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# LOOP STUDIES ON THE BEHAVIOR OF PREFORMED $ZrO_2$ IN URANYL SULFATE SOLUTION

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J. R. Bowen
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CHEMICAL TECHNOLOGY DIVISION

Unit Operations Section

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Date Issued

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## ABSTRACT

Results reported for the behavior of preformed $ZrO_2$ in 0.04 m $UO_2SO_4$ solution circulated in three pumped loops showed deposition on surfaces dependent on loop geometry, flow conditions, surface conditions, and other parameters. Because of the great importance of geometry effects, the results do not permit prediction of the behavior of $ZrO_2$ in the HRT.

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## 1.0 INTRODUCTION

Studies were made to characterize the behavior of zirconium corrosion products in 0.04 m uranyl sulfate at the conditions existing in an aqueous homogeneous reactor fuel system. Temperatures of 250-300°C were of primary interest but studies at lower temperatures permitted visual observation, simplified experimental procedures, and demonstrated the effects of temperature and Reynolds number variations.

The fission products produced in the aqueous Homogeneous Reactor Test will be removed by an adjacent chemical processing plant. A small fraction of the reactor circulation will bypass the heat-exchanger and pass through the chemical processing plant, where a hydroclone will remove fission-and corrosion-product solids. Currently available information indicates that more than 80% of the solids will be corrosion products[1] and that zirconium oxide from corrosion of the core tank will constitute the bulk of the solids circulating in the core loop. Experimental data from previous circulating loops studies[2-4] indicated that solids formed in the reactor may deposit on the walls of the system. If the behavior of these solids can be determined, the operation of the chemical processing plant and of the reactor system may be improved.

The studies reported here were made to correlate the deposition rate of these solids in dynamic systems with the temperature, flow rate, system geometry, particle size distribution, and solids concentration.

To simplify the data analysis, tests were conducted with simulated zirconium oxide corrosion products. Two separate methods were used during the courses of the tests to introduce the zirconium oxide into the loop; hydrolysis of zirconium sulfate and injection of preformed zirconium oxide.

## 2.0 FACTORS AFFECTING $ZrO_2$ DEPOSITION

The deposition of $ZrO_2$ on loop walls was a function of the geometry of the loop, the temperature, the flow conditions, the type of surface, the initial concentration of $ZrO_2$, and the previous history of the loop. These effects could not be completely separated and studied individually. Since none of the effects could be shown to be negligible, only qualitative evaluations of individual contributions were practical.

The results imply that removal of solids by the hydroclone will compete with solids deposition in the HRT at least in the transient period in which the equilibrium solids concentration at the wall is approached. If the surfaces that receive particles do not become saturated, then deposition may be expected to continue after the system solids concentration has reached a steady state, and deposition will continue to compete with the hydroclone in

removing solids from the bulk flow.

## 2.1 Effect of $ZrO_2$ Concentration

For otherwise identical conditions, the fraction of the initial solids remaining in circulation increased consistently with increasing initial concentration (Fig. 1). This relation could be deduced less directly from the results for multiple additions of solids (Fig. 2). While variations in other parameters tended to control the deposition rates during the first few hours, the relation between the concentration of circulating $ZrO_2$, $C_t$, and the initial concentration, $C_o$, became evident as the rate of deposition approached zero (Fig. 1b).

The decrease in the slope of the deposition curve during successive additions of $ZrO_2$ (Fig. 2) may be attributed to an approach of the solids concentration at the wall to the loop initial concentration and possibly to saturation of the surfaces trapping the solids. Once the wall concentration approximates that of the loop, no further deposition should occur with continued solids additions unless the amount of solids trapped by the phenomena occurring at the wall is proportional to the concentration at the wall.

## 2.2 Effect of Temperature

Temperature appeared to be the least important of the variables studied. Increased temperatures gave increased initial deposition rates for constant flow rates (Fig. 3a,b); but the effect may have been due to increased turbulent flow resulting from decreased viscosity. When the Reynolds number was held constant by decreasing the flow rate while the temperature was increased, the effect of temperature was reversed (Fig. 4). Generally, when temperature and another parameter were varied to give opposite effects, the effect of the other parameter was dominant.

## 2.3 Effect of Flow Rate

The fraction of the initially present $ZrO_2$ remaining in circulation after the deposition rate approached zero increased consistently with increasing volume flow rates as indicated by increasing Reynolds numbers for otherwise identical conditions (Fig. 4). The initial deposition rates were sometimes increased by increased flow rates; i.e., the more turbulent flow apparently resulted in a more rapid approach to steady state conditions.

The variations of concentration at a given time with Reynolds number were very similar when the Reynolds number changes were caused by either changes in temperature or changes in flow rate. However, when temperature and flow rate were varied together to hold a constant Reynolds number, the concentration curve was changed (Fig. 3a).

