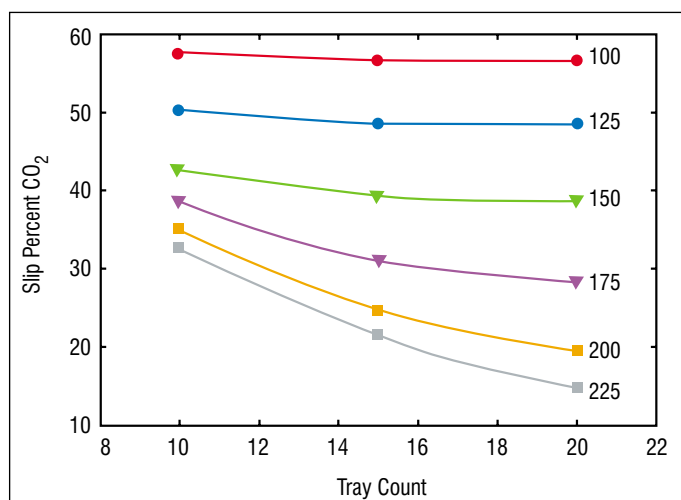
Higher solvent flows always produce pipeline quality gas even at low tray counts and that the treated gas quality falls off rapidly as the tray count is reduced beyond a certain minimum (Figure 2). Very low solvent flows are incapable of meeting pipeline specifications at all.

CO<sub>2</sub> slip is rather insensitive to tray count at low solvent rates, and increasing the solvent rate in order to meet an H<sub>2</sub>S specification causes reduced CO<sub>2</sub> slip (Figure 3). This results in regenerator off-gas of lower and lower quality because CO<sub>2</sub> pickup in the contactor increases (Figure 4). For this particular gas and solvent combination, trays typically slip only 30 percent to 50 percent of the CO<sub>2</sub> and produce a marginal quality Claus feed.

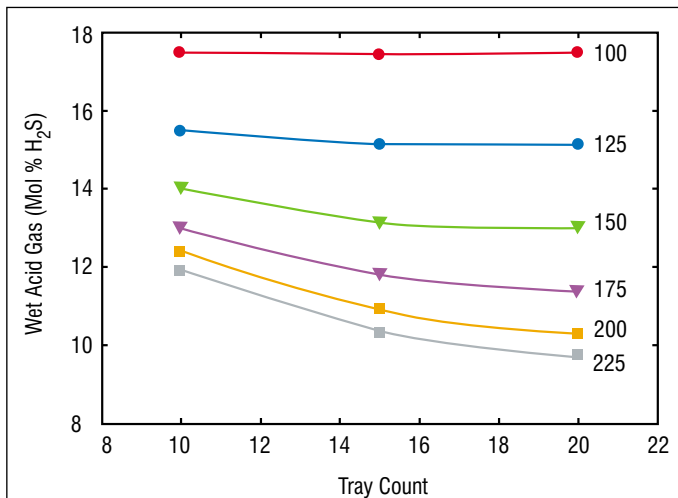
The effect of packed bed depth on the treated-gas H<sub>2</sub>S content, CO<sub>2</sub> slip, and regenerator off-gas quality can all be illustrated using the simulator. Greater depths of packing (2-inch Raschig rings) and higher solvent flows



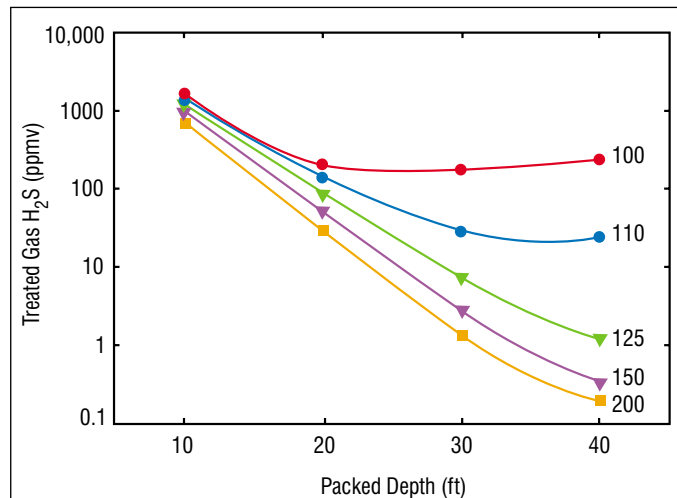
**Figure 2: Effect of Number of Trays and Circulation Rate on Treated Gas H<sub>2</sub>S Content**



**Figure 3: Effect of Tray Count on CO<sub>2</sub> Slip (Circulation Rate in US gpm)**



**Figure 4: Increasing Tray Count Lowers Acid Gas Quality**



**Figure 5: Effect of Packed Bed Depth on Treated Gas Residual H<sub>2</sub>S**

are needed to produce a treated gas containing less than 4 ppmv H<sub>2</sub>S (Figure 5). This despite the fact that CO<sub>2</sub> slip with packing is very much higher than with trays, typically 70 percent to 90 percent (Figure 6). However, this is only first stage treating, and it may be quite satisfactory to leave considerably more H<sub>2</sub>S in the treated gas, especially if the second stage regenerator off-gas is to be flared. At low solvent rates, as the

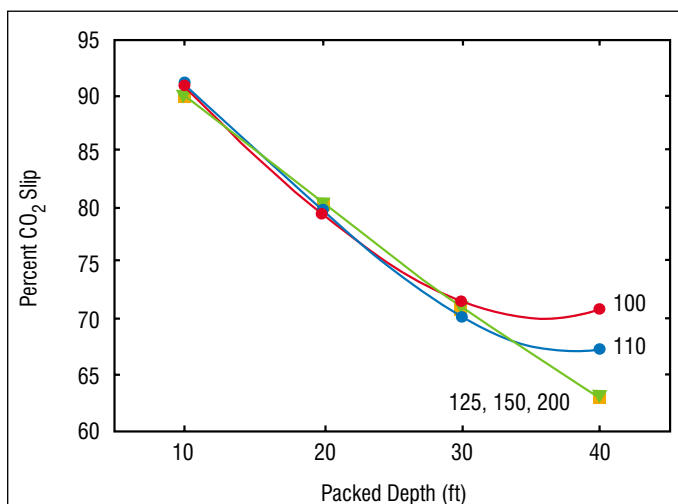
packed depth is increased, the sweet gas H<sub>2</sub>S content first falls, reaches a minimum, and then begins to increase. The increase at higher bed depths is caused by increased absorption of CO<sub>2</sub> which prevents H<sub>2</sub>S absorption.

Figure 7 shows a much improved quality of Claus plant feed (regenerator off-gas H<sub>2</sub>S content). Note that the absorber packed bed depth affects enrichment, but solvent rate does not. Solvent rate affects only treated gas

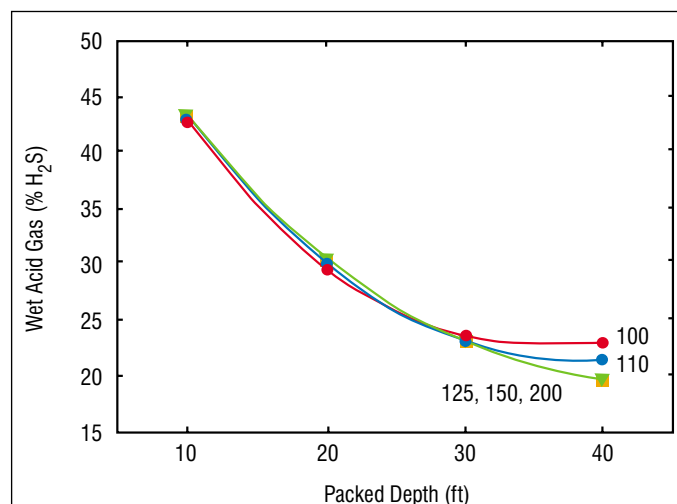
purity. Obviously, packing produces a much better quality Claus plant feed.

### Case Study 2: Regenerator Off-Gas Concentration

This case study depicts the enrichment of a dilute regenerator off-gas to make it a suitable sulfur-plant feed. The gas being enriched is essentially a wet H<sub>2</sub>S-CO<sub>2</sub> stream at 110°F and 10 psig, flowing at 10 MMSCFD. The solvent is 40 wt percent MDEA at 120°F. A

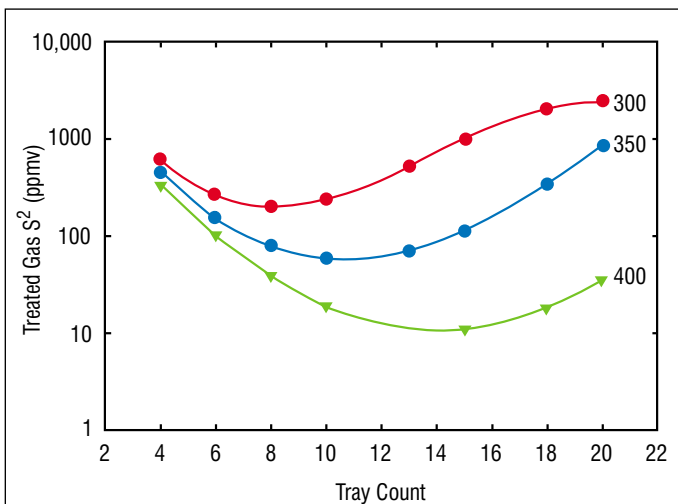


**Figure 6: CO<sub>2</sub> Slip Improved by Shorter Packed Beds**

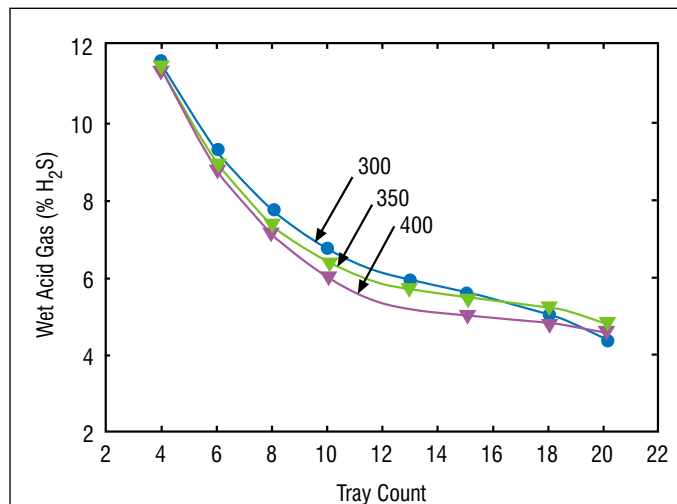


**Figure 7: Quality of Wet Acid Gas at Various Circulation Rates**





**Figure 8: Residual H<sub>2</sub>S in Treated Gas at Three Circulation Rates (Feed Gas is Being Enriched From 1% H<sub>2</sub>S in CO<sub>2</sub>)**



