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ABSTRACT

Over the past 14 years, the Gas Technology Institute and jointly with Uhde since 1997 developing Morphysorb[®] a new physical solvent-based acid gas removal process. Based on extensive laboratory, bench, pilot-plant scale experiments and computer simulations, DEGT Gas Transmission Company, Canada (DEGT) has chosen the process for use at its Kwoen processing facility near Chetwynd, British Columbia, Canada as the first commercial application for the Morphysorb process. DOE co-funded the development of the Morphysorb process in various stages of development. DOE funded the production of this report to ensure that the results of the work would be readily available to potential users of the process in the United States. The Kwoen Plant is designed to process 300 MMscfd of raw natural gas at 1,080-psia pressure. The sour natural gas contains 20 to 25 percent H₂S and CO₂. The plant reduces the acid gas content by about 50% and injects the removed H₂S and CO₂ into an injection well. The Kwoen plant has been operating since August 2002.

Morphysorb® is a physical solvent-based process used for the bulk removal of CO_2 and/or H_2S from natural gas and other gaseous streams. The solvent consists of N-Formyl morpholine and other morpholine derivatives. This process is particularly effective for high-pressure and high acid-gas applications and offers substantial savings in investment and operating cost compared to competitive physical solvent-based processes.

GTI and DEGT first entered into an agreement in 2002 to test the Morphysorb process at their Kwoen Gas Treating Plant in northern BC. The process is operating successfully without any solvent related problems and has between DEGTC and GTI. As of December 2003, about 90 Bcf of sour gas was processed. Of this about 8 Bcf of acid gas containing mainly H_2S and CO_2 was injected back into the depleted reservoir and 82 Bcf sent for further processing at DEGTC's Pine River Plant.

This report discusses the operational performance at Kwoen plant during the performance test as well as the solvent performance since the plant started up. The Morphysorb performance is assessed by Duke Energy according to five metrics: acid gas pickup, recycle gas flow, total hydrocarbon loss in acid gas stream, Morphysorb solvent losses and foaming related problems. Plant data over a period of one year show that the Morphysorb solvent has performed extremely well in four out of five of these categories. The fifth metric, Morphysorb solvent loss, is being evaluated over a longer-term period in order to accurately assess it. However, the preliminary indications based on makeup solvent used to date are that solvent losses will also be within expectations. The analysis of the solvent samples indicates that the solvent is very stable and did not show any sign of degradation. The operability of the solvent is good and no foaming related problems have been encountered. According to plant operators the Morphysorb unit runs smoothly and requires no special attention.

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INTRODUCTION

Natural gas at the wellhead often contains unacceptably high levels of acid gases such as hydrogen sulfide (H_2S) and carbon dioxide (CO_2) that degrade the heating value and are not permissible in the pipeline transmission network for reasons of corrosion, safety and economics. About half of all natural gas produced in the United States requires processing to remove CO_2 , H_2S and/or nitrogen (1.2). Cost-effective removal of these undesirable gases makes more gas available to consumers at an affordable price. Gas Technology Institute (GTI) has pursued research on gas processing technology since the mid-1970s, and again in the 1990s, and has identified the morpholine chemical family as promising candidates for use in acid gas removal. The use of the morpholine family as physical solvents for the removal of acid gas compounds like H₂S and CO₂ from sour natural gas is the result of extensive research by GTI and Uhde GmbH (Uhde) (3.4). Uhde has successfully used morpholine derivatives like N-formyl morpholine (NFM) for the recovery of high-purity aromatics and for pentane/butane separation since 1968, which resulted in commercial applications, like Morphylane[®], Morphylex[®], Octenar[®] and Butenex[®]. Morphysorb[®] technology is the use of mixtures of NFM and N-acetyl morpholine (NAM) as a solvent for the removal of CO₂ and/or H₂S from subquality gas which use is covered by one or more patents. The Morphysorb[®] process is characterized by the following general properties:

- High CO₂ and H₂S loading, further increased at low absorber temperatures resulting in lower solvent circulation
- Low co-absorption of C₁ to C₃ hydrocarbons
- Very high selectivity with good solubility of aromatics
- Selectivity for H₂S acid gas suitable for Claus sulfur recovery or sulfuric acid production
- Good removal of organic sulfur compounds
- Chemical and thermal stability
- Environmentally compatible biological degradable, non-toxic solvent
- Low capital investment smaller vessels and rotating equipment, all with carbon steel construction
- Low operating costs due to lower circulation rate, lower recycle gas flow and lower energy requirements compared to other physical solvents

Earlier work that identified NFM as a leading candidate for acid gas removal for coal gasification processes ₍₃₎ was used as the basis for starting work for GRI in 1990 to explore NFM's application in upgrading subquality natural gas. Over 200 vapor-liquid equilibrium experiments were conducted, with the gas phase containing pure components and mixtures with carbon dioxide, hydrogen sulfide, methane, ethane, and carbonyl sulfide ₍₄₎. The vapor-liquid equilibrium (VLE) data formed the basis for intensive regression analysis with the Aspen PlusTM process simulation software for further use in thermodynamic modeling. The simulation data was later validated by field tests using a

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pilot plant capable of processing up to 1 MMscfd of gas at approximately 1,000 psig (~70 bar). For that purpose, a process development unit was constructed by GTI as a skid-mounted gas processing pilot plant for field-testing at gas wellhead sites to evaluate the improved solvent technologies. Both physical and chemical solvents can be tested in this facility, which has an eight inch-diameter absorption column and eighteen inch diameter regenerator, with either structured or random packing, at feed gas rates up to 1 MMscfd. Over 100 tests were performed with the morpholine family of solvents, processing a total of 50 MMscf of sour gas, with acid gas concentrations up to 45% (5.6.7).