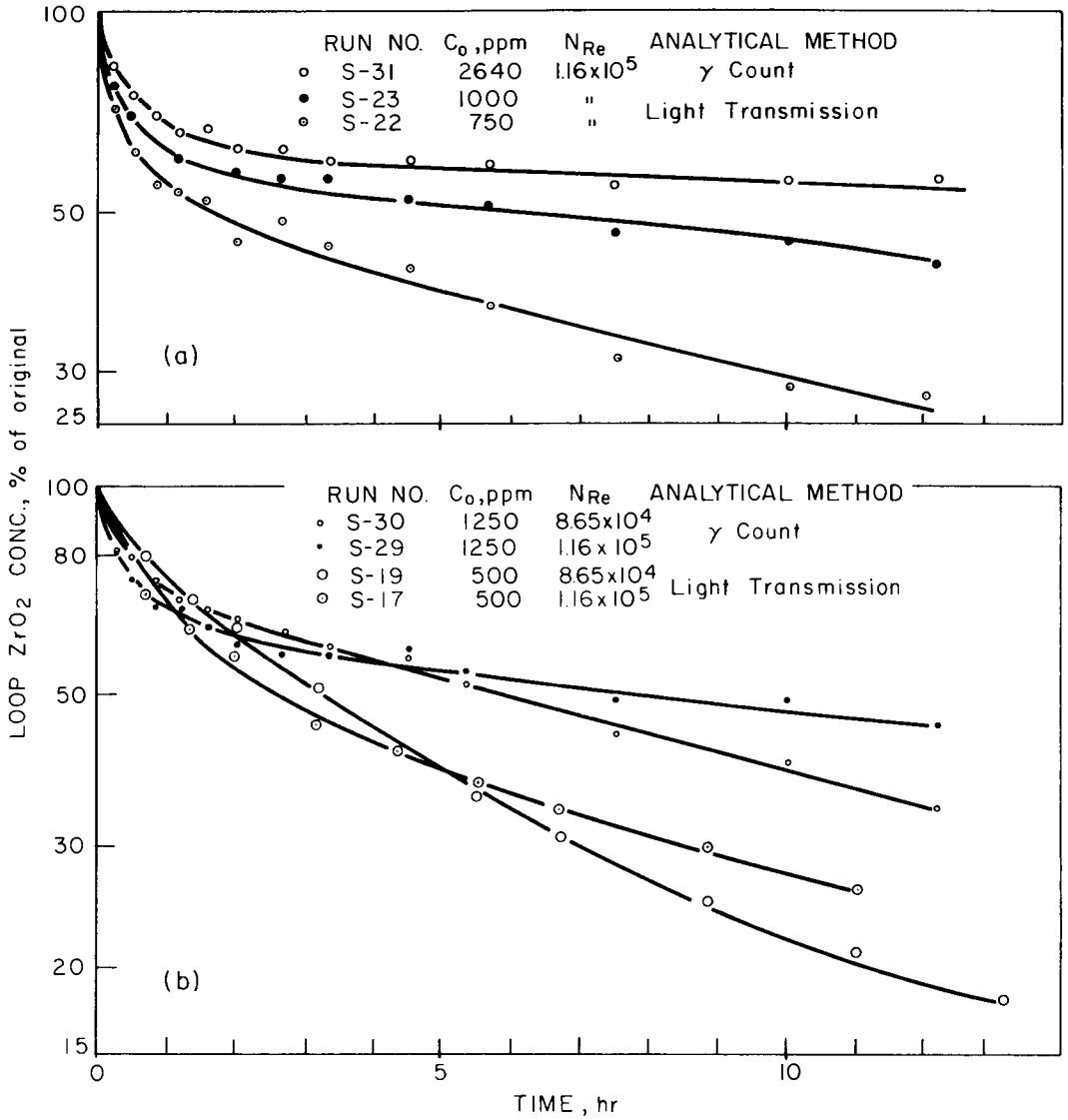


Fig. 1. Concentration of $ZrO_2$ in circulating A-loop as a function of time with varying initial concentration. Temperature: 115 °C. $N_{Re}$ based on flow through 1/2-in.-dia. pipe; $C_0$ based on volume of solids suspension added.

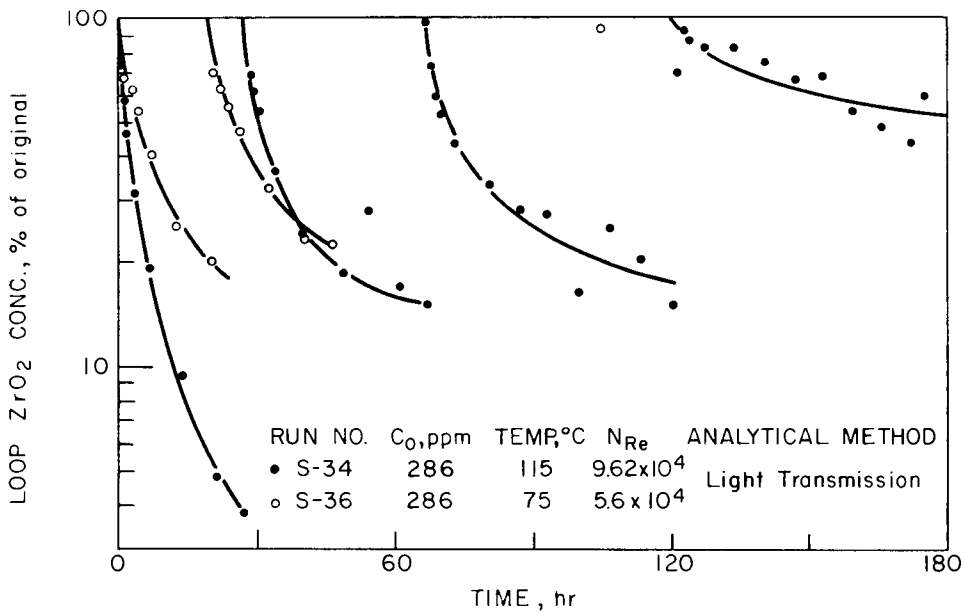


Fig. 2. Concentration of $ZrO_2$ in circulating A-loop as a function of time with successive additions of 312 mg of solids. $N_{Re}$ based on flow through 1/2-in.-dia. pipe; $C_0$ based on volume of solids suspension added.

