R & D facts

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Separations and Fuels Processing

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Selective Catalytic Oxidation of Hydrogen Sulfide (SCOHS)

High Performance Reactor (HPR) Test Facilities

CONTACTS

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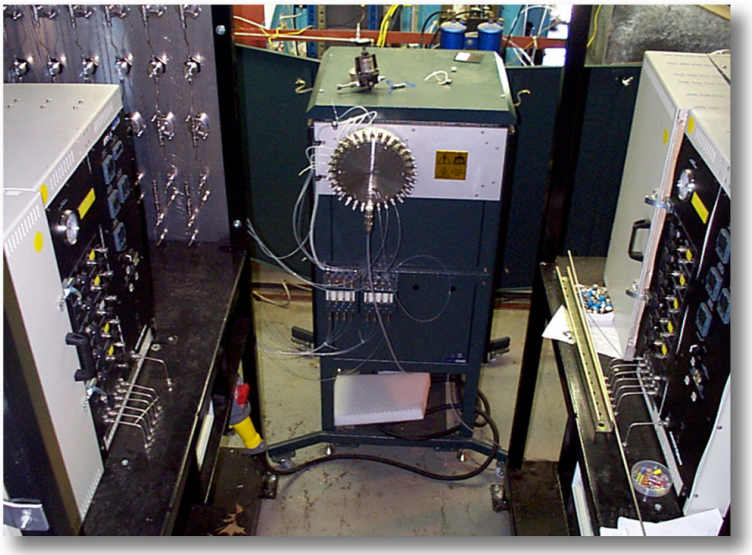
Description

The HPR (High Performance Reactor) system consists of two bench-scale test units that are designed for catalytic, continuous flow, vapor phase reaction analysis. Each facility consists of three sections: (i) feed delivery, (ii) reactor, and (iii) analysis. The feed delivery section consists of reactant gas cylinders, a series of mass flow controllers (Brooks, model 5850i) to accurately deliver respective gas flows, and a syringe pump (Teledyne-Isco, model 100DM) for precise liquid flow delivery. The reactor section consists of a BTRS-Jr® (Autoclave Engineers) reactor body. The analysis section of the test facility includes an online Prima B (Thermo Electron Corporation) scanning magnetic mass spectrometer and a gas sampling station for offline analysis via gas chromatography.



High Performance Reactor Test Facility

The BTRS-Jr® reaction system features a mixing/vaporization manifold and a temperature-controlled 5 ml Incoloy fixed-bed tubular reactor that is heated via an isothermal furnace (Industrial Heater Corporation, Model B56448). The manifold provides blending of incoming gaseous/liquid feed to create a single homogeneous stream feed to the reactor. The Incoloy construction of the reactor provides resistance to the corrosive gaseous sulfur species and/or condensates. The temperature of the reactor is regulated via a single-zone heater that is controlled from a thermocouple located within the catalyst bed. The inlet gas feed line is heated via a forced convection blower assembly that minimizes condensation. System pressure is controlled by a pneumatic back-pressure control valve. Each HPR unit is equipped with switching valve features that allow feed gases to bypass the reactor for direct inlet gas reactant analysis. HPR system control consists of three tunable proportional integral derivative (PID) controllers maintaining reactor and furnace temperature, as well as operating pressure. Safety features include a rupture disc assembly to eliminate the risk of over-pressurization, and power termination to any heater station when a failed/open sensor is detected via its respective temperature controller.

The HPR facilities are remotely operated via a Micro Clientpro computer (933 MHz, 1GB Ram, Windows 2000 SP4), using ProcidiaTM software and process automation hardware. System temperature, pressure, and feed flow rates are programmed from a remote control room. Process variables (i.e., temperature, pressure, and reactant gas flow rates) are logged as a function of time over the course of each experiment, while reaction performance trends are simultaneously logged via the mass spectrometer. Gas samples are taken at defined intervals and analyzed via gas chromatography for accurate quantification of the effluent reaction product gas phase species.