Based on field-test experimental data, GTI/Uhde developed a computer simulation model to verify the benefits of the Morphysorb process. The solvent has a high relative absorption of acid gas compounds as compared to competing solvents, which results in lower circulation rates, and it has lower relative absorption of higher hydrocarbons, which results in less losses and more heating value in the sales gas. The simulation results indicate that the new solvent system will significantly outperform the commercial physical solvent applications for gas sweetening processes (8,9,10). Additionally, the Morphysorb process is capable of at least partially removing organic sulfur compounds like COS, CS₂, and mercaptans. Because of its hydrophilic characteristic, the solvent can be used for simultaneous dehydration. On the other hand, no additional dehydration step is required downstream from a Morphysorb unit if the feed gas is already dehydrated, such as the case in the Kwoen plant discussed herein.

GTI has negotiated an agreement Duke Energy Gas Transmission, Canada (DEGT) is a wholly owned subsidiary of Duke Energy) to test the Morphysorb process in a field experiment in their Kwoen plant, near Chetwynd BC, Canada, which, if successful, can be the first commercial application of Morphysorb. This plant processes 300 MMscfd of sour gas (over 23% acid gas concentration). In that plant the target is to remove 30 MMscfd of sour gas for reinjection. The balance of the sweetened gas, which is still quite sour, is sent to another plant for final acid gas removal and sulfur recovery using conventional technology. This field experimental test of Morphysorb was carried out at Duke Energy's Kwoen Plant in November 2002. The test objectives were to demonstrate Morphysorb operability in a commercial unit and to confirm that the advantages observed in the pilot plant could be achieved on a commercial scale and could meet the specific performance targets as agreed in GTI's demonstration agreement with Duke Energy. The process is commercially licensed to DEGTC under a separate agreement with Uhde.

GTI's role was to develop test criteria for a validation test (such as length of the test, criteria for steady state), parameters measured, determine the sampling location and sampling frequency analysis of data, set up a lab trailer, design sampling loops, observe and conduct performance test and sample collection, analysis of test data and report preparation.

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EXECUTIVE SUMMARY

GTI and Duke Energy Gas Transmission Company, Inc. Canada (DEGT) have entered into an agreement to test the performance of Morphysorb[®] solvent (technology jointly owned by Uhde and GTI) in DEGT's Kwoen Sour Gas Upgrader plant near Chetwynd, B.C., Canada. As part of that agreement, GTI was required to conduct a performance test at the Kwoen plant of 72 hours duration. Gas sampling and analytical equipment in a laboratory trailer was set up in anticipation of this requirement. The parties as per the agreement have established certain performance metrics. The metrics are acid gas pickup per unit volume of liquid, recycle rate as volume rate of gas, methane losses in the reinjection gas as mole percent, solvent losses as mass of solvent per volume rate of gas, and absence of any problems related to foaming.

The test was conducted over the period Nov.12 – Nov.15, 2002. The testing resulted in two useful "steady-state" periods (which differ in conditions significantly from each other) for the purpose of computation of the adjusted performance specification metric values and analysis of results. Material balances were computed for these two periods that were sufficiently close to balance, and the resultant values for the performance specification metrics were computed. In all cases the performance metrics satisfied the established criteria by being either less than adjusted* maximum values or greater than minimum adjusted values.

GTI has continued monitoring the Kwoen plant performance and collecting crucial design data for future commercial applications of the Morphysorb process during 2003. These data were quite helpful in ensuring the smooth running of the plant and in avoiding potential operational problems associated with corrosion, foaming, off spec gas and reduced performance. GTI has collected a total of seven samples for year 2003 and analyzed solvent composition and degradation products for these samples.

The choice of proper solvent depends upon obvious practical attributes such as chemical stability, corrosivity and economic considerations. The first commercial application of the Morphysorb process confirmed these outstanding features of the Morphysorb solvent. The Kwoen plant started up in August of 2002 and the process is operating successfully. As of December 2003, about 90 Bcf of sour gas was processed. The solvent shows a higher relative absorption of acid gas compounds as compared to competing solvents, which results in lower circulation rates, and it has lower relative absorption of higher hydrocarbons, which also results in further reduced circulation rates, lower recycle gas compression costs, and more heating value in the sales gas and less losses.

The Morphysorb performance is assessed according to five metrics: acid gas pickup, recycle gas flow, total hydrocarbon loss in acid gas stream, solvent losses and foaming related problems. Plant data over a period of sixteen months show that the Morphysorb solvent has performed extremely well and exceeded its targets in all of these categories.

^{*}Adjustment to account for deviations from assumed baseline conditions were expressly permitted.

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Morphysorb Process First Commercial Application--Project Opportunity

DEGT Gas Transmission, Canada (DEGT), is a gas gathering and processing service provider to the oil and gas producers in the northeast region of British Columbia, Canada, and operates a sour natural gas gathering system in the Pine River area. The original system was comprised of a 260 MMscfd gas plant and an 83-mile gas gathering trunk line and laterals (Figure 1) where the gas is desulfurized, dehydrated and sulfur is recovered. The plant is capable of removing 50 MMscfd of acid gas (H₂S and CO₂) and can produce 1,050 long tons (LT)/day of elemental sulfur at a recovery rate of 99%. The plant sulfur capacity was nearly doubled to 2,000 (LT)/day after an expansion in 1994.

Since the start up of the 1994 Pine River Expansion, the acid gas quantity has trended higher than the original design, 21.0% versus 16.8%, which was the expansion design basis composition. The plant capacity was fully utilized due to the maximum amount of total acid gas and H₂S that flowed into the plant; however, from a raw gas flow perspective, the raw feed was 120 MMscfd less than the plant design. To process an additional 120 MMscfd of sour gas, DEGT could either add more gas-treating and sulfur-conversion capacity at Pine River or install processing capacity upstream in the Pine River gathering system that would off-load acid gas prior to reaching the Pine River Plant (11,12). Due to significant cost advantage, DEGT decided to pursue the latter option. However, by the year 2000, new sources of sour gas in excess of 130 MMscfd flow capacity were in need of transportation and processing service.