**Figure 9: Effect of Tray Count on Enrichment of a 1% H<sub>2</sub>S - in - CO<sub>2</sub> Feed Gas at Three Circulation Rates**

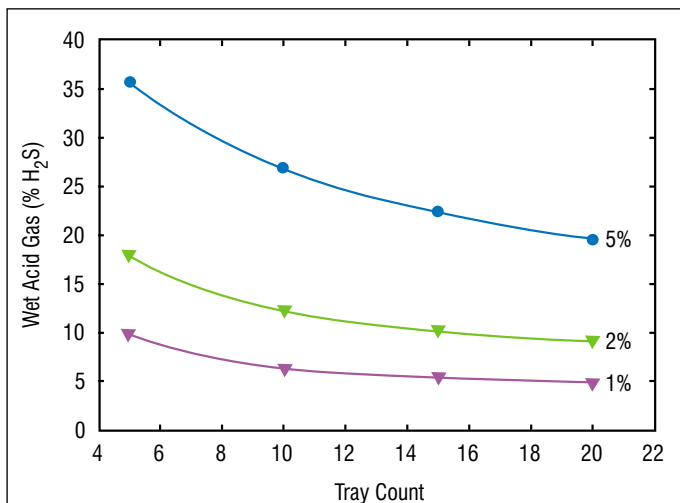
conventional absorber-regenerator flowsheet is used with a 5-ft diameter regenerator containing 20 two pass Koch FLEXITRAYS. Note that the regenerator duties for trays and packing were 25 MMBtu/hr and 15 MMBtu/hr, respectively. These were chosen to give similar regenerator reflux ratios under worst-case conditions.

Three feed gas dry-basis analyses are considered: 1, 2, and 5 volume

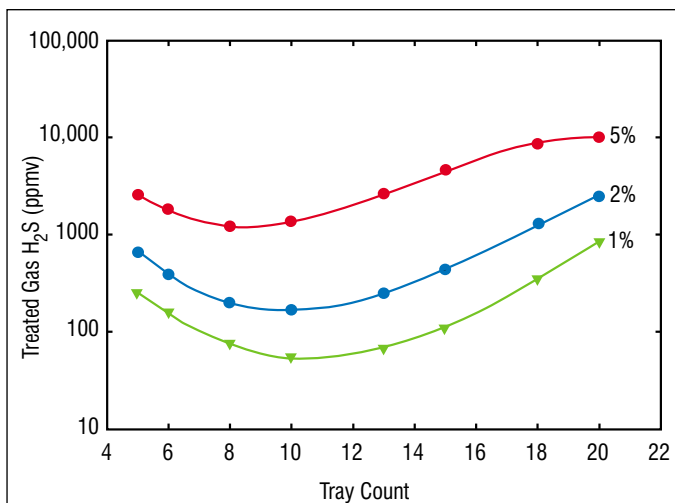
percent H<sub>2</sub>S with the balance CO<sub>2</sub> and, for the case of 1 volume percent H<sub>2</sub>S in the feed gas, three solvent rates are examined: 300, 350, and 400 US gpm. Both the trayed and packed contactors were sized for 80 percent flood as determined for the highest circulation rate.

Figure 8 shows the effect of tray count (Glitsch V-1 trays) and solvent flow on the residual H<sub>2</sub>S in the treated

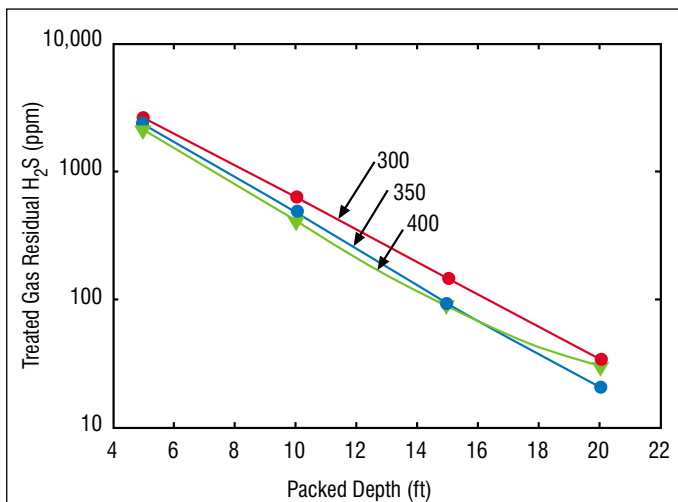
gas. The absorber here contained two-pass Glitsch V-1 valve trays in a 5.5-ft diameter shell. Higher solvent rates give lower residual H<sub>2</sub>S levels, as expected. However, in each case there is an optimal number of trays at which the H<sub>2</sub>S content is minimum. For very small tray counts there is insufficient contact to get to very low H<sub>2</sub>S levels and as the number of trays is increased, H<sub>2</sub>S pickup improves; however, as the



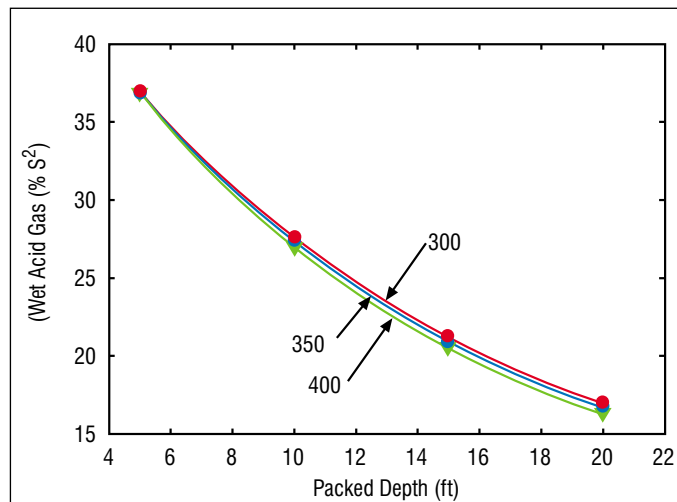
**Figure 10: Effect of Tray Count on Enrichment of 1%, 2%, and 5% H<sub>2</sub>S Feed Gas Streams at 350 US gpm**



**Figure 11: Treated Gas H<sub>2</sub>S Content After Enrichment of 1%, 2%, and 5% H<sub>2</sub>S Feed Streams with Solvent at 350 US gpm**



**Figure 12: Effect of Bed Depth on Treated Gas Quality After Enrichment at Three Solvent Rates**



**Figure 13: Enriched Wet Acid Gas Stream Obtained at Various Packed Bed Depths and Solvent Rates (Feed Gas Was 1% H<sub>2</sub>S)**

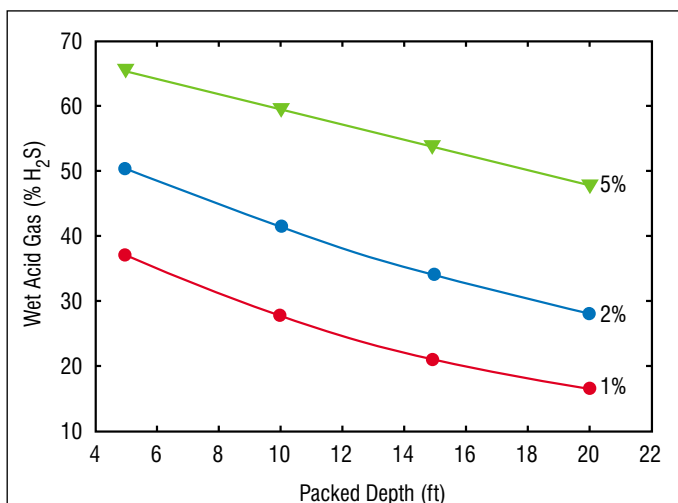
number of trays continues to increase, performance starts to deteriorate. This is because the increased contact results in increased CO<sub>2</sub> pickup which is detrimental to H<sub>2</sub>S removal.

This can be seen in deteriorating sulfur-plant feed quality as the tray count is increased (Figure 9). Because higher solvent flows can pick up more acid gas, the optimum tray count for

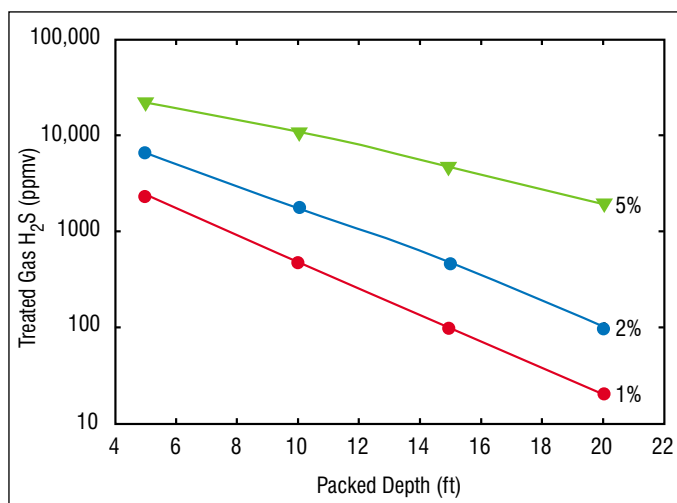
best H<sub>2</sub>S removal is an increasing function of solvent flow. Of course, the asymptotic limit is zero enrichment which occurs when the tray count and solvent rate becomes high enough for total acid gas pickup.