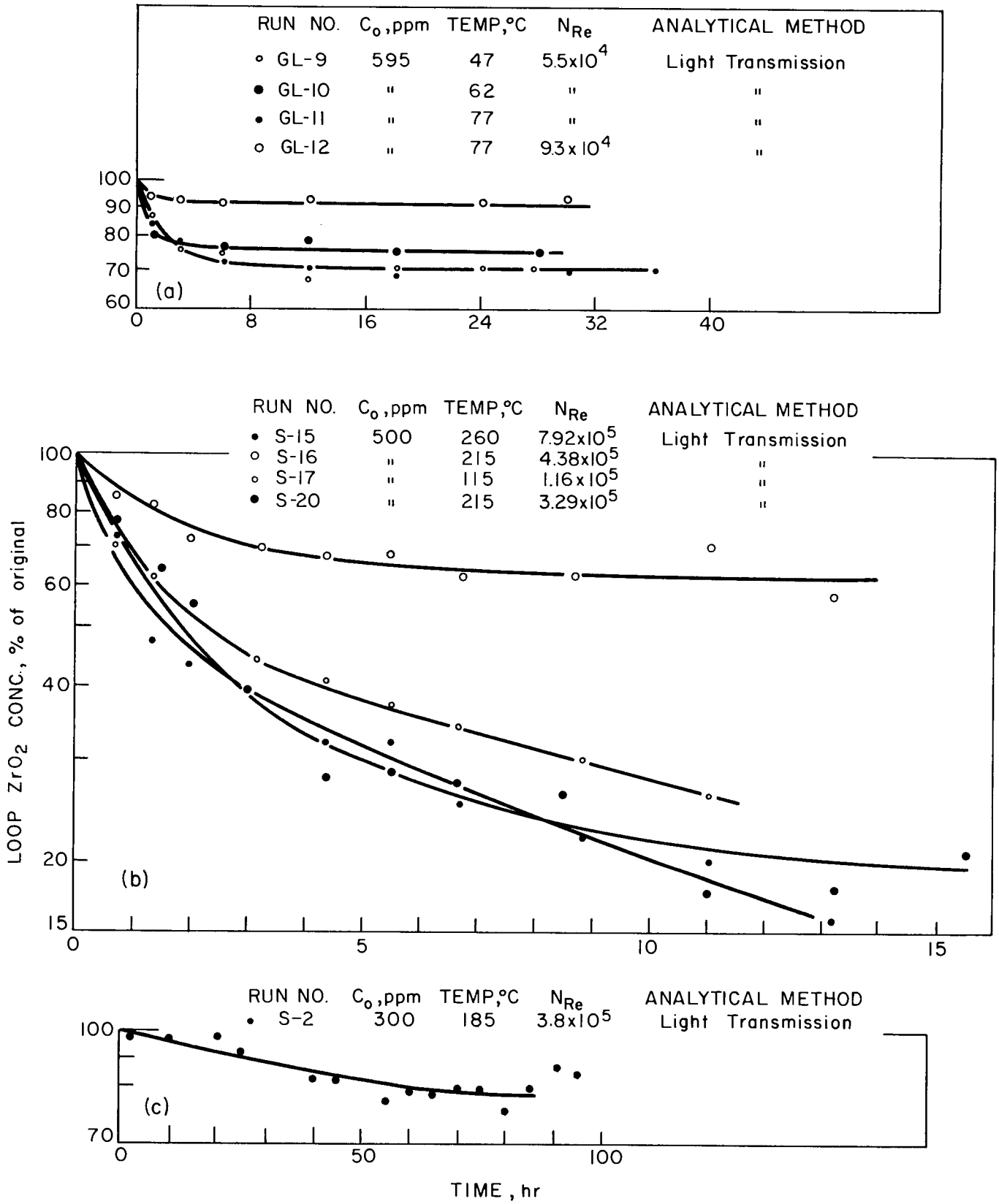


Fig. 3. Concentration of $ZrO_2$ as a function of time with varying temperatures in (a) and (b) A-loop and (c) B-2 loop. $N_{Re}$ based on flow through 1/2-in.-dia. pipe in A-loop and through 1 1/2-in.-dia. pipe in B-2 loop; $C_0$ based on volume of solids suspension added.

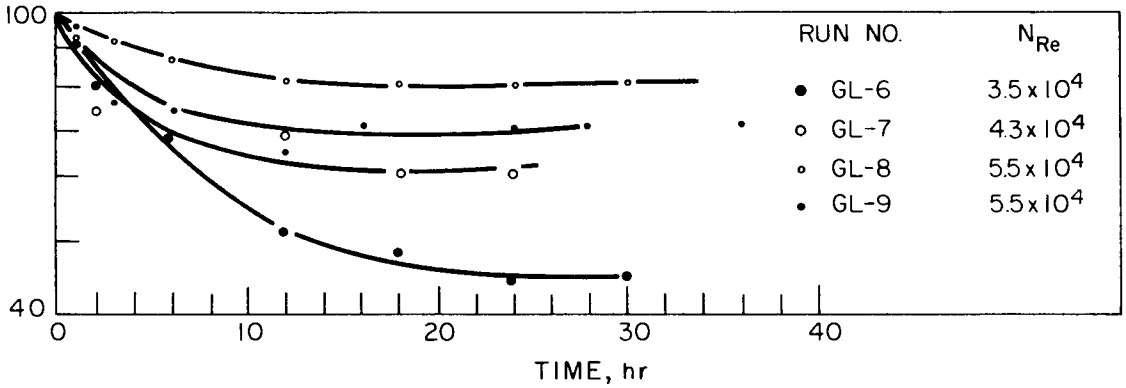


Fig. 4. Concentration of $ZrO_2$ as a function of time with varying Reynolds number in the glass loop. Temperature: 47°C; initial $ZrO_2$ concentration, 595 ppm; analytical method, $\gamma$ analysis. $N_{Re}$ based on flow through 1/2-in.-dia.; $C_0$ based on volume of solids suspension added. Heated metal section added between GL-8 & 9.