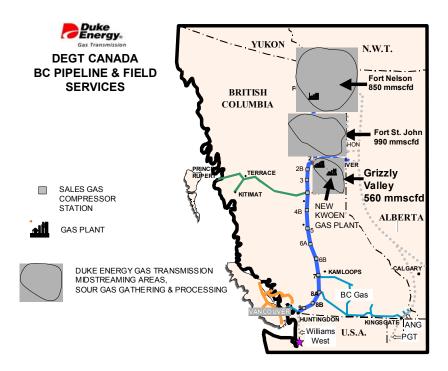


Figure 1. DEGT Canada Midstreaming Areas

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The location of new sour gas wells was as much as 69 miles beyond the current Pine River gathering area. To gain access to the gas production areas, the installation of the new processing capacity, sour gas compression, and a sour gas pipeline were required. Thus, a new Pine River expansion project was commissioned.

The new Kwoen upgrader gas plant was designed to fully utilize the remaining capacity of the Pine River Plant in terms of hydraulic throughput, sour gas removal, and sulfur recovery.

The processing plant consists of a bulk acid gas removal unit and an acid gas re-injection facility. The plant does not produce a sales gas specification product; it simply upgrades the feed gas to the extent of allowing the maximum sour gas flow through the Pine River Plant (see Figure 2).

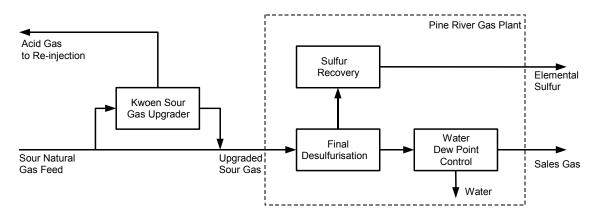


Figure 2: Pine River block flow diagram

Task 1:Morphysorb Demonstration Plant

The overall objective of the Morphysorb demonstration project is to validate earlier laboratory and simulation predictions for overall performance at the Duke facility by removing acid gas from subquality natural gas by utilizing a new physical solvent system.

The role of GTI is limited to assist in basic design engineering services based on GTI's knowledge acquired through lab, bench-scale and pilot plant experiments, in procuring plant facilities, plant start-up, establishing analytical set up for evaluating plant/technology performance, data collection, analysis and report preparation.

Task 1.1.Basic Design Engineering Assistance

The objective of this task is to assist Duke Energy's design engineering firm in basic design engineering for Morphysorb demonstration facilities in general and more

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specifically plant analytical and instrumentation requirements. GTI/Uhde are to share their pilot plant operating experiences in terms of selecting suitable equipment compatible with the Morphysorb process.

GTI and Uhde, the licensor of the process, jointly investigated the solvent change and entered into a demonstration agreement with DEGT. As a part of agreement, GTI/Uhde carried out an engineering review task before the solvent was purchased and loaded into the system. GTI/Uhde has provided DEGT with its review and recommendations regarding the suitability of the "as built facility" for use with the Morphysorb process.

The engineering review consisted of the following:

- Update of the process simulation
- Review of P&I diagrams
- Rating check of tower, including internals, heat exchangers, vessels, pumps and compressors including update of process data
- Update of process data for in-line instruments (control valves, orifices, level transmitters) and safety valves
- Hydraulic check of main pipes
- Specification of anti-foam dosing device
- Definition of performance test procedure and analytical standards for its evaluation, and
- Solvent handling instructions for use in operating manuals.

The engineering review of the Duke Energy Kwoen plant by GTI/Uhde resulted in the following minor recommendations:

(a): Sealing Material Selection

Several screening tests were conducted to determine the compatibility of standard seal materials for the Morphysorb process. It was determined that several seals needed to be replaced due to incompatibility with the solvent, but the situation was judged non-critical so this was deferred until a suitable turnaround opportunity.

(b): Existing Equipment

According to the process design check, the capacities of all compressors (recycle and acid gas compressors) were sufficient. GTI/Uhde recommended that all air coolers are to be rechecked by the air cooler vendor especially those with an indicated under design. According to the process design check, the capacities of all vessels were suitable.

(c): Hydraulic Check

No limitations regarding piping hydraulics were found.

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Task 1.2. Procurement Assistance and Emission Permits

The Gas Technology Institute has provided necessary information on the Morphysorb process solvents to Duke Energy to obtain required environmental permits and, MSDS information and the like. The Material Safety Data Sheet was prepared and submitted to Duke Energy for submission to regulatory authorities such as National Energy Board of Canada.

Task 1.3.Pilot Plant Start-up, Analytical Equipment Set-up and Data
Collection

(a): Description of Kwoen Plant Operations during Performance Test

As agreed in a kick-off meeting prior to the performance test, the plant was operated with one contactor in operation with a feed gas flow of ~150 MMscfd with fixed solvent circulation rate due to limited availability of gas. This was deemed acceptable since single contactor would behave identically to the idled contactor and the downstream consequences of 50% less flow were expected to be minor. Any deviations from the contractually agreed design basis (e.g. feedstock composition/characteristics) were noted for recalculation purpose. A total actual test run of 53 hours was achieved as against 72 planned hours due to equipment problems at the site.

(b): Analytical Equipment Set Up at Plant site

The GTI analytical lab trailer was setup for gas and liquid analysis at the site during the performance test. Two GC's, one dedicated to gas sampling and the other dedicated to liquid sampling were installed. A Karl Fischer titrator was installed for water content analysis; and a ductless fume hood was installed to prevent any H₂S exposure in the lab (figure 3).



Figure 3. Analytical lab area

The ductless fume hood (figure 4) was installed and the sampling systems for both GC's were installed inside the hood for protection against any possible H_2S leaks. Both GC's were installed with packed columns connected to thermal conductivity detectors.

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Figure 4. Fume hood

A Varian Star 3600 GC (figure 5) was used for gas sample analysis. Gas samples that were kept inside the fume hood were directed to a 6-port sampling valve on the GC and back into the fume hood. The vent from the 6-port valve was passed through a solution of NaOH, to oxidize H_2S , and then vented into the fume hood. The H_2S concentration at this point was 1-2 ppmv while sampling a calibration gas containing 10.01% H_2S .



