DOE Contract No. DE-AC21-93MC31258 -20
RTI Project No. 93U-5869
July 1, 1998 to September 30, 1998

ADVANCED SULFUR CONTROL CONCEPTS FOR HOT GAS DESULFURIZATION TECHNOLOGY

Quarterly Technical Progress Report

Submitted to

U.S. Department of Energy
Federal Energy Technology Center
3610 Collins Ferry Road
P.O. Box 880
Morgantown, WV 26507-0880

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Submitted by

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1. CONTRACT OBJECTIVE:

The objective of this project is to develop a hot-gas desulfurization process scheme for control of H_2S in HTHP coal gas that can be more simply and economically integrated with known regenerable sorbents in DOE/METC-sponsored work than current leading hot-gas desulfurization technologies. In addition to being more economical, the process scheme to be developed must yield an elemental sulfur byproduct.

2. TECHNICAL APPROACH:

The Direct Sulfur Recovery Process (DSRP), a leading process for producing an elemental sulfur byproduct in hot-gas desulfurization systems, incurs a coal gas use penalty, because coal gas is required to reduce the SO_2 in regeneration off-gas to elemental sulfur. Alternative regeneration schemes, which avoid coal gas use and produce elemental sulfur, will be evaluated. These include (i) regeneration of sulfided sorbent using SO_2; (ii) partial oxidation of sulfided sorbent in an O_2 starved environment; and (iii) regeneration of sulfided sorbent using steam to produce H_2S followed by direct oxidation of H_2S to elemental sulfur. Known regenerable sorbents will be modified to improve the feasibility of the above alternative regeneration approaches. Performance characteristics of the modified sorbents and processes will be obtained through lab- and bench-scale testing. Technical and economic evaluation of the most promising processes concept(s) will be carried out.

3. CONTRACT TASKS:

Phase I - Concept Assessment:

Completed.

Phase II:

<u>Economic Analysis and Process Simulation</u>

The presentation is being prepared for the Pittsburgh Coal Conference being held in September, 1998. The primary topic will be the engineering analysis conducted at North Carolina State University (NCSU).

Work continued on preparation of the presentation for the Pittsburgh Coal Conference being held in September, 1998. The primary topic will be the engineering analysis conducted at North Carolina State University (NCSU).

A paper was presented at the 15th Annual International Pittsburgh Coal Conference held in September, 1998. The paper title was "Advances in Hot Gas Desulfurization with Sulfur Recovery," with the primary topic being the engineering analysis conducted at North Carolina State University (NCSU).

The topical report that was previously submitted for DOE review was finalized and submitted in electronic form (.PDF file format) so that it can be easily entered into the NTIS document system. Bibliographic data are as follow: Gangwal, S. K., J. W. Portzer, G.W. Roberts, and S.C. Kozup. "Engineering Evaluation of Hot-Gas Desulfurization with Sulfur Recovery." Topical Report. May 1998.

Bench-Scale Sorbent Testing

Based on the favorable desulfurization results that were obtained with sorbent FHR-8 (described in the Quarterly Technical Progress Report covering April through June, 1998), two larger samples, designated AHI-1 and AHI-2 were prepared with only slight variation according to the same recipe. Both samples were tested in the atmospheric thermogravimetric analyzer (TGA) using a combination of gases and temperatures that simulated the complete Advanced Hot Gas Process (AHGP): sulfidation, SO_2 regeneration, and O_2 regeneration. Variations in specific conditions, and multiple cycles with constant conditions were run in the TGA in order to determine the preferred conditions to use for bench-scale testing.

Figure 1 shows the temperature profile and describes the cycle conditions that were used in cycles 3 and 4 to test AHI-1, and also reports the weight change (as a fraction of the initial sample weight). It is hypothesized that the initial weight loss with introduction of coal gas for the sulfidation (step 2) is caused by the reduction of Fe_2O_3 to Fe_3O_4. Simultaneously with this reaction, the sulfidation reactions of Fe_2O_3 to FeS and ZnO to ZnS are also occurring, and these reactions eventually result in a weight gain.

The regeneration with SO_2 in Step 4 results in a weight loss, as expected, as the FeS is changed to FeO, but no regeneration of the ZnS occurs. With the introduction of air in step 6 there is a rapid weight increase, as it is hypothesized that the FeO is oxidized to Fe_2O_3, and then there is a subsequent weight loss as the slower reaction of ZnS to ZnO takes place.

Figure 2 compares the weight change data for Cycle #3 with Cycle #4, which was run at identical conditions. Comparable results were obtained.

Figure 3 shows the temperature profile and gas conditions that were used for a 5-cycle test of AHI-2. The initial weight gain of AHI-1 is greater than that for AHI-2 and suggests that AHI-1 will have greater sulfidation capacity.

