CO₂ Hydrate Process for Gas Separation

Technical Progress Report

Quarterly

Reporting Period Start Date: July 1, 2003

Reporting Period End Date: September 30, 2003

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Issued:
January, 2004

Contract:
DE-AC26-99FT40248

Submitted by:
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Abstract

Modifications were implemented to the hydrogen flow test rig per safety review comments, and the apparatus was tested for leaks. Tests were then done using Helium/CO₂ mixtures to re-verify performance prior to hydrogen testing. It was discovered that hydrate formation was more difficult to initiate, and new initiation methods were developed to improve the tests. Delivery of ETM hardware continued and buildup of the ETM system continued, the ETM is now mechanically complete. The STU (pilot plant) site selection process was resumed because Tennessee Eastman declined to participate in the program. Two potential sites were visited: The Global Energy/Conoco-Phillips Wabash River Plant, and the Tampa Electric Polk Power Plant.

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EXECUTIVE SUMMARY

This document reports on the activities of the third quarter, July thru September, of 2003 for phase 2 of the project on syngas upgrading, a low temperature approach.

Relocation of Test Apparatus

The phase 1 test apparatus has been relocated into an area that can safely contain the hydrogen/H₂S. Safety-related modifications included installing and testing H₂S sensors in the fume hood, installing a pressure vessel catch tank for the separator, and a general revision of the hazards control plan. Prior to the H₂/H₂S/CO₂ runs, the system was operated using Helium/CO₂ mixtures to test the performance with less hazardous gas. Tests were done at low pressure. Difficulties in hydrate reaction initiation were encountered, so new procedures were developed to initiate hydrate formation more reliably.

Engineering Test Model

Delivery of ETM hardware continued and buildup of the ETM system continued. Facility modifications included an outdoor station for two tube trailers and structural supports for the pressure vessels. The test system plumbing was completed, insulation applied and instrumentation installed and calibrated. The ETM is now mechanically complete.

Slipstream Test Unit

The STU (pilot plant) site selection process was resumed because Tennessee Eastman declined to participate in the program. Two potential sites were visited: The Global Energy/Conoco-Phillips Wabash River Plant, and the Tampa Electric Polk Power Plant.

MILESTONE PROGRESS

The following table illustrates progress made toward reaching project milestones.

Date	Milestone Title
September 2003	ETM Mechanically Complete Task 2.3B
August 2003	Milestones are in progress
July 2003	Milestones are in progress
June 2003	Milestones are in progress
May 2003	Milestones are in progress
April 2003	Milestones are in progress
March 2003	Milestones are in progress
February 2003	Milestones are in progress
January 2003	Milestones are in progress
December 2002	Milestones are in progress
November 2002	Milestones are in progress
October 2002	Validation Tests Complete, Task 2.2B

September 2002	Milestones are in progress
August 2002	Literature Review Complete and Viscometer Operational, Task 2.1B
July 2002	Milestones are in progress
June 2002	Milestones in progress
May 2002	Milestones in progress
April 2002	Engineering Test Model System Design Complete Task 2.3A
March 2002	Task 2.1A Optical Cell Fabricated
February 2002	All milestones are in progress
January 2002	All milestones are in progress

TECHNICAL ACTIVITIES PERFORMED DURING THE REPORTING QUARTER

The major activities for the quarter include:

- Testing of the Relocated phase 1 flow test apparatus
- Buildup of the Engineering Test Model flow test apparatus
- Discussions with two possible sites for the STU tests.

Relocation of Phase 1 Test Apparatus

Modifications were implemented to the hydrogen flow test rig per safety review comments. This included leak detection and modifications to the catch basin collecting the separator liquid. Systematic helium leak checks were performed and the system was verified to be leak-tight. Reverification runs were done on the hydrogen flow test rig using helium/CO₂ mixtures. Tests were done at low pressure. Difficulties were observed in the hydrate formation initiation, so new procedures were developed to facilitate the start of hydrate formation.

Engineering Test Model Design

Delivery of ETM hardware continued and buildup of the ETM system continued. Facility modifications included an outdoor station for two tube trailers and structural supports for the pressure vessels. The test system plumbing was completed, insulation applied and instrumentation installed and calibrated. The ETM is now mechanically complete.

Subsystems were tested and the ETM appears to work very well. Since some of the operational approvals were still incomplete, the systems were tested at low-pressure only. The chiller units perform very well.

Initial plans were developed for testing with larger-diameter flow systems when the current ETM tests are concluded. The large capacity of the feed systems and chillers would enable testing with 3/8-inch ID tailtubes. A 1/2-inch Venturi reactor has already been fabricated, and alternate reactor designs are being considered as well.

Slipstream Test Unit

The STU (pilot plant) site selection process was resumed because Tennessee Eastman declined to participate in the program. Two potential sites were visited: The Global Energy/Conoco-Phillips Wabash River Plant, and the Tampa Electric Polk Power Plant.

Meeting notes of the visits are available on request. The Wabash River facility is appealing because it has a high-pressure syngas available. However, the Wabash River plant currently uses a feed of 100% petroleum coke. The Tampa Electric plant uses a mixture of petcoke and coal, which is closer to the objective of the slipstream unit demonstration. However, a larger syngas compressor would be required because of the lower pressure available.

PROBLEMS/ISSUES

Mechanical completion of the ETM in September 2003 puts the program 15 months behind the schedule at the start of Phase 2. Resumption of the ETM and Hydrogen/H₂S flow testing are on hold pending the approval by the lab safety authorities to use these systems at high pressure.