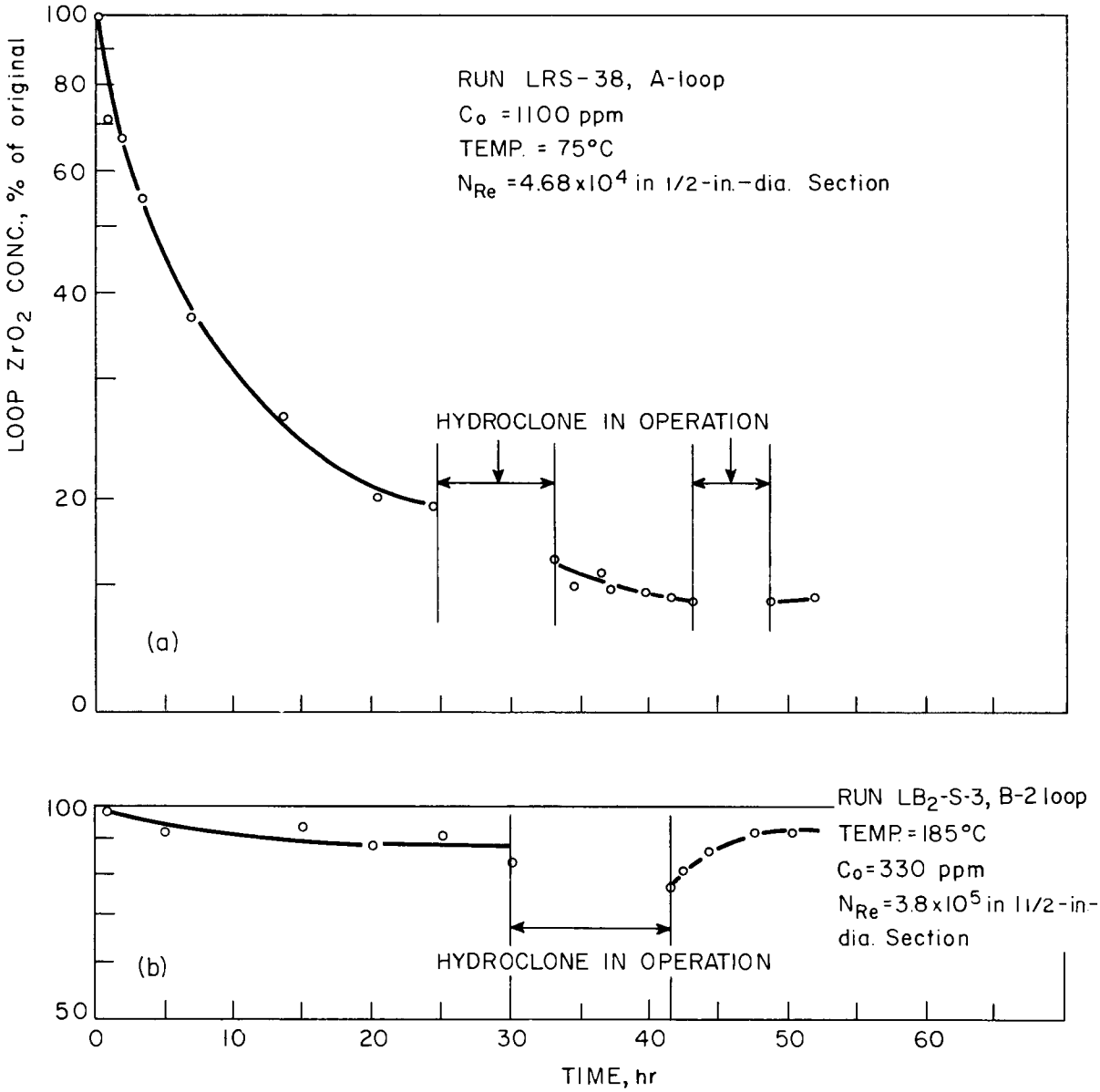


Fig. 5. Concentration of $ZrO_2$ in circulating loop as a function of time during intermittent hydroclone operation.

## 2.4 Effect of Other Loop Parameters

The effects observed during limited studies of loop surface and history variations appear logical, but were not complete enough to justify firm conclusions. Addition of a heated section resulted in lower concentration of circulating solids as compared to the same average loop temperature without a heated section (Fig. 4). This effect had been observed previously, especially for rare earth sulfates.[5] The studies with multiple additions of solids (Fig. 2) are in effect a varying loop history test and show that differences in the previous history of the system resulted in large variations in the behavior of newly added material. The effect of periods of hydroclone operation was not great (Fig. 5).

## 2.5 Application of Experimental Results to Other System

The above discussed results (Table 1) were examined with the intention of evaluating the effects of geometry. The most meaningful simple characterization of geometry was the ratio of surface area to circulating volume (Table 2). The fraction of the initial solids which remained in circulation appeared to increase as this ratio decreased. The solids deposition was influenced not only by the geometrical shape through which the bulk stream flowed, but also by the disturbances in flow patterns resulting from changes in geometry. The effect of these disturbances would be less significant in large loops and in the HRT than in these small loops studied unless the small loops were carefully designed to negate the effect.

The forces that may contribute to the transport of the suspended particles to the pipe walls are thermal, gravitational, diffusional, electrostatic, and inertial. Theoretical treatment of deposition is possible if inertial forces are assumed to transport most of the particles to a surface.[6] However, attempts to apply the approach of Friedlander to these systems have little experimental basis, unless the deposition of $ZrO_2$ particles is first studied in less complicated systems to determine the contributions of the various forces. Furthermore, qualitative analysis of the data indicated that the other forces may make significant contributions to the deposition of $ZrO_2$.

The fact that deposition occurred in A loop at temperatures varying from 216°C to 80°C with only slight changes in deposition characteristics indicated that the contribution of thermal gradients to the deposition was relatively small. The temperature range studied in the glass loop was only 30°C, and no effect of thermal gradient was apparent. Diffusional and gravitational effects could not be determined from the data.

The possibility of surface-electrostatic effects on deposition was indicated in the glass-loop tests when a metallic heating section was installed.