Figure 4 reports the results of all 5 cycles of the AHI-2 testing. There was an initial loss of activity between the first and second cycles, but in subsequent cycles , the amount of weight loss and gain in each step is approximately the same, showing reproducible multicycle performance.

Based on the results of the TGA testing, experimental conditions are being developed for multi-cycle microreactor testing of AHI-1. The existing microreactor setup will be modified to include SO_2 regeneration, as well as sulfidation and air regeneration.

Work continued on developing a detailed, correct set of process and instrumentation diagrams (P&IDs) for the laboratory AHGP bench unit. These documents will be useful for operator training and during the operation of the bench unit in planned sorbent testing.

PSDF Field Test

Design, engineering, and construction are continuing for the renovation of the Mobile Laboratory for the Advanced Hot Gas Process (AHGP) field test. The new thermocouple readout panel was installed in the control room side of the Mobile Laboratory. Bundled, multi-conductor thermocouple wire was connected to the panel terminal strips and was routed to the equipment room for eventual connection to the field terminal strips. Work on revamping the process equipment to match the new piping and instrumentation (P&ID) drawings continues.

The process and instrumentation diagrams (P&IDs) were enhanced with additional detail to aid the fabrication and construction operations. The preheater furnace and control box was remounted. A new preheater coil (that will be used to provide the hot SO_2 regeneration gas) was fabricated and installed. A small sulfur trap vessel, similar to the one that was used successfully on the laboratory bench-scale test rig, was fabricated and installed in the AHGP bench unit in the Mobile Lab.

The piping around the fluid bed reactor was reconfigured so that an existing orifice flow meter and high-temperature control valve system could be used to control the flow rate of actual coal gas.

Contractors Conference

The presentation 2A.5 “Hot Gas Desulfurization with Sulfur Recovery” was made as planned at the Advanced Coal-Based Power and Environmental Systems ‘98 Conference.

Miscellaneous

RTI received an inquiry from a foreign entity for additional information about the AHGP process for direct regeneration of sorbents to elemental sulfur. The inquiry was apparently based on the 1997 FETC conference proceedings. The entity is currently partnering with a consortium engaged in constructing a 500 MW IGCC project at a European refinery. RTI's response to this inquiry will be based on consultation with DOE.

4. OPEN ITEMS

None.

5. PLANS FOR NEXT QUARTER:

- Continue the engineering design effort for refurbishing the Mobile Laboratory.
- Continue the construction activities in the Mobile Laboratory.
- Continue preparation for microreactor testing of a promising sorbent formulation with multiple cycles of sulfidation and regeneration, prior to bench-scale testing.
- Prepare the presentation for the Pittsburgh Coal Conference (to be held in September, 1998).

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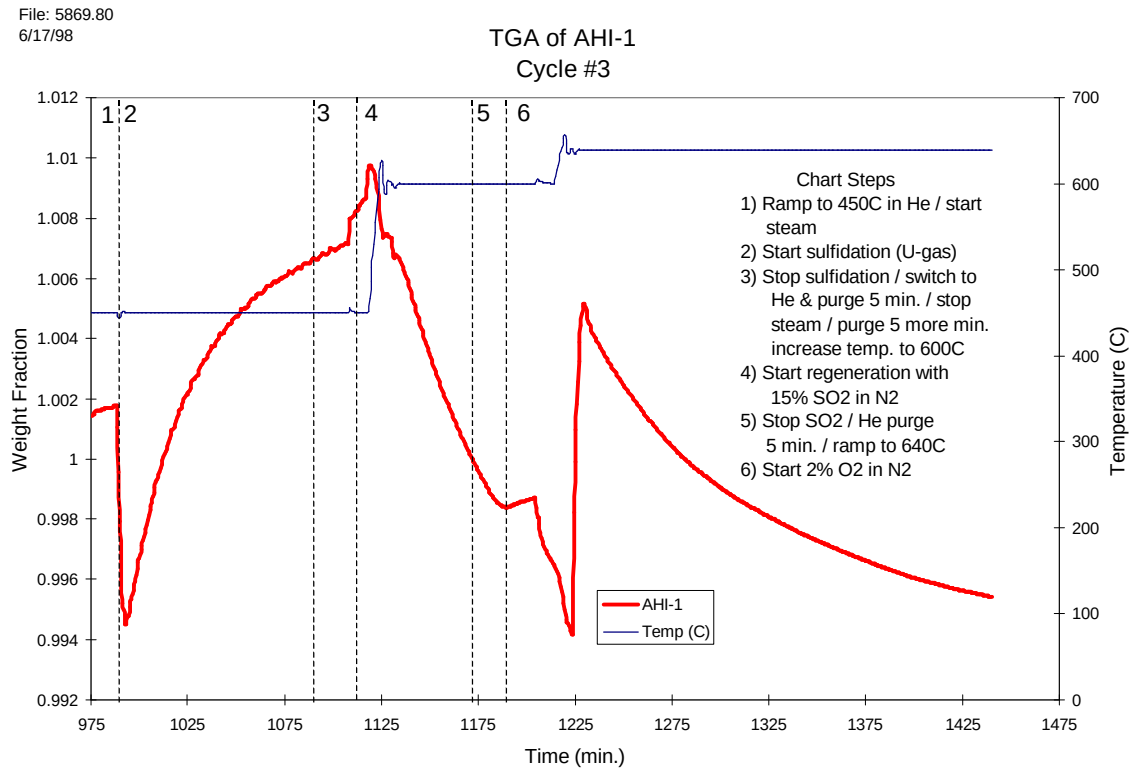

