DOE/PC/90177--T10

EVALUATION, ENGINEERING AND DEVELOPMENT OF ADVANCED CYCLONE PROCESSES

QUARTERLY TECHNICAL PROGRESS REPORT

Quarterly Report #14
For The Period January 1, 1994 to March 31, 1994

Work Performed Under DOE Contract # DE-AC22-90PC90177

For

U.S. Department of Energy
Office of Fossil Energy
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By

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DISCLAIMER

EXECUTIVE SUMMARY

The project goal is to develop an advanced coal beneficiation technology that can achieve high recovery of the parent coal's calorific value, while maximizing pyritic sulfur removal. Coal cleaning is to be accomplished by physical means incorporating an advanced form of cycloning or gravimetric process. Evaluation of different media types and their attendant systems for recovery, concentration, and regeneration is to be completed.

Phase I, Media Evaluation, now completed, involved a paper study and a number of laboratory tests to eliminate all but the best media options. Phase II, Media Testing, involved detailed testing of the more promising media and separators in a closed–loop pilot facility.

In the final phase, Phase III, it is proposed to test individual components of the process using the optimum medium, separator, and medium recovery system(s) selected in the prior phases.

Major activities during this reporting period are reviewed below.

It became evident that the remaining funds would not allow completion of the remaining scope of work. Major tasks remaining were; construction and commissioning of the Bench Scale Circuit (BSC), Process Optimization and Testing, and preparation of the Final Report.

CTC requested additional funding of approximately 300 K to complete the scope of work; however, DOE made it clear there was no additional funding available. Therefore, it became necessary to modify the Work Plan. Two alternatives were proposed:

1) Construct the fully integrated circuit (BSC), and test one project coal. CTC initially supported this option but would have had to absorb any and all costs over the contract amount if this alternative was performed.

2) Perform closed loop component testing of critical process components, i.e., the filter, decanter, evaporator, and grinding system. DOE supported this option. Under this option, a fully integrated circuit would not be built, but a test stand would be required.

CTC agreed to the second option and submitted a new Work Plan revision that reflected component testing. CTC also requested a time extension until 12/31/94. Work Plan approval is pending, but the time extension was granted.

Hot water wash experiments were performed in the laboratory with filter leaf tests to determine whether hot water would recover significantly greater amounts of calcium

nitrate than previously obtained and thereby lower process costs. No significant improvement in recovery of calcium nitrate was found, but there was some questions concerning the results.

Triple S Corporation has fabricated and delivered the structural steel for the BSC. Steel remaining to be delivered includes the platework, chutes, sumps and tanks. The steel was a long lead item and had been ordered earlier. While BSC construction is no longer planned, approximately half of the steel can be used to construct a test stand for the planned Component Test program.

INTRODUCTION

"Evaluation, Engineering and Development of Advanced Cyclone Processes" is a research and development project aimed at reducing pyritic sulfur in coal products. Minimum project goals are to produce a 6% product ash and 85% pyritic sulfur rejection while retaining 85% of the parent coal's heating value. A number of media and separator options are to be evaluated and tested and performance characteristics of the critical process components investigated in a 1,000 lb/hr closed–loop test stand constructed on a site provided by Coal Technology Corporation (CTC) in Bristol, Virginia.

The project involves the physical beneficiation of coal based on the density differential that exists between clean coal and its impurities, i.e., pyrite and ash bearing minerals. Coal may be beneficiated by employing a parting liquid or pseudo–liquid with a specific gravity between that of coal and its impurities. A number of parting liquids (separating media) were considered for evaluation and testing in this program. They represented three families of liquids: aqueous solutions, organic liquids, and aqueous suspensions. The aqueous suspensions of starch and solutions of sugar, though environmentally benign, were dropped from consideration early in the program because of their high viscosity and handling difficulties. Micronized magnetite, an aqueous suspension, was also dropped because another firm plans to develop it to commercialization.

