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ANNUAL REPORT FY 1978

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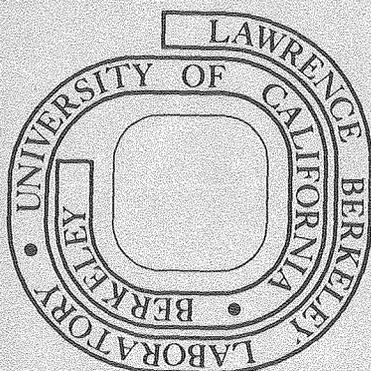
A. Levy, W. Lochmann, and I. Cornet

November 1978

Prepared for the U. S. Department of Energy  
under Contract W-7405-ENG-48

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COAL LIQUEFACTION ALLOY TEST PROGRAM

A. Levy, W. Lochmann, I Cornet

ANNUAL REPORT

FY 1978

Prepared for the U. S. Department of Energy  
FE-8807



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FOREWORD

This report covers work performed under Contract FE-8807 for the 1 August, 1977, to 30 September, 1978. The project was administered by the Fossil Energy Division of the U. S. Department of Energy with Wate Bakker as project manager. The report was prepared by Alan V. Levy of the Materials and Molecular Research Division of the Lawrence Berkeley Laboratory (LBL).

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ABSTRACT

The degradation of the surfaces of metals used in contact with non-aqueous liquid-solid particle slurries in coal liquefaction systems occurs by combined erosion-corrosion mechanisms. The resistance of commercial alloys to surface attack as a function of the environmental variables of slurry composition and flow is being investigated. Four different test devices, two of which recirculate the slurry resulting in some particle breakdown, and two which direct the solid particles at the specimens only once, are being used to determine material loss rates after exposures ranging from several minutes to several hundred hours. It has been determined in ambient temperature exposures that the erosion rates of mild steel specimens vary significantly with slurry viscosity, velocity, solids loading, impingement angle, Reynolds number of the flow, type and size of impacting particles, and composition of the liquid component of the slurry.

## INTRODUCTION

The objective of the program is to determine the erosion/corrosion behavior of materials used in the flow passages of liquid slurries under conditions representative of those in coal liquefaction systems. From the understanding gained from testing a number of different materials over a range of controlled operating conditions within and beyond those of currently acceptable operating practice, slurry flow operating parameter guidelines and improved performance materials selection and design criteria will be developed.

The program is being carried out by personnel from the Lawrence Berkeley Laboratory who are responsible for program management and for materials testing and analysis and from the Ralph M. Parsons Company who are responsible for slurry loop design and selection of loop operating conditions and who will contribute to behavior analyses. The Pittsburgh and Midway Coal Mining Co., SRC pilot plant at Tacoma, Washington, will also participate in the program as operators of a materials test side loop on a cooperative basis.

The program is structured to investigate the major variables inherent in the design and operation of non-aqueous liquid-solid particle slurry systems. These are:

1. flow passage geometry
2. materials of construction
3. slurry composition and properties
4. operating conditions.

A major consideration in the simulation of once-through solid particle-liquid slurry systems is the breakdown of the solid particles in

the slurry recirculated in the laboratory devices for economic reasons. A sequence of tests in recirculating and once-through test devices will be performed to determine the degree of particle comminution in recirculating slurry tests, the effect on the erosion-corrosion rates of test materials, and the correlation with erosion-corrosion rates in once-through slurry tests.

#### PROGRAM PLAN SUMMARY

The program will be conducted in six overlapping tasks. These are:

1. state-of-the-art determination
2. development and evaluation of test devices
3. determination of the effects of flow passage geometry on erosion-corrosion of materials
4. determination of the behavior of commercial and experimental materials
5. development of an understanding of erosion-corrosion mechanisms
6. establishment of system operating parameter guidelines and materials selection and design criteria.

#### Task 1 - State-of-the-Art Determination

A literature review has been conducted and visits made to current slurry test facility and coal liquefaction pilot plant operators. Concentration has been on non-aqueous slurry experience.

#### Task 2 - Development and Evaluation of Test Devices

Four different test systems have been designed and constructed. The erosion-corrosion data being generated from each in a series of coordinated

tests on common materials using the same test conditions will be compared. The effect of recirculation of the slurry on the size and configuration of the solid particles with time and operating conditions and the resulting effect on erosion of the test materials will be determined. These data will be compared with erosion rates of specimens exposed in slurry flows where an individual particle only impacts on the specimen once. Two test systems will operate in a recirculating mode and two systems in a once-through mode. In addition, the effect of test system size and the nature of the slurry flow against the test materials surface will be studied.

The solid particles used in the test slurries are pulverized coal bought to the same requirements and from the same source as that used at the SRC, Wilsonville, Alabama, pilot plant,  $\text{SiO}_2$ , and silicon carbide. Particle sizes range from sub-micron up to as large as 1/8" diameter with different size fractions being selected for individual tests. The  $\text{SiO}_2$  and SiC particles are used in limited tests where accelerated erosion is desired or less variation in particle geometry than occurs in coal is necessary to better define an observed behavior. The slurry liquids used are the SRC-Wilsonville pilot plant creosote oil starter solvent, water, kerosene and process solvent from the SRC-Tacoma pilot plant. This variety of liquids provides a large variation in viscosity and lubricity which are important variables in determining the relative erosion force of slurries at the initial, ambient temperature testing conditions.

The four test systems being operated are:

1. Two-liter slurry pot with circulating cylindrical specimens 1/8" diameter by 2" long suspended vertically in the pot at selected radii from

the central stirring shaft. This is a recirculating slurry mode.

2. Slurry flow from a 1/8" diameter nozzle, pressurized gas fed, impinging a jet stream on a flat surface specimen held at a fixed distance and angle from the nozzle. This is a once-through slurry mode.

3. A two-inch diameter pipe recirculating loop system with recirculating pump and metering valves capable of long-time operation utilizing an 800 gallon capacity tank of stirred slurry; capable of pumping slurry through all of the piping configurations used in a coal liquefaction system. This is a recirculating slurry mode.

4. A two-inch diameter pipe system operating as a materials test side loop at the Solvent Refined Coal (SRC)-Tacoma, Washington, pilot plant of the Pittsburgh and Midway Coal Mining Co. (P&M); utilizing elbow, tee, bend and straight pipe sections as well as various valve and pump configurations. This is a once-through slurry mode.

### Task 3 - Effects of Flow Passage Geometry

The erosion-corrosion of A53 mild steel, cast iron and type 304 stainless steel components as a function of passage geometry variations will be determined in this task. Both the recirculating slurry loop and the SRC side loop will be utilized for this purpose.

The operating conditions of slurry flow passages in the major coal liquefaction systems under development have been determined. Piping systems, pumps, valves, hydroclones, filters and other components were studied. Reports on process development operation have been studied and the analysis of plant operations by Ralph M. Parsons Company (RMP) used to define current and desired flow conditions and flow passages, configurations of

system components. Experience to date of erosion-corrosion of materials in plant operations has been analyzed.

The resulting bank of data was used to establish the slurry test loop design, select the slurry and define the loop operating conditions. Measurement techniques to determine loop operating conditions, condition of the slurry with time, and the degradation of containment surfaces will be investigated and the most appropriate instruments incorporated into the loop design.

Methods for handling quantities up to 800 gallons of solvent-pulverized coal at a time were established. Safety and environmental control practices have been developed for receiving, handling, circulating and disposing of the test slurries.

The test loop was designed so that various test components such as pipe elbows, loops, straight pipe run spools, pump valve, and particle separation device components could readily be removed for inspection and analysis-

The initial design operates at essentially ambient pressure and temperature. The capability for elevated temperature and pressure operation can be incorporated in subsequent years of the program with design changes for additions of insulation and elevated temperature pump, valve tankage and instrumentation components.

The first test series in the two-inch diameter slurry loop will determine the effect of varying slurry velocity, solids loading and viscosity on mild steel and 304 stainless steel components. The test duration is at least 400 hours. Material removal is determined during flow by ultrasonic thickness gauge determination.

In addition to varying the gross geometry of the piping components such as elbow radius, straight run length, and diameter transitions from the nominal 2 inches, discontinuities are being investigated such as weld joint mismatch, weld joint discrepancies, and circumferential grooves. The selection and dimensions of the discrepancies are selected to represent typical occurrences in actual plant construction.

Upon completion of each test the components are removed, cleaned and measured. Key components may be sectioned and their surface changes studied by light microscope and microscope and SEM. Grooving, corrosion, and other surface discontinuities are documented.

#### Task 4 - Behavior of Materials

The effect of material composition, structure, properties and surface condition on erosion-corrosion behavior will be determined. Slurry operating conditions will be varied and all four of the test devices will be utilized to determine behavior. Both commercial and experimental materials will be used. Weight changes, surface changes as measured by Tallysurf indicators, and microscope and microscope examinations will be made.

Three materials will be investigated in the initial group: A53 mild steel, 2 1/4Cr-1Mo steel, and type 304 stainless steel. Tests will be performed using different fluids and particles, flow velocities, particle loadings in the slurry, particle size distributions, exposure times and impingement angles. Subsequent material and test condition variables will be selected based upon the knowledge gained from the first test series. The effect of temperature on behavior is an important variable that will be

studied.

#### Task 5 - Erosion-Corrosion Mechanisms

The mechanisms of surface deformation and material loss by the impingement of solid particles in a slurry will be investigated in this task. The slurry pot tester, the 1/8" diameter nozzle jet impingement tester, and the two-inch diameter pipe recirculating loop will be used to determine on a macroscopic and microscopic level how material is removed as a function of impingement conditions. Pilot plant starter and process solvent will be used with and without solid particles to determine the role that corrosion plays in surface removal. The small radius elbow is the principal test specimen in the recirculating loop.

Weight measurements as a function of flow conditions and time will be determined for the smaller specimens in addition to the thickness change measurements. Metallographic examination using optical and SEM equipment will be conducted to observe the behavior of surfaces. An attempt will be made to model the observed mechanisms of material loss as a function of material properties and slurry variables.

#### Task 6 - Operating Guidelines and Materials Criteria

As data is obtained and analyzed, information will be prepared in formats that can be used by systems designers and materials developers. The interplay between slurry flow conditions and component geometry on erosion rates will be defined for combinations of these factors that are beyond the current state of the art used in the design of slurry systems. The effects of materials variables such as hardness, strength, microstruc-

ture and surface composition and condition on erosion rates will be defined.

#### PROGRESS SUMMARY

Progress on the program is summarized in Figure 1.

##### Task 1 - State-of-the-Art Determination

Completed.

##### Task 2 - Development and Evaluation of Test Devices

The slurry pot, jet impingement tester, and two-inch diameter pipe slurry loop tester have all been designed, constructed and are in operation. The data produced on all three devices appears to be satisfactory for conducting a program of investigation on liquid slurry erosion-corrosion behavior. Comminution of particles does occur in the recirculating slurry test devices, but the effects of particle change on erosion behavior are not excessive and can be accounted for in the analyses of data. The side loop to be installed by PAMCO at their SRC pilot plant has been designed and construction has not been completed.

##### Task 3 - Effects of Flow Passage Geometry

The two-inch diameter pipe slurry loop has been operated at ambient temperature and 40 fps velocity for 1000 hours and mild steel pipe component behavior has been observed. Maximum erosion, up to 0.007 inch, occurred after an 800-hour exposure on the sharp angle pipe elbow in the loop.

##### Task 4 - Behavior of Materials

Not initiated.

Task 5 - Erosion-Corrosion Mechanisms

Not initiated.

Task 6 - Operating Guidelines and Materials Criteria

Not initiated.

TECHNICAL PROGRESSTask 1 - State-of-the-Art Determination

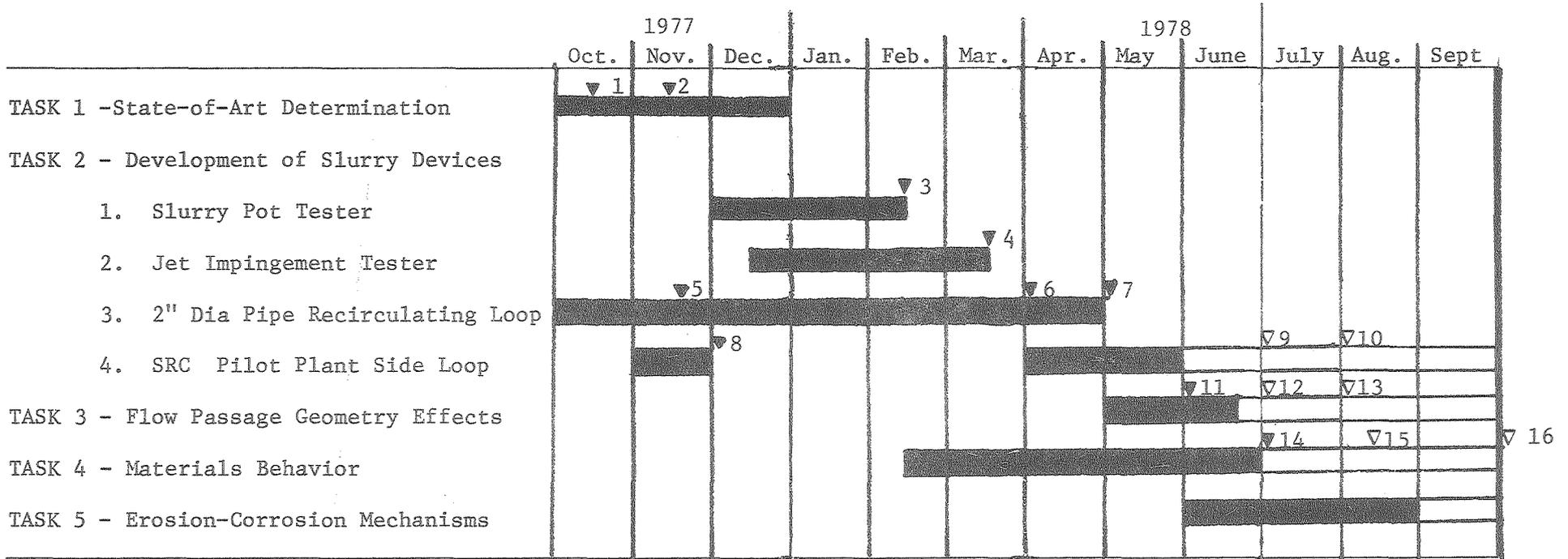
Task 1 has been completed. A review is contained in the first quarterly progress report. The reference list from the review is on pages            of this Annual Report.

Task 2 - Development and Evaluation of Test Devices

The four test devices that will be used in the program have been designed based upon their use with slurries of up to 50% by weight solids at room and elevated temperatures. The high viscosity of the coal-solvent slurries at room temperature was the determining factor in the selection and sizing of the methods of mixing and moving the slurries over the test specimen surfaces. The three laboratory devices are being operated at room temperature for their initial period of operation. Elevated temperature operation will be introduced in the slurry pot and jet impingement testers in the next year of operation. Elevated temperature operation will be incorporated into the recirculating test loop in its second year of operation. The SRC pilot plant side loop will operate at the temperature of the slurry at the point of takeoff from the main stream.

FIGURE 1.

FY 1978 PROJECT SCHEDULE



MILESTONES:

- |   |   |
|---|---|
| 1. Plant Visits Completed                                 | 9. SRC Plant Side Loop Construction Completed |
| 2. Initial Literature Review Completed                    | 10. SRC Plant Side Loop Operational           |
| 3. Slurry Pot Tester Operational                          | 11. Test 1 Completed                          |
| 4. Jet Impingement Tester Operational                     | 12. Test 2 Completed                          |
| 5. 2" dia. Pipe Recirculating Loop Design Completed       | 13. Test 3 Completed                          |
| 6. 2" dia. Pipe Recirculating Loop Construction Completed | 14. A53 Mild Steel Evaluated                  |
| 7. 2" dia. Pipe Recirculating Loop Operational            | 15. 2 1/4 Cr-1Mo Steel Evaluated              |
| 8. SRC Plant Side Loop Design Completed                   | 16. 304 Stainless Steel Evaluated             |

## 1. Viscosity Tests

Measurements have been made of the kinematic viscosity of the slurries that will be used in the slurry pot, jet impingement tester, and two-inch diameter slurry loop test devices. A rotating concentric cylinder viscosimeter (Contrares model 15T) was used. The tests were made on slurries of creosote oil plus 0, 10, 30, 35, 40, 45 and 50 % by weight of ground coal of the type used in the Wilsonville SRC pilot plant (-200 mesh) at 5°, 22°, 53° and 80° C, and on slurries of kerosene plus 0 and 10 % by weight of ground coal at the same temperature. The weight per cent of solids in the slurry is calculated by dividing the weight of the solid particles by the total weight of the slurry (particles plus liquid).

The viscosities of the slurries were found to be quite sensitive to changes in temperature, the sensitivity being enhanced by the increasing concentration of solid particles. Temperature was controlled by means of a water bath. It was observed that there was a strong tendency for the coal particles to coagulate resulting in inhomogeneity of the slurry and difficulty in generating repeatable viscosity measurements at high particle concentrations and low temperatures.

The viscosity data obtained for kerosene agrees quite well with values from a standard handbook indicating the basic validity of the test procedure used. The rheograms, at 22°C, show a slight dilatant behavior of the coal slurries (Figures 2 and 3) at the lower concentrations and a pseudoplastic behavior for the higher concentrations. Dilatant behavior, where viscosity increases with increasing shear stress, is generally not encountered in solid particle slurries.

The viscosity versus particle concentration curves for coal-kerosene

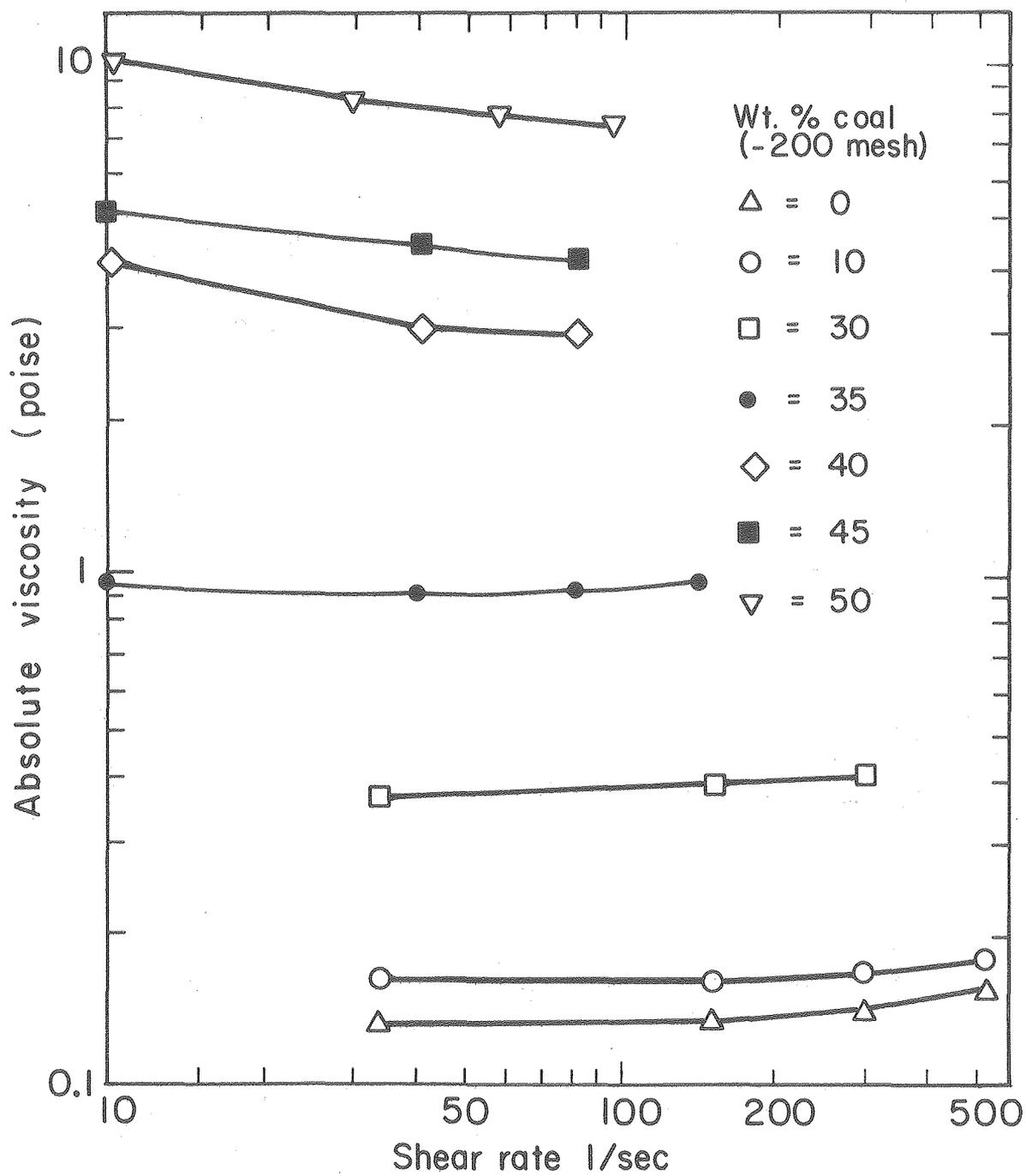


Figure 2. Viscosity vs shear rate at different concentrations for coal + creosote oil at room temperature ( $\sim 22^\circ \text{C}$ ).