Figure 5. Gas sample GC

(d): Frequency of Measurement

Electronic and manual methods were used to collect the process parameter data. The electronically–collected parameters were recorded using a computerized data acquisition system in place at the Kwoen plant. The process operating data, which can be read out on the data acquisition system, is recorded in a Microsoft Excel spreadsheet using macros and recorded every five minutes. Some process operating data related to the Performance Guarantee Test that were not available with the automatic data acquisition system were recorded manually every four hours.

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(e): Sample Collection

A total number of seven gas sampling points and two liquid sampling points were used for sample collection during the performance test. The locations of sampling points are shown in the following Figure 6.

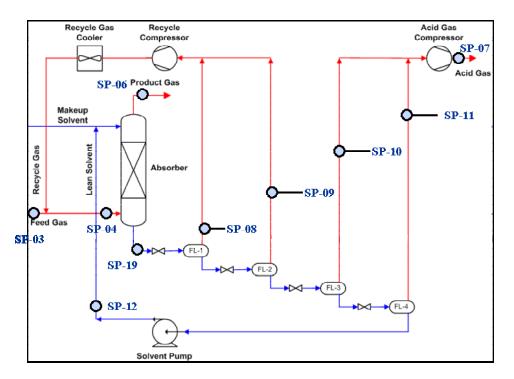


Figure 6: Kwoen Plant Sampling Points

A full set of gas samples consist of Feed, Product, Flash Gas 1 to 4 and Acid gas. Samples were obtained from each of the sample locations during collection of a full set of samples (three times for entire duration of the test) and partial samples (Feed, Product and Acid Gas collected in four other instances). Thus, we have collected a total of seven sample sets during the 72-hour performance test period. The samples are collected based on GTI standard operating procedure for sample collection. GTI established a temporary laboratory at the site to analyze the gas and liquid samples collected during the performance test. In addition to the GTI onsite analysis, Duke had its own independent online analyzers for measuring CO_2 , H_2S in feed, product and CO_2 , H_2S and CH_4 in acid gas streams. A typical sampling location is shown in Figure 7.

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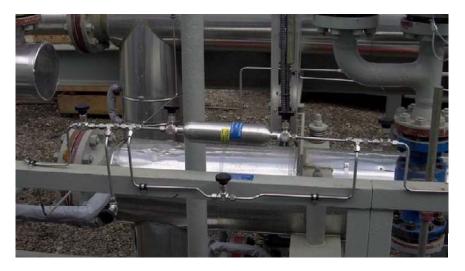


Figure 7. Typical Sampling arrangement at sample location

All the gas and liquid samples that were collected were analyzed immediately after sample collection.

EXPERIMENTAL

The Kwoen Gas Plant is located near Chetwynd, British Columbia in Canada. The plant was designed to reduce the acid gas load of the existing Pine River Gas Plant (PRGP), which is located about 18 miles upstream from Kwoen. The Kwoen processing plant, referred to by DEGTC as a sour gas upgrader, consists of bulk acid gas removal unit and an acid gas injection facility (11,12). The Kwoen plant will take a slipstream of the gas flowing to the PRGP and remove about 30 MMscfd of acid gas for downhole injection. A schematic of the Kwoen plant is given in Figure 8.

The acid gas is absorbed in Morphysorb solution in two parallel 150 MMscfd capacity absorbers. The rich Morphysorb solvent is consecutively flashed at 425 psia and 185 psia in Flash 1 and 2 Flash vessels. To minimize methane losses, the flash gas from these vessels is compressed and recycled to absorber feed. The Flash 3 and Flash 4 vessels, which operate at 65 psia and 25 psia respectively, will generate the acid gas stream that will be compressed and liquefied for downhole injection. The semi-lean Morphysorb solution flows from the Flash 4 vessel back to the absorbers.

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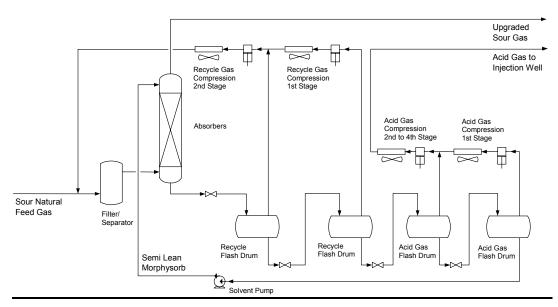


Figure 8: Process Flow Diagram for Kwoen Plant

A photograph of the Kwoen Gas Plant is shown in Figure 9.



Figure 9. Photo of Kwoen Plant – The Upgrader

The acid gas removed from the sour natural gas is not converted to elemental sulfur in a sulfur recovery unit as in other conventional sour gas plants. After compression in a fourstage acid-gas compressor, the acid gas is liquefied and fed to the nine mile, six-inch acid gas pipeline at 1,100 psia. The liquefied acid gas arrives at the injection well as a liquid and flows down the well tubing to a depth greater than 8,200 feet. A summary of the design operating stream conditions and compositions is shown in Table I.

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STREAM	SOUR	UPGRADED	ACID GAS
	FEED GAS	OUTLET GAS	
Flow, MMscfd	300	266	34
Pressure, psia	1085	1074	1015
Temperature, ^o F	63	55	120
mol%			
CO_2	8.60	7.21	19.60
H_2S	13.54	5.33	78.71
CH ₄	77.26	86.81	1.47
C_2H_6	0.21	0.23	0.09
C_3H_8	0.02	0.02	0.02
COS	0.02	0.02	0.05
CH_4S	0.01	0.00	0.04
N ₂	0.34	0.38	0.00
H ₂ 0	0.01	0.00	0.04

Table I: Gas Balance of Kwoen Plant Bulk Removal

Performance Test

The Kwoen plant was mechanically completed in early August 2002. Morphysorb solvent was delivered to the site, loaded into the process train, and brought up to operating pressure with sweet gas. Initial testing with sour gas began in early September. Initial planned flow rates of 90 MMscfd, utilizing gas that was available at that time, were achieved and initial process data and observations made. This was carried out in a single column that was running far below capacity. Originally, there was anticipated to be full flow in both contactors, but the availability of gas and bottlenecks downstream required that only one contactor be run at full flow. The test was to demonstrate that the Morphysorb process is capable of achieving performance on several metrics established between the process owners and DEGT, as described below,

