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IN A LOW TEMPERATURE CAPILLARY TUBE VISCOMETER
TO: Distribution
FROM: R. H. Nimmo and A. S. Kitzes

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I. Summary

A compilation is presented of the rheological data for thorium oxide slurries which were obtained in a capillary tube viscometer. Tests were made with eight slurries at solids concentrations of 400 to 1500 g Th/kg H₂O and at temperatures from 25 to 150°C.

The flow curves of the slurries approximated Bingham plastics and yielded estimates of the characteristic properties, yield stress, τ_y , and coefficient of rigidity, η . Both τ_y and η increase with concentration at constant temperature. The degree of flocculation as measured by the apparent yield stresses of the slurries appeared to be changed by heating or simply by standing. In some cases the slurries became thinner when heated, while other slurries became thicker.

End effects were found to contribute a measurable part of the pressure drop in the horizontal viscometer tubes. For the two slurries tested, single pseudo-shear diagrams were obtained by plotting $8V/g_c D$ versus $D(\Delta P_{\text{long}} - \Delta P_{\text{short}})/4(L_{\text{long}} - L_{\text{short}})$. It may be concluded that either the slurries were not thixotropic or that thixotropic equilibrium was quickly established near the entrance to the tube.

II. Introduction

The rheological data for thorium oxide slurries obtained by P. R. Crowley and A. S. Kitzes in a low temperature pipe line viscometer have been reviewed as a basis for future work. Some of these results have been previously reported.⁵⁻¹¹ The purpose of the present report is to make the collection of data more readily available. Although some of these data may be considered of a preliminary survey nature, their dissemination is desirable since they constitute a substantial part of the available information on the rheology of thorium oxide slurries. Correlations of these experimental data and relationships reported in the literature for other slurries will be presented in a forthcoming report.

III. Experimental

A. Equipment

The low temperature slurry viscometer is essentially a blow case-capillary tube type unit. The feed tank is a conical bottomed, stainless steel vessel having a capacity of about 4 liters which is fitted with an agitator, a thermowell, external electric heaters, and a special angle-type drain valve at the bottom. The fitting at the outlet of the drain valve permits the use of tubes of a range of diameters. Table A-1 of the appendix lists the tubes which were employed in this work. Gas pressure, applied above the liquid in the feed tank, is varied to effect different flow rates through the opened drain valve and viscometer tube. The receiver tank, connected to the end of the length of viscometer tubing, is positioned on a weigh scale. The receiver is pressurized with gas when it is necessary to prevent vaporization of water from hot slurry. With the steady state flow established, the times required for the collection of incremental weights of effluent are determined.

B. Scope of the Test Program

This report covers work done in the period, October, 1954 through December, 1955. Viscometer tests were made on eight thorium oxide slurries which had been prepared from several different thorium oxide powders and which had been subjected to different conditions of operation. The temperature, run time, solids concentration and ppm $\text{SO}_4^{=}$ for the slurries from the loop runs and the source and firing temperatures of the powders are listed in Table I. More detailed histories of the loop runs may be found in ORNL Quarterly Reports. 7, 8, 9, 11

TABLE I
THORIUM OXIDE SLURRIES TESTED
IN LOW TEMPERATURE VISCOMETER

Slurry History					ThO ₂ Powder	
Loop Run No.	Loop Temp °C	Run Time hr.	Avg. Conc. g Th/kg H ₂ O	SO ₄ ⁼ Conc. ppm (1)	Source (2)	Firing Temp. °C
S-52	250	189	1070	300	Lindsay No. 7	900
S-54	250	318	500	4000	Lindsay No. 12	650
S-55	250	309	1200	5600	Lindsay No. 13	650
S-59	290	335	486	1300	D-12	650
T-48	250	298	600	400	Lindsay No. 11	650
D-13	Agitated in Tank	100	1000	100	D-13	650
D-25	Agitated in Tank	100	1000	300	D-25	800
200A-1	300	234	500	700	D-27	800

(1) SO₄⁼ Conc. ppm based on water

(2) Lindsay No. 7 Oxide was prepared from oxalate precipitated at 75°C and then fired at 900-950°C.