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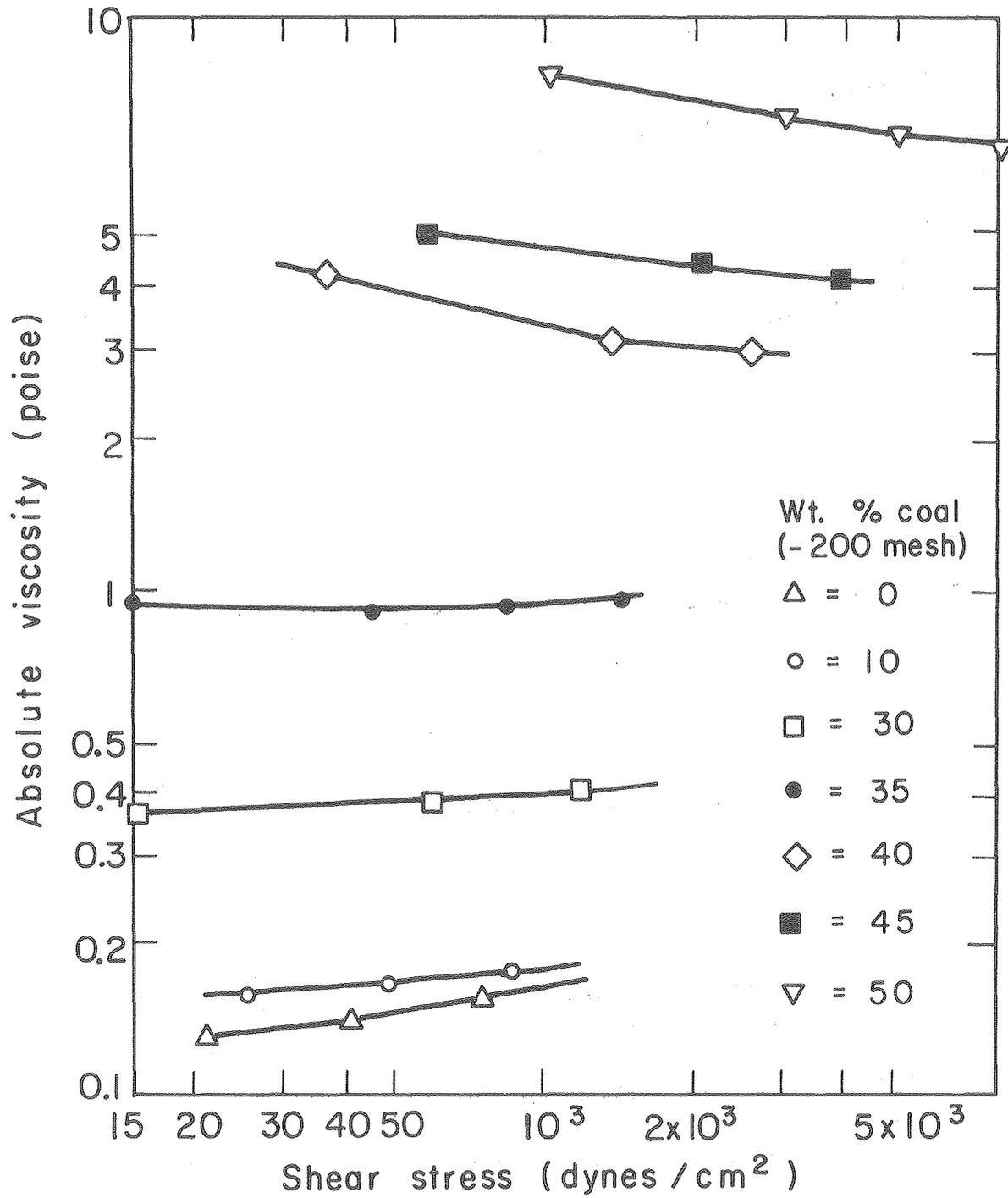


Figure 3. Viscosity vs shear stress at different concentrations for coal + creosote oil at room temperature ( $\sim 22^\circ \text{C}$ )

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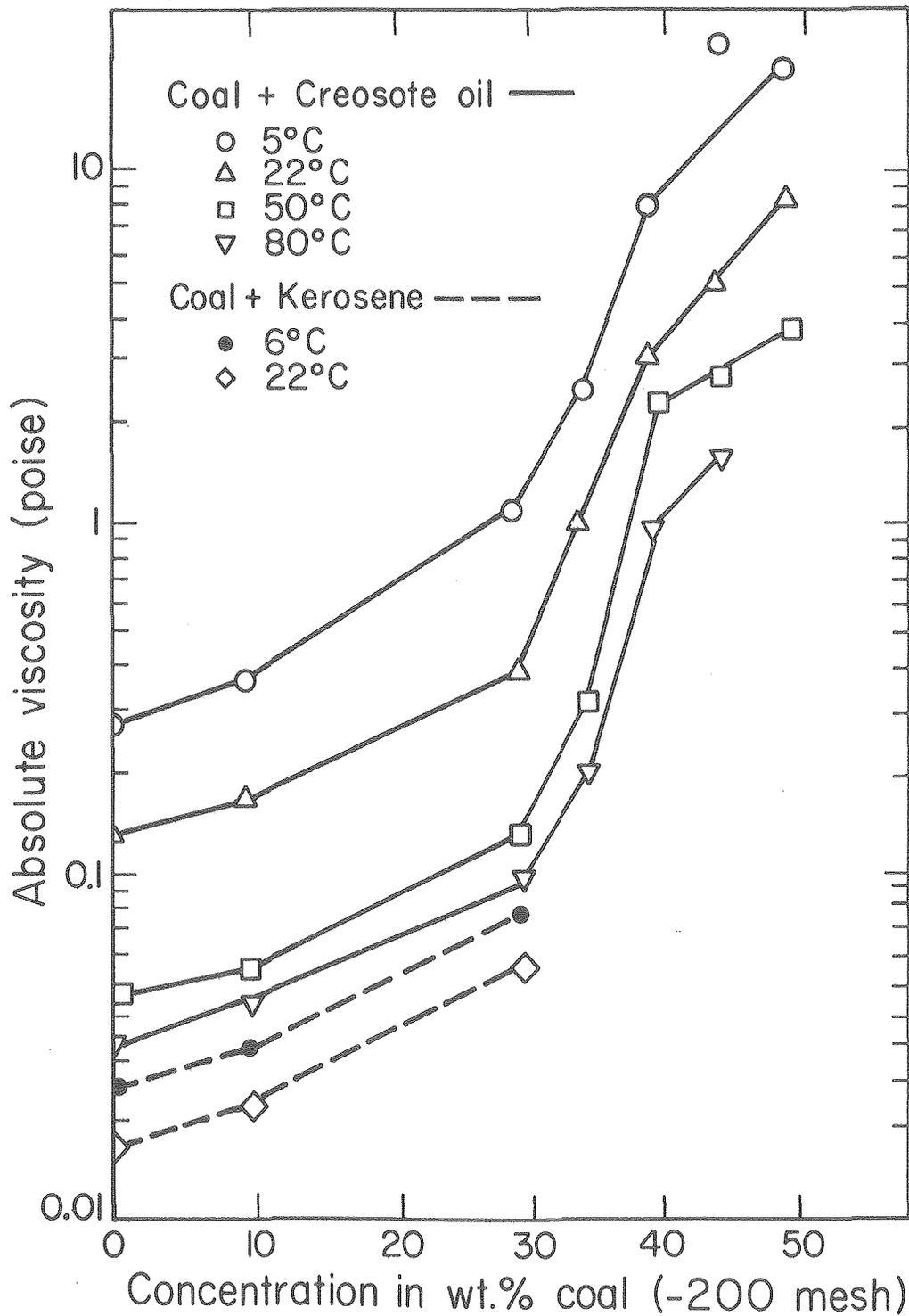


Figure 4. Viscosity vs concentration at different temperatures and fixed shear rate of 100 (1/sec).

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and coal-creosote oil (Figure 4) show that the increase in viscosity with solids concentration is dramatic. The increase is most pronounced between 30 and 40 wt % coal concentration. The increase from 40 to 50 wt % coal shows a reduction in the increase compared to that between 30 and 40 wt %, but a greater increase than occurred at the lower solids loadings. There was significant settling of solids and coagulation during the measurements at 5°C for the 50 wt % concentration. The final direction of the curve above 40 wt % concentration must still be determined by further testing. The viscosity increases about fifty-fold as the solids concentration increases from zero to 50 wt % as shown in Figures 4 and 5. In the viscosity-temperature plots of Figure 5 a and b, the shape of the curves seem to be unaltered when the solid is added to the liquid. The only effect is to shift the entire curve upward, to greater viscosities. This indicates that the variation in viscosity with temperature in the range studied is due entirely to the liquid phase of the slurry. Curve 5a is expressed in poises while curve 5b is plotted on an ASTM standard viscosity-temperature chart for liquid petroleum products (D 341-39).

## 2. Slurry Pot Tester

The problem of erosion-corrosion in a slurry pipeline can be studied in the laboratory by exposing the specimens of the same material as the pipeline material to the slurry by rotating them on arms around a shaft connected to a vari-drive motor in a pot of slurry. Figures 6, 7 and 8 show the pot, baffles, rotation system and the complete assembly in its first version. Erosion-corrosion effects are measured by weight loss and cross sectional change.

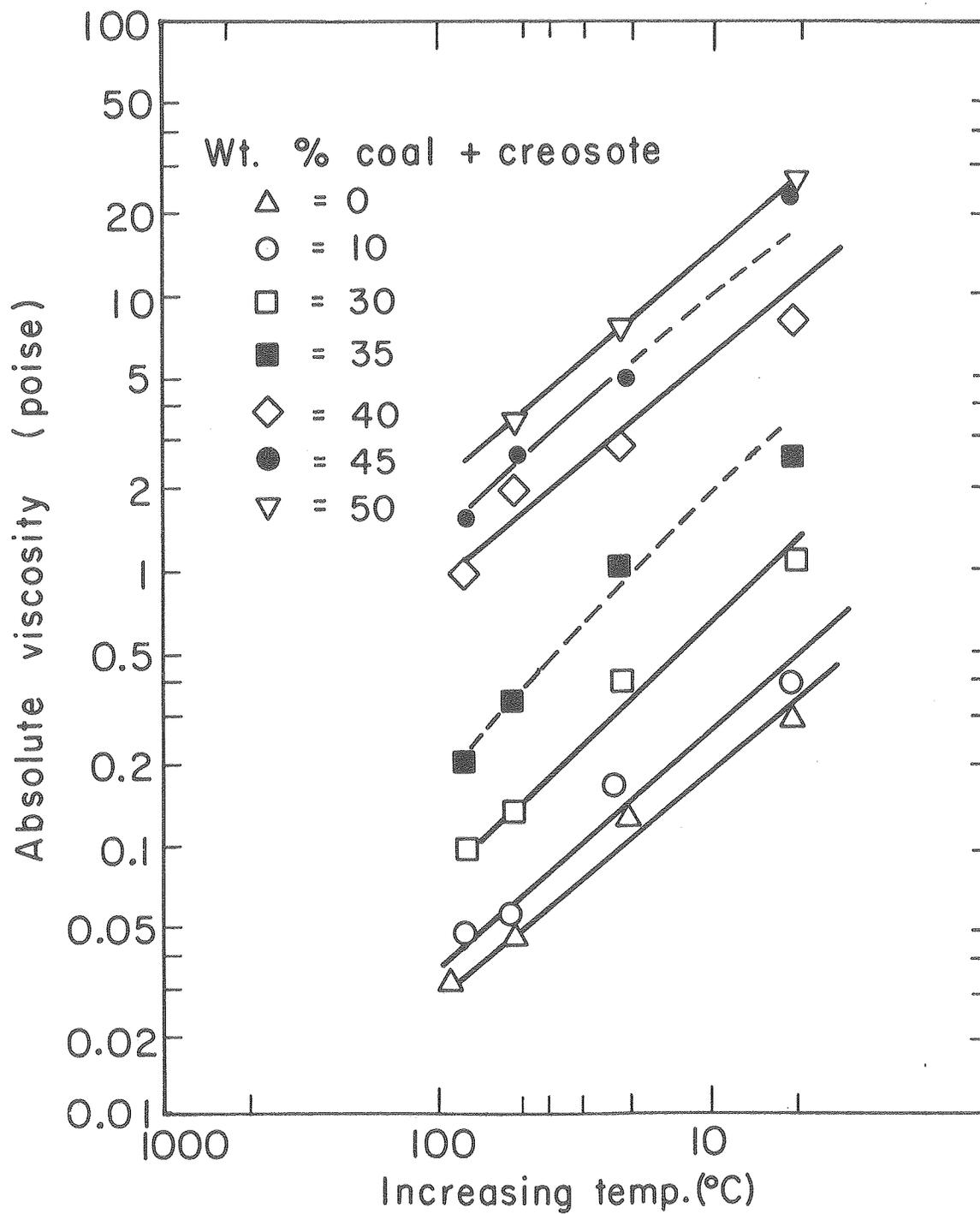
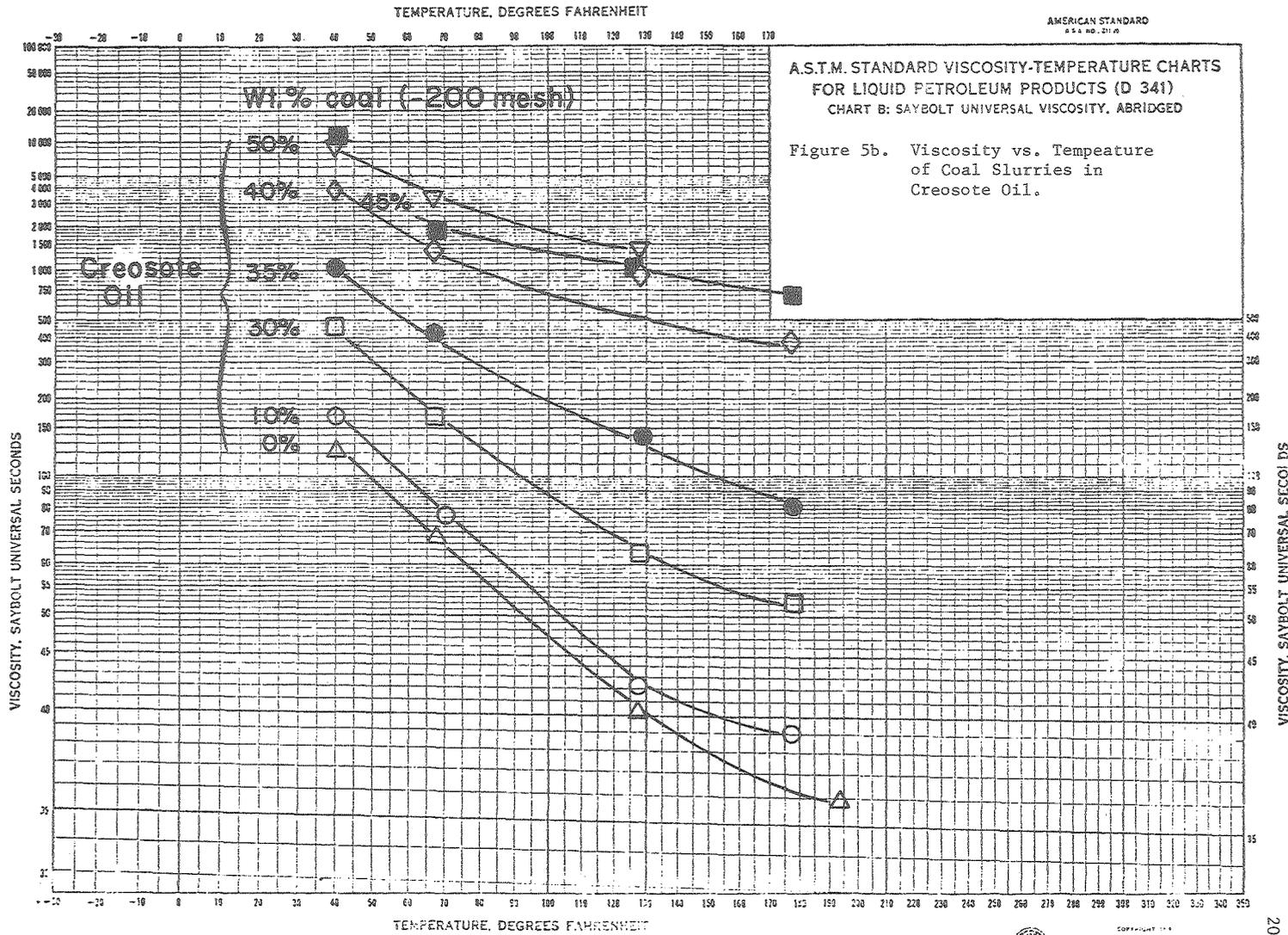


Figure 5a Viscosity vs temperature at different concentrations for coal + creosote oil at fixed shear rate of 100 (1/sec).