As part of that agreement, GTI/Uhde was required to conduct a performance test at the Kwoen plant of 72 hours duration $_{(13)}$. Gas sampling and analytical equipment was set up in a laboratory trailer for the performance test. The parties, as per the agreement, established certain performance metrics. The metrics are acid gas pickup per unit volume of liquid, recycle rate as volume rate of gas, methane losses in the reinjection gas as mole percent, solvent losses as mass of solvent per volume rate of gas, and absence of

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any problems related to foaming. A further provision of the agreement stipulates that GTI may adjust the specific values of the metrics if the feed gas conditions (composition, flow, temperature, or pressure) are not substantially as specified in the agreement. The test data discussed is this paper was collected during the performance test period of November 12 – November 15, 2002. Electronic and manual methods were used to collect the process parameter data. The electronically collected parameters were recorded using a computerized data acquisition system that is in place at the Kwoen plant. The process operating data, which can be read out on the data acquisition system, is recorded every five minutes in a Microsoft Excel spreadsheet using macros.

The liquid circulation rate was set at a fixed value for these tests. Material balances were computed for those test periods that were sufficiently close to balance, and the resultant values for the performance specification metrics were computed. These are summarized in Table II below. Table III provides information on gas composition of individual streams.

Feed Gas Conditions: Flow 146.8 MMscfd, Pressure 1084.9 psig, Temperature 51.4 °F Acid Gas Composition in Feed Gas: 14.8 mole% H ₂ S, 12.6% CO ₂						
Feed Gas Flow, MMscfd	Acid Gas Flow, MMscfd	Recycle Gas Flow, MMscfd	CH ₄ Losses in Acid Gas Stream, Mole%	Overall Mass Balance (Based on GTI Analysis)	Overall Mass Balance (Based on Duke's Analyzers	
146.8	<u>3 22.35 11.0 1.0 105.99 106.35</u>					

Table II. Performance Test Data

The test run was maintained for 53 hours, and during that time, process data required for evaluation were recorded every five minutes. Seven gas sampling points and two liquid sampling points were used during the performance test. In all cases the metrics satisfied the criteria established by DEGT. All performance targets for Morphysorb were met or exceeded and solvent losses were to be determined separately. The measurements were well in line with earlier computer simulations. Because of the low co-absorption, only 0.95 to 1.2 mole-% of the hydrocarbons are lost to the acid gas stream. The recycle gas stream was 50% lower than predicted for the conventional physical solvent originally considered for the plant.

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		Flash 1	Flash 2	Flash 3	Flash 4	Acid Gas	Product Gas
	Feed Gas						
CH ₄	72.96	54.08	21.81	2.70	2.61	1.16	82.16
CO ₂	10.88	21.01	31.90	25.72	10.09	17.30	10.00
C ₂ H ₆	0.17	0.23	0.03	0.10	0.02	0.05	0.19
H ₂ S	15.68	24.40	46.19	71.29	87.12	81.13	7.47
C ₃ H ₈	0.01	0.01	0.01	0.02	0.01	0.01	0.01
i-C ₄ H ₁₀	0.00	0.00	0.00	0.00	0.00	0.00	0.00
n-C ₄ H ₁₀	0.00	0.00	0.00	0.00	0.00	0.00	0.00
i-C ₅ H ₁₂	0.00	0.00	0.00	0.00	0.00	0.00	0.00
n-C ₅ H ₁₂	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N ₂	0.30	0.27	0.06	0.17	0.15	0.35	0.36
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00

 Table III. Gas Composition (vol. %) of Gas Streams (GTI analysis)

The Morphysorb process at the Kwoen plant was formally accepted by DEGT in December 2002, and has been in operation ever since. In December 2002, the gas volumes at the Kwoen plant were available at design capacities. The typical operating data at design feed flow is given in Table IV.

Feed Gas Pressure: 1136 psig, Feed Gas Temperature: 60.2 °F					
Acid Gas Composition in Feed Gas: 17.90 mole % H ₂ S, 7.8 mole% CO ₂					
Feed Gas Flow, MMScfdAcid Gas Flow, Flow, MMscfdRecycle Gas Flow, MMscfdCH4 Losses in Acid Gas Stream, Mole%					
292	28.7	13.96	0.97		

Table IV. Test Data at Designed Feed Gas Flow

Though the Morphysorb plant would be able to produce an acid gas stream of 36 MMscfd or more at designed capacities, the acid-gas removal capacities at the Kwoen plant are dependent on the requirements of the Pine River plant and in no case should exceed 29 MMscfd until a regulatory approval for a new acid gas injection well is obtained. As of December 2003, about 90 Bcf of sour gas was processed. Of this about 8 Bcf of acid gas containing mainly H_2S and CO_2 was injected back into the depleted

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reservoir and 82 Bcf was sent for further processing at DEGT's Pine River plant. Duke's incentive for this project was to produce more gas without needing to add sulfur processing capacity at Pine River, which would have been prohibitively expensive. The process operation was uncomplicated and stable; no special attention to process control was necessary once the steady-state condition was reached. No solvent-related process upsets (e. g., excessive foaming) were reported since start-up of the plant. Due to leakages of hydraulic oil from the compressors to the process, only one addition of antifoam agent was temporarily required. During 2003, GTI monitored the solvent performance and observed no solvent degradation or undesired side reactions with gas constituents. The solvent losses to the process gas due to evaporation are within the expected range. Because the solvent contains no water, carbon steel is generally used as the material of construction. (See the corrosion section following)

The Kwoen plant debottlenecked the gas going to Pine River in terms of acid gas content, so more natural gas could be produced for sale without any additional sulfur production. The weekly averages of the feed, product, recycle, and acid-gas flows are shown in Figure 10. As can be seen from this figure, the plant operated close to its full capacity 300 MMscfd throughout 2003. Figure 11 shows the gas composition in the acid gas injection stream.