Lindsay No. 11 Oxide was prepared from formate calcined at 650°C.

Lindsay No. 12 Oxide was prepared from oxalate precipitated at 40°C and then calcined at 650°C.

Lindsay No. 13 Oxide was prepared from hot precipitated oxalate and fired at 650°C.

D-13, D-25, and D-27 Oxides were prepared from oxalate precipitated at 40°C and fired as shown in table.

The program included studies of the effects of solids concentration, temperature, and tube dimensions on the rheological properties of these thorium oxide slurries. Tests were made at 25°C with five of the slurries at solids concentrations ranging from 500 to 1500 g Th/kg H₂O. One slurry, 200A-1, was examined over the concentration range of 400 to 1000 g Th/kg H₂O at each of the temperatures 45, 55, and 75°C. Temperature effects were also observed on another slurry, at substantially one concentration, at 53, 75, 100, 125 and 145°C. The effects of tube length were investigated in tests at 25°C with each of the tubes in the two sets having inside diameters of 0.097 and 0.130 inches.

The viscometer tests are listed in Table A-2 of the appendix according to the tube employed, the slurry tested, the solids concentration, and the viscometer temperature. The letter-number references indicate the log book and the page where the individual run data are summarized.

C. Experimental Data

The experimental data consisted of the weight and volume of the slurry charge (from which the density and solids concentration were calculated), the gas pressure applied to the feed tank, and the time intervals for the collection of given weights of effluent in the receiver. The pressure drop across the capillary tube was the sum of the applied gas pressure and the average head of slurry existing above the entrance to the tube during each test interval. The latter was figured from a feed tank volume - height calibration and the running inventory of effluent collected.

IV. Experimental Results

A. Treatment of Data

The experimental data are presented in the form of pseudo-shear diagrams which customarily show the shear stress at the tube wall, $D\Delta P/4L$, plotted against a factor proportional to the average rate of shear, $8V/g_c D$. It should be noted that in all these data the pressure drops, ΔP , are the uncorrected total pressure differences across the tubes affecting flow and include end and kinetic energy effects. Flow curves have been drawn by eye through the data, with more concern being given to the points which exhibit a linear relationship between $8V/g_c D$ and $D\Delta P/4L$ than to the points at low rates of shear which form a curve towards the shear stress coordinate. Measurements at low rates of shear may be subject to phenomena such as settling, slip, and seepage^{3,4}.

Most of these flow curves indicate that the thorium oxide slurries possess yield stresses and that they approximate Bingham plastic type non-Newtonian behavior. Two exceptions are the flow curves for pumped slurries containing about 1000 g Th/kg H₂O which appear to go to the origin, characteristic of a pseudo-plastic material, see Fig. 1. For purposes of the aforementioned review the linear portions of the flow curves were extrapolated to the shear stress axis. According to a simplified form of Buckingham's integration of Bingham's differential equation for plastic flow through circular conduits, this intercept is $4/3\tau_y$ and the reciprocal of the slope, $\Delta(D\Delta P/4L)/\Delta(8V/g_c D)$ is the coefficient of rigidity, η .

$$\text{Bingham's equation}^3 - \frac{du}{dr} = \frac{g_c}{\eta} (\tau - \tau_y)$$

$$\text{Buckingham's equation}^4 \quad \frac{8V}{g_c D} = \frac{\tau_w}{\eta} \left[1 - \frac{4}{3} \frac{\tau_y}{\tau_w} + \frac{1}{3} \left(\frac{\tau_y}{\tau_w} \right)^4 \right] + \frac{8u_R}{g_c D}$$

$$\text{Simplified form} \quad \frac{8V}{g_c D} = \frac{1}{\eta} \left[\frac{D\Delta P}{4L} - \frac{4}{3} \tau_y \right]$$

where,

u = point linear velocity, ft/sec.

U_R = slip velocity, at wall, ft/sec.

V = average linear velocity, ft/sec.

r = any radius within tube, ft.

D = inside diameter of tube, ft.

g_c = conversion factor, (lb mass)(ft/lb force)(sec²).

η = coefficient of rigidity, lb mass/sq ft.