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## SLURRY POT TEST DEVICE

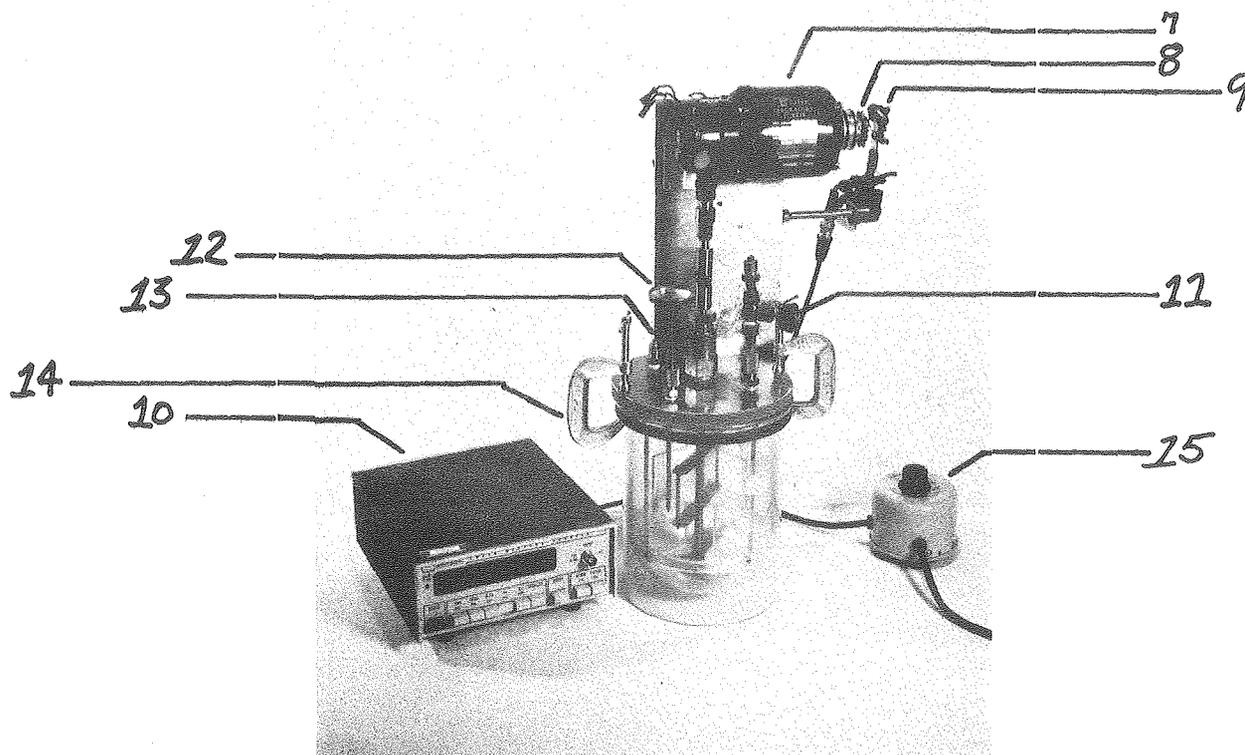


FIGURE 6

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1. 200 ml Pyrex Pot (Fig. 2-A)
2. Pot Stand (Fig. 2-A)
3. Baffles (Fig. 2-A)
4. Propeller (Fig. 2-B)
5. Sample (Fig. 2-B)
6. Pin to hold Sample (Fig. 2-B)
7. Vari-drive Motor (1/18 hp.)
8. 10-tooth Gear
9. Magnetic Pick-up
10. Digital Counter
11. Outlet Valve
12. Thermometer
13. Nitrogen Inlet
14. Clamp
15. RPM Regulator

SLURRY POT TEST DEVICE

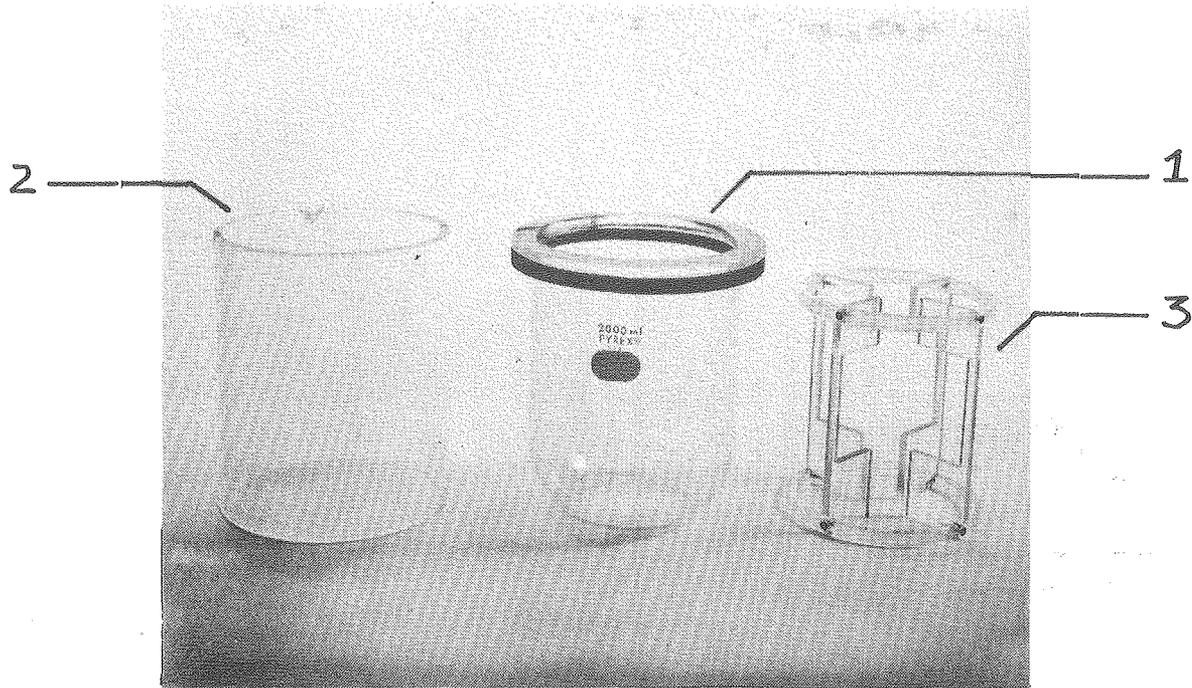


FIGURE 7

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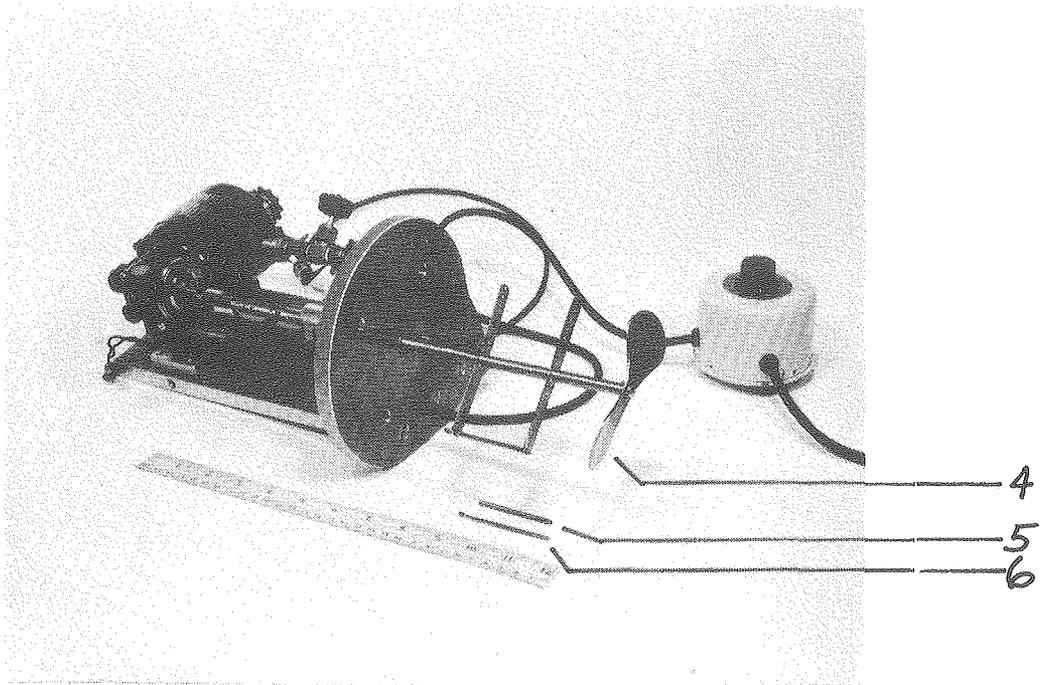


FIGURE 8

XBB 782-2133

Variables of the experiments are: Particles (coal, SiC, SiO<sub>2</sub>), liquid (water, kerosene, creosote oil, SRC process solvent), solids concentration (0 - 50 wt %), particle size (-200 mesh for coal, -60/+70 mesh for SiC), flow velocity (10 - 30 fps), time (2 - 40 hours), and temperature (room temperature to 400°F). The maximum temperature that can be reached is limited by solvent flash point and the maximum velocity that can be imposed is limited by interfering effects of cavitation which will make considerable error at high velocities. The minimum velocity is limited to the minimum velocity which keeps the particles in suspension during an experiment.

A rubber seal (Wilson seal) is provided around the shaft at the pot lid in order to prevent slurry contained in the pot from being exposed to air, rather the slurry is exposed to nitrogen gas, the pressure of which is kept slightly higher than the atmosphere in order to prevent air from getting into the pot.

The side arms on the shaft consist of two parallel thin sheets 1/4" wide which hold vertically oriented samples in between them by means of pins in different locations. The edges of the sheets are well-rounded so that they make a minimum disturbance in the slurry. Four baffles 90° apart are placed inside the pot in order to prevent a vortex while the shaft is rotating. A thermometer is mounted in the lid which measures the slurry temperature. In order to measure the rpm of the shaft a ten-tooth gear is mounted on the shaft which is rotating in front of a magnetic pickup connected to a digital counter. The digital counter indicates the shaft rpm to a high accuracy (+ 1 count error).

Samples are made of 1/8" o.d. by 2" long tubes of various materials

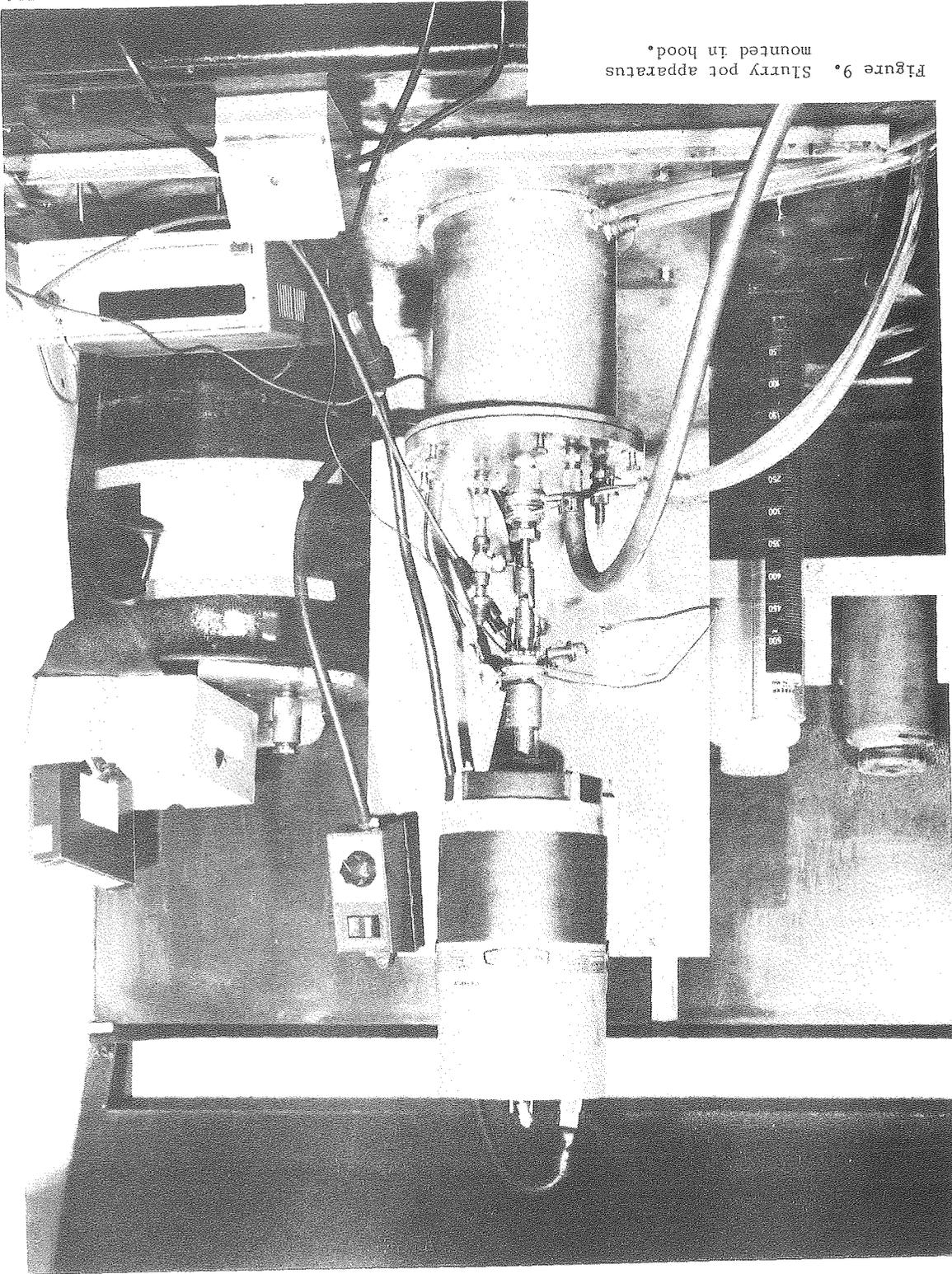
and are attached to the side arms of the main shaft. In order to have a balanced shaft it is necessary that symmetry of sample location on the shaft be achieved. Velocity is determined by the distance out from the shaft that the specimens are mounted. Erosion is obtained by measuring the weight loss of each sample. A stirrer is attached to the shaft to fluidize coal particles at various shaft speeds.

The slurry pot tester has been installed in a hood and is operating with SRC-Tacoma process solvent and pulverized coal of -200 mesh particle size, as well as with other slurries made up of water, creosote oil and SiC in addition to the SRC process solvent and coal in all combinations. A new, larger size motor has been mounted in order to overcome difficulties resulting from stirring 50 wt % coal plus SRC process solvent. The new motor is 3/4 hp and is equipped with a variable speed regulator. The plastic baffles have been replaced by stainless steel baffles in order to increase the strength to resist the forces generated in mixing the slurry. The glass container has been replaced with a steel container. Figure 9 shows the revised slurry pot apparatus mounted in the hood.

Initial experiments with the slurry pot tester containing coal plus SRC process solvent generated an irregular increase in the temperature of the slurry in a relatively short period of time. The temperature increase is due to dissipation of frictional heat because of the relative high viscosity of the moving slurry. A cooling system was installed which consisted of running cold water around the pot tester. This has proved to be effective in keeping the temperature constant during the test period. The bimetallic strip thermometer initially mounted on the slurry pot tester has been replaced by a thermocouple in order to avoid errors made by the stirring motor vibrations.

BBC 789-10554

Figure 9. Slurry pot apparatus mounted in hood.



Early experiments with 23 wt %, -200 mesh coal plus SRC process solvent did not show any weight loss for the 1/8" o.d., 2" long, mild steel specimens after 20 hours of testing at 30 fps velocity at room temperature. In order to evaluate the ability of the slurry pot to generate measurable erosion in a reasonable time period, several experiments were run incorporating particles more abrasive than coal in different base fluids with different viscosities. These "accelerated" tests which have the same dynamic characteristics as the original experiments with coal and SRC process solvent are performed at room temperature. They have the advantage that the erosion mechanism can be analyzed more readily with respect to governing conditions and erosion rates can be correlated to such different variables as viscosity of the fluid, particle size distribution and hardness as well as solids concentration, velocity and the geometry of the eroded metal, all in a relatively short laboratory time scale. Another advantage of performing tests with solids more abrasive than -200 mesh coal particles is to show the capability and the consistency of performance of the slurry pot tester when an abrasive medium is incorporated. Silicon carbide particles -60/+70 mesh size were used in water and SRC process solvent.

Comparing the results of the SiC plus water and SiC plus SRC process solvent show the significance of the viscosity of the fluid on the erosion rate, presumably because of the effect of the boundary layer around the specimens. Similarly, the experiments with coal and water and SiC and water emphasize the effect of particle size and hardness on the erosion rate. Some of these tests have been performed at different velocities in order to analyze the velocity effects, with the solid particle

concentration being kept fixed at 30 wt %. Solids concentration effects will be considered in future work.

The slurry pot tester appears to be an effective and useful device for studying the mechanism of erosion in slurries.

#### Slurry Pot Tester - Results

Early experiments with thin-walled tube specimens resulted in pounding and deformation of the specimens instead of erosion. Tube wall thickness was then increased. All of the experiments to date have been performed at room temperature (25°C). The relative flow of the slurry is perpendicular to the long axis of the rotating tubes. Experiments with 33 wt % coal plus SRC process solvent did not give any erosion that could be weighed. A measure of erosion capability of coal plus SRC process solvent could be obtained by using much longer runs, by resorting to Tallysurf meter rather than weight measurements, by increasing velocity, or by changing the test specimen material. An alternative to the long-time tests required with coal is to use a more erosive particle such as SiC or SiO<sub>2</sub> and a less viscous fluid in shorter time tests to determine erosion mechanisms. It is necessary to understand erosive behavior before introducing elevated temperature corrosion behavior into the slurry pot test series in order to be able to differentiate the two mechanisms in the subsequent combined erosion/corrosion tests.

In order to study the mechanism of erosion of materials in non-aqueous slurries using relatively short time tests, it was decided to run some of the tests with fluids other than SRC process solvent and solid particles other than coal. Comparing the results of the 23 wt % SiC plus water and the 23 wt % SiC plus SRC process solvent showed the major effect of changing viscosity of the fluid component of the slurry on erosion rates. Figure 10 plots the cumulative erosion vs time for water and SRC process solvent slur-

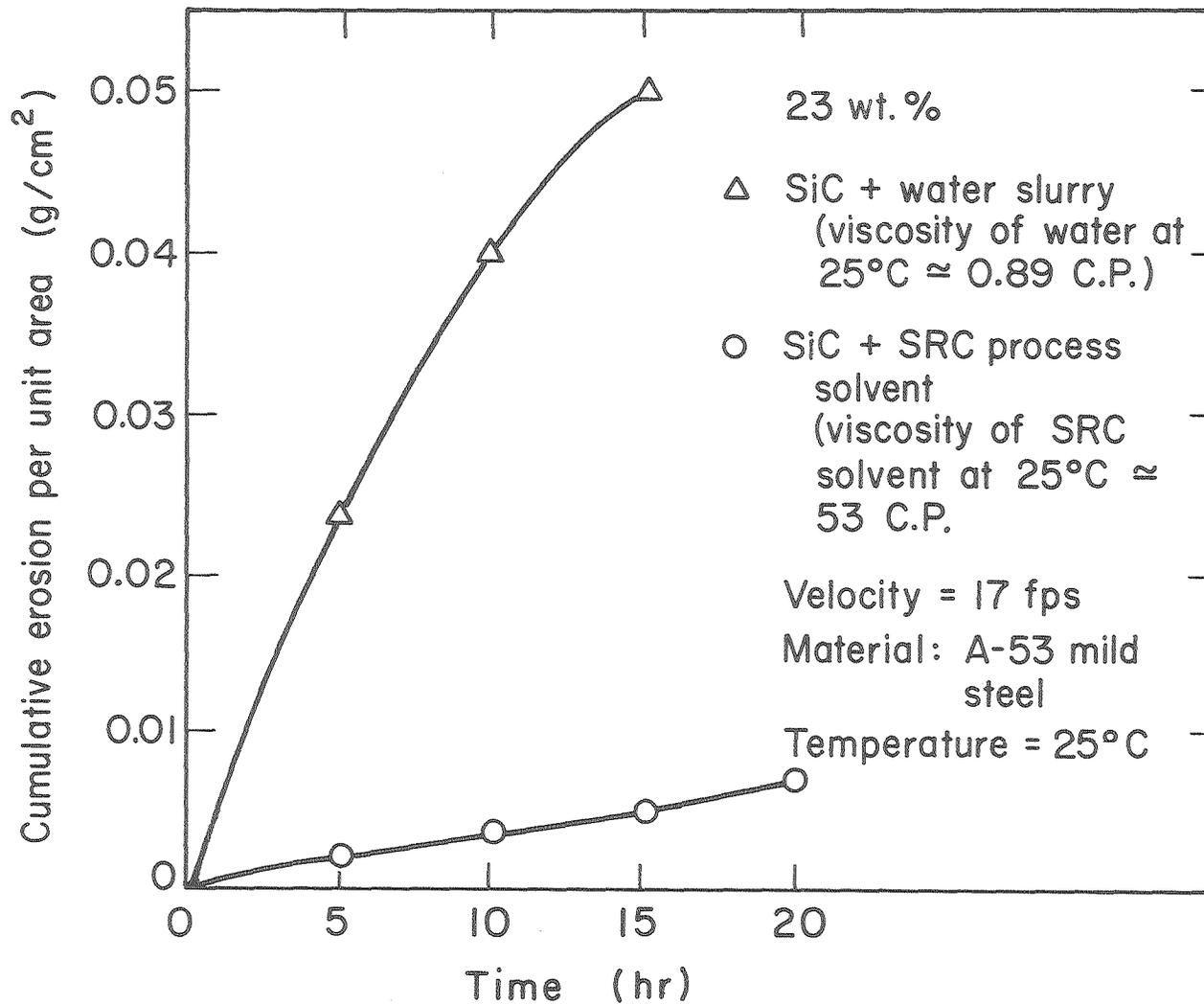


Figure 10. Effect of change in viscosity on erosion of 2" long 1/8" o.d. A-53 mild steel specimens.