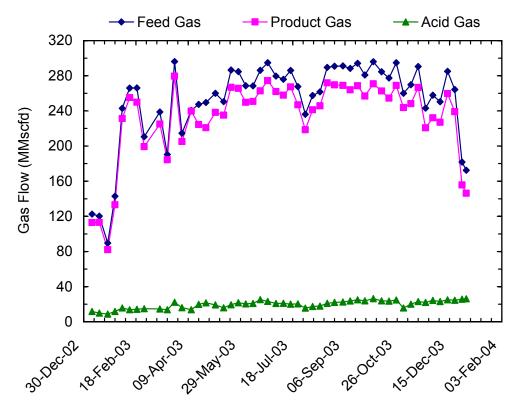


Figure 10: Weekly Averages of the Feed, Product and Acid Gas Flows

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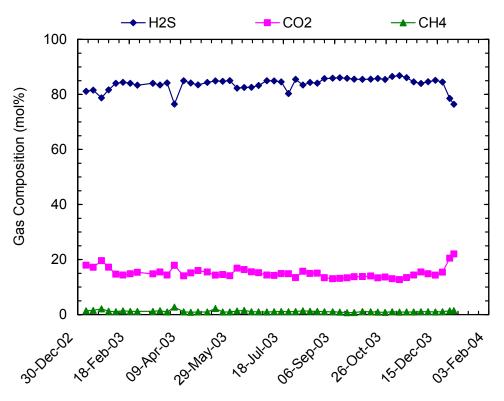


Figure 11: Weekly Averages of the Acid Gas Composition

SOLVENT OPERATIONAL ISSUES:

As part of the agreement with Duke Energy, GTI is obligated to analyze the solvent samples once a month for a period of one year (2003) and keep track of solvent stability and performance. A total of seven sample sets have been analyzed as per details provided in the following Table V.

(a): Solvent Thermal and Chemical Stability

The solvent analyses performed at GTI indicate that the Morphysorb solvent is very stable after 16 months of operation. No solvent degradation has been observed, and the solvent composition is similar to the initial startup value. Total makeup solvent added to date is less than predicted initially. Figure 12 indicates the solvent component ratio deviation compared to the initial solvent loading. The difference in solvent component ratio varies only +/-4 % from the value at the initial filling.

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a 1	D.	
Sample	Date Collected	Remarks
Virgin	July 2002	This sample was collected soon after the solvent was loaded.
1	Nov.15, 2002	This sample was collected on November 15, 2002 during plant performance testing.
2	Nov. 28, 2002	This sample was collected on November 28, 2002 during GTI staff visit to the plant.
3	March 14, 2003	This sample was collected by Duke Energy and shipped to GTI in April 2003.
4	April 20, 2003	This sample was collected by Duke Energy and shipped to GTI in May 2003.
5	June 6, 2003	This sample was collected by Duke Energy and shipped to GTI in June 2003.
6	July 25, 2003	This sample was collected by Duke Energy and shipped to GTI in August 2003.
7	Oct. 26, 2003 Filter Upstream	This sample was collected by Duke Energy and shipped to GTI in November 2003.
8	Oct. 26, 2003 Filter Downstream	This sample was collected by Duke Energy and shipped to GTI in November 2003.

Table V. Morphysorb Solvent Samples

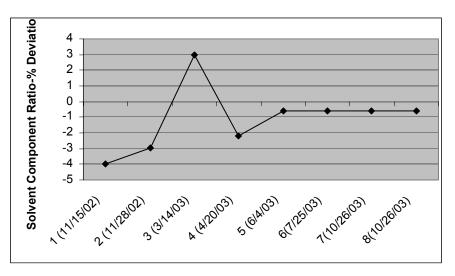


Figure 12. Morphysorb Solvent Component Ratio Deviation

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NFM and NAM are the products of the reaction of morpholine with formic and acetic acid respectively. The solvent samples were analyzed for thermal and chemical degradation products such as formate, acetate, sulfate, oxalate and chloride. Frequent solvent sampling showed nearly the same composition as the fresh solvent, indicating the excellent stability of Morphysorb. A total of ten solvent samples were collected since plant start up. The details regarding possible degradation products are listed in Table VI.

		%WT OF SAMPLE				
Date	Sample	Acetate	Formate	Chloride	Sulfate	Oxalate
7/1/2002	Virgin	0.0226	0.0051	0.0010	0.0005	0.0049
11/15/2002	Sample 1	0.0273	0.0080	0.0096	0.0093	0.0083
11/28/2002	Sample 2	0.0333	0.0112	0.0073	0.0081	0.0064
3/14/2003	Sample 3	0.0313	0.0106	0.0119	0.0152	0.0057
4/20/2003	April 2003	0.0324	0.0071	0.0173	0.6233	0.0190
6/4/2003	June 2003	0.0364	0.0112	0.0221	0.6569	0.0129
7/25/2003	July 2003	0.0392	0.0100	0.0126	0.4772	0.0092
10/26/2003	Filter Upstream	0.0696	0.0105	0.0027	0.1679	0.0113
10/26/2003	Filter Downstream	0.0699	0.0090	0.0033	0.1441	0.0072

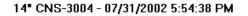
As seen from the above table, sulfate concentration rises unexpectedly and then decreases with the later samples. This is most probably due to the co-elution of SO₃ and SO₄ during the analysis. Unfortunately, a pure SO₄ peak was not be seen, due to the dissolved H₂S and, instead a combination of two peaks observed. The samples in which sulfate concentration measurement was a problem also had high levels of H₂S degassing from the samples. Therefore, the sulfate concentration values reported not correct and ideally should be determined after degassing all H₂S from the sample. Chloride concentration was seen to increase around June. This might be a result of Morphysorb addition to the system. There might be some salt-water trapped somewhere between the storage tanks and the system which was released during addition, and this might have contributed to the increase in chloride concentration. As seen on the later samples, the concentration decreased as time passed. Oxalate and formate concentrations remain almost constant. This indicates that the Morphysorb is not degrading to oxalate or formate.