Table 1. Deposition of $ZrO_2$ During Loop Runs

| Run No. | Initial $ZrO_2$ Concentration, mg/l | Temp., °C | Reynolds No. | Length of Run, hr | Solids Deposition, mg/ft$^2$ By Material Balance | From Coupon |
|---|---|---|---|---|---|---|
| | | | A and B-2 Loops | | | |
| S-17 | 550 | 115 | $1.16 \times 10^5$ | 11 | 64.3 | |
| S-22 | 825 | 115 | $1.16 \times 10^5$ | 12 | 69.6 | |
| S-23 | 1100 | 115 | $1.16 \times 10^5$ | 12 | 83.0 | |
| S-29 | 1375 | 115 | $1.16 \times 10^5$ | 12 | 101.0 | |
| S-31 | 2900 | 115 | $1.16 \times 10^5$ | 12 | 173.0 | |
| S-19 | 550 | 115 | $8.65 \times 10^4$ | 13 | 65.1 | |
| S-30 | 1375 | 115 | $8.65 \times 10^4$ | 12 | 98.5 | |
| S-16 | 550 | 215 | $4.38 \times 10^5$ | 13 | 46.5 | |
| S-15 | 550 | 260 | $7.92 \times 10^5$ | 13 | 74.5 | |
| S-34 | 1260 | 115 | $9.62 \times 10^4$ | 180 | 135.0 | |
| S-36 | 1260 | 75 | $5.60 \times 10^4$ | 45 | --- | |
| S-38 | 1210 | 75 | $4.68 \times 10^4$ | 52 | 156.0 | |
| $B_2$-S-2 | 3620 | 185 | $3.80 \times 10^5$ | 96 | 45.6 | |
| $B_2$-S-3 | 3620 | 185 | $3.80 \times 10^5$ | 51 | 2.4 | |
| | | | Glass Loops | | | |
| Gl-6 | 274 | 47 | $3.5 \times 10^4$ | 30 | 108.6 | 113.3 |
| Gl-7 | 274 | 47 | $4.3 \times 10^4$ | 24 | 74.1 | 75.2 |
| Gl-8 | 274 | 47 | $5.6 \times 10^4$ | 36 | 53.4 | 67.8 |
| Gl-9 | 274 | 47 | $5.6 \times 10^4$ | 27.5 | 60.0 | 36.2 |
| Gl-10 | 274 | 62 | $5.6 \times 10^4$ | 30 | 51.1 | 41.8 |
| Gl-11 | 274 | 77 | $5.6 \times 10^4$ | 36 | 59.2 | 34.3 |
| Gl-12 | 274 | 77 | $9.3 \times 10^4$ | 30 | 13.3 | 615.8 |

The installation decreased the glass surface from 67% to 38%, and deposition curves made under similar conditions before and after the installation were significantly different. Further tests would be required to determine the nature of the electrostatic forces.

Table 2. Loop Parameters

| System | Circulating Vol. $ft^3$ | Approx. Wetted Surface Area, $ft^2$ | Ratio of Surface Area to Vol, $ft^{-1}$ | Pump | Circulation Rate, gpm | Temp, °C | Maximum Pressure psi |
|---|---|---|---|---|---|---|---|
| A loop | 0.035 | 3.3 | 100 | Canned rotor | 3.6-4.8 | 60-260 | 2000 |
| B-2 loop | 0.37 | 7.2 | 20 | Canned rotor | 25. | 185 | 2000 |
| Glass loop | 0.022 | 1.2 | 55 | Mechanical seal | 1.2-2.4 | 47-77 | 30 |
| HRT | 18. | 600. | 33 | Canned rotor | 400 | 250-300 | 2000 |

## 3.0 EXPERIMENTAL METHODS

### 3.1 Apparatus

The studies were made in the A, B-2, and glass loops (Fig. 6). Important system parameters are listed in Table 2. The A and B-2 loops are high-pressure circulating loops that can be maintained at temperatures and pressures corresponding to HRT operating conditions. They differ in volume, flow rate, and geometry. In addition, the B-2 loop has no primary heater; all the heat required to maintain the system temperature of 300°C is supplied by the circulating pump. The A loop has a primary heater with a high heat flux through the loop wall.

The principal disadvantage of these loops was that deposited solids cannot be easily recovered. The glass loop was designed for operation at low temperatures and pressures of 1 to 2 atm. A metal heating section was installed to permit operation at temperatures above ambient. The principal advantage of the glass loop was that it could be easily disassembled for cleaning and modification, the deposited solids could be more easily

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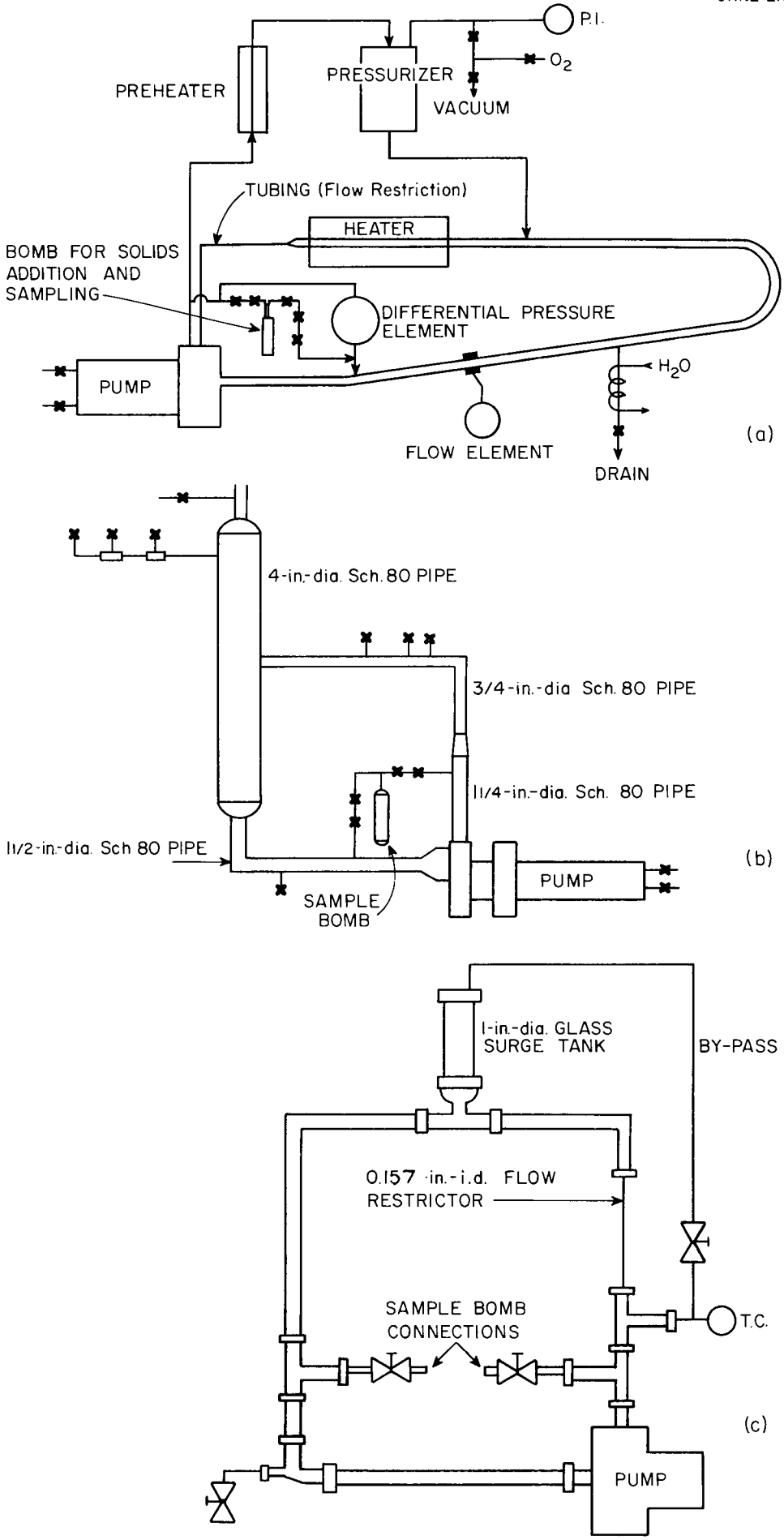