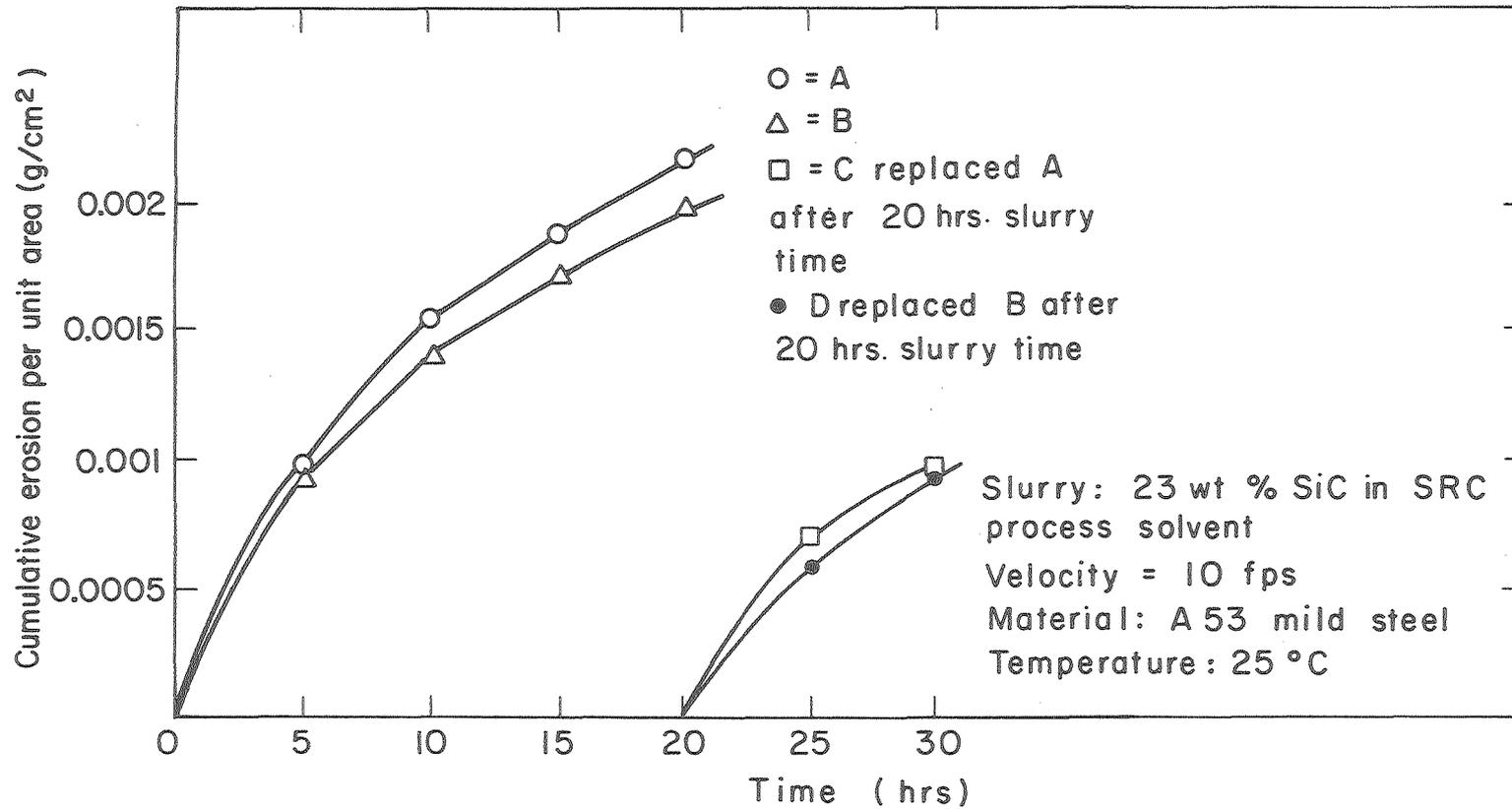
XBL 789 - 1773

ries and shows the effect of the change of viscosity of the fluid on the erosion by the solid particles. The viscosity of the carrier fluid has been changed from the SRC process solvent viscosity at room temperature of  $\approx 53$  cp to the water viscosity at room temperature of  $\approx 0.89$  cp. Reducing the fluid's viscosity from 53 cp to 0.89 cp appears to have increased the erosion rate more than ten times. The possible contribution of the corrosive ability of the water to the increased material loss in the water slurry must also be considered.

Viscosity affects the boundary layer around the solids. In some circumstances, the effect of boundary layer is to deflect solid particles and change the impingement angle as well as the impact velocity. This is particularly important in erosion analysis in slurry systems. Boundary layer effects bring up the dependency on geometry of the metal under attack. This kind of analysis becomes especially important at critical points like bends, tees, valves, and places of changing cross section. The linear nature of the SRC process solvent slurry curve in Figure 10, compared to the water slurry curve, is another indication of the effect of viscosity on the slurry behavior that will be investigated later.

Figures 11 and 12 give the results of tests with 23 wt % SiC particles in SRC process solvent and show the cumulative erosion versus time at 10 fps and 17 fps at room temperature. All the specimens show a decrease in erosion rate versus time. This decrease is due to particle breakdown and/or change in the geometry of the test specimens because of erosion. Change in the slopes of the cumulative erosion-time curves A and B with time gives an indication of particle breakdown after 20 hours in the SRC process solvent.

In order to verify the particle breakdown after 20 hours of testing, new cylindrical specimens replaced the old specimens in the used slurry and



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Figure 11. Erosion of 2" long, 1/8" o.d. A-53 mild steel tubular specimens.

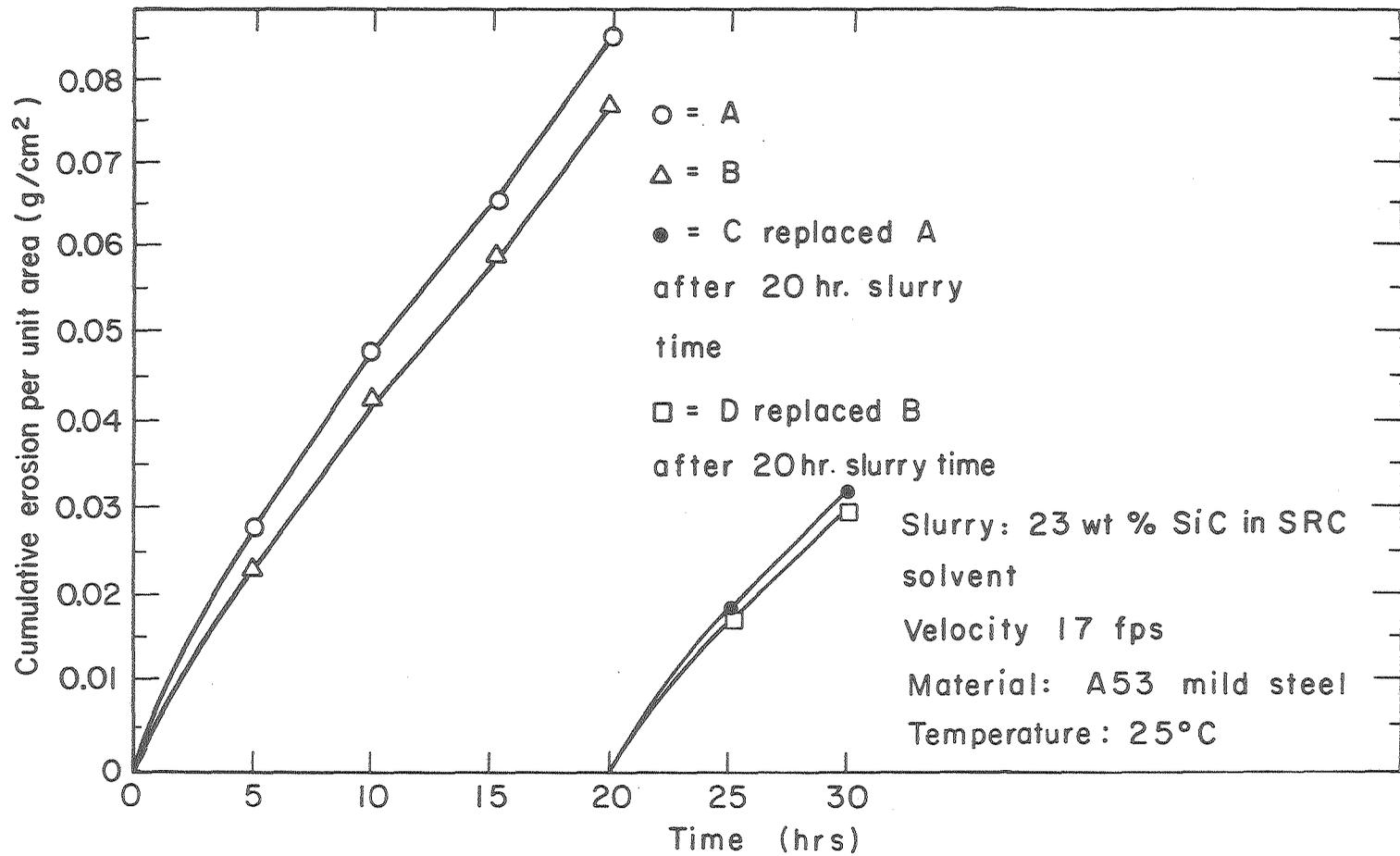
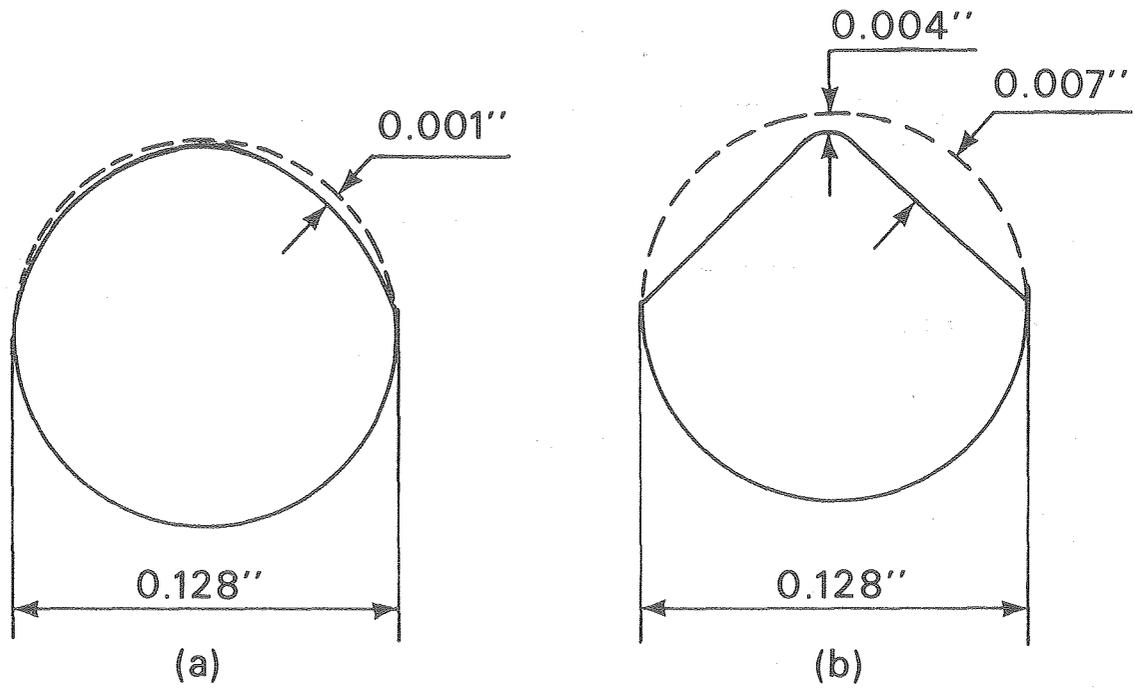


Figure 12. Erosion of 2" long, 1/8" o.d. A-53 mild steel tubular specimens.

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running continued for 10 additional hours. In Figures 11 and 12, comparing the slopes of curves A with C and B with D at the origin shows that there has been substantial particle breakdown, comminution and/or rounding of corners and edges, since the slurry which has been used for 20 hours is less erosive to a freshly exposed test specimen than the fresh slurry was. Comparing the instantaneous slopes of the curves for samples A and B at 20 hours with the instantaneous slopes of curves for samples C and D indicates that changes in erosion rate are not solely due to comminution of the SiC particles. The slurry condition is the same at the end of curves A and B and the beginning of curves C and D, yet the slopes are different. The rate differences observed are due to the different specimen geometry of the already eroded specimens A and B compared to the new specimens C and D, and/or to work hardening of the specimen surfaces of A and B due to erosion. For the system SiC plus water, shown in Figure 10, a measurable change in specimen geometry was observable after five hours erosion. Figure 13 shows the cross section of the SiC plus SRC process solvent and the SiC plus water specimens.

Figure 14 shows an interrupted test series for a 30 wt % -200 mesh coal in water slurry. The curves again show by the marked differences in their initial slopes the effect of the comminution of the particles as occurred in the SiC tests, Figures 11 and 12. Because coal is softer, less angular and breaks down easier than SiC, it is not as efficient an erodent. The erosion rate is several times less than that for SiC in water, Figure 10. The slope of the curve for the new specimen, B, in Figure 14, compared to that of the original specimen which continued to run in the same test (unlike the SiC solvent tests where both specimens



Cylindrical samples cross sections after:

- a) 20 hours in 23% SiC + SRC process solvent at the velocity of 17 fps.
- b) 4 hours and 20 minutes in 23% SiC + deaerated water at the velocity of 20 fps.

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Figure 13. Cross sections of o.d. of tubular specimens after exposure in slurry pot tester.

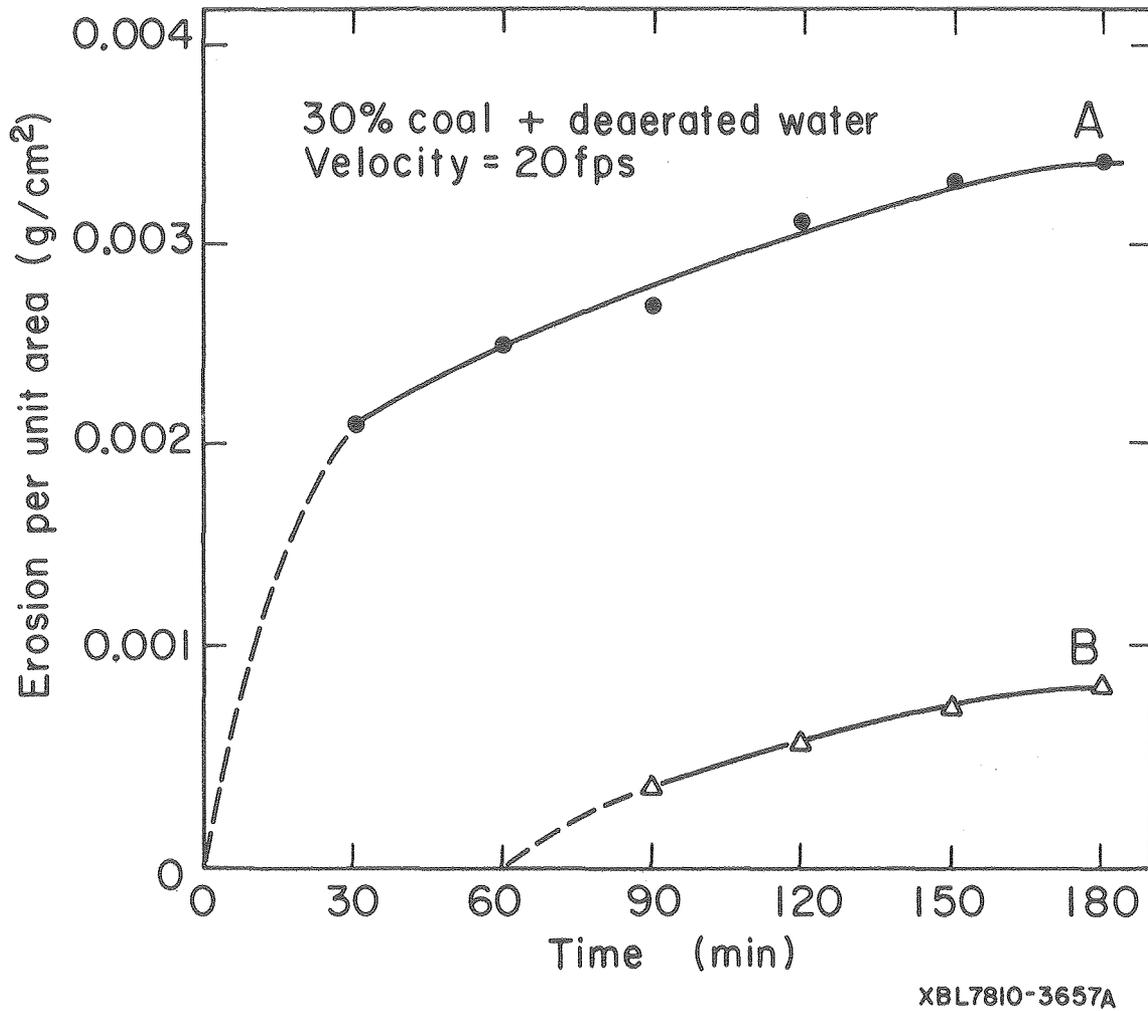


Figure 14. Erosion of 2" long, 1/8" o.d. A-53 mild steel tubular specimens. 30 wt. % coal plus deaerated water. Note that B replaced A after 60 minutes exposure time.

were replaced at 20 hours exposure) is very nearly the same. This indicates that deal did not cause as severe a change in the geometry of the specimen as SiC did.

Figure 15 shows the effect of velocity on the cumulative erosion. It can be seen that doubling the velocity changes the erosion by only 25 - 30 %. Also all three velocity curves have the same slopes at any point on the curve indicating the same effect of particle comminution on erosion over the range of velocities tested. Figure 16 shows how increasing velocity increases the erosion rate.

Figure 17 plots the erosion rate for a total of 30 minutes of testing with the change in Reynolds number due to changing the velocity. It can be seen that the erosion rate increases with increasing Reynolds number, indicating that the fluid flow field around the specimen has a significant effect on erosion behavior.

The initial test series performed in the slurry pot apparatus was intended to establish the nature of the device for studying slurry erosion-corrosion. The data obtained to date demonstrates the suitability of the apparatus for the intended purpose. The magnitude of the changes in erosion behavior as a function of the variations in the test conditions are adequate for carrying out more extensive investigations in Tasks 4 and 5 of how and to what degree alloys and their protective coatings can resist surface degradation from non-aqueous slurries. The section on future work (page 69 of this Annual Report) will define in some detail the use that will be made of the slurry pot tester in the coming year.