The simulation can also show the effect of feed-gas H<sub>2</sub>S content on the degree of enrichment as a function of tray count (Figure 10) and the

corresponding treated gas residual H<sub>2</sub>S concentration (Figure 11). Again, there is an optimal number of trays for maximum H<sub>2</sub>S recovery. However, as expected from the kinetic preference for H<sub>2</sub>S versus the thermodynamic selectivity for CO<sub>2</sub>, the highest degree of enrichment corresponds to the fewest number of trays, but then the H<sub>2</sub>S recovery is poor. Thus, when it comes to



**Figure 14: Effect of Packed Bed Depth on Enrichment Achievable From Feed Streams Containing 1%, 2%, and 5% H<sub>2</sub>S in CO<sub>2</sub> (Solvent Flow of 350 US gpm)**



**Figure 15: Residual H<sub>2</sub>S in Treated Gas for Various Feed Gas H<sub>2</sub>S Concentrations in a Packed Contactor With Solvent Flow of 350 US gpm**

tray count, there is always a tradeoff between fractional recovery of H<sub>2</sub>S and degree of enrichment.

As an alternative, the same acid gas enrichment operation was simulated using 2-inch steel Pall rings with 40 weight percent MDEA. In this case, the tower diameter required for 80 percent flood at the highest flows was only 4.5 feet. The residual H<sub>2</sub>S in the treated gas decreases exponentially with increasing depth of packed bed (Figure 12). Solvent circulation rate appears to have very little effect on treated gas quality.

The quality of the enriched gas stream is shown in Figure 13. The improvement that packing affords over trays is remarkable. Referring to Figure 9, the best that could be achieved under these same circumstances using trays was about a ten-fold enrichment. Packing produced a thirty-fold enrichment, taking a stream of 1 percent H<sub>2</sub>S in 99 percent CO<sub>2</sub> and enriching it to about 30 mol percent H<sub>2</sub>S. Depending on the solvent rate, trays may or may not achieve a cleaner treated gas; however, the cost is always a tremendously reduced quality of Claus sulfur plant feed.

Figures 14 and 15 show the enrichment possible with packing from acid gas streams containing 1 percent, 2 percent and 5 percent H<sub>2</sub>S in CO<sub>2</sub>, and the concomitant level of treating, respectively. Figure 14 shows that production of sulfur plant feed of outstanding quality from problem off-gas streams is quite achievable. As shown in Figure 15, residual levels of H<sub>2</sub>S in the treated gas comparable to those produced with trays can also be reached.

What has been done here is to take a problem off-gas stream containing levels of H<sub>2</sub>S that are hopelessly low for use in a sulfur plant (and probably

too high for flaring or to make a sulfur scavenger economic) and produce a very high quality sulfur plant feed.

The residue is a very dilute stream of H<sub>2</sub>S in CO<sub>2</sub> which must be disposed of, but at these small concentrations of H<sub>2</sub>S, both flaring and scavengers become more attractive.

### Case Study 3: Performance Test of Commercial Absorber

The final example is a comparison of a ProTreat simulation with field performance data collected from a commercial contactor removing H<sub>2</sub>S and CO<sub>2</sub> with 46.3 weight percent DGA. The contactor is 66 inches in diameter and contains 16 Nutter trays on 2 foot spacing. The plant performance test data (taken in 1982) included measured temperatures on three trays and the tower sump. Table 2 shows the feed stream conditions.

This column produced better than 1 ppmv H<sub>2</sub>S treated gas, but unfortunately the gas was not analyzed for CO<sub>2</sub>. The ProTreat simulation gave a treated gas containing 0.05 ppmv H<sub>2</sub>S and 1.8 ppmv CO<sub>2</sub>. These are very encouraging numbers, particularly in view of the fact that the model was not tweaked into agreement with any of the data—the simulated quality of the treated gas is a pure prediction.

No parameters have been fine-tuned to achieve agreement and the close match is completely natural. Even more impressive, however, is the agreement between the measured and simulated column temperature profiles (Figure 16). This plot shows the (unequal) vapor and liquid temperatures on each real tray in the column. The large square symbols are measured data.

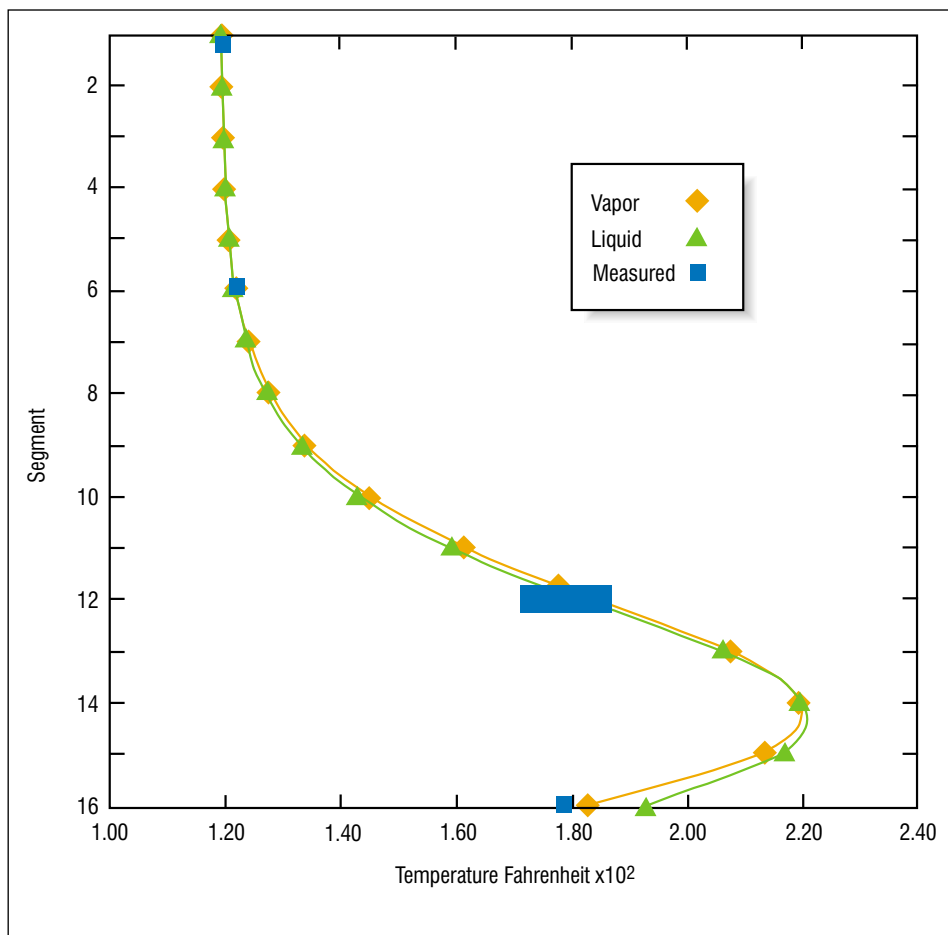
For this performance test, the column was operating in a severely turned down condition, at 14.4 percent of jet flood and 11.3 percent of downcomer flood. Given that the tower was probably originally designed for 80 percent flood, this represents a 5.5 to 1 turndown ratio. Operation was certainly outside the recommended operating range and there was likely substantial weeping and possibly tray blow-dry.

### Using ProTreat™

These three case histories provide support for the notion that rigorous simulation of amine treating units can be very important in maximizing their performance and that the ProTreat simulator can be used to uncover ways to maximize CO<sub>2</sub> slip and relate the findings to column internals details and actual column structure.

**Table 2: Feed Stream Data for Performance Test**

	Sour Gas (Inlet 1)	Lean Solvent (Stream 2)
Temperature (°F)	88	119
Pressure (psig)	980	980
Flow (MMscfd or US gpm)	17.22	
H <sub>2</sub> S (vol% or loading)	0.38	0.0001
CO <sub>2</sub> (vol% or loading)	5.82	0.034
Methane (vol% or loading)	93.80	
DGA (wt%)		46.3



**Figure 16: ProTreat™ Simulated Vapor and Liquid Temperature Profiles Compared With Performance Test Data**

ProTreat is a Microsoft Windows® based, flexible-flowsheeting package for amine gas treating designed to be run with Windows 95, 98 and NT. Data input is through Windows dialogs and includes on-line help features as well as extensive preprocessing validation of input data.

The model is available for licensing and currently is in use by a number of major oil and gas producing companies and amine suppliers. ■

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*ProTreat simulation software was developed with the partial financial support of the Gas Research Institute, Dennis Leppin, GRI Project Manager. ProTreat is a trademark of Optimized Gas Treating, Inc. For more information related to the use of this product contact the author at 281-496-2729 or via e-mail at [jdingman@ogtrt.com/](mailto:jdingman@ogtrt.com/).*

# Developing A Cost Effective Composite Drill Pipe

by Dr. James C. Leslie,  
*Advanced Composite Products  
and Technology, Inc.*

*Greater depths, deeper water and horizontal drill paths are pushing the limits of steel drill pipe. Composite pipe, light but strong, may provide a solution if it can be manufactured cost effectively.*

The projected future demand for natural gas is one of the important drivers for offshore exploration efforts moving towards deeper and deeper water depths. Deepwater Gulf of Mexico, a promising new area for increasing US gas production, now accounts for 20 percent of total Gulf of Mexico natural gas output, versus 5 percent only five years ago. This trend is expected to continue.

One constraint on drilling depth (and hence water depth) that also affects the practical distance to which horizontal laterals can be drilled, is drill pipe weight. This fact has driven research into the use of composite materials (see sidebar on page 34) to replace steel drill pipe. Composite drill pipe (CDP) is projected to have a weight less than half that of its steel counter part, significantly increasing the lateral distance which can be reached from an offshore drilling platform and the depth of water in which drilling and production operations can be carried out.

Other potential benefits include the capability to carry real time signal and power transmission conduits within the pipe walls, the ability to accommodate a much shorter turn radius, and an increase in the pipe storage capacity of floating offshore drilling platforms. It is anticipated that

commercial CDP will be available in the near future at 2 to 5 times the cost of comparable steel pipe. To further rapid development of cost-effective composite drill pipe manufacturing capability in the U.S., a three year development program is being sponsored by the National Energy Technology Center (NETL) of the U.S. Department of Energy. The work is being carried out by *Advanced Composite Products and Technology Inc.* (ACPT), an established producer of custom composite parts. During the first two years of this NETL/DOE supported program, specifications for both 5½ inch and 3¾ inch composite drill pipe have been finalized, materials for the composite tubing, adhesives, and abrasion coatings have been selected based on laboratory testing, and a composite tube/metal tool joint interfacial connection has been successfully tested (the joints on CDP are metal).