Fig. 6. (a) A-loop, (b) B-2 loop, and (c) glass loop as used in $ZrO_2$ behavior studies.

recovered, and visual observation of deposition was possible while the loop was in operation.

### 3.2 Operating Procedures

A typical run in any one of the loops was made by filling the loop with the necessary volume of simulated HRT core solution and bringing the loop to test conditions. Zirconium was then injected into the system, either as zirconium sulfate or as preformed zirconium oxide, by a bomb sampler. The bomb sampler was connected into the loop for a time long enough that the loop solids concentration was that of the bomb. At this point, the bomb sampler was disconnected from the loop and emptied to give the initial sample. Samples were taken during the course of the run in a similar way. To maintain constant loop volumes, the bomb was filled with the proper volume of simulated HRT core solution before each sample was taken. Results were corrected for the dilution.

## 4.0 REFERENCES

1. "Homogeneous Reactor Project Quarterly Progress Report for Period Ending January 31, 1956," ORNL-2057, p. 41.

2. "Chemical Technology Division - Unit Operations Section Monthly Progress Report, November, 1956," CF-56-11-143, p. 37-48.

3. J. R. Engel, "Preliminary Solids Injection Experiments on the HRT Mock Up at Y-12," CF-56-12-115 (Dec. 18, 1956).

4. "Homogeneous Reactor Project Quarterly Progress Report for Period Ending January 31, 1957," ORNL-2272, p. 37, 133, 136.

5. P. A. Haas et al., "HRP-CP Conclusions Concerning the Predicted Behavior of Insoluble Corrosion and Fission Products in the HRT," CF-58-1-97, (Jan. 23, 1958).

6. S. K. Friedlander, H. F. Johnstone, "Deposition of Suspended Particles from Turbulent Gas Streams," Ind. Eng. Chem. 49, p. 1151-1156 (1957).

## 5.0 APPENDIX

### 5.1 Analytical Methods

The deposition of preformed $ZrO_2$ solids in a loop is difficult to analyze without accurate information on the characteristics of the solids at the temperature and pressure of the loop. In particular, the light transmission

methods used to analyze samples during most of the tests is valid only when the sample has the same particle size distribution as the standard. If the particles undergo any process, either physical or chemical, that results in a change of particle size distribution, the analytical results may be anomalous. In the experiments reported here the results were not consistent, and a better understanding of the nature of the physical or chemical transitions that the particles underwent during the test may reconcile the contradictions.

During the tests in the A-loop, two sets of bomb samples were submitted for particle size distribution analysis by neutron activation techniques. The analytical results (Fig. 7) showed a growth in particle size during the course of a run or possibly preferential deposition of smaller particles. Thus under the conditions of the tests (115°C and 215°C, 6.5 fps flow, and 200 psig $O_2$ overpressure), particles may have agglomerated. However, tests in the A loop with activated solids indicated that under these conditions, there was no significant difference between the light transmission and $\gamma$ counting analytical methods. It should be cautioned that two separate runs are being compared and that the results obtained might compare favorably because of fortuitous circumstances. The results were even more surprising in the glass loop. If agglomeration occurs, the light transmission analytical method should yield a value below the actual suspended solids concentration. Analyses performed during the final glass loop runs indicated that the solids concentration as determined by light transmission were greater than that determined by $\gamma$ counting (Fig. 8). Under the glass loop test conditions (47°C, 3 fps flow, and 5 psig $O_2$ pressure), therefore, a process, either physical or chemical, occurred that resulted either in the destruction of large particles or possibly the formation of $ZrO_2$ colloids in the system. Until the nature of these changes are better understood so that their effect on light transmission analytical techniques can be predicted, the information collected with this analytical technique merits only qualitative treatment.

## 5.2 Calculated Loop Parameters and Results

Flow velocities, Reynolds numbers, and areas for the various-diameter sections in each loop were calculated (Table 3, 4) and material balances for the studies with preformed $ZrO_2$ were made (Table 5).