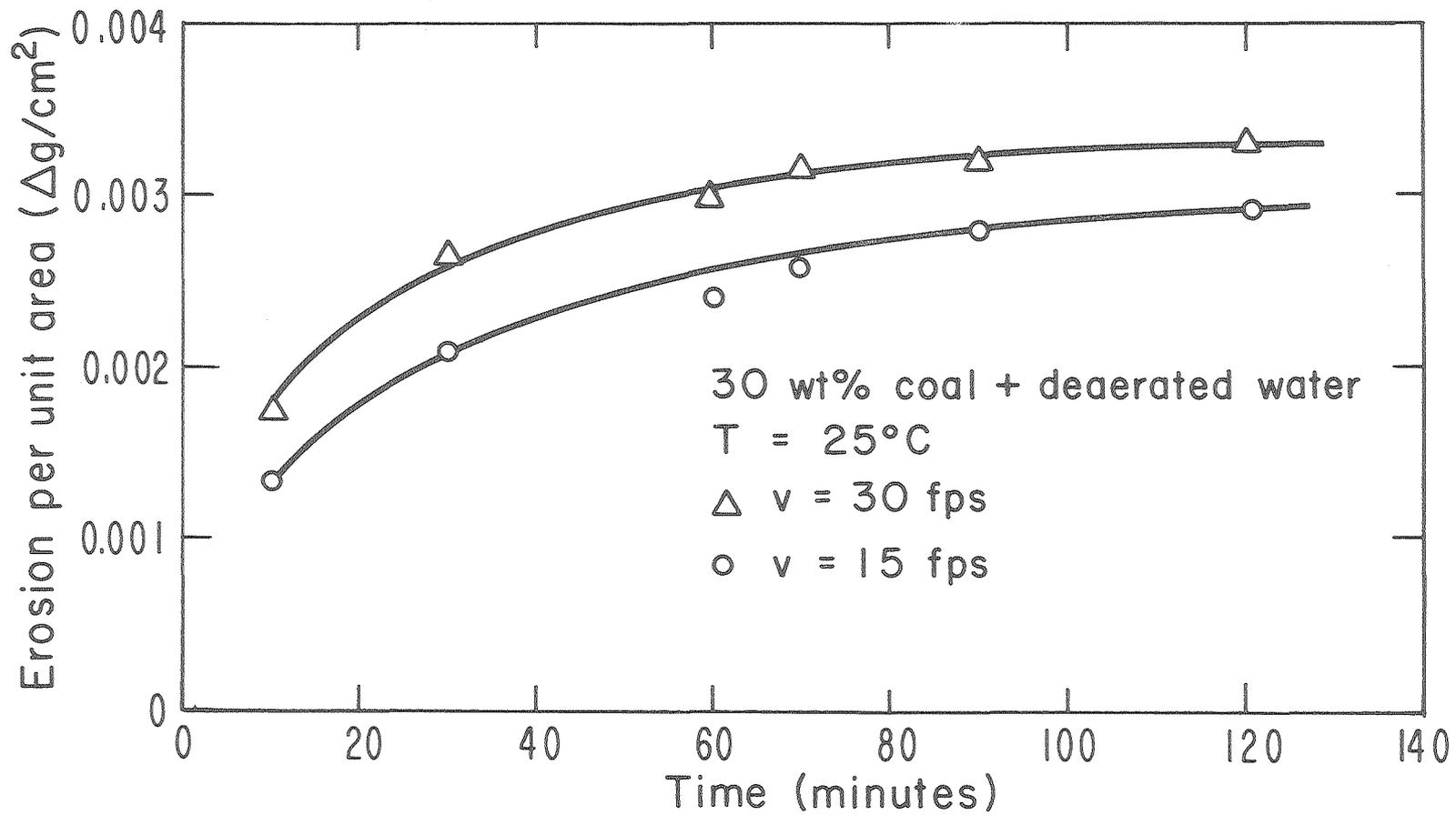
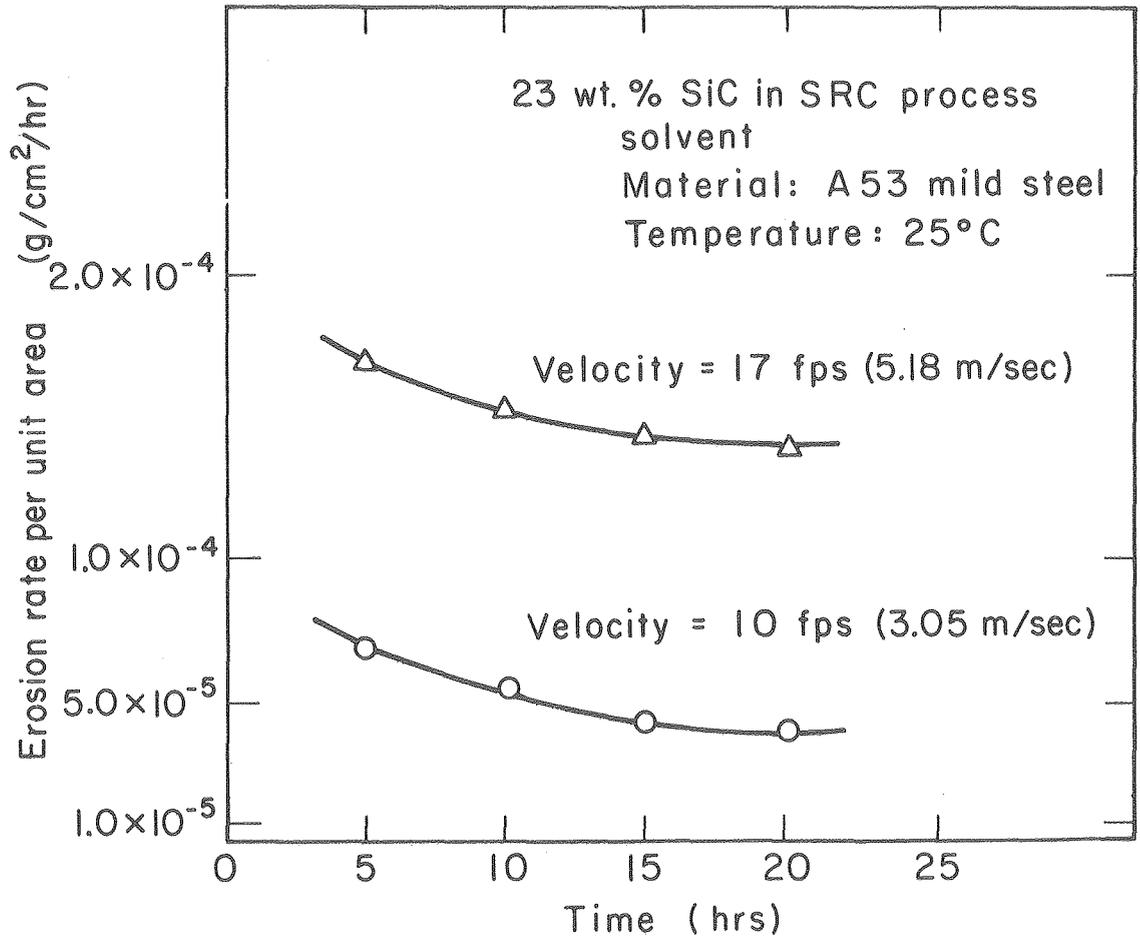


Figure 15. Effect of velocity on erosion of A-53 mild steel tubular specimens.

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Figure 16. Effect of change in velocity on erosion rate of 2" long, 1/8" o.d. A-53 mild steel tubular specimens.

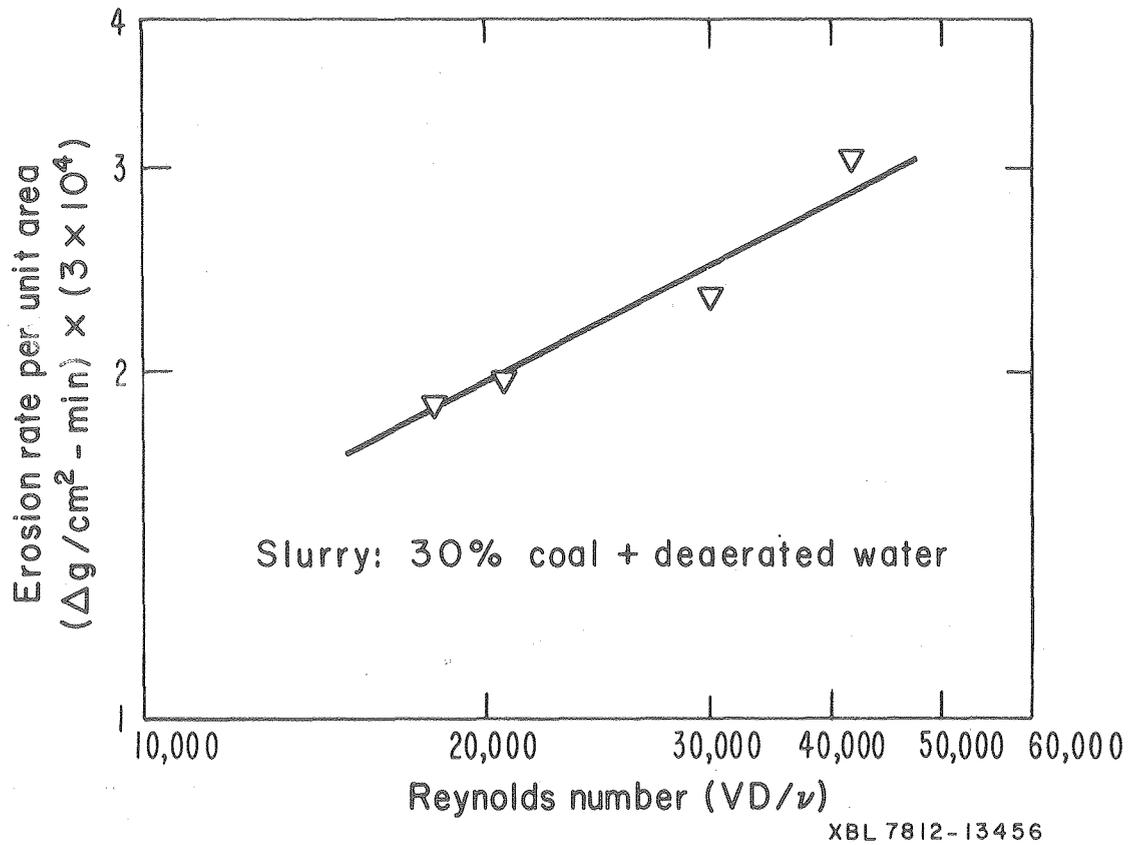


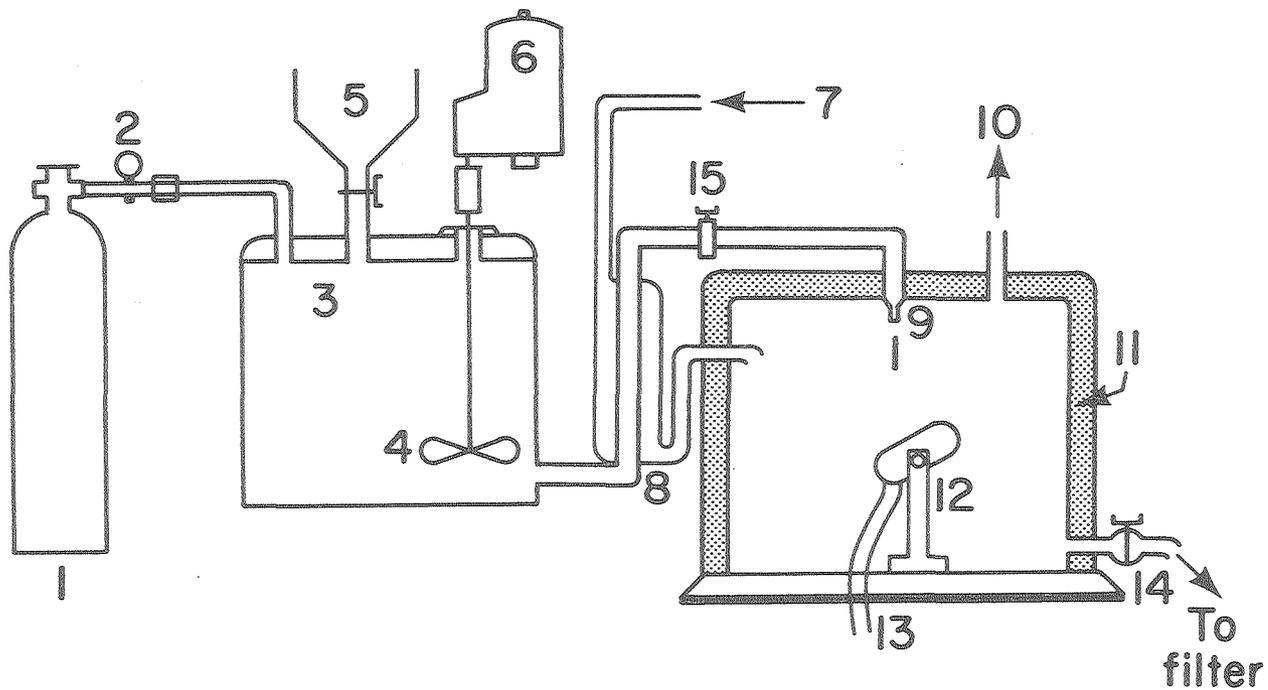
Figure 17. Erosion rate of A-53 mild steel tubular specimens as a function of Reynolds number in the slurry pot tester.

### 3. Jet Impingement Tester

In order to investigate the impingement on a metal specimen by a coal-solvent oil slurry without the complication of coal particle breakdown, it is desirable to have a stream of the slurry impacting on the specimen without driving the coal particles against the specimen more than once in order to prevent comminution of the particles. The velocity must be in the desirable range and the angle of impingement of the particle on the specimen should be controlled. Also, since this is a once-through test for the slurry in a laboratory operation, the amount of materials involved must be small enough to be practical and the test duration reasonably short.

An apparatus has been constructed that produces a small jet stream driven to sufficiently high velocity by pressurized gas to impinge on the surface of a flat metal specimen with the desired velocity. Figure 18 is a schematic of the test apparatus and Figure 19 is a photograph of it.

A slurry pre-mixed to the desired composition is put into the main tank, which is then closed and pressurized with air or nitrogen from a pressurized bottle supply. The particles are kept in total suspension by a stirrer. When the test begins the slurry is forced through the piping under a driving pressure of air or nitrogen through a stainless steel nozzle with a 1/8" diameter opening to impinge on a specimen supported on a rigid mounting. For tests at elevated temperatures, the slurry will be preheated before reaching the nozzle and the test specimen heated as well. The initial test series was carried out at ambient temperature.



## DIRECT IMPINGEMENT TEST SET-UP

FIGURE 18 Components

XBL 783-452

- |                         |                         |                              |
|-------------------------|-------------------------|------------------------------|
| 1. Propellant Gas       | 6. Stirring Motor       | 11. Insulation               |
| 2. Pressure Gauge       | 7. Heating Gas Inlet    | 12. Rotatable Specimen Mount |
| 3. Pre-Mix Slurry       | 8. Pre-Heating Exchange | 13. Thermocouple             |
| 4. Stirrer              | 9. Nozzle               | 14. Drain                    |
| 5. Slurry Re-Fill Inlet | 10. Gas Exhaust         | 15. Secondary Valve          |

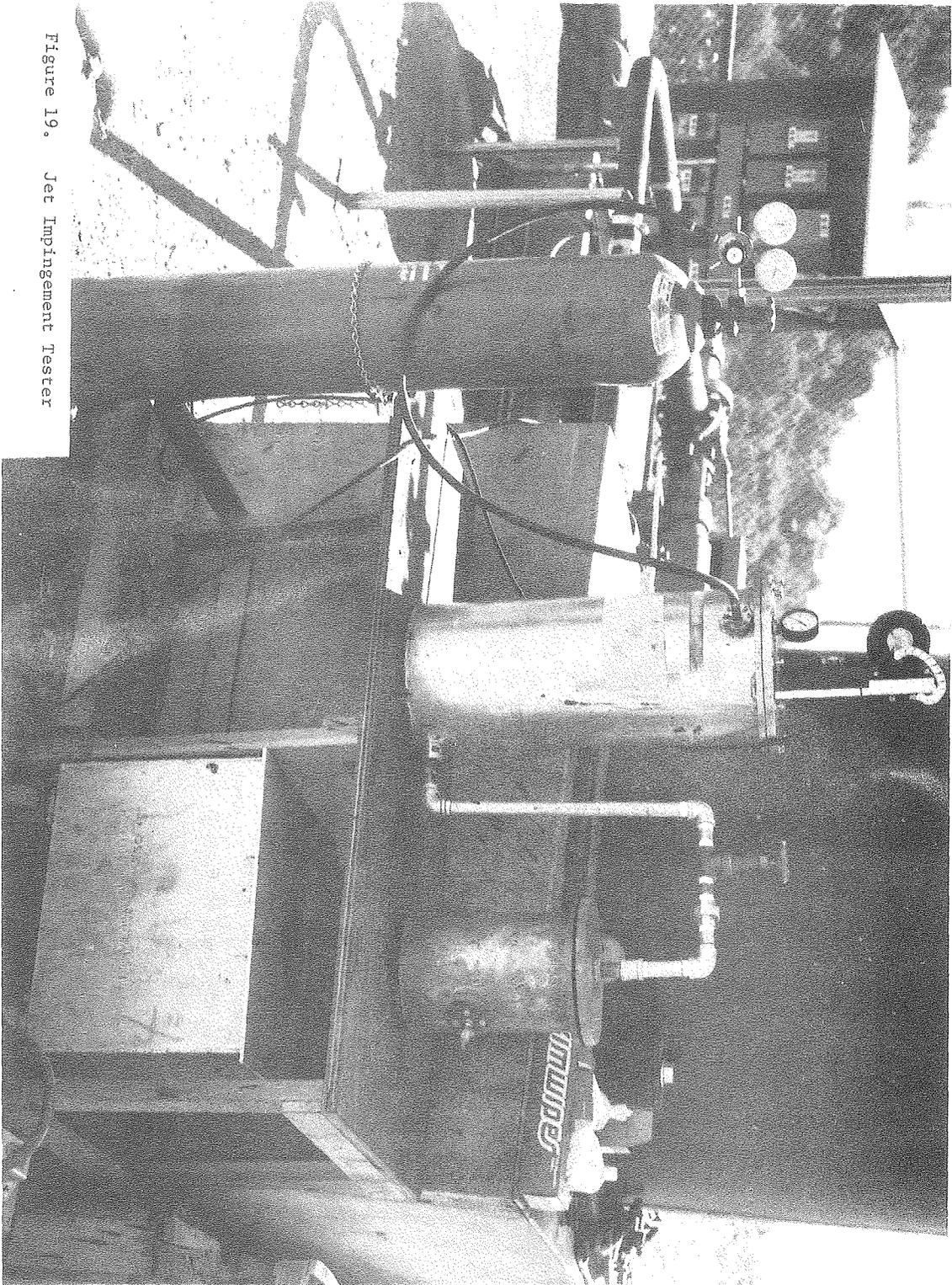


Figure 19. Jet Impingement Tester

BBC 789 10550

The nozzle is made of hard stainless steel, easily replaceable, and designed to minimize jet spread. Also a transparent lucite window is included in the test chamber to allow visual inspection of the flow regime.

Initial operation of the tester used unloaded kerosene and creosote oil for velocity calibration purposes and for familiarization with the equipment. The first slurry test runs used slurries of coal or SiC in kerosene to test the effects of various solids concentration, velocities and impingement angles on the ability of the tester to produce measurable test data in reasonable test times. A-53 mild steel was used as the target material. The specimen size was 1.5" x 0.5" x .125". The surfaces of the specimens were burnished before testing.

The test conditions were:

Velocity	=	30 fps
Angle of Impingement, $\alpha$	=	45°, 90°
Solids loading	=	10 wt % coal or 10 wt % SiC
Fluid	=	kerosene

The velocity was calculated by recording the time for a measured quantity of slurry to discharge through the nozzle. Tests with kerosene at such velocities, which are higher than those used in actual plants, are intended to give accelerated erosion rates that can produce data in laboratory scale time periods. Along with conducting accelerated tests, it is planned to test at lower velocities that more closely match pilot plant velocities.

### Jet Impingement Tester - Results

Four different tests at a velocity of 30 fps with 10 wt % coal in kerosene did not result in any measurable weight loss. The average duration of these tests was ten minutes at a 90° angle of impingement. Longer test runs will be made. However, there were optical effects on the specimen surfaces that showed some erosion had occurred. A profilometer was used to analyze these specimens. Table I shows the results of one of the four tests plus three tests that used a more abrasive particle, SiC. It can be seen that the coal slurry produced a minor, but measurable difference in surface roughness. The SiC-containing slurries produced a much greater change in the surface. The effect of velocity on the surface roughness is shown by test 5 and 6 results. In subsequent testing, a surface analysis device will be used that measures the depth of the affected zone.

Tests with 10 wt % SiC in kerosene at ambient temperature resulted in erosion weight losses that were measurable using test durations that required a reasonable number of loadings of the slurry tank. Figure 20 shows that a constant erosion rate was achieved, indicating that there was no breakdown of the particles' effect in this once-through slurry tester. The effect of the increasing surface roughness of the test specimen as erosion occurred did not change the rate of erosion for the test duration used.