Existing facilities are being modified to allow pilot plant production of up to 10, 30-foot sections of CDP per day. It is believed that this production rate will allow production of enough pipe for an initial market evaluation. Arrangements are in progress to have samples of 3¾ inch CDP used in a short radius well drilling operation early in 2002. It is planned to have 5½ inch composite

drill pipe ready for initial drilling operations by Spring 2003.

## Composite Drill Pipe Manufacturing

Composite drill pipe consists of a composite material tube with steel box and pin connections. The tube is manufactured by winding a composite material consisting of graphite fibers and epoxy resin around a metal mandrel and the metal box and pin connections. This length of fresh composite tube must then be cured before the mandrel can be removed and reused. The cured pipe section is finish machined and coated for abrasion resistance. Final preparation, normally done in the field, involves the addition of standard elastomeric centralizers before the pipe is run in the hole. Both the centralizers and the abrasive resistant coating can be repaired in the field. More extensive wear, as long as it is not too severe, can be repaired at the factory.

## Mechanical Strength Specifications

Initial work on this project concentrated on specifying the requirements for a "typical" drill pipe as a target for the CDP. These requirements have been refined during this program and will be upgraded as experience in the manufacture and use

**Table 1: CDP Specifications**

Case	Initial Requirements 5%	Revised CDP 5%	Revised CDP 5 <sup>5</sup> / <sub>16</sub>	CDP 3%
Tension (1000 lb. load)	20,000 TVD plus 133 Applied 199.5 Test 399 Ultimate	20,000 TVD plus 133	20,000 TVD plus 133	75
Compression (1000 lb. load)	30 Applied 45 Test 90 Ultimate	30	30	50
Torsion (1000 ft.-lb. load)	30 Applied 45 Test 90 Ultimate	56.25	37.5	6
Internal Pressure (psi)	3,500 Applied 5,000 Test 10,500 Ultimate	11,875	11,000	2,000
External Pressure Differential (psi)	4,500 Applied 6,750 Test 13,500 Ultimate	4,500	6,500	
Temperature (°F)	-67 to 250 Applied -67 to 250 Test -67 to 350 Ultimate	350	350	325

of CDP is obtained. Initially, industry partners supplied mechanical requirements identical to those of 5% inch high strength steel drill pipe (Table 1).

These were reviewed and modified through open forum industry discussions. Revised mechanical requirements were then converted to conform to the mechanical/weight characteristics possible with low cost graphite/epoxy materials. More recently, the required mechanical specifications have been exhaustively analyzed through joint efforts with a commercial pipe supplier. The resulting mechanical specifications, currently in use for design of 5<sup>5</sup>/<sub>16</sub> inch CDP are shown in Table 1.

Further consultation with industry

contacts defined an immediate need for 3% inch drill pipe to be used in short radius applications. Initial 1/8-scale testing demonstrated that ACPT designs can meet the requirements for this size pipe. The final specifications, arrived at through discussions and analysis with a drilling contractor, are also shown in Table 1. Short and full length sections designed to these specifications are being manufactured and will be tested in preparation for actual drilling operations scheduled for the spring of 2002.

### **Transfer of Electrical Signal or Power Across Joints**

A significant feature of CDP is that it can be designed to carry electrical power and/or real time communication

lines embedded in the composite walls. The problem to be solved is reliably transmitting signal or power through the metal joints connecting individual CDP sections. Several approaches to solving this problem are currently being examined elsewhere.

- **Direct Connect.**

This has been tried unsuccessfully numerous times. Several revised concepts are being investigated for ACPT by *Maurer Engineering* and if successful, *Maurer* and ACPT will submit a proposal to reduce this concept to practice.

- **Acoustic Transmission.**

This concept is being explored with DOE funding by another contractor.

- **Inductive Transmission.**

This approach shows positive

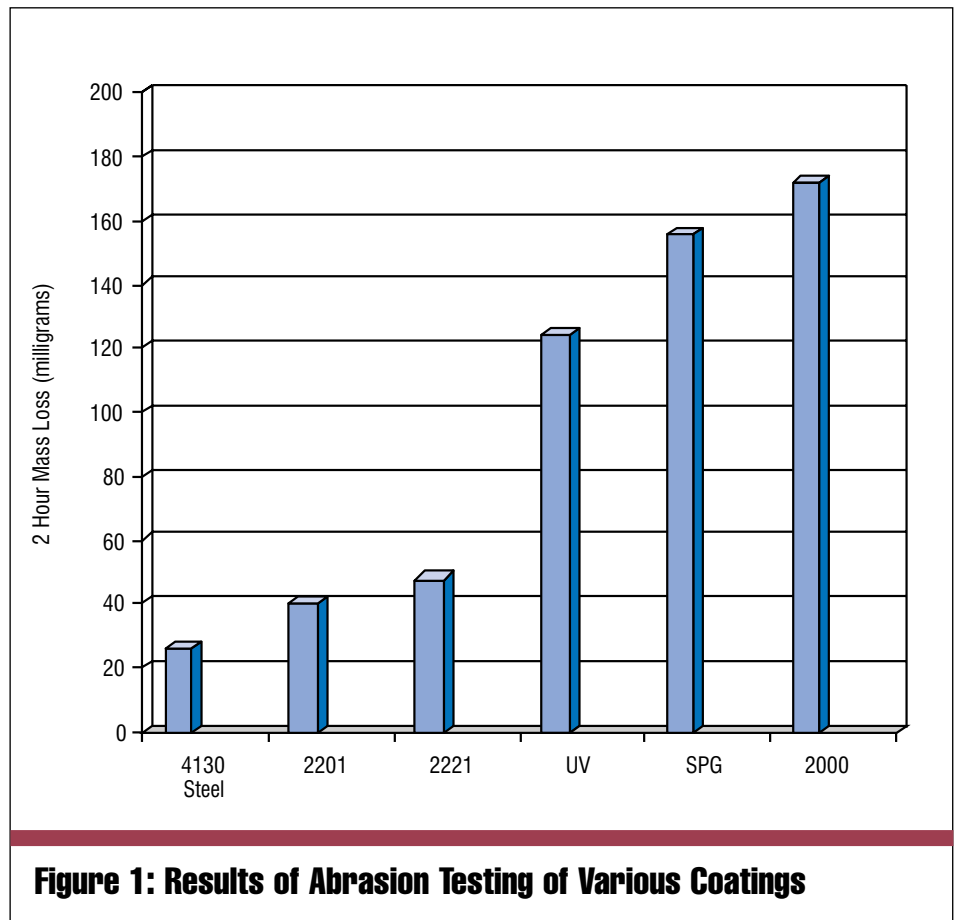
potential. Sandia National Laboratory is continuing to investigate inductive transmission. Inductive coupling has been considered and will be further investigated if the conceptual demonstrations show sufficient merit.

In anticipation of the development of a successful method for transmitting signals across metallic joints, Sandia National Laboratory has been tasked to measure signal loss/transmission characteristics in CDP with wires incorporated into the walls of the pipe.

### Screening and Testing of CDP Material

The testing portion of this project includes initial material screening through final in-ground evaluation of market-ready CDP. The material screening and material properties verification portions are complete. Laboratory testing included verification of mechanical, thermal and environmental properties of resins, fibers, and adhesives, and measurement of erosion and mechanical abrasion characteristics of interior and exterior coatings for CDP.

Temperature capability and environmental resistance were evaluated through short beam shear (SBS) and in-plane shear tests. SBS testing provides an excellent screening tool for evaluating the mechanical relationship between the resin and the fiber in composite structures. Short beam shear tests were run on the selected materials after exposure to a wet and dry temperature environments ranging from ambient to 350°F. SBS tests were also performed after temperature and pressure exposure to water base and oil base drilling muds at a similar range of temperatures and simulated downhole pressures for 10 days.



**Figure 1: Results of Abrasion Testing of Various Coatings**

These tests proved that, as anticipated, the graphite fiber/epoxy matrix experienced a reduction in high temperature shear strength after exposure to moisture. It was postulated that the strength degradation was caused by hydrolysis of the resin. However, this does not constitute a fatal flaw. Resin softening is a diffusion controlled phenomena and the very small (¼ inch x ¼ inch x 1 inch) SBS specimens present the absolute worst case exposure conditions. Actual CDP will be a continuous tube with walls on the order of 0.56 inch thick and with environmental protection on both the inside and outside surfaces. In addition, drill pipe does not experience long term continuous exposure at the most extreme environmental conditions. Therefore, a second set of 100 hour boiling water exposure tests were run with in-plane shear

specimens and with ½ scale pipe. The results of these environmental exposure tests showed that the current composite matrix can be used in downhole conditions up to 350°F.

As composites are much more susceptible to wear and abrasion than steel, it was recognized at the beginning of this program that CDP would have to be protected from mechanical wear. A dual approach was planned for protecting the exterior of CDP from abrasion: a highly wear resistant coating plus centralizers.

ACPT screened more than 20 potential coating systems for external abrasion protection and evaluated five selected systems through Slurry Abrasion Resistivity (SAR) testing. SAR is a standard wear test used to measure wear resistance within slurry pumps and is accepted by the oil industry. Results showed that at least

one coating system (numbered 2201 in Figure 1) compares favorably with 4130 steel relative to other coatings. ACPT has also evaluated “off-the-shelf” centralizers and determined that in addition to the abrasion resistant coating, high durometer elastomeric centralizer units will need to be utilized with CDP.

### Testing of One Third-Scale Pipe

The major difficulty in producing a commercially useful composite drill pipe has always been recognized as the interface between the composite tube (pipe) and the steel joints. In order to reduce developmental costs ACPT broke the CDP development and testing into two distinct areas: subscale design and testing and full-scale design and testing. One-third size (diameter) was chosen for the small-scale effort and the full-scale work was broken into full diameter pipe in 10-foot sections and full diameter pipe at



**Figure 2: Tension Test on 1/3-Scale, 10-Foot Section of CDP**

the full length of 31.5 feet (shoulder-to-shoulder) of the metal joints.

To date, the 1/3 scale testing is complete; 10-foot sections of full diameter CDP have been fabricated and tested; and tooling, fabrication

equipment, and procedures are being prepared for building the 31.5-foot test units. The 1/3-scale test specimens are 1.417 inch ID and have 12 inches of composite tube between the steel joints.