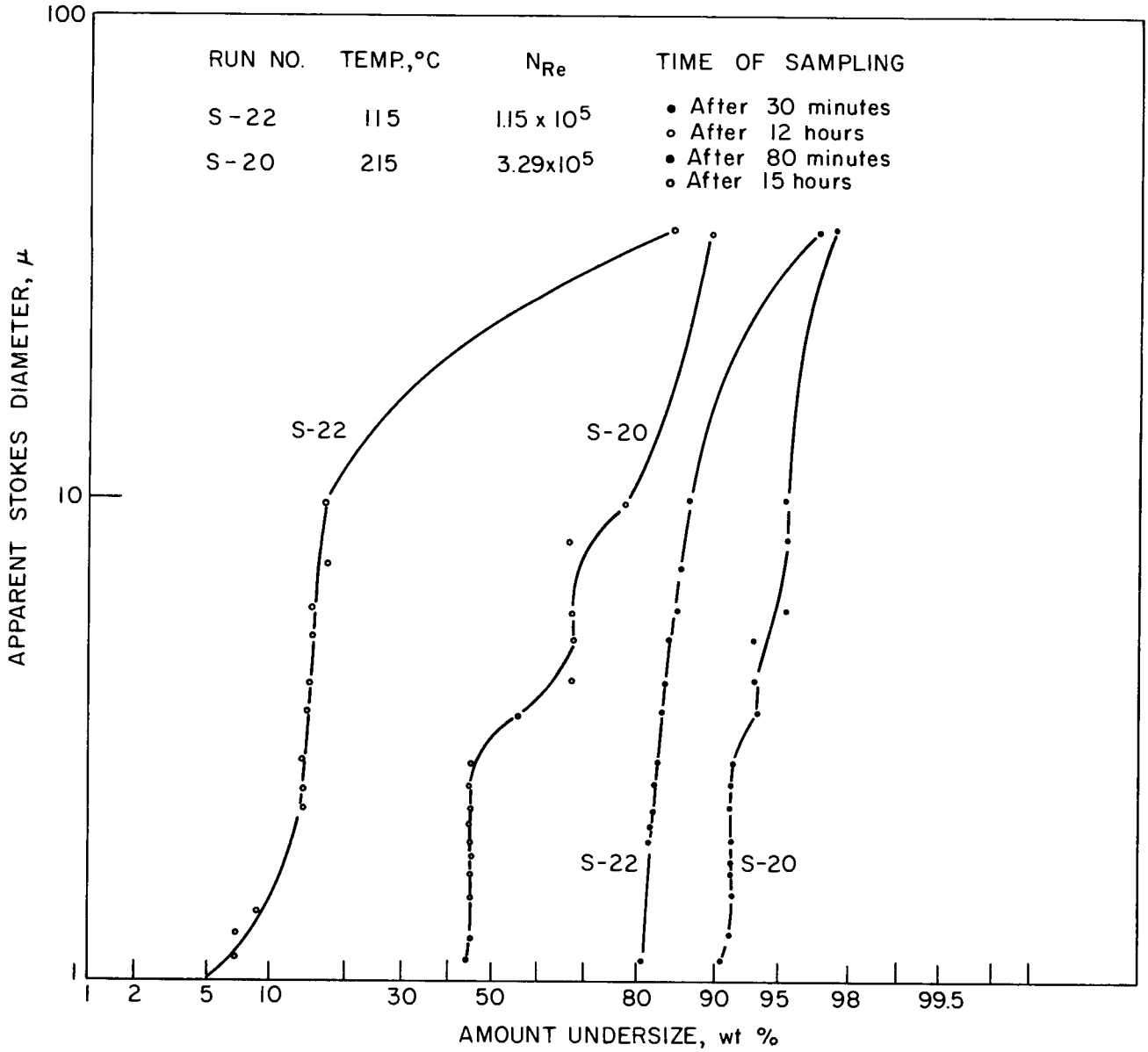


Fig. 7. Change in $ZrO_2$ Particle size during run.

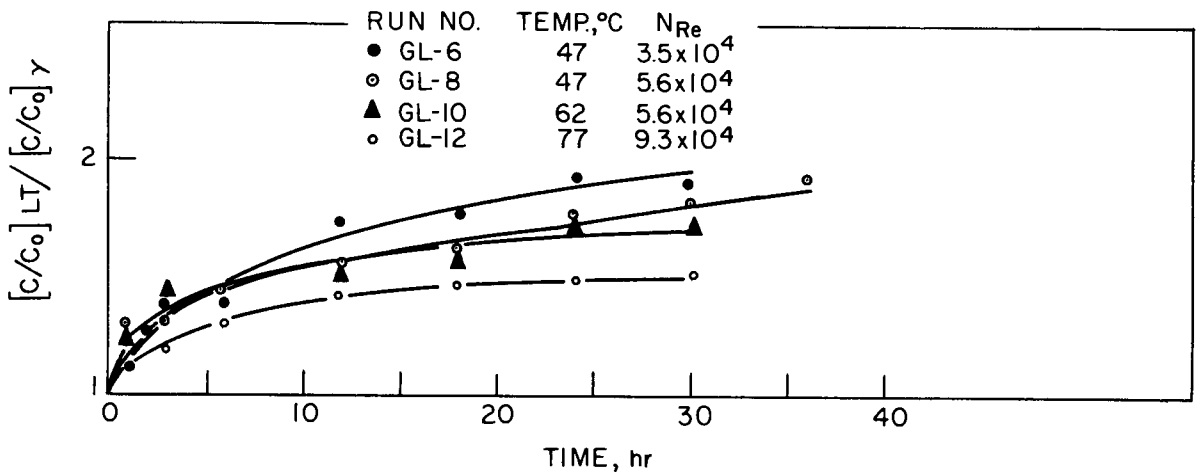


Fig. 8. Comparison of analytical techniques. Initial $ZrO_2$ concentration = 595 ppm.