Figure 21 indicates the effect of increasing velocity on the amount of erosion at 45° and 90° impingement angles in the initial tests with a 10 wt % SiC-kerosene slurry at ambient temperature. While more data is needed, there is a definite trend of increasing erosion with velocity and

TABLE I

Jet Impingement Tester  
Results

Profilometer Measurements on Sample Specimens

Test #	Slurry	$\alpha$	Velocity	Roughness/RMS micro-inches	
				Finish Surface	Wear Spot
1	10 wt % coal in kerosene	90°	37 fps	15-19	21-26
5	10 wt % SiC in kerosene	90°	50 fps	16-19	75-76
6	10 wt % SiC in kerosene	90°	37 fps	14-18	51-56
7	10 wt % SiC in kerosene	45°	53 fps	15-18	70-75

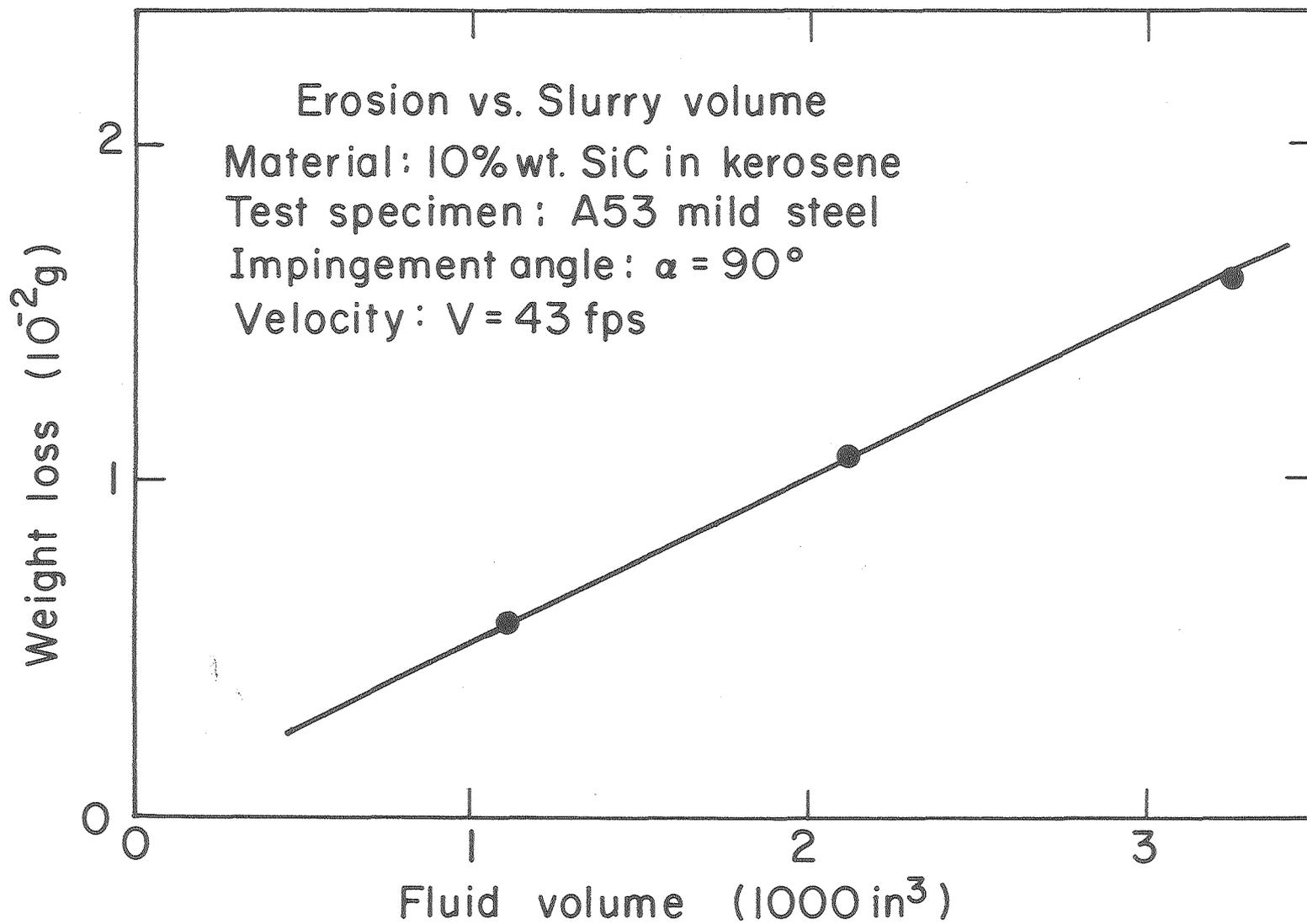


Figure 20. Erosion vs. slurry fluid volume for A-53 mild steel sheet specimen exposed in jet impingement tester.

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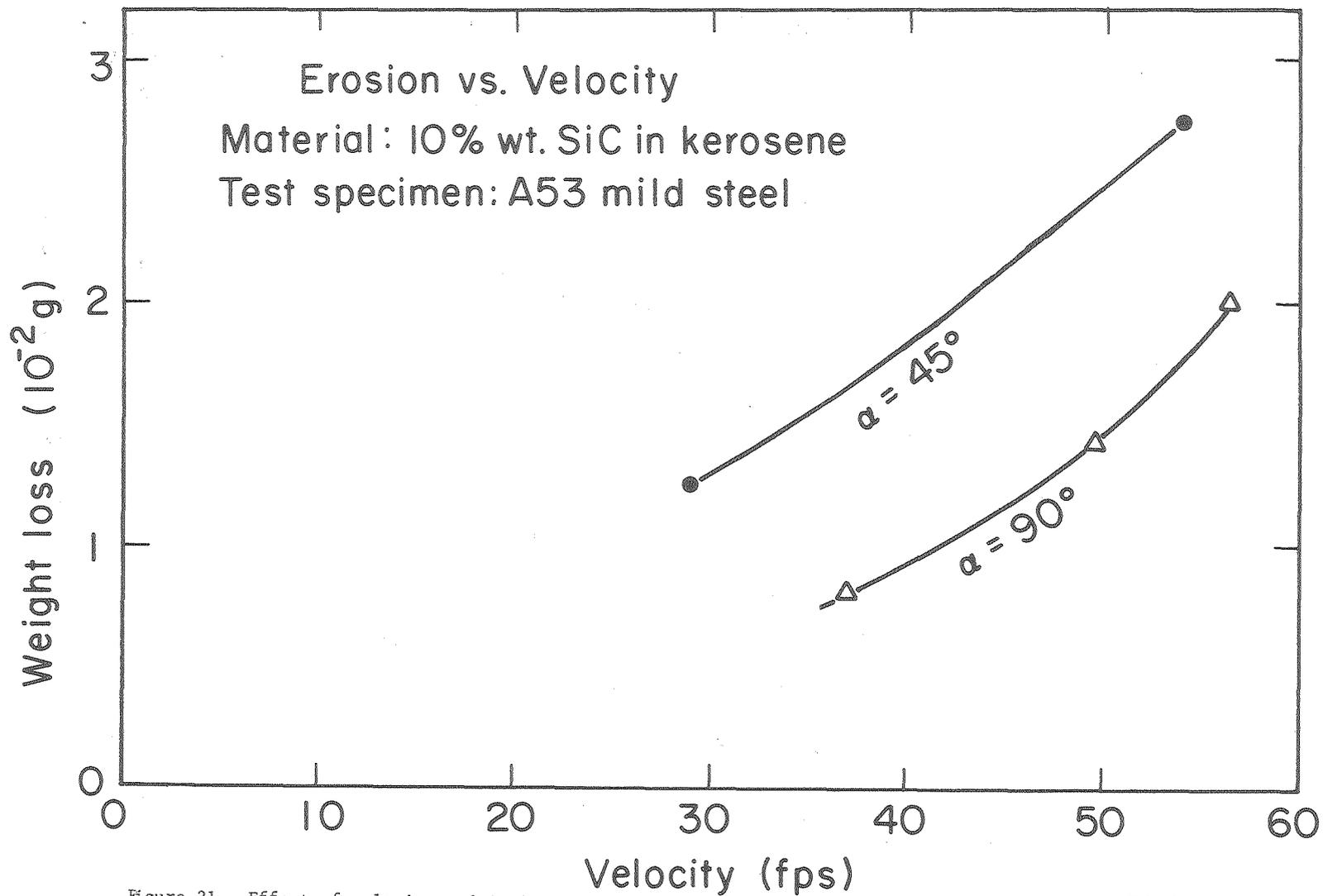


Figure 21. Effect of velocity and impingement angle on erosion of A-53 mild steel sheet specimens in jet impingement tester.

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at the lower impingement angle.

As in the case of the slurry pot tester, the initial test series in the jet impingement tester established the suitability of the apparatus to produce slurry erosion data in the laboratory. The elimination of the particle breakdown effect that occurs in recirculating type tests was achieved. The device will be utilized extensively in Tasks 4 and 5 to rate commercial alloys and to study erosion mechanisms. Possibly an analytical erosion rate prediction model similar to that developed for gas-solid particle erosion can be developed. The section on future work (page 69 ) will define in some detail the use that will be made of the jet impingement tester in the coming year.

#### 4. Two-Inch Diameter Pipe Recirculating Loop System

This apparatus was designed by the Ralph M. Parsons Company to simulate slurry flow through all of the piping configurations that are typically used in a coal liquefaction system. The 2" diameter pipe size was selected because it is thought to be the smallest size that would reflect scale-up characteristics of a full-scale plant.

Figure 22 is a piping isometric drawing of the test system. This design was selected after a number of earlier designs of greater complexity were discarded. Each of the elements of the system is, in itself, a test section and will be monitored intermittently during each test.

The piping components selected are: straight run section, screwed elbow, screwed tee, screwed flanges, five diameter bend, ten diameter bend, welded flanges, and weld ells. Table II lists the test elements that are included in the loop. The asterisked items are those that are incorporated

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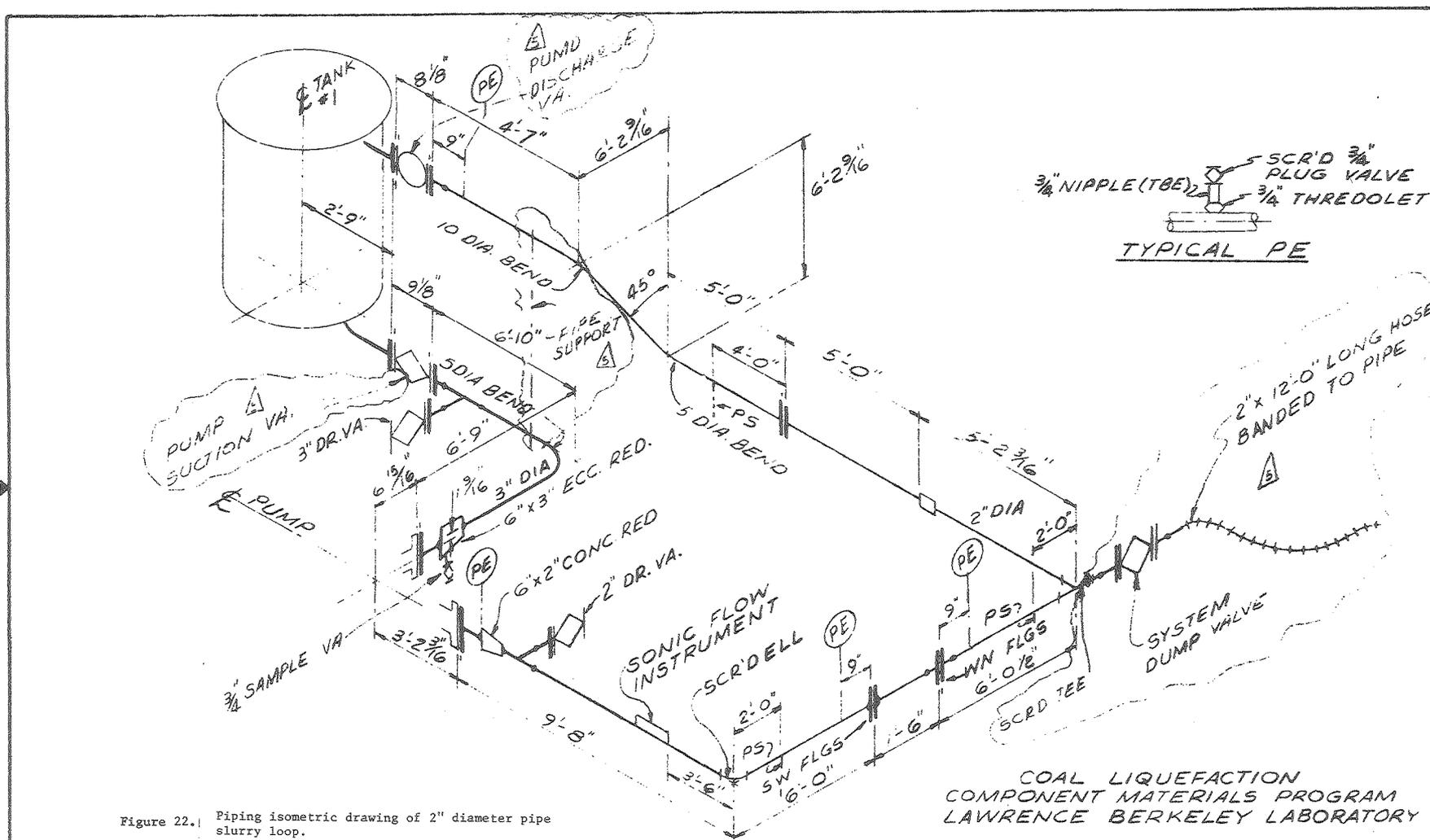


Figure 22. Piping isometric drawing of 2" diameter pipe slurry loop.

COAL LIQUEFACTION  
COMPONENT MATERIALS PROGRAM  
LAWRENCE BERKELEY LABORATORY

OPER TEMP	P&ID NUMBER	SECTION NO											RWP THE RALPH W. PARSONS COMPANY	ACCOUNT NUMBER		
PAIN	DESIGN TEMP	FIELD FAB												LINE NUMBER	UNIT/BASE OR AREA	
SPE PAIN SPEC	150°F												TEST LOOP			
INSULATION	DESIGN PRESS	SHOP FAB	5	7/1/78	JMA					ADD. P.S. & SYS DUMP VALVE		JOB NUMBER	DRAWING NUMBER	REV		
STRESS RELIEVE	COLOR CODE		4	1/23/79	JMA					REDESIGNED & REDRAWN		57521	LC-1	5		
			NO	DATE	BY	CHK	BIT	SEC	PRJ	APP	DESCRIPTION					

TABLE II

## TEST COMPONENTS

10 Bends	*
5 Bends	*
3R 90° W. Ells	
LR 90° W. Ells	
SR 90° W. Ells	
Scrd 90° Ells	*
SW 90° Ells	
45° W. Ells	
45° SW Ells	
Tee W.E.	
Tee SW	
Tee Scrd	*
45° Lat.(Fab)	
WN Flgs	*
SW Flgs	
Scrd Flgs	*
Butt Welds	
Weld Imperfections	
Ball Valves (T)	*
Plug Valves Rubber Lined	*

\* used in initial checkout test  
of slurry loop

into the initial loop test. Sufficient length of pipe, i.e. at least ten diameters, are used between pipe components to assure that there is laminar flow entering the component. A straight test section, 1-foot long, for precisely controlled flow conditions is preceded by an 8-foot long (50 diameter) section for flow control and is followed with a 2-foot long section to prevent flow perturbations in the test spool. Thickness of the pipe and fittings are measured by calipering and/or ultrasonic thickness gauge examination. Visual examination of internal smoothness is also made. Any examples of protuberances and discontinuities and their effect on erosion are noted. Figure 23 is a plan layout drawing of the test installation. Figure 24 is a view of the loop test system. Figure 25 is a closeup of the 2" diameter pipe components.

#### Ball valve -

The ball valve is full port with the internals coated with a Union Carbide erosion resistant material. The valve is a prototype, but has had good success in coal and water slurry applications. The valve gives tight shutoff service for the elevated temperature (to 400°F) phase of the test program, and provides evaluation of the Union Carbide coating for coal-solvent slurries. The maximum service temperature of the valve is limited by the seals and seat materials.

#### Plug valve -

The plug valve selected for pump and tank blocks is a full round port lubricated type. It operates either full open or closed. The valve in the full open position is equivalent to an equal length of pipe and produces a negligible amount of turbulence.

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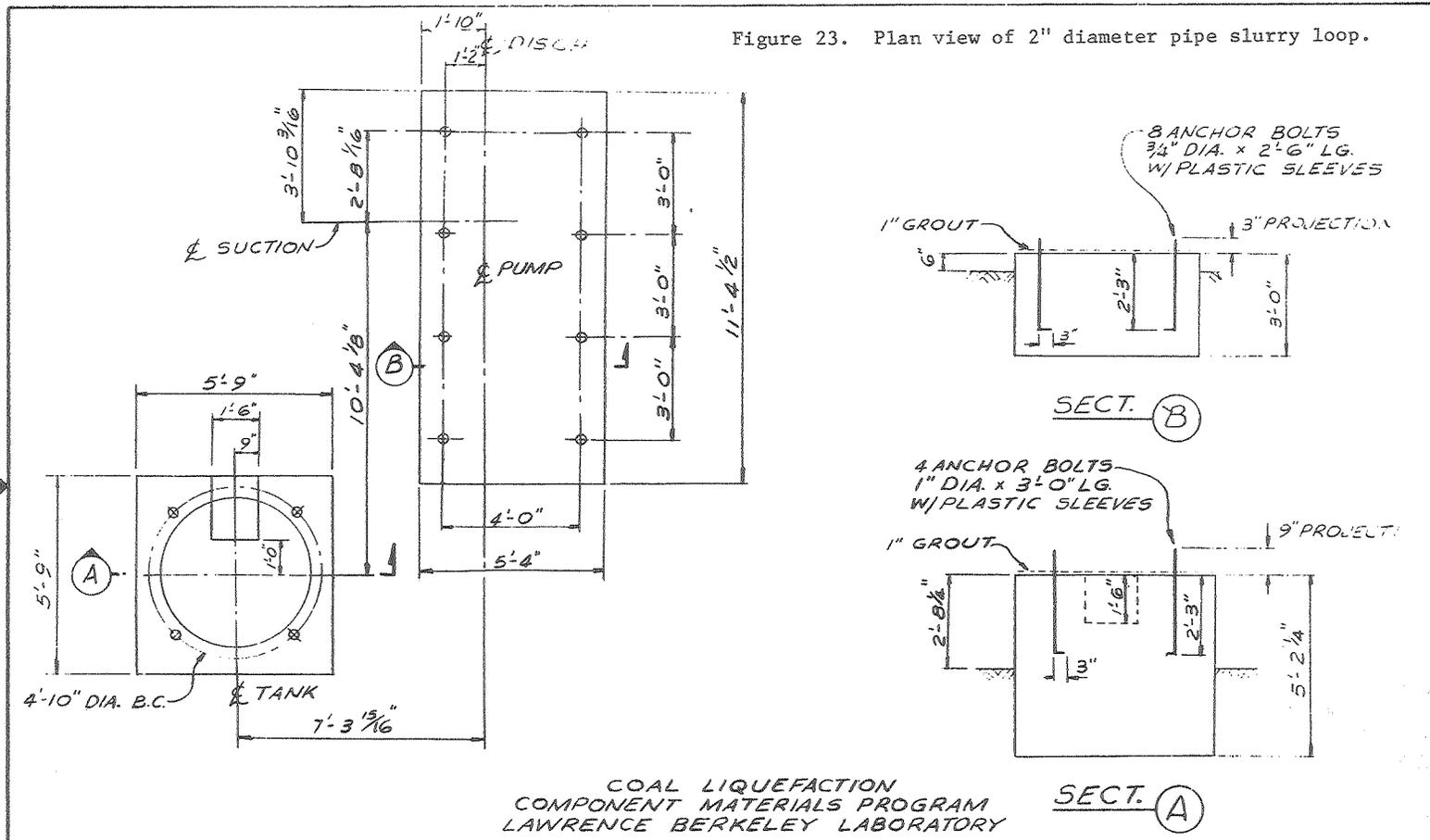


Figure 23. Plan view of 2" diameter pipe slurry loop.