Except for the aqueous suspensions, the candidate media may be classified as true heavy liquids. True heavy liquids are not affected by the multiple gravities (g) required in fine coal gravimetric separation processes, whereas suspensions may deteriorate if subjected to excessive g force. Multiple gravities in combination with true heavy liquids can be used to increase the speed and efficiency of separation of particles of small size having slight differences in density. Generally, the greater the number of gravities the more precise the separation. This implies the use of small diameter, high–pressure cyclones or high gravity centrifuges. Therefore, the term cycloning encompasses centrifuges and other enhanced–gravity devices where fluid motion or mechanical motion is converted to centrifugal force.

Task 1 - Project Planning and Management

Task Description or Objective(s): The objective of this task is good technical and fiscal control and management of this project, both internally and externally, by Coal Technology Corporation (CTC), the prime contractor. CTC is responsible for interfacing with the DOE and ensuring that all subcontractors fulfill their responsibilities and meet the milestones and goals of the Project Work Plan. The subcontractors are:

- Process Technology, Inc. (PTI) – providing analytical services and conducting Phases I and II laboratory and closed–loop media testing.

- ICF Kaiser Engineers, Inc. (ICF–KE) – performing detailed design of a 1000 lb/hr Bench Scale Circuit.

- Intermagnetics General Corporation (IGC) – providing media, separator, and technical service for magnetically enhanced media.

Project management is an ongoing effort designed to monitor the subcontractors, keep the project running smoothly, resolve problems, and in general ensure that the project is performed on a timely and cost–effective basis.

Activity: Due to the limited funds remaining in the project budget it was decided to pursue component testing rather than the construction and testing of a complete integrated circuit as originally planned. Critical components include size reduction, separation, and filtering systems. It is also proposed that evaporative studies investigating pH variation, corrosion potential, and medium condition be performed by a qualified vendor. A subcontract to erect the BSC was quoted earlier at $421,661 while erection of a test stand is estimated at $100,00 to $150,000. Revised Work, Cost and Labor Plans were prepared and submitted consistent with component testing. Approval of the revised Plans is pending.

Task 2 - Coal Procurement and Characterization (Three Phases)

Task Description or Objective(s): The objective of this task is to provide characterized feedstock for all three phases of the program. The three phases are associated with: (I) Separating Media Evaluation, (II) Separating Media Testing, and (III) Process Optimization Testing. A total of four coals have been selected for the program. The four coals constitute a substantial reserve, are technically difficult to clean, and contain significant amounts of pyritic sulfur. The characterization will determine the degree of liberation needed to reach the project goals. This information will be used as a database for the entire program and to measure the performance of individual tests.

The project's requirements for the four coals include:

- Raw coals must have moderate to high pyritic sulfur contents that are not sufficiently liberated in conventional cleaning.

- Precleaning operations must recover 90 to 95% of the parent raw coal's heating value while principally removing coarse rock and fine clays.

- Raw and clean coal handling systems must facilitate readily obtaining one–to–two ton samples of the raw and precleaned coals for Phase I and III characterization

Activity: No activity during this period.

Task 3 - Evaluation Plan and Test Plan Formulation

Task Description or Objective(s): Task 3 represents the planning stage of the work that will be conducted during Phases I through III of Task 6. It is the technical basis of the program and provides for evaluating the media by paper study supplemented by laboratory study, selection of medium and separator combination, and implementation of one medium/separator option for long–term, open loop testing. The three phases of this task are:

- Phase I Media Evaluation
- Phase II Separating Media Testing
- Phase III Process Optimization Testing

These plans detail Task 6, Scope of Work.

Activity: The Separating Media Evaluation Plan was approved by DOE in February, 1991. The Preliminary Separating Media Evaluation Report contains the Separating Media Test Plan, which was carried out during Phase II. A draft Process Optimization Test Plan for Phase III was submitted on December 18, 1992, intended as a plan for an integrated circuit. It may require revision to reflect the newly planned component testing program.