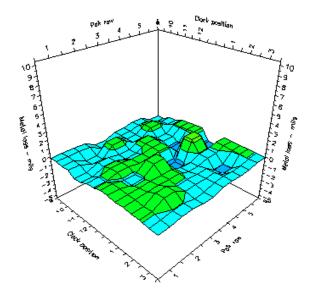
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(b): Corrosion

Corrosion monitoring during the first year of operation detected no measurable corrosion effect on the equipment. In the Kwoen plant, corrosion in the plant is monitored by an advanced FSM (Field Signature Method) as well as through a Microcor monitoring system. FSM is a non-intrusive technique for corrosion monitoring of process equipment and is currently being used in major oil and gas facilities. Any changes of the area as a result of general corrosion, erosion, fatigue or other cracking phenomena cause the change in the "fingerprint" or field signature. FSM generates graphical interpretation showing the severity and location of pits, cracks and corrosion. In the Kwoen plant, these FSM probes are installed in three different locations. The locations are in a rich solvent stream (downstream of the contactor), in a solvent stream between HP and IP flash vessels, and in a solvent stream between IP and MP flash vessels. Figures 13 to 16 show FSM graphical interpretation of corrosion in a location downstream of the contactor. Most of the metal loss at this location is due to erosion, as it is located in an area of highpressure, rich-solvent loaded with acid gas and hydrocarbon components. The erosion was observed immediately after the start of the plant but levelled off during continued operation.



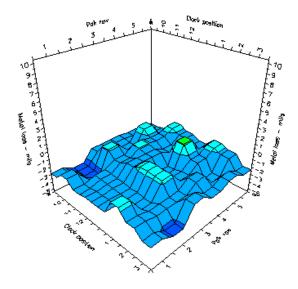




FSMTrend for Windows © 2002 Conficean ASA

Figure 13

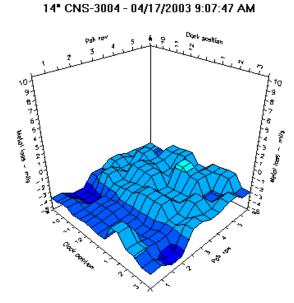
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FSMTrend for Windows © 2002 Conflicean ASA

Figure 14





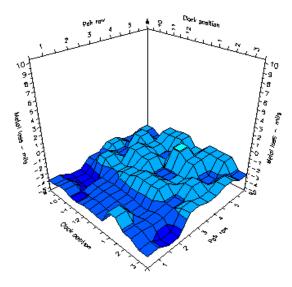
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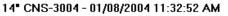
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Figure 15

Contract Number: DE-FC26-01NT41028

-1-3-4-5





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Figure 16

The Microcor corrosion monitoring technology has been installed at the Kwoen plant to increase the speed of response over conventional monitoring techniques such as coupons and electrical resistance probes (ER). There are about 10 probes installed throughout the plant, mostly in liquid streams with a few in the gas phase at location where corrosion monitoring is needed. Figures 17 to 20 show the corrosion data with Micocor probes for the absorber downstream stream, HP, IP and LP flash vessels, respectively.

The analysis of data from FSM probes as well as Microcor probes indicate that corrosion is very minimal (<0.24 mils/year), which is one of the strongest advantage of this process. The overall corrosion in the plant is very minimal, confirming earlier experience with NFM in aromatic removal plants [10].

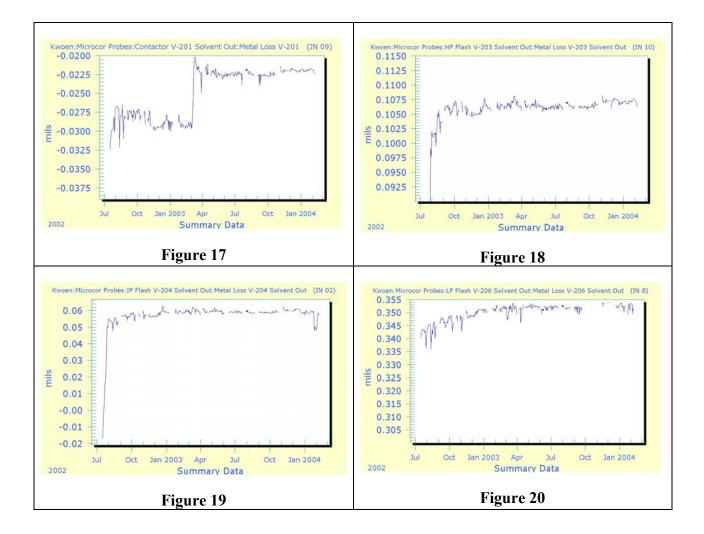
(c): Accumulation of Iron in Solvent

GTI also observed that the iron (Fe) content in the solvent is increasing over time. This is expected level off in the near future. A slow increase in iron concentration in the solvent following start-up to the present is likely the result of a typical passivation process at the carbon steel surface. The online corrosion monitoring system clearly confirmed that corrosion in the plant is very low which is less than 0.24 mils/year. GTI suspects and Duke agrees that the rise in iron concentration is due to iron sulfide content in the incoming sour gas. The details of hydrocarbon and iron content in solvent samples are given in Table VI.

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(d): Accumulation of Higher Hydrocarbons

GTI's analysis shows that the concentration of higher hydrocarbons in the solvent has increased over time (see Table VII). This phenomenon is quite common in any gastreating plant using physical solvents. The accumulation of "heavies" in the solvent and their effect on solvent performance should continue to be monitored closely. There are various procedures that can be implemented if the concentration of heavies increases beyond an acceptable limit. Duke has started skimming hydrocarbon layers from flash vessels to reduce the hydrocarbon content in the solvent.