## Composite Materials

In materials science, a composite is defined as: “a complex material, in which two or more distinct, structurally complementary materials combine to produce structural or functional properties not present in any individual component.” The materials (reinforcing elements or fibers, fillers and binders), differ in form or composition on a macro scale, and the combination results in a material that can be engineered to maximize specific performance properties. The constituents do not merge completely and therefore normally exhibit an interface between one another. Reinforced concrete, road asphalt aggregate, and fiberglass reinforced epoxy are simple, common examples of everyday composites.

Advanced composites usually contain materials such as metals, ceramics, glasses, polymers or graphite. Many of these have been developed for military or space applications where a combination of high strength and low weight is critical (e.g., satellite components, missile launch tubes, tank tracks, ordnance components, and radomes). The variety of commercial applications is

continually growing as well (e.g., drive shafts, high speed rotors and ultracentrifuge bodies, cryogenic tanks, and lightweight auto parts, and sporting equipment). Over the two decades, composite materials, along with ceramics, have become the dominant emerging materials in engineering design. The number of applications of composite materials has grown steadily, rapidly penetrating and conquering new markets.

Recently, the price of graphite fiber has been significantly reduced while its properties (strength, stiffness, and conductivity) have continued to be improved. As a result, new applications [including composite drill pipe (CDP)] are becoming more economically attractive. Also, as volume usage of specialty resins increases, their price also drops. As with graphite fibers, resin matrix characteristics are being improved so as to provide both easier processing and improved composite properties. These improved resins provide a higher temperature capability in composites, expanding the depth range of CDP to deeper wells.





**Figure 3: Torque Test on 1/3-Scale, 10-Foot Section of CDP**

Twenty six (26) different 1/3-scale tension tests were completed. These tests evaluated 15 different combinations of composite/metal joint interface and composite wall configuration. After testing showed that a successful composite/metal interface design had been achieved, full size, 10-foot sections of CDP were fabricated and tested (Figures 2 and 3). More recently, based upon the final 1/3 scale work and in order to qualify for short radius drilling, 3 3/8 inch sections have been tested. The results of these tests proved that the full-scale requirements will be met. Fabrication of specimens for “proof-prior-to-drilling” testing of the CDP, is underway. This will include laboratory testing of full size 5 1/2” and 3 3/8” CDP, and field testing of 3 3/8” CDP in drilling short radius wells.

### **Manufacturing of Full Scale CDP**

ACPT is presently completing the modification of existing equipment to allow “pilot plant” production of up to 10 (possibly 15 with more upgrade and modification) 30-foot sections of CDP per day. It is believed that this production rate will permit enough production for an initial market evaluation. Additional capacity will require the incorporation of automation

and continuous operation to the winding, curing, and machining functions. ACPT is working closely with Omsco, a unit of ShawCor Ltd., to establish marketing levels and schedules. These results will determine the schedule and extent of pilot plant upgrade or the necessity to build a full scale, continuous operation CDP production unit. ■

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*Additional information of the progress of this project will become available as scaled-up CDP testing is carried out in the laboratory and the field during 2002. For more information on the progress of this project contact Gary Covatch, National Energy Technology Laboratory's Strategic Center for Natural Gas, at 304-285-4589, or via e-mail at [gcovat@netl.doe.gov](mailto:gcovat@netl.doe.gov).*

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# Producing Advanced Drill Bit Cutters Using Microwave Technology

by Dr. Dinesh Agrawal and  
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University*

*The search for natural gas is requiring drillers to reach deeper and deeper depths, economically. A new approach to the manufacturing of tungsten carbide bit cutters could help reduce the cost and improve the performance of bits.*

The paramount objectives of a drilling operation are to reach the target safely in the shortest possible time and at the lowest possible cost. One technology advancement focused on these objectives has been the evolution of tungsten carbide or diamond cutters to replace the hardened steel teeth of the drill bit. These specialized bits are well suited to drilling the harder formations typically found at greater depths. The deeper, tighter, often highly abrasive formations that are increasingly the target of domestic gas exploration are one important application.

The cutters on these bits remove rock by impact or shearing processes and thus their wear qualities, toughness, and hardness are important factors in the performance/cost ratio of the bits. Overall performance depends upon their design, composition, microstructure, and coating material.

Tungsten carbide (WC) based composites, due to their unique combination of hardness, toughness and strength, are universally used in cutting tools and drills, including cutters for hard rock bits. These composite cutters are manufactured by a sintering process (see sidebar). Conventional methods for sintering WC with cobalt (Co) as a cementing material involve high temperature and a lengthy processing

cycle (about 24 hours), making the production cost quite high. Furthermore, such conditions favor an undesired growth of WC grains during sintering, resulting in diminished mechanical strength and hardness.

Work done by The Pennsylvania State University (PSU), with support by the Department of Energy (DOE), has focused on the development of a microwave sintering process that does not require long processing times and produces higher performance tungsten carbide products. The PSU project team has designed and built a prototype microwave sintering system to produce WC/Co samples. These samples show a significant increase in wear performance, erosion resistance, and corrosion resistance over conventionally produced WC/Co parts. The technology has been transferred to a commercial enterprise for the manufacturing of tungsten carbide drilling tools.

## Microwave Material Processing Technology

Conventional heat processing involves radiant/resistance heating followed by transfer of thermal energy via conduction to the inside of the body under process. Microwave processing is fundamentally different. Microwaves are a small portion of the electromagnetic spectrum with

wavelengths ranging from 1 millimeter to 1 meter in free space and a frequency between 300 gigaHertz and 300 megaHertz, respectively. While it is well recognized that bulk metals are opaque to microwaves and good reflectors, metallic materials in powder or porous form are very good absorbers of microwaves and can be heated very rapidly. The absorption of microwave energy involves a conversion of electromagnetic energy into thermal energy, making the process instantaneous. With microwave heating the heat is generated internally within the material and flows towards the outside. The advantage for material processing is the very rapid heating rate (>400°C/min), resulting in considerably reduced processing time.

Conventional methods for sintering WC with Co as a binder phase involve high temperature (up to 1500°C) and sintering cycles on the order of one day in order to achieve a high degree of sintering (Schwarzkopf, 1960). Such conditions favor undesirable WC grain growth. It is generally recognized that finer microstructures provide superior mechanical properties and longer life of the product. Often, additives such as titanium carbide (TiC), vanadium carbide (VC) and tantalum carbide (TaC) are used to prevent grain growth of WC grains. Unfortunately such

additives deleteriously affect the mechanical properties of the product. Since microwave heating requires very little time to obtain nearly full sintering, the grain growth is relatively suppressed and finer microstructure is generally obtained. Consequently, the mechanical strength and hardness of the tools are improved.

### PSU Microwave Material Processing Research

It has been well recognized that microwave heating does not work in metals and is good only to oxide ceramics and semi-metals like carbides and nitrides. However, PSU researchers have found that microwave sintering can be applied as efficiently and effectively to powdered metals as to many ceramics (Roy *et al.*, 1999). The microwave sintering of PM green bodies comprising various metals and metal alloys (Fe-Ni-C and Fe-Cu-C systems) at PSU produced highly sintered bodies in a very short period of time (Saji, 1995). Mechanical properties such as the

### Powder Metallurgy

Powder metallurgy (PM) is a highly developed method of manufacturing ferrous and nonferrous metal parts. The PM process is cost effective in producing mechanical parts very close to final dimensions, requiring little in the way of further machining to obtain a part that meets dimensional specifications. The basic procedure in the manufacture of PM parts is:

1. Mix elemental or alloy powders with a suitable lubricant. The most important metal powders in use are iron and steel, copper, aluminum, nickel, molybdenum, tungsten, tungsten carbide, tin and their alloys.
2. Load the mixture into a die or mold and apply pressure. This gives what is called a "compact" or "green body" which requires cohesion only sufficient to enable it to be handled safely.
3. Heat the compact, usually in a protective atmosphere, at a temperature below the melting point of the main constituent so that the powder particles weld together. This is called sintering. If stages 2 and 3 are combined the process is termed hot pressing, or pressure sintering.

In many cases the sintered part is then subjected to additional processing (minor machining, plating, etc.). A very comprehensive description of the entire process and its variations is available at the web site for the European Powder Metallurgy Association ([www.epma.com](http://www.epma.com)).

modulus of rupture (MOR) and hardness, of microwave processed samples, were much higher than for conventional samples. The densities of

microwave processed samples were also better than conventional samples. Figure 1 shows some of typical powder metal steel parts microwave sintered at PSU.

J. P. Cheng, in a Ph.D. thesis (Cheng, 1991), first showed that WC/Co composites could be sintered in a microwave field. Gerdes and Willert-Porada also reported the sintering of similar WC objects from normal size powders, but they followed reactive sintering route using a mixture of pure W, C and Co instead of normal sintering (Gerdes *et al.*, 1994). More recently (Cheng *et al.*, 1997), Cheng and others at the PSU, using a newly designed microwave apparatus (Figure 2), were able to fully sinter WC commercial green bodies containing 12% and 6% Co. They observed that microwave processed WC/Co bodies exhibited finer and more uniform microstructure (~ 1 micron size grains) with very little grain growth, and nearly full density without adding any grain-growth