τ = shear stress at radius r , lb force/sq ft.

τ_w = shear stress at tube wall, lb force/sq ft; equals $D\Delta P/4L$.

τ_y = yield stress, lb force/sq ft.

L = tube length, ft.

ΔP = pressure difference along tube, lb force/sq ft.

The simplified form results from assumptions that there is no slip at the wall (U_R is zero) and that in the linear relationship region the contributions of the term, $\frac{1}{3} (\tau_y/\tau_w)^4$, are small. Babbitt and Caldwell² pointed out that the error in calculating velocity with the simplified form is 5.9% when τ_y/τ_w is 0.5, and 1.8% when τ_y/τ_w is 0.4. Lyon¹² has calculated this % error over the range τ_y/τ_w equals 0.1 to 1.0 and the error increases rapidly above 18% at τ_y/τ_w of 0.6; high τ_y/τ_w ratios correspond to points on the lower curved portions of the flow curves. Hence, the estimated value of $\frac{4}{3} \tau_y$ lies between τ_y and $\frac{4}{3} \tau_y$ and the expected error in τ_y does not exceed 25%.

B. Influence of Solids Concentration at 25°C

The pseudo-shear diagrams for the run made with the five different slurries, at solids concentration of 500-1500 g Th/kg H₂O are shown in Fig. 2,

3, 4, 5 and 6. The values of yield stress, γ_y , and of the coefficient of rigidity, η , for each of these curves are listed in Table II. The five sets of curves show the same general pattern in that the slopes of the straight lines gradually decrease as they become displaced to the right with increased solids concentration. Thus, both the yield stress and the coefficient of rigidity increase with increase in solids concentration.

C. Viscometer Tests at Temperatures above 25°C

The two slurries whose properties were measured at temperatures greater than 25°C were (1) 200A-1, an 800°C fired oxide the slurry from which was pumped 234 hours at 300°C and (2) D-13, a 650°C fired oxide which was agitated in a tank at room temperature for 100 hours. These will be considered in turn.

1. 200A-1 Slurry

Tests were made with this slurry at solids concentrations ranging from 400 to 1000 g Th/kg H₂O at temperatures of 25, 45, 55 and 75°C. Pseudo-shear diagrams for the data obtained in these tests are plotted in Fig. 4, 7, 8 and 9 respectively. The individual results are given in Table III. Despite the scatter of the data, the results for this thorium oxide slurry indicate that the yield stress, γ_y , increases and the coefficient of rigidity, η , decreases with increase in temperature, in the range 25 to 75°C.

2. D-13 Slurry

The pseudo-shear diagrams for the tests made with this slurry at 53, 75, 100, 125, and 145°C are shown in Fig. 10 and the calculated results are given in Table IV. The positions of the pseudo-shear curves indicate that the yield stress, γ_y , decreases with increase in temperature; this is contrary to the effect found with slurry 200A-1. The slopes (and, hence, the coefficients of rigidity) do not form a regular pattern. It may be noted that repeat runs, made at 25°C tests when the concentration was varied from 500 to 1500 g Th/kg H₂O.

TABLE II
 INFLUENCE OF SOLIDS CONCENTRATION ON YIELD STRESS, τ_y ,
 AND COEFFICIENT OF RIGIDITY, η , AT 25°C

Figure No.	Slurry	Concentration g Th/kg H ₂ O	Yield Stress lb _F /sq ft	Coeff. of Rigidity	
				lb _M /ft sec	Centipoise
2	S-59	450	0.097	0.00223	3.32
	Pumped	667	0.375	0.00333	4.96
	335 hr	790	0.622	0.00444	6.60
	at 290°C	877	0.930	0.00416	6.19
3	S-54	400	0.053	0.00166	2.47
	Pumped	610	0.188	0.00262	3.90
	318 hr	715	0.300	0.00303	4.51
	at 250°C	864	0.558	0.00344	5.12
		1072	1.245	0.00489	7.27
4	200A-1	308	0.009	0.00156	2.32
	Pumped	493	0.053	0.00217	3.23
	234 hr	670	0.135	0.00275	4.09
	at 300°C	830	0.277	0.00326	4.85
		920	0.368	0.00352	5.24
		1075	0.638	0.00371	5.52
5	D-13	490	0.037	0.00166	2.47
	Agitated	687	0.143	0.00227	3.38
	in Tank	848	0.262	0.00254	3.78
	100 hr	965	0.425	0.00299	4.45
		1150	0.788	0.00323	4.80
		1330	1.356	0.00469	6.98
		1535	2.040	0.00678	10.09
6	D-25	820	0.075	0.00213	3.17
	Agitated	1095	0.180	0.00284	4.23
	in Tank	1498	0.547	0.00398	5.91
	100 hr				