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Sample	Total Hydrocarbons	Fe Content,
	μg/g	μg/g
Virgin, (July 02)	940	4.0
Sample 1 (11/15/02)	3261	78.2
Sample 2 (11/28/02)	2634	105
Sample 3 (3/14/03)	4929	124.2
Sample 4 (4/20/03)	4137	149.5
Sample 5 (6/04/03)	7594	188.0
Sample 6 (7/25/03)	5065	187
Sample 7 (10/26/03)	14,720	259
Filter Upstream		
Sample 8. (10/26/03) Filter Downstream	15,114	239

Table VII. Hydrocarbon and Iron Content in Morphysorb Solution

(e): Solvent Foaming

So far no foaming incidents or process-related upsets have occurred. As indicated earlier, about 90 Bcf of gas has been processed (as of December 2003) and no anti-foam agent has been used except in one instance, in which leakages of hydraulic oil from the recycle compressors to the process solvent stream caused foaming and the minor addition of an anti-foam agent was temporarily used.

(f): Total Hydrocarbon Losses in Acid Gas Stream

The methane content in the acid gas is continuously monitored using an online analyzer. The total methane losses are in the range of 0.95 to 1.2-mole%. This is another significant advantage of this process. By GTI/Uhde calculations methane losses are 3 to 4 times lower than the competing physical solvent process.

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CONCLUSION

The Morphysorb has the potential to reduce the cost of gas treating significantly, with a resulting potential increase in natural gas supply. This has benefit to the natural gas industry and the consumers. This report is intended to document the success of the process in its commercial debut to facilitate adoption of the process by gas producers/processors.

The Kwoen plant started up in August of 2002. The process is operating successfully without any solvent related problems. As of December 2003, about 90 Bcf of sour gas was processed. The solvent shows a higher relative absorption of acid gas compounds as compared to competing solvents, which results in lower circulation rates, and it has lower relative absorption of higher hydrocarbons, which also results in further reduced circulation rates, lower recycle gas compression costs, and more heating value in the sales gas and less losses.

The Morphysorb performance is assessed by DEGT according to five metrics: acid gas pickup, recycle gas flow, total hydrocarbon loss in acid gas stream, Morphysorb solvent losses and foaming related problems. Plant data over a period of sixteen months show that the Morphysorb solvent has performed extremely well and exceeded its targets in all of these categories and validated GTI/Uhde's simulation models.

These results indicate the Morphysorb process is ready for widespread use in bulk acid gas removal service and also reduced the risk in applying the process for pipeline specification-level and other similar treating applications.

ACKNOWLEDGEMENT

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REFERENCES

- "Chemical Composition of Discovered and Undiscovered Natural Gas in the U.S. Lower-48, March 1993, Hugman, V. and Springer, Energy and Environmental Analysis, Inc.
- 2. "Gas Processing Industry Lower 48 States," Tannehill, C. C. and Gibbs, J., July 1991, Purvin & Gertz, Inc., GRI-91/0232.
- 3. "Acid-Gas and Trace Impurity Removal," Macriss, R. A., and Duncan, D., Annual Report for 1975, A.G.A Project No. IU-4-10, 1976.

Contract Number: DE-FC26-01NT41028

- "Subquality Natural Gas Sweetening and Dehydration Potential of the Physical Solvent N-Formyl Morpholine," J. T. Semrau, N. Palla, and A.L.Lee. Paper presented at 45th Annual Laurance Reid Gas Conditioning Conference, February 26-March 1, 1995
- "An Acid-Gas Removal System for Upgrading Subquality Natural Gas," N. Palla, A. L. Lee, D. Leppin, H. D. Shoemaker, H. M. Hooper and T. F. Moore. Paper presented at the XII Jornados De Gas, Puerto La Cruz, Venezuela, May 8-11, 1996.
- "Acid Gas Removal for Upgrading Natural and Synthesis Gas" Part One;
 M. Groß, J. Menzel; Krupp Uhde GmbH; *Hydrocarbon Engineering*, November 1997
- "Field Test Results of the Physical Solvent N-Formyl Morpholine at Shell's Fandango Plant", Dr. M. Gross. H. Max Hooper, J. Menzel, N. Palla, A. Lee, D. Leppin. Paper presented at IGT's Natural Gas Quality Conference, Orlando, Florida, Dec'1998.
- 8. "Advancements in Treating Subquality Natural Gas Using N-Formyl Morpholine", Nagaraju Palla and Anthony L. Lee, Dr. Manfred Gross, H. Max Hooper, and Johannes Menzel, Dennis Leppin. Paper presented at 77th Gas Processors Association annual meeting, Dallas, March 16-18, 1998.
- "A New Gas Treating Process for Upgrading Subquality Natural Gas using N-Formyl Morpholine", N. Palla, A. Lee, ", Dr. M. Gross. H. Max Hooper, J. Menzel, O. Tondorf, D. Leppin. Paper presented at International Gas Research Conference, San Diego, and Nov 8-11, 1998.
- "Acid Gas Removal for Upgrading Natural and Synthesis Gas" Part Two;
 M. Groß, J. Menzel, O. Tondorf; Krupp Uhde GmbH; *Hydrocarbon Engineering*, June 1998
- "Large Scale Demonstration of Morphysorb Technology on Natural Gas Treating". Kowalsky, Glenn, Leppin, D., Palla, R., Jamal, A, a paper at GTI's first Natural Gas Technologies Conference held at Orlando, FL during September 30-October 2nd, 2002.
- 12. "Performance of Morphysorb in a Commercial Acid Gas Treating Plant"; Glenn Kowalsky, DEGT Gas Transmission; Dennis Leppin, Nagaraju Palla, and Dr. Aqil Jamal, Gas Technology Institute USA; Max Hooper, Krupp Uhde GmbH; paper presented at 53 rd Laurance Reid Gas Conditioning Conference Norman, Oklahoma, February 2003

Contract Number: DE-FC26-01NT41028

13. "An Ace at Removal"; Olaf von Morstein, Johannes Menzel, Uhde GmbH, Nagaraju Palla, Dennis Leppin, Gas Technology Institute USA, *Hydrocarbon Engineering*, February 2004

LIST OF ACRONYMS AND ABBREVIATIONS

- NFM-N-Formyl Morpholine
- NAM—N-Acetyl Morpholine
- Scf—Standard Cubic Feet
- Gal—Gallons
- MM—Million
- DEGT—Duke Energy Gas Transmission Company
- GTI-Gas Technology Institute
- KU—Krupp Uhde