Task 4 - Bench Scale Test Circuit Design

Task Description or Objective(s): This completed task provided the design of a fully integrated bench scale advanced cycloning test circuit (BSC). The design of the advanced cycloning test circuit was based on the Separating Media Evaluation and Testing results (Phases I and II) and the detailed characterization of the four proposed test coals. ICF–KE was the lead team member for Task 4.

The BSC design fully integrates all pretreatment, cleaning, and post–cleaning operations necessary to allow continuous steady–state operation including at least one (1) uninterrupted run of 100 hours duration for each of the four test coals.

Activity: No activity during this period.

Task 5 - Bench Scale Test Circuit Set-Up and Commissioning

Task Description or Objective(s): This task covers the functions necessary to construct and commission the bench scale circuit module at CTC in Bristol, Virginia. The construction will be performed by an experienced contractor with construction management provided by CTC. The start–up will be supervised by CTC and performed by craft labor supplied by the construction contractor.

Activity: This activity is on hold pending approval of the revised Work Plan. It is proposed to construct a test stand for critical components instead of the fully–integrated BSC as originally planned.

Received Equipment and Materials
Triple S Corporation has fabricated and delivered the structural steel for the BSC. Steel remaining to be delivered includes the platework, chutes, sumps and tanks. Though there are no longer plans to construct the BSC, the steel had been ordered earlier as a long lead item and could not be canceled. Fortunately, approximately half of the steel can be used to construct a test stand for the planned Component Test program.

Task 6 - Evaluation and Test Plan Implementation

Task Description or Objective(s): This task consists of the technical implementation of plans produced and approved under Task 3. Please refer to the project Separating Media Evaluation Plan and Separating Media Test Plan.

Phase I - Media Evaluation

Activity: The media evaluation has been completed and reported in the revised Preliminary Separating Media Evaluation Report (PSMER) during a prior reporting period. In the PSMER, methylene chloride/perchloroethylene, calcium nitrate/water, MEM (Magnetically Enhanced Media), and water were selected as media for inclusion in the test matrix for performance testing during Phase II.

Phase II - Separating Media Testing

Activity: Experiments were performed on February 23 & 24, 1994 to determine if a hot water wash could improve calcium nitrate recovery. Anticipating greater solubility, it was theorized that hot water would recover significantly greater amounts of calcium

nitrate in comparison to cold water used in prior experiments. The BSC and conceptual process would have hot water returning from the evaporator/condenser circuit during normal operation. This hot water would be utilized for rinsing and residual calcium nitrate on those cakes needed to be determined. Washing experiments were conducted using rinse water at 100 and 150 °F on ceramic filter leaves. The procedure used to–date for measuring calcium, the indicator for calcium nitrate, required boiling the solids in deionized water for several hours, followed by filtering, and then analysis of the filtrate using an atomic absorption spectrometer. This method assumes that all of the non–naturally occurring calcium (medium) reports to the water phase. Results are shown in Table 1 and related Figures 1 and 2 with cold water results from 1992 taken from Tables 3 and 4 of the FSMER.

The hot water wash did not provide significant improved recoveries compared to those achieved with cold water; however, there is some doubt as to the validity of these experiments.

It was noted by Outomec personnel that the more recent solutions did not behave as they did in 1992. During hot water wash experiments, it was discovered that on the first pass fresh calcium nitrate solutions filtered significantly slower through both ceramic and GORE–TEX materials used in the filter system. Refiltered solutions filtered quickly. Experiments conducted in 1992 used actual products of separation from PTI's Phase II closed–loop circuit. Fresh medium had first been passed through a vacuum filter prior to introduction into the closed–loop circuit, presumably removing any contaminants. Fresh solutions are cloudy in appearance while filtered ones are clear. There appears to be a contaminant in the calcium nitrate that is removed upon the first filter pass. The contaminant appears to retard flowrate through the filter surface. If so, it is expected that a commercial process circuit would utilize a pre–filter to remove the contaminant from make–up medium prior to introduction into the process circuit. It is planned to conduct additional experiments using test slurries made up of coal and pre–filtered medium in the next Quarterly Period.

Phase III - Process Optimization Testing

Activity: It is proposed that component testing replace Process Optimization Testing.