TABLE III

YIELD STRESSES AND COEFFICIENTS OF RIGIDITY OF 200A-1 SLURRYAT VARIOUS TEMPERATURES AND CONCENTRATIONS

Figure No.	Temp °C	Concentration g Th/kg H ₂ O	Yield Stress lb _T /sq ft	Coeff. of Rigidity	
				lb _M /ft sec	Centipoise
4	25	308	0.0086	0.00156	2.32
		493	0.0525	0.00217	3.23
		670	0.135	0.00275	4.09
		830	0.277	0.00326	4.85
		920	0.368	0.00352	5.24
		1075	0.638	0.00371	5.52
7	45	393	0.0225	0.00202	3.00
		545	0.0825	0.00223	3.32
		782	0.247	0.00360	5.36
		892	0.364	0.00385	5.73
		1030	0.570	0.00610	9.08
		1038	0.630	0.00510	7.59
		1175	0.930	0.00810	12.05
8	55	423	0.045	0.00184	2.74
		575	0.098	0.00229	3.41
		700	0.2025	0.00235	3.50
		782	0.281	0.00254	3.78
		867	0.4575	0.00334	4.97
		1060	0.728	0.00392	5.83
9	75	425	*	*	*
		465	0.060	0.00148	2.21
		547	0.097	0.00217	3.23
		572	0.090	0.00216	3.21
		708	0.210	0.00227	3.38
		863	0.394	0.00265	3.94
		945	0.518	0.00294	4.42
		1070	0.750	0.00410	6.10

* Insufficient Data

TABLE IV

INFLUENCE OF TEMPERATURE ON D-13 SLURRY

Temp °C	Concentration g Th/kg H ₂ O	Yield Stress lb _F /sq ft	Coeff. of Rigidity		Ref.
			lb _M /ft sec	Centipoises	No.
A. Tests Above 25°C (Fig. 10)					
53	820	0.372	0.00211	3.14	A-228
75	850	0.270	0.00236	3.51	A-241
100	860	0.090	0.00300	4.46	{A-231 A-258
100	856				
125	875	--	Insufficient Data		A-244
145	890	0.075	0.00225	3.35	A-237
B. Tests at 25°C (Fig. 11)					
25	687	0.1425	0.00227	3.38	A-138
25	783	0.0900	0.00340	4.06	A-252
25	824	0.2325	0.00264	3.93	A-234
25	848	0.2625	0.00254	3.78	A-148
25	855	0.1725	0.00283	4.20	A-247
25	856	0.0825	0.00283	4.20	A-255
25	965	0.420	0.00299	4.45	A-163

These irregular results are shown in the flow curves in Fig. 11 and in the values listed in Part B of Table IV. This behavior may be attributed to the influence of temperature and ageing on the flocculation of these slurries which, at the present time, are not clearly understood.

D. Effect of Tube Dimensions

For a Bingham type plastic the yield stress, γ_y , and the coefficient of rigidity, η , are characteristic properties of the material and are independent of the dimensions of the measuring system. However, this is not true of non-Newtonian materials possessing time-dependent properties. Thixotropic fluids, for example, show decreases in consistency with increased times of shear at a given shearing stress, $D\Delta P/4L$. Hence, the flow curves determined in a number of capillary tubes of the same diameter but of different lengths will show increased values of $8V/g_c D$ with increased tube length at a given shear stress. The figure by Alves¹ shows this trend and also the decrease in $8V/g_c D$ with increase of tube diameter at constant values of tube length and shear stress $D\Delta P/4L$.