Table 3. Velocities in the Various Sections of the Loops

| Pipe Size, in. | Surface Area, % of total | Velocity, fps 2 gpm | 4.8 gpm | 4.06 gpm | 3.6 gpm | 25.0 gpm |
|---|---|---|---|---|---|---|
| A loop, total surface area 3.26 $ft^2$ of which 1.43 $ft^2$ is in preheater and pressurizer circuit | | | | | | |
| 1/2 | 46.6 | | 6.6 | 5.5 | 4.9 | |
| 1/8 | 28.9 | | 42.4 | 35.3 | 31.8 | |
| 1/4 | 2.4 | | 20.4 | 17.0 | 15.3 | |
| 3 | 22.1 | | - | - | - | |
| B-2 loop, total surface area = 7.2 $ft^2$ | | | | | | |
| 4 | 59.0 | | | | | 0.59 |
| 1-1/2 | 18.8 | | | | | 4.53 |
| 3/4 | 16.4 | | | | | 18.54 |
| 1-1/4 | 4.7 | | | | | 6.26 |
| 3/8 | 1.1 | | | | | 72.50 |
| Glass loop, total surface area = 1.2 $ft^2$ | | | | | | |
| 1/2 | 65.7 | 3.28 | | | | |
| 3/8 | 11.8 | 5.84 | | | | |
| 1 | 8.5 | - | | | | |
| 1/4 | 7.8 | 13.1 | | | | |
| 0.197 | 6.2 | 20.2 | | | | |

Table 4. Reynolds Numbers Under Various Operating Conditions

| Pump Electrical Supply Frequency | Dia., in. | Velocity | Temp, °C | Viscosity, Centipoises | Vol., $ft^3$ | Re |
|---|---|---|---|---|---|---|
| A-loop | | | | | | |
| 80 | 1/2 | 6.57 fps | 260 | 0.026 | 0.0198 | $7.92 \times 10^5$ |
| | | | 215 | 0.049 | 0.01894 | $4.38 \times 10^5$ |
| | | | 115 | 0.21 | 0.01692 | $1.15 \times 10^5$ |
| 50 | 1/2 | 4.94 fps | 260 | 0.026 | 0.0198 | $5.95 \times 10^5$ |
| | | | 215 | 0.049 | 0.01894 | $3.29 \times 10^5$ |
| | | | 115 | 0.21 | 0.01692 | $8.65 \times 10^4$ |
| | | | 75°C | 0.40 | 0.01643 | $4.68 \times 10^4$ |
| $B_2$-loop | | | | | | |
| 60 | 1-1/2 | 25 gpm | 185 | 0.077 | | $3.8 \times 10^5$ |
| | 3/4 | | | | | $7.2 \times 10^5$ |
| | 1-1/4 | | | | | $4.4 \times 10^5$ |
| | 4 | | | | | $1.32 \times 10^5$ |
| | 3/8 | | | | | $15.6 \times 10^5$ |

Table 5. Material Balances

| Run No. | Solids In, (mg) A | Solids Recovered, mg Drain B | Wash C | Scrub D | Samples E | Total F | % Recovery G | Solids Circulating at End of Run H | % Circulating Solids Recovered by Loop Drain | Solids Deposited, mg/ft$^2$ $\frac{A-(E+H)}{\text{Surface Area}}$ (c) |
|---|---|---|---|---|---|---|---|---|---|---|
| S-15 | 550 | ND | ND | - | 86 | 86 | - | 26 | - | 74.5 |
| S-16 | 550 | ND | ND | - | 185 | 185 | - | 91 | - | 46.5 |
| S-17 | 550 | ND | ND | - | 118 | 118 | - | 53 | - | 64.3 |
| S-19 | 550 | ND | ND | - | 135 | 135 | - | 31 | - | 65.1 |
| S-20 | 550 | ND | ND | - | 117 | 117 | - | 35 | - | 67.5 |
| S-22 | 825 | ND | ND | - | 323 | 323 | - | 88 | - | 69.6 |
| S-23 | 1100 | ND | ND | - | 447 | 447 | - | 164 | - | 83.0 |
| S-29 | 1375 | ND | ND | - | 560 | 560 | - | 222 | - | 101.0 |
| S-30 | 1375 | ND | ND | - | 628 | 628 | - | 168 | - | 98.5 |
| S-31 | 2900 | ND | ND | - | 1380 | 1380 | - | 503 | - | 173.0 |
| S-34 | 1260 | 601 | ND | - | 383 | 908 | 72 | 80 | 750 | 135.0 |
| S-38 | 1210 | 120 | 474 | - | 271 | 939[a] | 77 | 45 | 267 | 156.0 |
| $B_2$-S-2 | 3620 | 2280 | 1456 | - | 358 | 4094 | 113 | 2810 | 81 | 45.6 |
| $B_2$-S-3 | 3620 | 2415 | 687 | - | 419 | 3686[b] | 102 | 3060 | 79 | - 2.4 |
| G1-6 | 274 | 68 | 19 | 52 | 55 | 194 | 71 | 88 | 77 | 108.6 |
| G1-7 | 274 | 116 | 14 | 31 | 57 | 218 | 79 | 128 | 91 | 74.1 |
| G1-8 | 274 | 135 | 26 | 23 | 79 | 263 | 96 | 131 | 103 | 53.4 |
| G1-9 | 274 | 212 | 14 | 23 | 64 | 313 | 114 | 138 | 154 | 60.0 |
| G1-10 | 274 | 151 | 23 | 17 | 66 | 257 | 94 | 147 | 103 | 51.0 |
| G1-11 | 274 | 140 | 25 | 21 | 70 | 256 | 94 | 133 | 105 | 59.2 |
| G1-12 | 274 | 164 | 13 | 14 | 76 | 267 | 98 | 182 | 90 | 13.3 |

a. 74 mg recovered from hydroclone underflow receiver
b. 165 mg recovered from hydroclone underflow receiver
c. Total surface area including pump rotor cavity of 5.9 ft$^2$ for Loop A, 9.9 ft$^2$ for Loop B, and 1.2 ft$^2$ for Glass Loop

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