Task 7 - Data Analysis and Reporting

Task Description or Objective(s): This task takes place throughout the project to keep up with the day–to–day logging of data and reporting requirements. Dissemination of data to the Project Team members is vital to the project. Analysis and interpretation of the data are critical to this task. Numerous reports are required during the life of the project. Technical reports required under the contract include the following: Biweekly and Quarterly Progress/Status Reports, Washability Analyses Report,

Preliminary Separating Media Evaluation Report, Final Separating Media Evaluation and Test Report, and the Final Report.

Activity: The Biweekly and Quarterly reports have kept DOE informed concerning the progress of the project. Other reports and plans are covered under their applicable Tasks.

Task 8 - Conceptual Design

Task Description or Objective(s): This task is performed with the objective of providing DOE with a conceptual description and detailed estimate of the cost to construct and operate a 20 tph advanced cycloning test module. This is a modification to the contract, which originally called for detailed design of a 3 tph circuit. CTC will be the lead team member for Task 8. This Task involves the conceptual design of a fully integrated, continuous operation, advanced cycloning test module. The conceptual design will be sized for 20 tph feed rate and will include all necessary pre–treatment, cleaning and post–treatment unit operations. The conceptual design will be based on the results of the Process Optimization Tests performed under Phase III of Task 6.

Activity: No activity during this period.

Task 9 - Final Reporting

Task Description or Objective(s): The Project Team members will submit a Draft Final Technical Report in the 41st month of the project. This report will be preceded by a detailed outline to be reviewed by the DOE. The final report will meet contract requirements as stated in the Project Work Plan and will comply with DOE Order 1332.1A (Uniform Reporting System).

Activity: No activity during this period.

Task 10 - Decommissioning

Task Description or Objective(s): CTC shall be responsible for decommissioning, protecting, removing, and disposing of all contractor installed property encompassed by the contract. Contractor procured Government property shall be protected and dispositioned as directed by the DOE Contract Officer. This is strictly limited to the cost of decommissioning, removal, protection, and shipment from CTC to PETC.

Activity: No activity during this period.

Table 1

RESIDUAL CALCIUM NITRATE MEASUREMENTS OF WASHED FILTER CAKES

Outomec Cold Water Wash, 1992

Clean Coal	Wash Rate*	% Soluble Calcium
	1.24	0.748
	2.07	0.174
	2.13	0.177
	2.29	0.205
	2.80	0.369

Reject	Wash Rate*	% Soluble Calcium
	0.00	3.838
	0.36	3.024
	0.42	3.143
	1.41	0.881
	1.37	1.312
	1.32	1.296

*Wash Rate = wash water weight/dry solids weight.

Eimco Cold Water Wash, 1992

Clean Coal	Wash Rate*	% Soluble Calcium
	0.00	5.620
	0.67	0.981
	1.33	0.685
	2.00	0.619
	3.33	0.412
	6.67	0.365

Reject	Wash Rate*	% Soluble Calcium
	0.00	5.230
	1.00	0.903
	2.00	0.621
	4.00	0.520
	5.67	0.467
	10.00	0.458

Table 1 (Continued)

Outomec Hot Water Wash, February 23–24, 1994

Clean Coal	Wash Rate*	% Soluble Calcium
	3.05	0.18
	3.99	0.17
	4.20	0.15
	3.37	0.17
	3.04	0.19
	3.62	0.17
	2.58	0.22
	3.55	0.15
	3.10	0.18
	2.58	0.22
	3.54	0.18

Reject	Wash Rate*	% Soluble Calcium
	4.55	0.74
	3.48	0.77
	3.07	0.64
	3.36	0.81
	2.95	0.80
	3.27	0.76
	3.31	0.78
	2.76	0.80