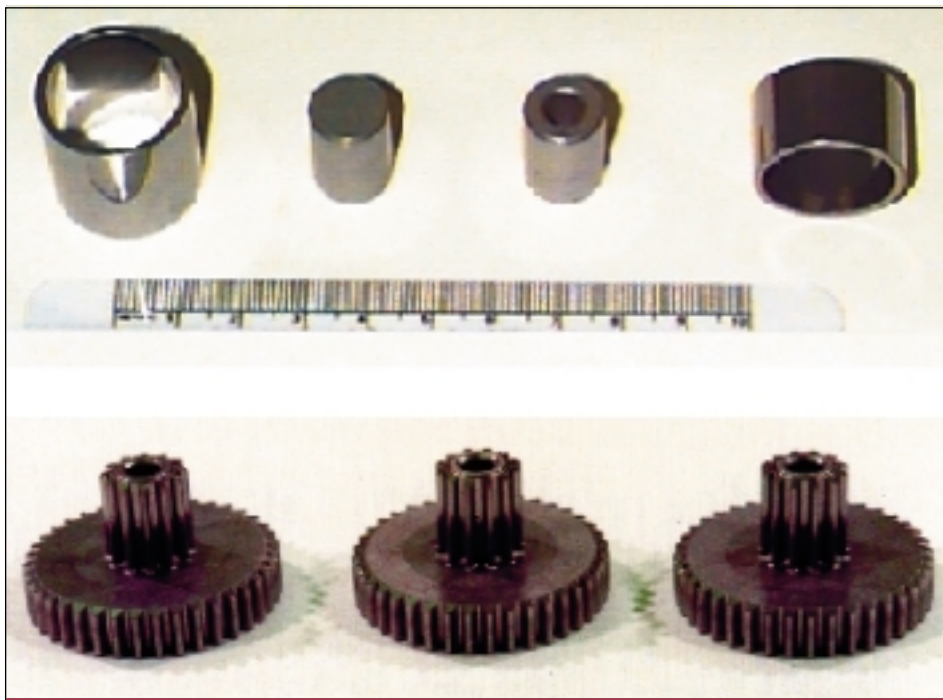
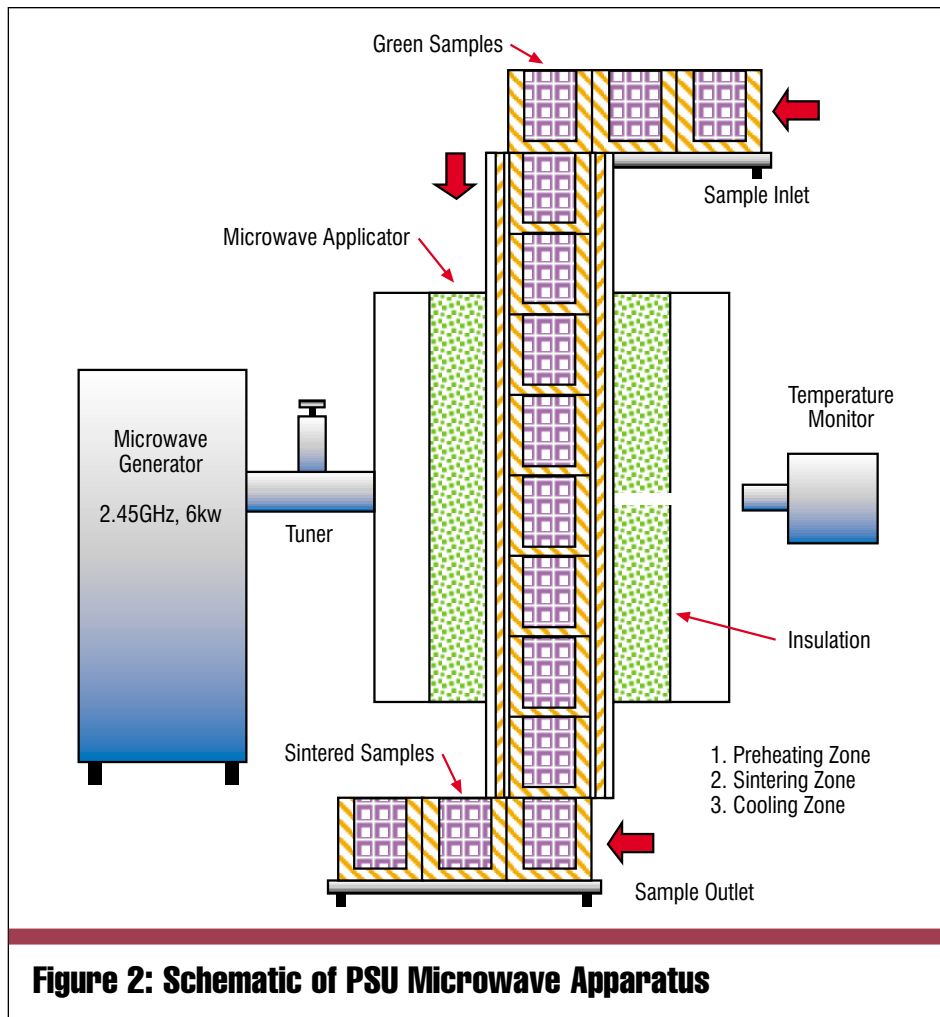


Figure 1: Powder Metal Parts Microwave Sintered at Penn State



**Figure 2: Schematic of PSU Microwave Apparatus**

WC/Co surface have encountered adherence problems. Commercial diamond tools including PDC are basically composites of 2 to 35% volume natural or synthetic diamond embedded in a metal, alloy or WC matrix. Conventional methods to make PDC involve very high temperatures and pressures, and hence the production cost is high.

Currently, PSU is exploring the following approaches using microwave processing:

- Metal (Si, Co, WC/Co) and diamond composites
- Diamond + WC/Co on steel substrates
- WC/Co + TSP (Thermally Stable Polycrystalline Diamond Compact)
- Diamond ceramics
- Encapsulation of steel with diamond composite
- *In-Situ* brazing between diamond composite and steel

The results of exploratory research to develop diamond composites with WC/Co as the matrix have been highly encouraging. PSU researchers are using nickel, titanium and chromium-coated diamond powders because pure diamond powder is sensitive to Co attack and does not form a bond with the matrix. The highlight of this research so far is that PSU has successfully obtained about 96% density of the diamond composites at ambient pressure without damaging the diamond. This has been achieved on small laboratory type samples.

Numerous attempts involving high temperature, hot press and hot isostatic press conditions have been made to develop cutting tools with diamond as an active phase in the composite instead of as a coating material, but these efforts have met with little success. PSU's microwave technology are enabling the processing of diamond-metal composites at lower sintering temperatures and ambient

inhibitors when sintered at 1250°-1320°C for only 10-30 minutes (Clark *et al.*, 1993; Roy *et al.*, 1999; Argawal *et al.*, 1997).

The microwave-processed parts (Figure 3) were tested to determine their strength relative to conventionally produced parts. Some of the highlights of these tests are as the following:

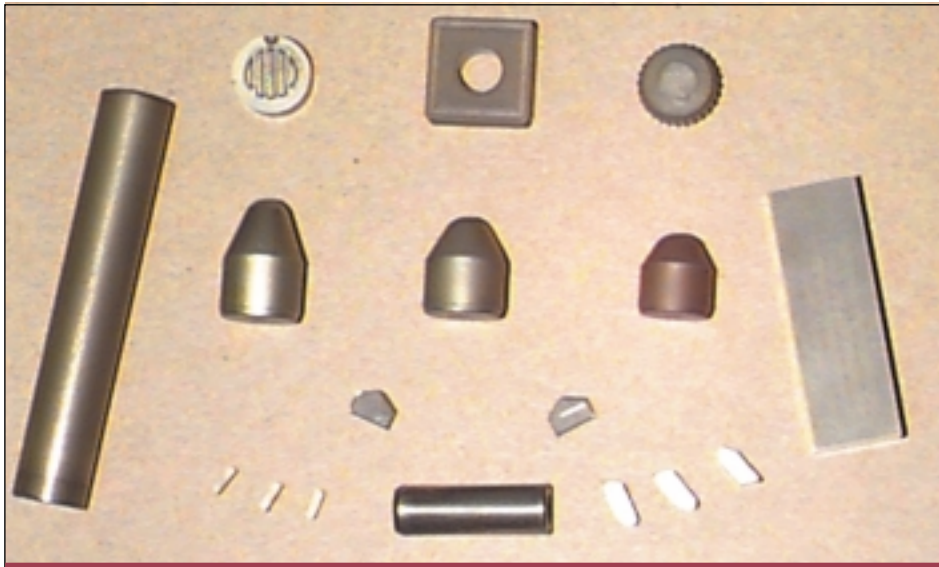
- 20-30% increase in wear performance
- Three to four times more resistant to cobalt leaching by acid treatment.
- Superior resistance to nitric acid corrosion test performed on WC/13%Co substrates. (Less than 1/7 the weight loss.)
- 15% better erosion resistance from dry blasting at 100 psi.

The prototype microwave systems designed, built and tested for their

capability to produce parts with reproducible and consistent properties are now ready for commercialization. PSU has successfully transferred the technology to an industrial partner (Dennis Tool Co.), and has signed an agreement with Valenite, a leading producer of cutting and drilling tools and wear parts, to push the new technology in the marketplace.

### Future Research Directions

The performance of hard rock bit cutters can be improved if diamond is incorporated as a coating material, as TSD (thermally stable diamond) compact or PDC (polycrystalline diamond compact) imbedded on the WC/Co cutter surface, or as an active phase within the WC/Co matrix. Efforts to develop a diamond coating on a



**Figure 3: WC/Co Tools Processed in the PSU Microwave Systems**

the success of microwave sintering of WC/Co parts and initial positive results in the exploratory experiments conducted so far in the diamond composites area. ■

*For more information on the results of this research contact Dr. Dinesh Agrawal, at the Pennsylvania State University, 814-863-8034, or via e-mail at [dxa4@psu.edu](mailto:dxa4@psu.edu)/. For additional information on the Department of Energy's drilling related research efforts, contact William Gwilliam, National Energy Technology Laboratory's Strategic Center for Natural Gas, at 304-285-4401, or via e-mail at [wgwill@netl.doe.gov](mailto:wgwill@netl.doe.gov)/.*

pressure without causing graphitization of the diamond.

Future research on the development

of diamond composites should be able to exploit the inherent advantages of

microwave technology by building on

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# New PRODUCTS, SERVICES & OPPORTUNITIES

## Alliance Advances Natural Gas Technology Transfer in Canada

Petroleum Technology Alliance Canada (PTAC) and the Gas Technology Institute (GTI) are entering into the second year of a three-year agreement for PTAC to provide a Canadian Technology Transfer Agent (CTTA) program to the upstream natural gas industry. Funded primarily by GTI, this program provides the required technology transfer capability and resources to enable technology providers to commercialize natural gas technologies needed within the Canadian natural gas industry.

As part of its mandate, the CTTA program hosted or co-sponsored a number of events late last year, including the Third Annual Canadian Coalbed Methane Conference and the Drilling Waste Management Forum. In addition, the CTTA has established the Technology Centre for Natural Gas (TCNG), a growing collection of natural gas-related technical information supplied by GTI and other PTAC members.