OPER TEMP	P&ID NUMBER	SECTION NO.											<b>RJP</b> THE RALPH M. PARSONS COMPANY	ACCOUNT NUMBER	51	
PAINT SEE PAINT SPEC	DESIGN TEMP	FIELD FAB											LINE NUMBER	UNIT/BASE OR AREA		
INSULATION	DESIGN PRESS	SHOP FAB											<b>FOUNDATION DET.</b>			
STRESS RELIEVE	COLOR CODE												JOB NUMBER	DRAWING NUMBER	REV	
													5752-1	LC-5	2	
REVISIONS			NO.	DATE	BY	CK	PD QC CK	SEC	PROJ	CLIENT	DESCRIPTION					
			2	1/23/78	JMA						REDESIGNED & REDRAWN					

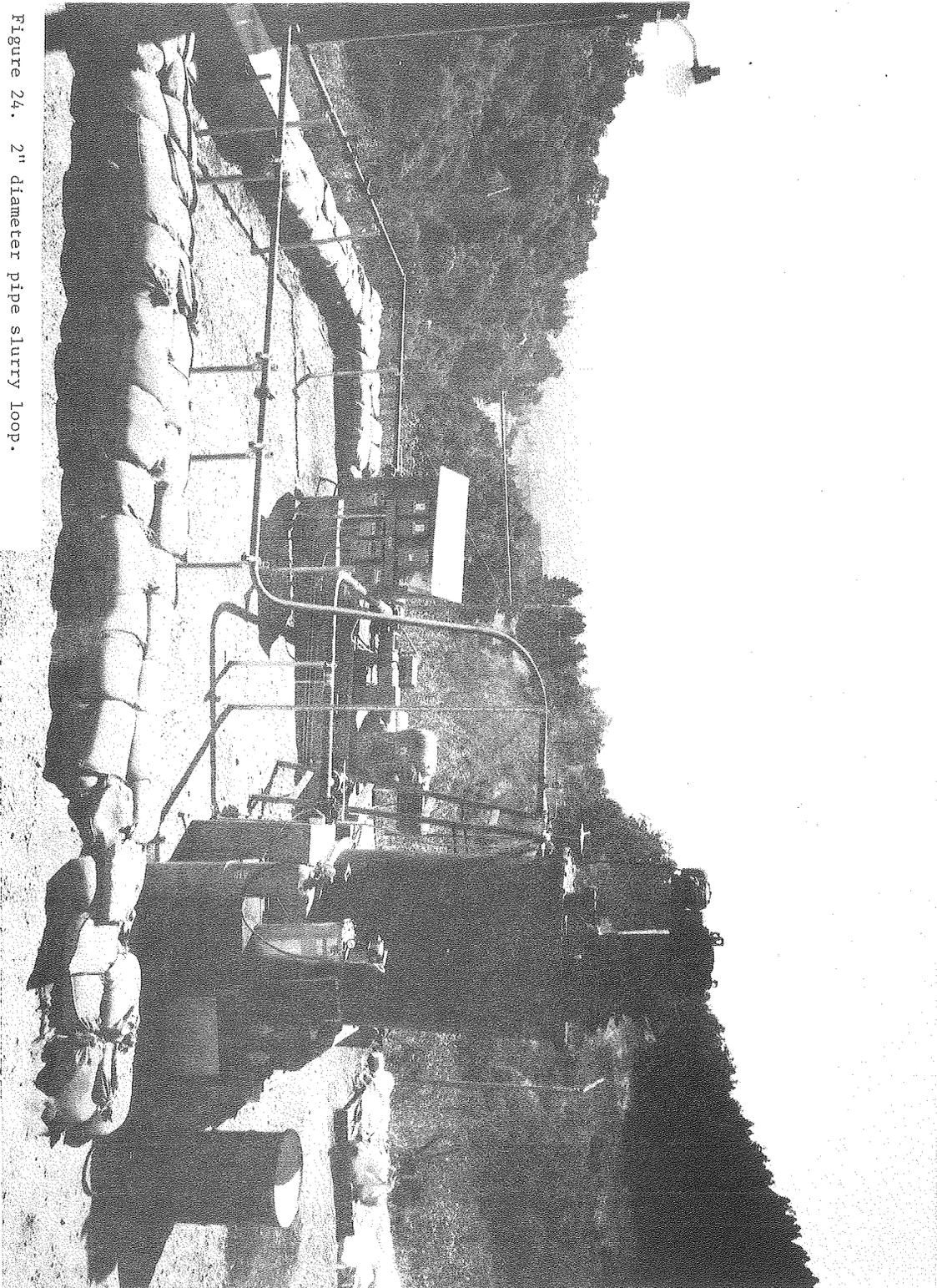


Figure 24. 2" diameter pipe slurry loop.

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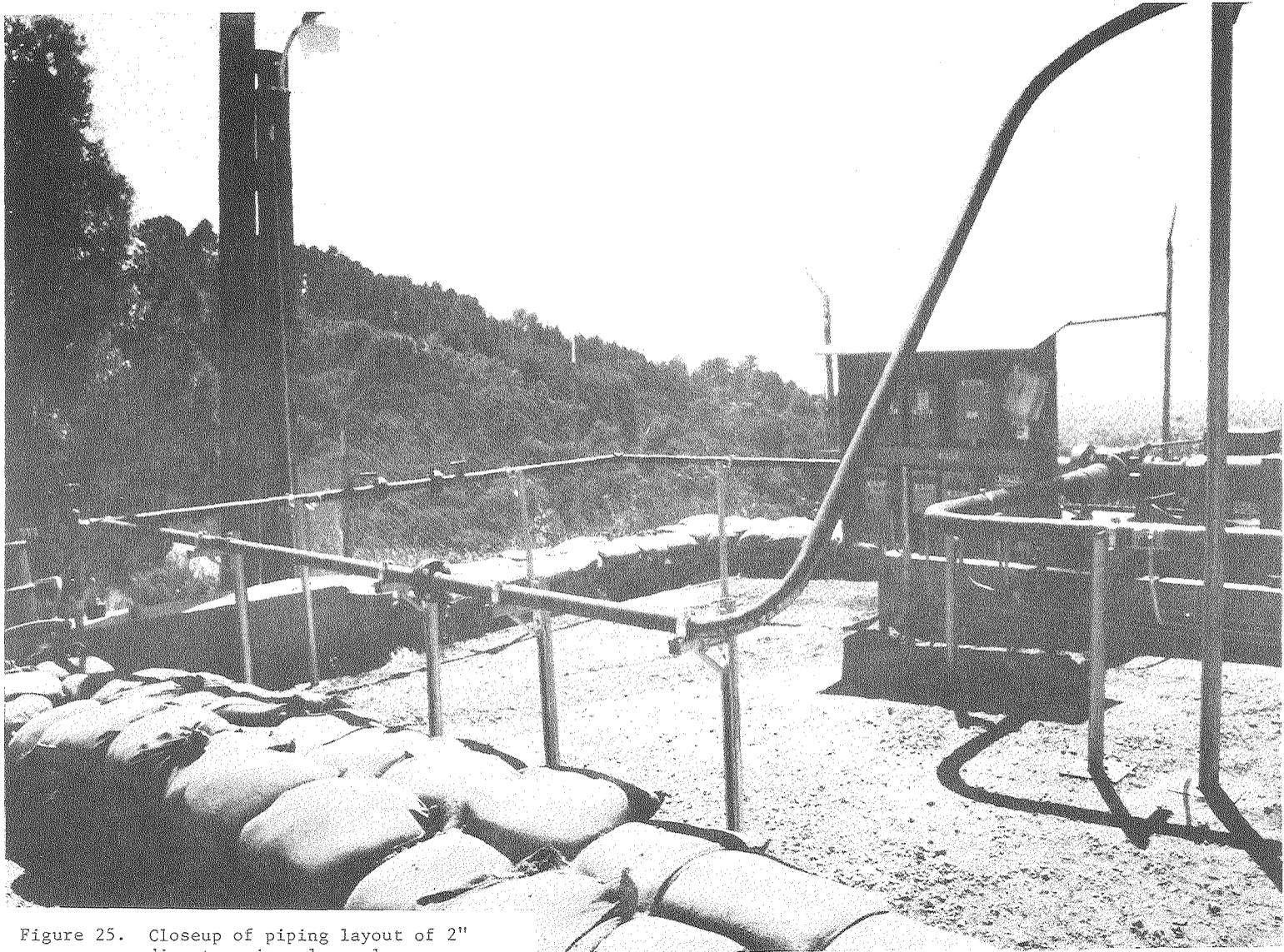


Figure 25. Closeup of piping layout of 2" diameter pipe slurry loop.

BCC 789-10544

Tank -

The tank selected for the initial phases of the test program is of carbon steel, has a flat bottom with flat plate cover, and is vented to the atmosphere. It is fitted as follows:

- 2" side inlet near top of shell
- 2" bottom outlet
- 3/4" thermowell in shell
- 8" mixer connection in cover
- 10" fill connection in cover
- 3/4" level gauge
- 2" atmosphere vent connection
- 6" spare connection with blind flange.

The tank is also fitted with four internal baffles designed to assure even mixing of slurry and solvent. The tank is attached to the foundation with four anchor bolts each 7/8-inch in diameter and is designed for seismic zone 3. The tank capacity is approximately 800 U.S. gallons and is 4' 6" in diameter by 6', 0" high.

The tank will provide a sufficient volume of test fluid to give a residence time of 5 to 12 minutes, depending on the velocity selected for the test. The cover is removable for tank cleaning and maintenance and is self-supporting for loads imposed by the mixer.

Tank agitator -

Selection of the slurry tank agitator was based on achieving the following:

1. The agitator and drive had to be large enough to maintain the

proper suspension and distribution of coal particles as a uniformly mixed slurry under the expected viscosity conditions. Violent agitation was not desirable.

2. The agitator speed of rotation had to be as low as possible in order to minimize abrasion due to contact with the coal particles, but still achieve the required mixing as set forth above.

Accordingly, a two-impellor (4 blades, 28-inch outside diameter) system, operating at 84 rpm, and driven by a 5 hp - 1750 rpm explosion-proof motor through a speed reducer was specified. The agitator diameter being close to half the diameter of the 4' 6" tank insures mixing at the higher viscosities where a small diameter propeller type agitator would merely form a hole. The 5 hp loading is sufficient for mixing the 800 gallons of slurry volume. This approaches a power loading approximately 6 hp per 1,000 gallons which is in the range for good, but not violent, agitation.

#### Pump -

A positive displacement pump was selected because it is not greatly affected by changes in viscosity or pressures in the ranges the loop will be operating. Centrifugal pump performance, particularly pump capacity and efficiency changes greatly with variations in viscosity, especially in the higher viscosity ranges that will be experienced in room temperature operation.

Of the positive displacement pumps, the progressive cavity pump is the most acceptable because it should be relatively unaffected by abrasive solids in the slurry. It is also expected that this type pump will cause

less coal particle degradation than centrifugal type pumps because of the appreciably lower velocities within the pump and elimination of particle impingement against a volute wall and impellor surfaces.

Of the progressive cavity pumps, the Robbins and Myers "Moyno" has the best record of service and experience. Moyno pumps have been employed successfully in coal/sulfuric acid slurry service by TRW in San Clemente and in coal/water slurry service by Lunday-Thaggard Oil Company in Los Angeles.

For use on this recirculating loop, a pump designed by Frederick Pump Company was selected. This pump, designated as Moyno 2SWG10H, Type CDQ, consists of a hard chrome-plated tool steel rotor, Viton Stator and double Durametalllic mechanical seals. It is mounted on a fabricated steel drip rim baseplate and driven by a 20 hp, 3 phase, 60 H, 230/460 volt TEFC 520-130 RMP US vari-drive motor. The system also includes a seal support system consisting of a 30 gallon seal liquid tank, centrifugal pump and a one hp, 3 phase TECF motor, radiator, valves, gauges and piping necessary to pressurize the seal chamber.

### Task 3 - Effects of Flow Passage Geometry

#### Equipment Operation -

The two-inch diameter pipe recirculating loop system is used in the program to determine the effects of flow passage geometry on metal erosion rate. The loop was pressure-checked with water. Kerosene, rather than creosote oil the starter fluid used in the Wilsonville SRC pilot plant, was selected for the first test in the loop because it is easier to handle while operating experience is being gained. Creosote oil was procured for



Figure 26. Ground elbow from 2" diameter pipe slurry loop.

subsequent tests.

A 200-hour duration shakedown run was conducted at the top speed of the pump, which resulted in a calculated slurry velocity of 40 fps, using a slurry of -200 mesh coal at 23 wt % in a kerosene liquid. All elements of the system performed satisfactorily except for the ultrasonic flow meter which gave inconsistent readings. In discussion with the supplier and D. Canfield of P & M Coal Mining Co. at Ft. Lewis, Washington, it was decided that the instrument was not suitable for use in slurries that contained the amount of solid particles that are planned for use in the 2" diameter test loop. It was decided that since the pump was of a positive displacement type and the system was a closed loop of fixed dimensions of flow passage throughout, that a calculation based on the pump rpm and its void volume would determine the velocity satisfactorily.

The other problem that occurred in the shakedown run concerned the measurement of wall thickness loss. Initially, an ultrasonic thickness gauge, inside and outside micrometers and a specially made jaw device with a dial indicator gauge on it were planned to measure the material loss of the test components in the loop. Since the most severe conditions occurred at the first elbow downstream from the pump discharge, it was measured during and after the first test run. It was determined that precise information on thickness changes of the order of a few thousandths could not be obtained because the inside surface of the as-fabricated elbow were too rough to measure small differences. After the first run, the elbow was, therefore, ground smooth over its interior surface. Figure 26 shows the ground elbow.

Other components measured mechanically after the first run also did

not show measurable material loss, primarily, it is thought, because of the difficulties of trying to obtain small differences in before and after readings. No measurable gouging or streaking loss of material had occurred, even downstream of protruding weld beads. However, visual smoothing of all surfaces had occurred.

Based on the smooth functioning of the loop in the 200-hour shakedown run and the desire to determine as soon as possible whether the loop could produce measurable erosion of surfaces at ambient temperature, it was decided to add more coal to the existing slurry in the loop to bring it up to 30 wt % coal in the kerosene and to run the loop for 400 hours. The amount of breakdown in the coal that had already been used for 200 hours was not thought to be important enough to change the 300 gallons of slurry in the tank, an arduous undertaking, because all we were trying to learn was whether the loop could produce measurable differences in wall thickness, not necessarily how much loss would occur.

The first sustained duration test was conducted at the maximum capacity of the pump to have the most severe conditions possible in order to give the system the best chance to produce measurable material loss at ambient temperature. The resultant velocity was 12 fps. It was determined from charts prepared by the Ralph M. Parsons Company and contained in their design manuals. The flow rate of the pump at maximum capacity is 100 gpm at a pump discharge pressure of 25 PSIG, resulting in a slurry flow velocity of 12 fps in a two-inch diameter pipe.

Samples of the slurry were taken through the sample valve periodically and kept for analysis of particle breakdown. The temperature of

the slurry in the test loop was measured at the end of the run and found to be 60°C. The air temperature in the area varied from 10°C at night to 32°C in the daytime. The higher slurry temperature was due to frictional heating.

Mechanical and ultrasonic thickness gauge measurements of the as-ground elbow were taken before the test. Measurements were made at twelve locations along the outside of the elbow, as shown in Figure 27. Shortly after the test was initiated, the ultrasonic thickness gauge broke down and a replacement for it could not be obtained immediately. The instrument was returned to the supplier. It was decided to rely on the mechanical measurements of the elbow and forego measurements of other components which could only be obtained using an ultrasonic gauge in order to expedite determining whether the loop would produce usable data.

#### Two-Inch Diameter Pipe Slurry Loop Results

##### Metal Erosion

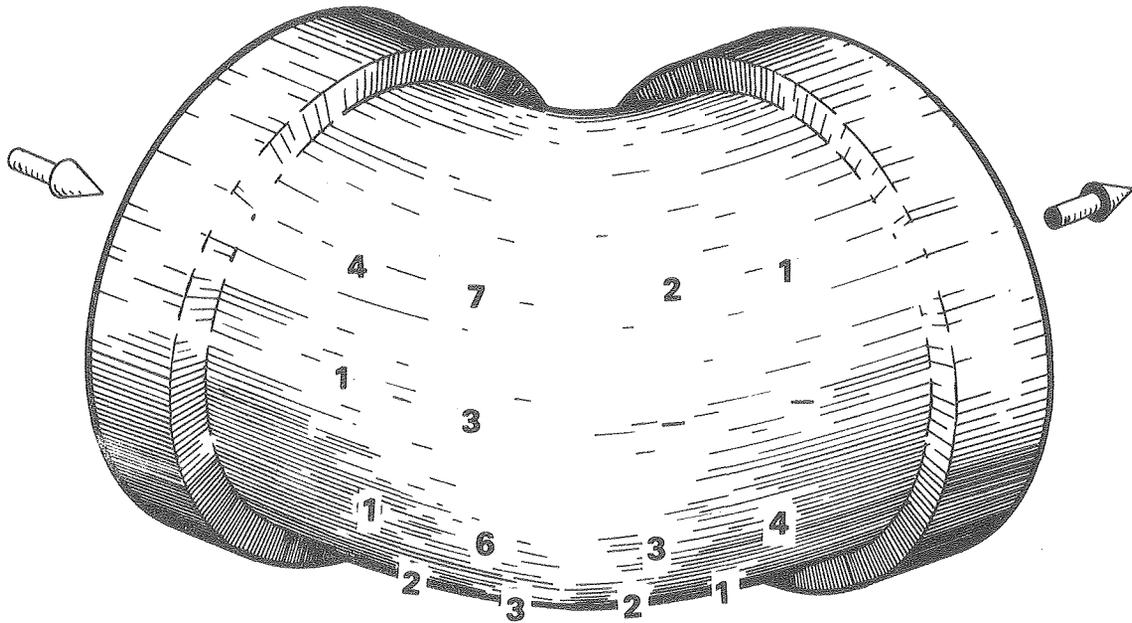
After 400 hours of continuous operation, the pump was turned off, the loop was drained of slurry and the elbow and the one-foot long straight test section were removed and analyzed. Measurements of the elbow showed that the loop is capable of eroding components to a measurable degree in a 400 hour exposure at ambient temperature. It was determined that the mechanical dial gauge system used to reach into the elbow to obtain measurements is not satisfactory as it produces somewhat erratic data. This is because it cannot be placed precisely in the same location for each measurement. An ultrasonic thickness gauge from another

manufacturer has been successfully evaluated on the loop and has been procured. It will be used in all subsequent tests. A simple fixture will be attached to each measurement position on all components to assure that the same, precise location is used for each measurement. The use of an inside micrometer on the one-foot-long straight test section did not result in usable data, another indication that the ultrasonic thickness gauge is necessary to result in reliable test data from the system.

To further test the loop, an additional 400 hours of operation was obtained, using the same slurry as was used in the first 400 hours. Figure 27 shows the total differences in the measurements obtained after the total of 800 hours of operation. Because of the problems of measuring the elbow wall with the dial gauge, only the trends in the amount of erosion will be discussed. The data from the future planned tests will be more suitable to establish geometry and material behavior as a function of the test conditions.

It can be seen in Figure 27 that, generally, more erosion occurred in the center of the elbow where the higher impingement angles are. Also, more erosion occurred in the lower region of the horizontally oriented elbow than in the upper region because of the effects of gravity which increased the solids loading in the lower part of the slurry. There is also some indication that more erosion occurred in the first 400 hours of operation than in the second 400 hours. This is due to comminution of the coal particles.

The appearance of the eroded surface of the elbow was smooth with no indication of localized gouging. However, there was indication of localized removal of material downstream from a weld protuberance in the



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Figure 27. Isometric drawing of pipe elbow showing number of thousandths of erosive wear after 800 hours of slurry loop operation.

one-foot straight pipe test section. The shallow streak in the pipe wall was too far from the section's attach flange to be mechanically measured. In future tests, it is planned that the ultrasonic thickness gauge which can measure to ten thousandths of an inch will pick up such changes. Other components of the loop, such as the 5D and 10D bends either could not or were not measured or observed in this first feasibility test.

#### Particle Comminution

The slurry was sampled periodically during the 800 hours of testing. Each sample removed from the loop via the sampling valve on the suction side of the pump was approximately one liter and has been stored in plastic flasks until it can be analyzed. A technique was developed for sizing the particles in the samples and SEM was used to observe the change in particle shape.