FIGURE 1

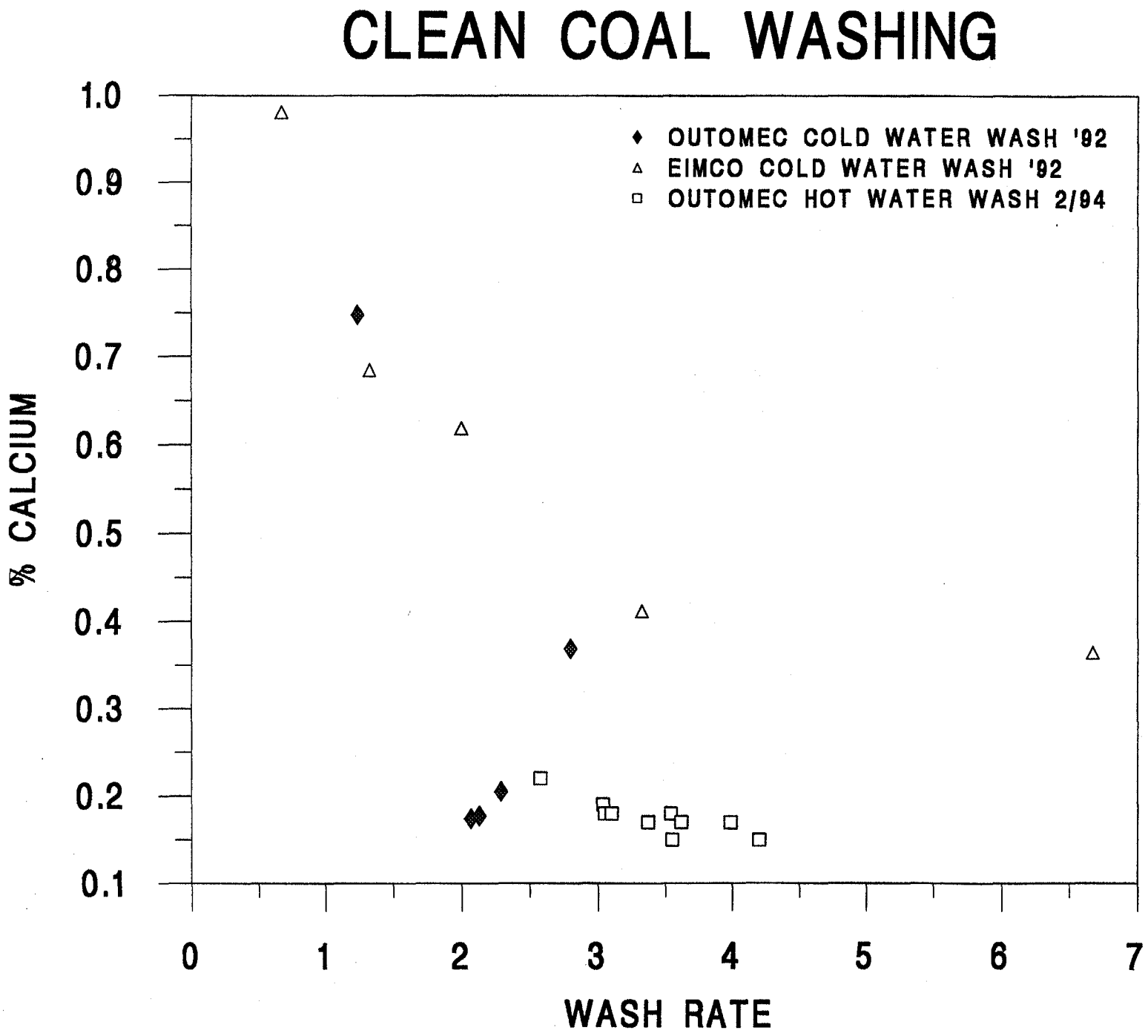
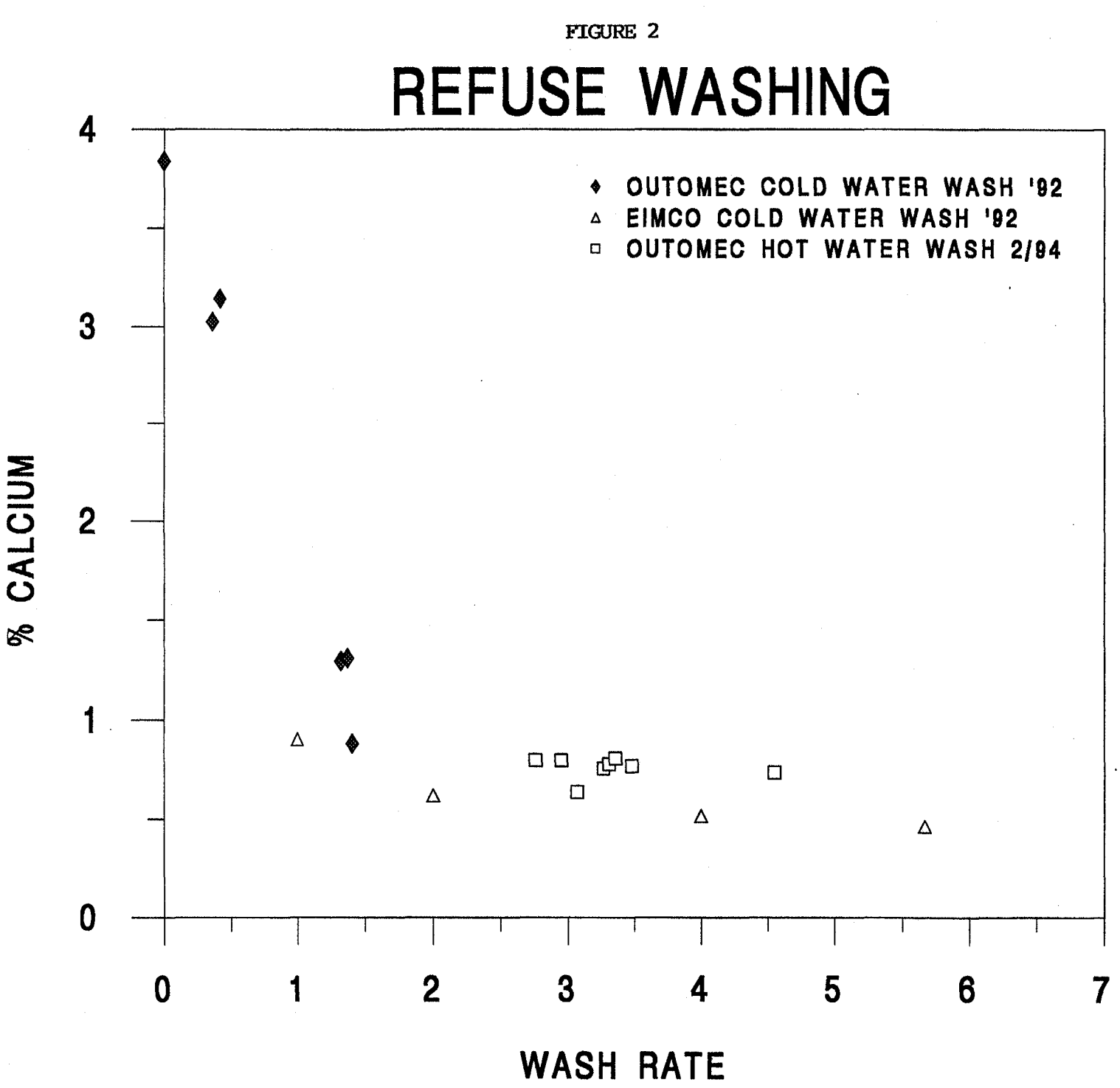

CLEAN COAL WASHING
OUTOMEC COLD WATER WASH '92
EIMCO COLD WATER WASH '92
OUTOMEC HOT WATER WASH 2/94
% CALCIUM
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
1
2
3
4
5
6
7
WASH RATE

FIGURE 2


REFUSE WASHING
OUTOMEC COLD WATER WASH '92
EIMCO COLD WATER WASH '92
OUTOMEC HOT WATER WASH 2/94
% CALCIUM
WASH RATE
0
1
2
3
4
5
6
7