Figure 1.

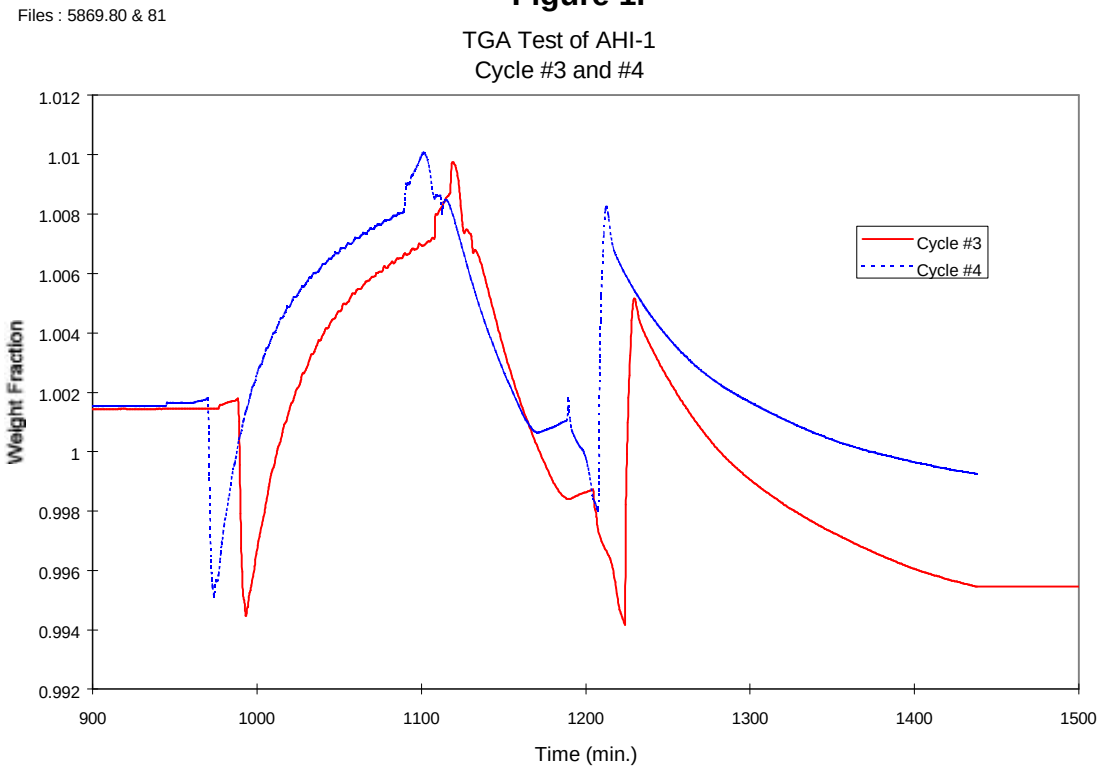

Figure 2.