Over 400 delegates attended the 2001 Canadian Coalbed Methane Conference last October in Calgary. Three full days of informative presentations on coalbed methane were offered by leading representatives from both industry and government organizations, followed by a field trip to the Horseshoe Canyon Coals. Over 40 presentations were made during the Conference, on topics such as:

- “Characterizing CBM and Gas Shale Reservoirs for Reserves Booking – Implications for Western Canada.”

Dr. Marc Bustin, University of British Columbia.

- “CBM Potential in Alberta – Geological Challenges and Opportunities.” Rick Richardson, Alberta Geological Survey.
- “Update on US CBM Economics vs. Canadian Economics.” Dr. John Seidle, Sproule Associated Inc.
- “Gas Storage Characteristics of Fractured Shale Reservoirs.” Dave Hill, TICORA Geosciences, a subsidiary of GTI.

Conference Proceedings are available (at a cost of \$195 + shipping and handling) by contacting Brenda Belland, Information Specialist, Technology Centre for Natural Gas (TCNG), phone (403) 218-7712, e-mail [bbelland@ptac.org/](mailto:bbelland@ptac.org/).

At the Drilling Waste Management Forum held last December, 110 attendees listened to presentations on alternate drilling mud systems, on-site waste handling and disposal techniques, treatment of oil-contaminated drill cuttings and bioremediation of drill fluids and cuttings. Forum Proceedings are available by contacting Kerri Markle, Technology Transfer Coordinator, PTAC-CTTA, phone (403) 218-7711, e-mail [kmarkle@ptac.org/](mailto:kmarkle@ptac.org/).

The Technology Centre for Natural Gas is located in the PTAC offices at Suite 750, Hanover Place, 101 – 6 Ave. S.W., Calgary, AB T2P 3P4. For access to TCNG materials and services, please contact Brenda Belland, Phone (403) 218-7712, email [bbelland@ptac.org/](mailto:bbelland@ptac.org/). For additional information about the Canadian Technology Transfer Agent

(CTTA) program, please contact Denis Gaudet, Director, phone (403) 218-7710, e-mail [dgaudet@ptac.org/](mailto:dgaudet@ptac.org/).

## EPA Natural Gas Industry Partners Save \$102 MM by Reducing Methane Emissions

The U.S. Environmental Protection Agency’s (EPA’s) Natural Gas STAR Program recently announced its results for calendar year 2000, showing that its industry partners reduced methane emissions from unit operations and equipment leaks by 34 billion cubic feet (Bcf). At a gas value of \$3 per Mcf per thousand cubic feet, these gas savings are worth approximately \$102 million.

The Natural Gas STAR Program is a voluntary partnership between EPA and the natural gas industry, focused on identifying and implementing cost-effective technologies and practices to reduce emissions of methane, a potent greenhouse gas. The program has more than 90 partners across all of the major sectors of the gas industry—production, processing, transmission, and distribution.

The Gas STAR Program’s mission is to reduce gas losses through market-based, voluntary activities that are both profitable for industry partners and beneficial to the environment. Industry partners choose among a number of best management practices recommended by EPA for minimizing equipment leaks, reducing gas releases from unit operations, and improving equipment efficiency. Partners

implement only those practices that are cost effective for their particular operations.

EPA also encourages companies to discover and implement new practices for reducing gas losses, and such partner-initiated practices now account for over 70 percent of the program's methane reductions.

In addition to achieving record emission reductions in year 2000 (the most current available data), the Program is attracting more partners, with 11 companies signing on over the past year. Currently, the program's production sector partners represent 40 percent of domestic gas production, and the transmission and distribution partners represent 77 percent of transmissions mileage and 51 percent of distribution service connections. The program's partnership with gas processing companies, which was launched in 2000, already represents nearly 60 percent of industry throughput.

For more information on the Gas STAR Program, or to find out about the benefits of becoming an industry partner, call Program Manager Carolyn Henderson at 202-564-2318 or visit the program's website at <http://www.epa.gov/gasstar/>.

role of natural gas in the National Energy Policy and how to strike a balance among policy, regulation, and technology.

Conference highlights include plenary sessions with top energy leaders in the government, a keynote panel session with leaders in the natural gas industry, a panel discussion about natural gas technology investment incentives and benefits, and a future directions panel session on natural gas policy development and implementation. An informal technology information exchange, where government research strategists discuss the latest advancements in natural gas technology, is also scheduled.

This conference will bring together natural gas industry leaders, top regional and national government officials, and state and federal lawmakers seeking a better understanding of the synergies that are possible in a rapidly changing natural gas marketplace. Please watch the SCNG website ([www.fetc.doe.gov/scng](http://www.fetc.doe.gov/scng)) for updated information (agenda, registration forms, etc.), or contact NETL's event management office at Phone: (412) 386-6044, FAX: (412) 386-6486, E-mail: [kimberly.yavorsky@netl.doe.gov/](mailto:kimberly.yavorsky@netl.doe.gov/).

## **SCNG and GTI Sponsor Joint Conference on Natural Gas Technology**

A conference titled "Natural Gas Technology — Investment in a Healthy U.S. Energy Future" will be held May 14-15, 2002 at the Adams Mark Hotel in Houston, Texas. Co-sponsored by the National Energy Technology Laboratory's Strategic Center for Natural Gas & the Gas Technology Institute, the conference will provide a unique opportunity for individuals to express their views on the

# New PUBLICATIONS

## **Canada's Technology Centre for Natural Gas (TCNG) Energy Update**

The TCNG Energy Update is a complimentary monthly report for Canada's the oil and gas industry designed to provide timely and pertinent information on gas and oil prices, national events, and local meetings. If you are interested in receiving copies of this monthly e-mail update, in listing an event or meeting, or would like information on advertising in the TCNG Energy Update, please contact Kerri Markle, Technology Transfer Coordinator, at Phone (403) 218-7711, Fax (403) 920-0054, Email [kmarkle@ptac.org/](mailto:kmarkle@ptac.org/).

## **Coalbed Methane Evaluation Available from Alberta Geological Survey**

A new report titled "Regional Evaluation of the Coalbed Methane Potential of the Foothills/Mountains of Alberta" was published September 2002 and is available from the Alberta Geological Survey. Copies of this 55 page report are available on CD for \$20 plus shipping by calling Sarah Boisvert at (780)-422-3767, Fax (780)-422-1918, e-mail [adm06@ags.gov.ab.ca/](mailto:adm06@ags.gov.ab.ca/). Ask for Earth Science Report 01-19.

## **Committee Updates Assessment of Canadian Gas Resources**

The Canadian Gas Potential Committee (CPGC) has completed their four-year examination of the size of Canada's total gas resource and published the results

in a comprehensive, 570-page report. According to the CPGC's findings, future natural gas supplies will cost more to find and produce as the average size of new gas pools, primarily in Western Canada, shrinks. In addition, frontier production from remote locations in Canada's north and offshore will cost more to develop and operate than supplies from the Western Canada Sedimentary Basin.

The CPGC, an independent group of senior earth science professionals, estimated the potential for Canada's undiscovered gas resources at 233 trillion cubic feet, although they did not assess how much may be economic and accessible to producers. Rather, the report is a compilation of detailed information on pool sizes and reservoir parameters, data that will permit users to conduct further analyses of how much gas may be available for production from each of Canada's sedimentary basins.

The report also states that non-conventional gas sources, such as coalbed methane, may provide important gas supplies, and if the pilot projects in progress or planned prove successful, commercial coalbed methane production may be achieved in the next 10 years.

The 2001 report includes every known gas pool in Canada and uses geological judgment and statistical methods to assess the undiscovered gas potential in all of Canada's sedimentary basins, based on data to the end of 1998. Industry and government professionals reviewed the Committee's

preliminary work. These peer reviews enabled the Committee to refine its final assessments for the report.

The report is available in hard copy with color maps and graphs (US\$350) or as hard copy plus CD ROM (US\$1,700). Approximately 500 Excel workbooks containing all input, output and graphical information on all exploration plays is available on CD ROM, along with the report, for US\$10,000. Orders can be placed with the Canadian Gas Potential Committee at Phone (403) 607-8904, Fax (403) 268-7520, or via the website at [www.canadiangaspotential.com/](http://www.canadiangaspotential.com/).

## **More About Canadian Gas ...**

If you were unable to attend the 2001 North American Gas Strategies Conference held November 5 – 6 by Ziff Energy Conferences in Calgary, but would like to hear what was said and read what was presented, a CD of the conference is now available. The Gas Strategies Conference, now in its eighth year, highlighted issues surrounding the continued growth potential of the Western Canadian Sedimentary Basin. The CD contains all the PowerPoint® presentations as well as complete audio of all speeches. A total of 24 presenters included individuals from, El Paso Production, Aquila Capital & Trade, Husky Oil, Talisman, Petro-Canada, ATCO Pipelines, the National Energy Board, and many others. The CD can be ordered by calling Val Douglas at 1-800-853-6252. Cost is C\$535, or about US\$335, including tax.



## **New EIA Reserves Report Available**

The EIA posted the "U.S. Crude Oil, Natural Gas, and Natural Gas Liquids Reserves 2000 Annual Report" to their website on December 31, 2001. The Report can be downloaded at [www.eia.doe.gov/](http://www.eia.doe.gov/). The easiest way to find it is to simply type "2000 reserves annual report" into the search function.

## **Hart Publications Launches New Magazine for Pipeline and Natural Gas Industry**

Hart Publications recently announced that it will launch a new magazine, *Pipeline and Gas Technology*, for the worldwide pipeline construction, maintenance and rehabilitation business sectors, filling the void recently created when Gulf Publication's *Pipeline & Gas Industry* magazine suspended operations after 47 years. Publication of *Pipeline and Gas Technology* will commence with the April 2002 issue. *GasTIPS* readers with an interest in natural gas pipeline technology issues can obtain a complementary subscription by contacting M. Schroeder at 713-993-9325, ext 163 or by e-mail at [mschroeder@chemweek.com/](mailto:mschroeder@chemweek.com/).

## **PUMP III Solicitation Coming Soon From DOE**

The Preferred Upstream Management Practices III (PUMP III) Program continues an effort, begun in 2000, to encourage implementation of promising advanced technologies for optimizing domestic oil recovery by supporting their identification and demonstration. PUMP III will solicit proposals for work that focuses on one of two problem categories: (1) a regional technical barrier to production with a demonstrated solution, or (2) the

development of data, systems, or methodologies that enable oil permitting agencies to make decisions more quickly and/or that are based on better scientific data about environmental risks. This program funds selected projects where the proposing organizations provide between 20 percent and 50 percent (for field demonstrations) of total project funds.