In order to separate the agglomerated coal particles that constituted the settled-out slurry sludge after the kerosene was poured off, the sludge was stirred into one liter of acetone and allowed to settle. This was repeated several times. The final product was then dried overnight on a hot plate, and the dried coal particles sieved and the quantities measured on each sieve. Fresh coal was also exposed to the same kerosene, then acetone and then sieved. Table III shows the particle size distribution obtained for the fresh coal and for coal samples taken from the two-inch diameter pipe loop after 800 hours of operation. The table indicates that a breakdown of the larger particles had occurred over the 800 hours of testing. The reduction by more than a factor of two of the percentage of the  $> 180 \mu\text{m}$  particles is thought to be signifi-

TABLE III

## PARTICLE SIZE DISTRIBUTION OF COAL SLURRY

Particle Size ( $\mu\text{m}$ )	% of Fresh Coal	% of Sample After 400 hours	% of Sample After 800 hours
180-250	1.4	.59	.49
125-180	1.6	1.56	1.25
90-125	2.3	2.67	3.00
< 90	94.0	95.20	94.95

cant as the larger particles probably have the greatest potential to penetrate the slurry's boundary layer and impact the pipe wall surface. However, since the overall shift in particle size distribution is relatively small, more work will have to be done to determine the effect of particle comminution on the erosion characteristics of the slurry.

Samples of fresh coal and coal that had circulated through the loop for 800 hours were studied using the SEM. Figure 28 shows particles of fresh coal (a) and recirculated coal (b) of 180  $\mu\text{m}$ , among the larger of the particles in the slurry. It can be seen that the recirculated coal has undergone considerable rounding of the particle edges, probably decreasing its efficacy as an erodent. Figure 29 shows fresh coal (a), and recirculated coal (b) of smaller particle size, 90  $\mu\text{m}$ . The angularity of the recirculated coal appears more pronounced than in the larger size coal, which gives rise to the postulation that smaller particles are not as much of an eroding factor as large particles. More samples are being analyzed to determine coal particle modification with time of slurry loop operation.

The operation of the two-inch diameter pipe slurry loop will be utilized in the next year of the program to determine the effects of operating condition variations on the erosion of the various geometries built into the loop. The tests that are planned are discussed in the Future Work section, page 69.

FIGURE 28.

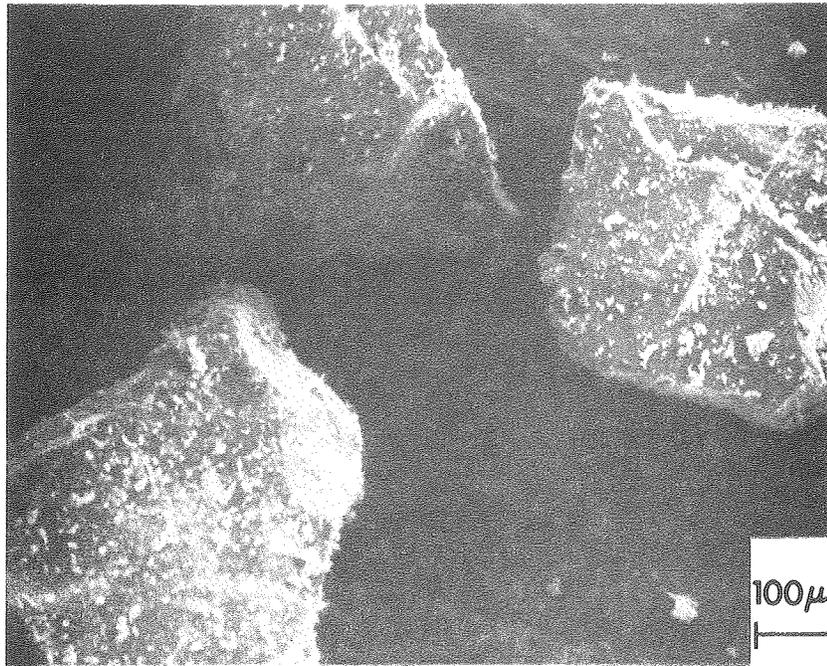
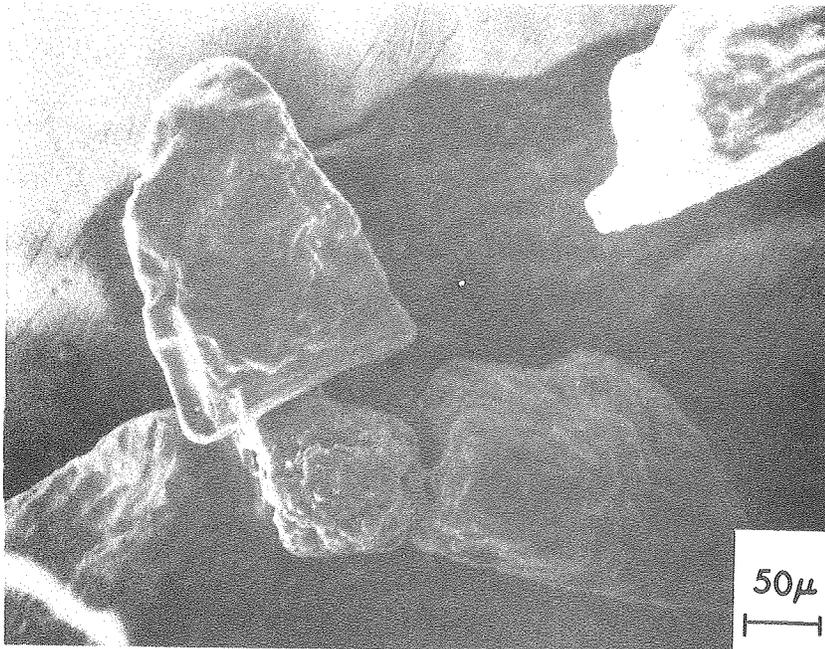
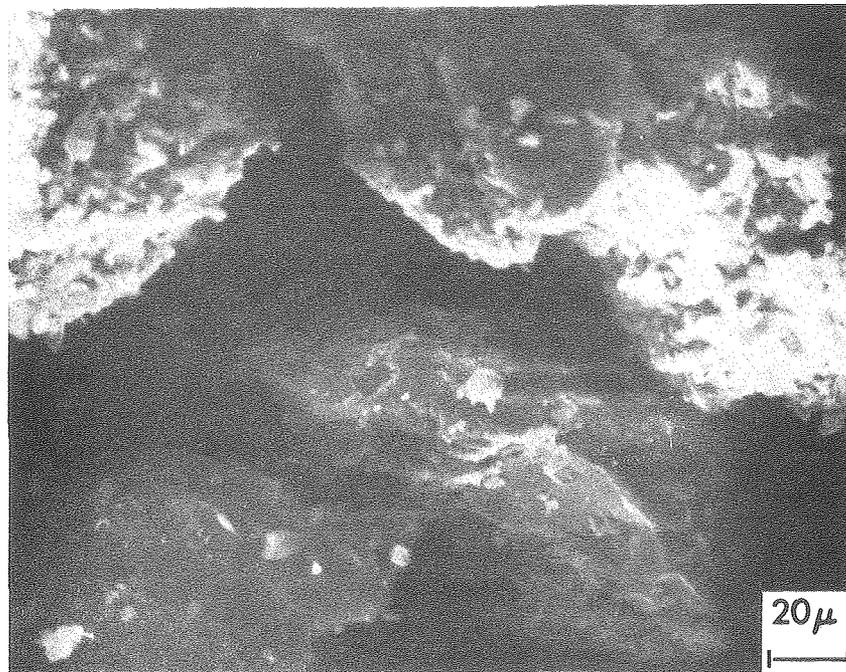
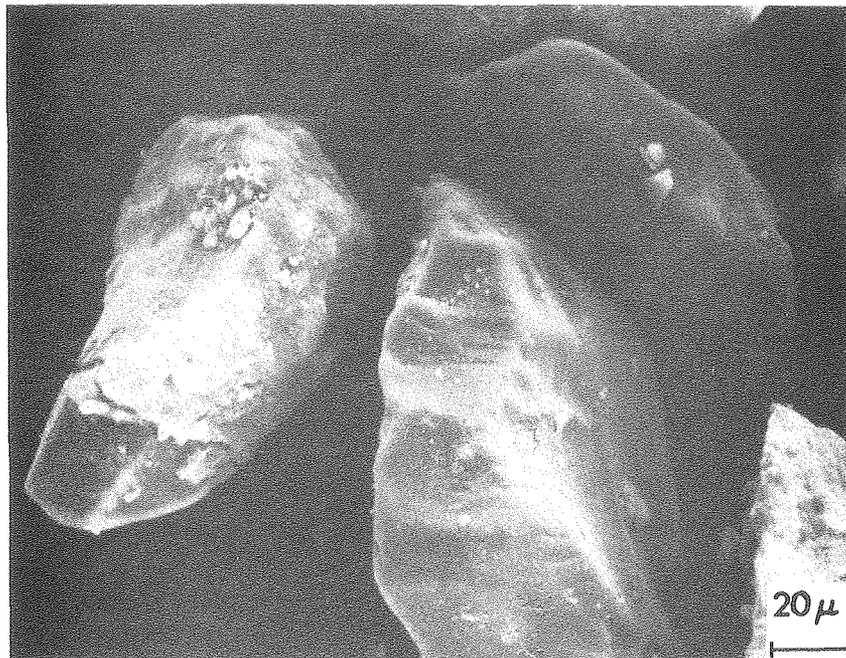
Fresh Coal Particles 180  $\mu\text{m}$ **A**Recirculated Coal Particles 180  $\mu\text{m}$ **B**

Figure 28. SEM photos showing change in recirculated 180  $\mu\text{m}$  coal particles after 800 hours slurry loop operation.

FIGURE 29.

Fresh Coal 90  $\mu$ m

A

Recirculated Coal 90  $\mu$ m

B

Figure 29. SEM photos showing change in recirculated 90  $\mu$ m coal particles after 800 hours slurry loop operation.

#### Task 4 - Behavior of Materials

The testing in the first year of the program was carried out to prove out the test devices. All tests were performed using A53 mild steel. The plans for the next year of the program involve the evaluation of several different steel compositions. Detailed plans are discussed in the future work section, page . . .

#### Task 5 - Erosion-Corrosion Mechanisms

The tests to prove out the equipment in Task 2 produced data reported in Task 2, that give some insight into the effects of viscosity, solids loading, velocity and angle of impingement on erosion of mild steel at ambient temperature. In the next year of the program considerably more knowledge will be gained of these effects and the added complexity of introducing corrosion into the test environment through the use of process solvents and elevated temperatures. In future reports, these investigations will be reported in Task 5.

#### Task 6 - Operating Guidelines and Materials Criteria

This task was not begun in the first year of the program.

PROGRAM PLAN FOR FISCAL YEAR 1979

Tasks 1 and 2 were completed in fiscal year 1978. A continuous review of the literature will occur throughout the program.

Task 3 - Effects of Flow Passage Geometry

## Viscosity Tests:

As viscosity is so important in determining the flow characteristics and, hence, the erosion potential of liquid slurries, additional viscosity tests will be carried out, particularly those at elevated temperature. Families of curves similar to those in Figures 4 and 5 will be generated for kerosene and at elevated temperatures to the maximum capability of the rotating concentric cylinder viscosimeter, not to exceed 400°F. The Ford cup test used for determining the viscosity of paints which are, in fact, liquid-solid particle slurries, will also be evaluated for use in determining viscosity variations in the slurries used in the program. The viscosities of slurries of other particle size distributions than the initial -200 mesh coal used will be determined. Viscosity data will be used in Tasks 3, 4, and 5.

## Two-inch Pipe Slurry Loop Tests:

It is planned to conduct eight to twelve 500-hour duration ambient temperature tests in the next year. The variables that will be investigated are listed below. All of the test components in Table II will be monitored for thickness changes. All tests will be carried out using A53 mild steel components except for one or two which will use type 304 stainless steel in some key components such as the elbow and one-foot straight

test section. For most of the tests a single set of loop hardware will be used with sequential erosion being measured for each test operation variation. If severe or localized erosion begins to occur, the affected component will be replaced prior to the succeeding test. If erosion cannot be measured in a severe or intermediately severe test condition, the planned milder condition will not be tested.

Test Conditions:

Velocity	10, 20, 40 fps
Particle Density	50 wt % coal, 30 wt % coal
Liquid (viscosity)	Kerosene, Creosote Oil

Jet Impingement Tests:

The test conditions used in the two-inch diameter pipe slurry loop will be used in the jet impingement tester at various impingement angles to relate erosion from specific geometries to the particle trajectories in those portions of the loop being analyzed. These results will be related to any predictive models that may be developed.

SRC-Ft. Lewis, Washington Side Loop Correlation:

The amounts of erosion measured at the various locations in the two-inch diameter pipe slurry loop will be compared to material losses in similar test components in the materials test side loop of the SRC pilot plant at Ft. Lewis, Washington. Since the operating conditions of the SRC side loop incorporate a major corrosion element from the process solvent that is circulated at elevated temperature and, generally, lower

velocities, the possibility of correlations occurring between the two test systems cannot be predicted. It is hoped that the erosion element in the combined erosion-corrosion material loss in the SRC side loop could be discerned by comparing the two-inch diameter pipe loop results at LBL with its results.

#### Design of Elevated Temperature Two-inch Diameter Pipe Slurry Loop:

Based on information coming from observations of corrosion in components of the SRC pilot plant, the two-inch diameter pipe loop at LBL need only be operated at temperatures up to 350-400°F to adequately simulate the erosion-corrosion that is occurring in the SRC pilot plant lines prior to the preheater. This temperature can be achieved by comparatively simple modifications to the current loop. The design of a 400°F capability loop will be undertaken in the next year of the program.

#### Task 4 - Behavior of Materials

The erosion-corrosion behavior of carbon, low alloy and stainless steel and cast iron compositions will be investigated. Alloy steels will include mild steel, plain carbon steels such as 1075 that can be heat-treated to provide markedly different microstructures for the same levels of hardness and the chrome-moly steels that are commonly used in chemical processing plant components. Stainless steels will include a ferritic type (410), austenitic types of lower and higher chromium contents (304, 310) and molybdenum content (316). Cast irons such as Ni-hard and white cast iron will also be tested.

### Slurry Pot Tests:

The test condition variables that will be utilized in the slurry pot tests will be selected from the following:

liquid	kerosene, cresote oil, SRC process solvent
particle	SiO <sub>2</sub> , coal
solids loading	0, 30 wt %
velocity	10, 20, 30 fps
temperature	RT, 200, 300, 400°F
test time	5, 10, 30, 60 minutes

The slurry pot tests will be used to determine the erosion and corrosion increments combining to cause material loss under combined conditions. This will primarily be done by subjecting the test specimens to fluids with and without solid particles in them with variations in the other test variables listed to determine their effect on the erosion and corrosion activity. The effectiveness of the erosion and corrosion elements in the environment on material removal rates as a function of testing variables and alloy composition, microstructure, and mechanical properties will be determined.

The cross flow characteristics of the specimens rotating in the pot will be studied to establish relationships between such flow factors as Reynolds number, effective impingement angles, changing specimen geometry as material loss occurs and resulting material loss rate.

Temperature will be a prime variant to produce desired changes in viscosity and corrosion activity. The selected temperatures will reach the region where corrosion has occurred in the SRC pilot plant.

#### Jet Impingement Tester:

Erosion tests will be performed on some of the metals studied in the slurry pot tests to determine the effect of fresh particles and varying the angle of impingement on erosion rate. These tests will be related to room temperature tests in the slurry pot tester. It is not expected that corrosion will be an element of the material removal process in these tests. The effect of microstructure differences in steels such as 1075 plain carbon steel at similar hardness levels on erosion resistance will be determined and compared to their behavior in gas-solid particle erosion.

#### Two-Inch Diameter Slurry Loop Tests:

As discussed in the section on Task 3, mild steel will primarily be used in the next year in the slurry loop tests. However, one or two tests of 304 stainless steel components will be conducted to determine how an austenitic steel compares to a ferritic steel.

#### SRC - Ft. Lewis, Washington, Side Loop Coorelation:

The materials that will be selected for testing by PANCO will be coordinated with compositions selected for tests at LBL and their test performance compared to LBL test results. The role of corrosion in material loss at the moderate elevated temperatures of the pilot plant side loop will be compared with corrosion losses in the slurry pot tests. Erosion from once-through coal particles in the side loop will be compared with results of the jet impingement tester. The complications arising from combined erosion and corrosion in the pilot plant side loop as compared to the more pure erosion in the jet impingement tester may make comparisons

between these devices difficult. It is felt that the low elevated temperatures, up to about 400°F, in the side loop will primarily affect corrosion and not erosion.

#### Task 5 - Erosion-Corrosion Mechanisms

The objectives of the next year's work to gain an understanding of mechanisms of material loss will be oriented toward erosion behavior. The effects of such operational variables as viscosity, solids loading, impingement angle and liquid composition on erosion will be investigated. Variables in materials such as hardness, crystallographic structure (BCC-FCC), presence of second phase particles and strength levels will be studied. The jet impingement tester will be the principal test device used in this task. The fluid mechanics of the liquid-solid particle system will be compared with that of the gas-solid particle system to determine whether erosion rate predictive models developed for the two phase gas system have applicability in the two phase liquid system.

#### Task 6 - Operating Guidelines and Materials Criteria

The information developed in Tasks 4 and 5 will be structured to provide guidelines to systems and materials designers.

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REFERENCES FROM LITERATURE STUDY

1. J. J. Mueller, et al; "Application of Advanced Materials and Fabrication Technology to Let-Down Valves for Coal Liquefaction Systems"; Battelle, Columbus Laboratories; EPRI AF-305; Project 458-1; Final Report; January, 1977.
2. S. Kimmel, et al; "Coal liquefaction Design Practices Manual"; Fluor Engineers & Constructors; EPRI AF-199; Project 409-1; Final Report; July, 1976.
3. W. Hong, The Fluor Corp; "Corrosion/Erosion Analysis of Construction Materials at the CRESAP Pilot Plant"; FE-1517-29; October, 1976.
4. Process Engineering Department, The Fluor Corp.; "Pipe System Erosion and Hydraulics Analysis at the CRESAP Pilot Plant"; FE 1517-32; May, 1976.
5. Pittsburg and Midway Coal Mining Co.; "Solvent Refined Coal (SRC) Process Annual Report"; 1975; FE-496-115; March, 1976.
6. Pittsburg and Midway Coal Mining Co.; "Development of a Process for Producing an Ashless, Low Sulfur Fuel from Coal"; R & D Report No. 53, Interim Report No. 22, June, 1974-April, 1977; FE 496-T13; June, 1977.
7. J. B. O'Hara, W. J. Lochmann, N. E. Jentz; "Coal Liquefaction: Materials Systems Design"; presented at ASM Symposium on Materials for Coal Conversion System Design; Pittsburgh, PA, April, 1976.
8. W. J. Lochmann, R. D. Howell; "Corrosion Design Problems in Coal Conversion Plants"; Materials Performance, NACE; July, 1977; P. 43-47.

9. T. L. Thompson, et al; "Slurry Pumps, A Survey"; Paper H1; Hydrotransport 2; University of Warwick, England; Sept. 1972.
10. G. Wilson, et al; "The Design Aspects of Centrifugal Pumps for Abrasive Slurries"; Paper H2; Hydrotransport 2; University of Warwick, England; Sept. 1972.
11. I. Tarjan, et al; "Theoretical and Experimental Investigation on the Wear of Pipeline Caused by Hydraulic Transport"; Page G1 Hydrotransport 2; University of Warwick, England; Sept. 1972.
12. D. F. Sambells; "A Practical Solution to Pumping on Abrasive Slurry"; Paper J1; Hydrotransport 3, Colorado School of Mines; May, 1974.
13. J. J. Vocadlo, et al; "Performance of Centrifugal Pumps in Slurry Service"; Paper J2; Hydrotransport 3; Colorado School of Mines; May, 1974.
14. M. L. Barker, G. F. Truscott; "The Development and Operation of a Test Facility for Pipeline Abrasive Wear Measurement"; Paper J3; Hydrotransport 3; Colorado School of Mines; May 1974.
15. R. R. Faddick; "Flow Properties of Coal-Water Slurries"; Paper H1; Hydrotransport 3; Colorado School of Mines; May 1974.
16. R. R. Faddick; "Pipeline Wear from Abrasive Slurries"; Internal and External Protection of Pipes Conference; University of Durham, England; Sept. 1975.
17. R. L. Gandhi, B. L. Ricks, T. C. Aude; "Control of Corrosion-Erosion in Slurry Pipelines; Paper G4; Internal and External Protection of Pipes Conference; University of Durham, England; September 1975.
18. T. L. Thompson, T. C. Aude; "Slurry Pipeline Design and Operation Pitfalls to Avoid"; ASME Paper 76-PET-12; Sept. 1976.



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