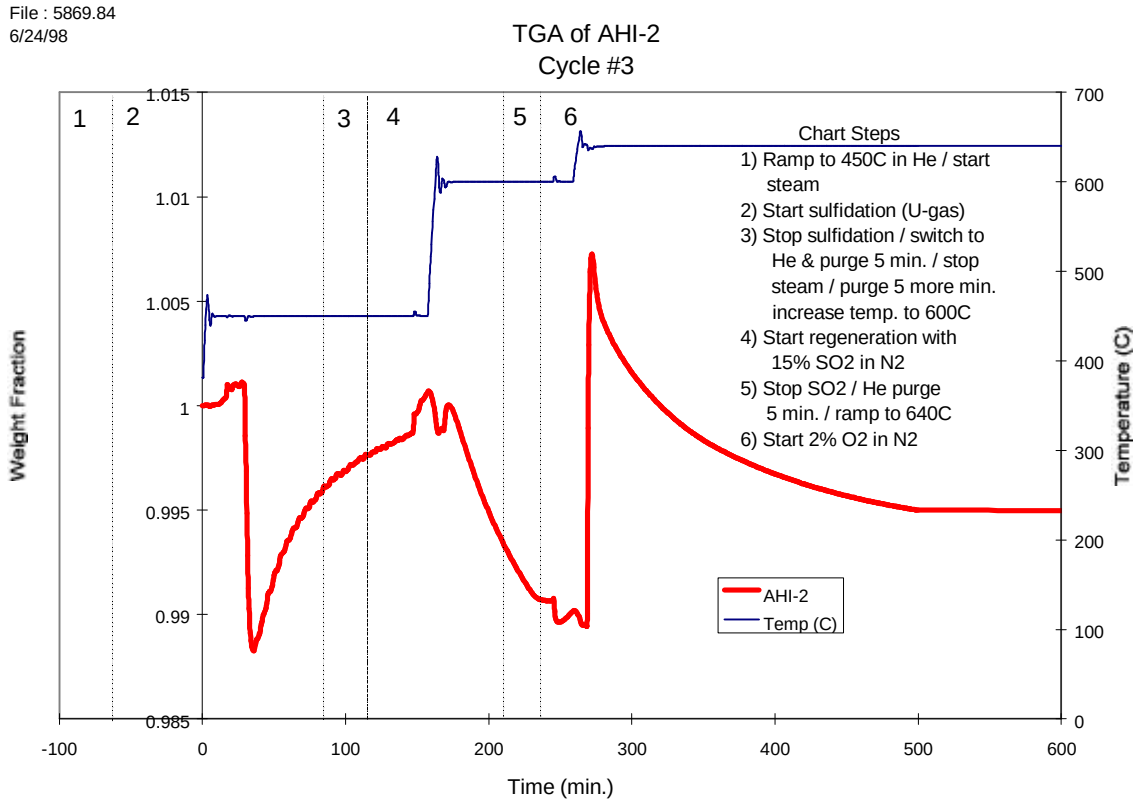

Figure 3.

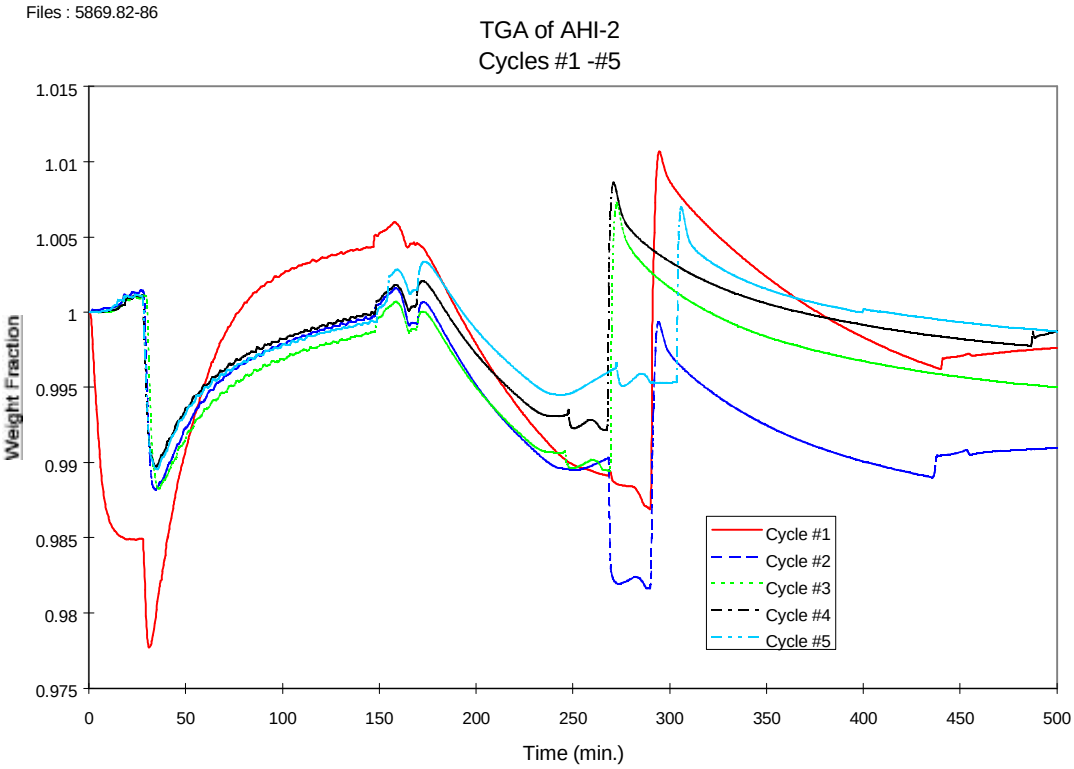

Figure 4.