In April 2001 DOE selected six projects in response to its PUMP I solicitation, and in September 2001 selected another four projects under PUMP II. The PUMP III program has been increased to provide DOE funding of up to \$1 million for each project.

Notification of the solicitation will be posted early in 2002 on the National Petroleum Technology Office website ([www.npto.doe.gov](http://www.npto.doe.gov)) and the solicitation will appear on the NETL site at <http://www.netl.doe.gov/business/solicit/>. The PUMP II solicitation required proposals to be submitted within two months of the solicitation's publication.

# CALENDAR

*Information related to workshops, short courses, and other industry meetings.*

## 2002

**March 10 - 13**

**AAPG Annual Meeting,  
George R. Brown Convention Center,  
Houston, TX.**

American Association of Petroleum Geologists (AAPG), Phone: 800-364-2274 or 918-584-2555. Fax: 918-560-2684. Email: [postmaster@aapg.org](mailto:postmaster@aapg.org). Internet: [www.aapg.org/](http://www.aapg.org/).

**April 25**

**IPAMS 2002 Rocky Mountain Energy  
Technology Conference,  
Adam's Mark Hotel, Denver.**

Independent Petroleum Association of Mountain States (IPAMS), Phone: 303-623-0987. Fax: 303-893-0709. Email: [ngarner@ipams.org](mailto:ngarner@ipams.org). Internet: [www.ipams.org/](http://www.ipams.org/).

**March 11 - 13**

**GPA Annual Convention,  
Wyndham Anatole Hotel,  
Dallas, TX.**

Gas Processors Association (GPA), Phone: 918-493-3872. Fax: 918-493-3875. Email: [gpa@gasprocessors.com](mailto:gpa@gasprocessors.com). Internet: [www.gasprocessors.com/](http://www.gasprocessors.com/).

**April 30 - May 2**

**SPE Gas Technology Symposium  
(with CERI), Calgary, Alberta,  
Canada.**

Society of Petroleum Engineers (SPE), Phone: 972-952-9353. Fax: 972-952-9435. Email: [bwright@spe.org](mailto:bwright@spe.org). Internet: [www.spe.org/](http://www.spe.org/).

**April 2 - 3**

**AADE Annual Technical Conference,  
Radisson Astrodome, Houston, TX.**

American Association of Drilling Engineers, (AADE-Houston Chapter), Phone: 281-922-7093. Fax: 281-922-6779. Internet: [www.aadehouston.net](http://www.aadehouston.net).

**May 1 - 4**

**SIPES Convention & Annual Meeting,  
Lafayette Hilton and Towers,  
Lafayette, LA.**

Society of Independent Professional Earth Scientists (SIPES), Phone: 214-363-1780. Fax: 214-363-8195. Email: [sipes@sipes.org](mailto:sipes@sipes.org). Internet: [www.sipes.org](http://www.sipes.org).

**April 9 - 10**

**SPE/ICoTA Coiled Tubing  
Conference and Exhibition,  
Houston, TX**

Society of Petroleum Engineers (SPE), Phone: 972-952-9353. Fax: 972-952-9435. Email: [bwright@spe.org](mailto:bwright@spe.org). Internet: [www.spe.org/](http://www.spe.org/).

**May 6 - 9**

**Offshore Technology Conference  
& Exhibition (OTC), Reliant Center  
at Reliant Park (formerly  
Astrodomain), Houston, TX.**

Society of Petroleum Engineers (SPE), Phone: 972-952-9353. Fax: 972-952-9435. Email: [bwright@spe.org](mailto:bwright@spe.org). Internet: [www.spe.org/](http://www.spe.org/).

**April 15 - 16**

**North American Gas Strategies  
Conference, Houstonian,  
Houston, TX.**

Ziff Energy Group, Calgary, Phone: 800-853-6252. Fax: 403-261-4631. Email: [gasconferences@ziffenergy.com](mailto:gasconferences@ziffenergy.com). Internet: [www.ziffenergyconferences.com](http://www.ziffenergyconferences.com).

May 14 - 15

**Joint SCNG-GTI Conference on Natural Gas Technology, Houston, TX.**

The conference titled "Investment in a Healthy U.S. Energy Future" will be held at the Adams Mark Hotel in Houston. Co-sponsored by the National Energy Technology Laboratory's Strategic Center for Natural Gas & the Gas Technology Institute, Please watch the SCNG website ([www.fetc.doe.gov/scng](http://www.fetc.doe.gov/scng)) for updated information or contact NETL's event management office at Phone: (412) 386-6044, FAX: (412) 386-6486, E-mail: [kimberly.yavorsky@netl.doe.gov](mailto:kimberly.yavorsky@netl.doe.gov).



August 6 - 9

**COGA Annual Rocky Mountain Natural Gas Strategy Conference & Rocky Mountain Energy Investment Forum, Colorado Convention Center, Denver, CO.**

Colorado Oil & Gas Association (COGA), Phone: 303-861-0362. Fax: 303-861-0373. Email: [Kdrew98103@aol.com](mailto:Kdrew98103@aol.com). Internet: [www.coga.org/](http://www.coga.org/).

August 27 - 29

**AAPEX - Prospect and Property Expo, Houston, TX.**

American Association of Petroleum Geologists (AAPG), Phone: 800-364-2274 or 918-584-2555. Fax: 918-560-2684. Email: [postmaster@aapg.org](mailto:postmaster@aapg.org). Internet: [www.aapg.org/](http://www.aapg.org/).

May 20 - 22

**SPE Western Regional Meeting (with AAPG), Anchorage, Alaska.**

Society of Petroleum Engineers (SPE), Phone: 972-952-9353. Fax: 972-952-9435. Email: [bwright@spe.org](mailto:bwright@spe.org). Internet: [www.spe.org/](http://www.spe.org/).

September 8 - 11

**AAPG Rocky Mountain Section Meeting, Laramie, WY.**

American Association of Petroleum Geologists (AAPG), Phone: 800-364-2274 or 918-584-2555. Fax: 918-560-2684. Email: [postmaster@aapg.org](mailto:postmaster@aapg.org). Internet: [www.aapg.org/](http://www.aapg.org/).

June 6 - 7

**CBM 2002 Conference, Complex, Gillette, WY.**

Powder River CBM Information Council and the Methane Operators Group, Phone: 307 265 5500. Email: [kit@roughriderpower.com](mailto:kit@roughriderpower.com). Internet: [www.wyomingcbm.com](http://www.wyomingcbm.com).

This will be the third annual information fair and trade show held in Gillette related to coalbed methane operations in the Powder River Basin.

September 29 - October 2

**SPE Annual Technical Conference and Exhibition, San Antonio, TX.**

Society of Petroleum Engineers (SPE), Phone: 972-952-9353. Fax: 972-952-9435. Email: [bwright@spe.org](mailto:bwright@spe.org). Internet: [www.spe.org/](http://www.spe.org/).

June 7 - 9

**IPAMS Annual Meeting and Summer Conference, Sonnenalp Resort, Vail, CO.**

Independent Petroleum Association of Mountain States (IPAMS), Phone: 303-623-0987. Fax: 303-893-0709. Email: [ngarner@ipams.org](mailto:ngarner@ipams.org). Internet: [www.ipams.org/](http://www.ipams.org/).

September 29 - October 3

**GTI Technology Transfer Conference, Wyndham Palace Resort Hotel, Orlando, FL.**

Gas Technology Institute (GTI), Phone: 847-768-0500; 847-768-0832. Fax: 847-768-0501. Email: [feingold@igt.org](mailto:feingold@igt.org). Internet: [www.igt.org](http://www.igt.org) or [www.gastechnology.org/](http://www.gastechnology.org/). New annual conference and exhibition cosponsored by the Strategic Center for Natural Gas of the U.S. Department of Energy's National Energy Technology Laboratory.



June 9 - 11

**GTI Natural Gas in the Americas 6 Conference, Republic of Trinidad and Tobago.**

Gas Technology Institute (GTI), Phone: 847-768-0500; 847-768-0832. Fax: 847-768-0842. Email: [education@gastechnology.org](mailto:education@gastechnology.org). Internet: [www.igt.org](http://www.igt.org) or [www.gastechnology.org/](http://www.gastechnology.org/).



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**GTI E&P Services Canada, Inc.**

Suite 720 101 6th Avenue SW  
Calgary, Alberta T2P 3P4  
Phone: 403/263-3000; Fax: 403/263-3041  
E-mail: [paul.smolarchuk@gastechnology.org](mailto:paul.smolarchuk@gastechnology.org)

**GTI E&P Services (Denver)**

19000 West Highway 72, Suite 100  
Arvada, CO 80007  
Phone: 720/898-8200 ext. 13; Fax: 720/898-8222  
E-mail: [dave.hill@gti-ticora.org](mailto:dave.hill@gti-ticora.org)

**GTI E&P Services (Houston)**

4800 Research Forest Drive  
The Woodlands, TX 77381-4142  
Phone: 281/363-7991; Fax: 281/363-7990  
E-mail: [terry.keane@gastechnology.org](mailto:terry.keane@gastechnology.org)  
TIPRO/GTI Phone: 281/363-7989  
TIPRO/GTI E-mail: [sbeach@tipro.org](mailto:sbeach@tipro.org)

**GRI/Catoosa<sup>SM</sup> Test Facility, Inc.**

19310 East 76th  
North Owasso, OK 74055  
P.O. Box 1590 Catoosa, OK 74015  
Phone: Toll-Free 877/477-1910; Fax: 918/274-1914  
E-mail: [ron.bray@gastechnology.org](mailto:ron.bray@gastechnology.org)

**U.S. Department of Energy (DOE)**

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[www.netl.doe.gov/scng](http://www.netl.doe.gov/scng)

National Energy Technology Laboratory (NETL)  
Strategic Center for Natural Gas (SCNG)  
626 Cochrans Mill Road  
Pittsburgh, PA 15236-0340

National Petroleum Technology Office  
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Tulsa, OK 74103-3519  
[www.npto.doe.gov](http://www.npto.doe.gov)

Office of Fossil Energy  
1000 Independence Ave., SW  
Washington, DC 20585  
[www.fe.doe.gov](http://www.fe.doe.gov)