SCOHS Catalyst Performance Assessment

During bench-scale performance evaluation of SCOHS catalyst materials, the catalyst is exposed to both desulfurization and regeneration, simulated, process plant operating conditions. While conducting catalytic desulfurization, 1 to 2 mm catalyst particles are exposed to a simulated fuel gas composition containing 4000 ppm H_2S. Testing is performed at 400 psig and temperatures of 120 to 250 °C, with 0.1 to 10 percent water vapor contained in the simulated fuel gas, representing both dry and humid gas cleaning Integrated Gasification Combined Cycle (IGCC) operations. Testing is typically conducted until 100 ppm total sulfur is detected in the effluent gas stream. Subsequently regeneration or reactivation of the catalyst is addressed, whereby the sulfided-catalyst/sorbent material is exposed to inert, steam-laden, or simulated fuel gas at temperatures of 500 °C for a period of 1 hour at atmospheric pressure. The temperature/pressure swing catalytic/sorption desulfurization/regeneration process is repeated, typically for three cycles with continuous monitoring of the effluent process gas composition.

Method of Catalyst Sample Containment within the HPR Test Units

A plug of quartz wool is initially placed within the reactor body. The catalyst sample is subsequently layered onto the quartz wool support bed. The simulated fuel gas flows downwardly into the reactor body, contacting the desulfurization/ sorption catalyst particles, passing through the particle bed and support quartz wool bed, and then exits the HPR reactor and subsequently, the resulting product gas is compositionally analyzed. The quartz wool is removed after each test, with fresh material inserted into the HPR prior to resuming subsequent testing.

Auxiliary System Capabilities

- On-line Mass Spectrometer Model: Thermo Electron Corp. Prima B (200 amu)
- Pump Model: Teledyne-Isco 100DM syringe pump
- Chemiluminesence Unit Model: Arnel Clarus 500 GC w/ model 4025 SCD analyzer

Supporting Analytical Equipment

- Gas Chromatograph Model: PerkinElmer Autosystem (characterization of low-H_2S)
- Gas Chromatograph Model: HP 5890 (characterization of high-H_2S)
- Gas Chromatograph Model: PerkinElmer Autosystem (characterization of COS)
- Gas Chromatograph Model: PerkinElmer 8500 (characterization of SO_2)
- Gas Chromatograph Model: Carle AGC-400 (characterization of fuel gases)
- LECO Sulfur Analyzer Model SC-432DR
- Quatachrome Autosorb 6: BET surface area, pore size distribution
- Metrohm 719S Titrino: Boehm titration

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HPR Test Facility & SCOHS Performance Operating Conditions

Flanged Reactor Design Criteria	**Body Materials of Construction:** Incoloy **Design Max Temperature Rating:** 982 °C up to 50 psig **Design Max Pressure Rating:** 1500 psig @ ambient temperature, 500 psig @ 650 °C **Materials of Construction for the Remainder of the System:** 316 SS
Flanged Reactor Dimensions	**OD:** 14.3 mm **ID:** 8 mm **Length:** 12.5 in. **Length of Heated Section:** 6 in.
Catalyst System	**Activated Carbon** **Mixed Metal Oxides**
Catalyst	**Quantity:** ~3 gm of material **Particle Size:** ~1-2 mm **Particle Shape:** Irregular
SCOHS Desulfurization/ Sorption Test Operating Conditions	**Temperature:** 120-250 °C **Pressure:** 400 psig **Flow Rate:** 350 sccm @ STP **GHSV:** 3800 hr^{-1}
Desulfurization/Sorption Simulated IGCC Fuel Gas Composition	~30% CO ~10-15% CO_2 0.1-0.4% O_2 20-30% N_2 ~23% H_2 0.1-10% H_2O 1000- 4000 ppm H_2S O_2:H_2S = 1 **Gas Delivery System: Blended Gas Tanks**
Desulfurization/Sorption Test Duration	1-9 hrs pending 100 ppm total sulfur concentration detected in the effluent gas stream
Regeneration Test Operating Conditions	**Temperature:** 400-500 °C **Pressure:** atm **Flow Rate:** 200 sccm **GHSV:** 2200 hr^{-1}
Regeneration Gas Compostion	N_2 N_2/H_2O Simulated Fuel Gas
Regeneration Duration	1 hr