Two slurries, T-48 and 200A-1, were tested at 25°C with tubes having inside diameters of 0.097-in. and 0.130-in., respectively, and different lengths, see Tables V and VI. The straight lines through the data from the tests with T-48 slurry, shown in Fig. 12, converge to substantially the same intercept but fan out with different slopes. In Fig. 13, the straight lines from the tests with the 200A-1 slurry show a similar pattern. As mentioned earlier, the pressure drops, ΔP , include end and kinetic energy effects in addition to laminar frictional resistance. It was shown¹¹ that when these effects were subtracted out, by plotting $8V/g_c D$ versus $D(\Delta P_L - \Delta P_S/4(L_L - L_S))$, a single flow curve for each slurry resulted. It may be concluded therefore that the slurries either were not thixotropic or that thixotropic equilibrium was quickly established near the tube entrance.

TABLE V

EFFECT OF DIFFERENT LENGTH TUBES WITH T-48 SLURRY

Tube No.	Tube Length in.	L/D Ratio	Slurry Concentration g Th/kg H ₂ O	Yield Stress lb _F /sq ft	Coeff. of Rigidity		Ref. No
					lb _M /ft sec	Centipoises	
A	60.09	620	596	0.323	0.00301	4.88	A-106
B	36.0	372	596	0.323	0.00366	5.44	A-115 A-119
C	24.25	250	596	0.323	0.00415	6.17	A-110

Inside Diameters, 0.097

TABLE VI

EFFECT OF DIFFERENT LENGTH TUBES WITH 200A-1 SLURRY

Tube No.	Tube Length in.	L/D Ratio	Slurry Concentration g Th/kg H ₂ O	Yield Stress lb _F /sq ft	Coeff. of Rigidity		Ref. No.
					lb _M /ft sec	Centipoises	
J	100	769	766	0.180	0.00271	4.03	A-210
I	60	461	775	0.175	0.00320	4.76	C-203
	60		772				C-220
H	36	277	772	0.176	0.00323	4.80	C-217
G	20	154	766	0.160	0.00405	6.02	C-209
F	8.9	69	770	0.160	0.00511	7.60	C-214

Inside Diameters, 0.130 in.

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APPENDIX

Table A-1	Viscometer Tube Dimensions
Table A-2	Slurries Tested in the Various Tubes
Figure 1	Pseudo-Shear Diagrams for Pumped Thorium Oxide Slurries at 25°C.
Figure 2	Influence of Concentration on Pseudo-Shear Diagrams for S-59 Slurry at 25°C.
Figure 3	Influence of Concentration on Pseudo-Shear Diagrams for S-54 Slurry at 25°C.
Figure 4	Influence of Concentration on Pseudo-Shear Diagrams for 200A-1 Slurry at 25°C.
Figure 5	Influence of Concentration on Pseudo-Shear Diagrams for D-13 Slurry at 25°C.
Figure 6	Influence of Concentration on Pseudo-Shear Diagrams for D-25 Slurry at 25°C.
Figure 7	Influence of Concentration on Pseudo-Shear Diagrams for 200A-1 Slurry at 45°C.
Figure 8	Influence of Concentration on Pseudo-Shear Diagrams for 200A-1 Slurry at 55°C.
Figure 9	Influence of Concentration on Pseudo-Shear Diagrams for 200A-1 Slurry at 75°C.
Figure 10	Influence of Temperature on Pseudo-Shear Diagrams for D-13 Slurry
Figure 11	Influence of Concentration on Pseudo-Shear Diagrams for D-13 Slurry at 25°C.
Figure 12	Influence of Tube Length on Pseudo-Shear Diagrams for T-48 Slurry at 25°C.
Figure 13	Influence of Tube Length on Pseudo-Shear Diagrams for 200A-1 Slurry at 25°C.

TABLE A-1

VISCOMETER TUBE DIMENSIONS

<u>Tube</u>	<u>Inside Diameter in.</u>	<u>Length in.</u>	<u>$\frac{L}{D}$ Ratio</u>
A	0.097	60.09	620
B	0.097	36.00	372
C	0.097	24.25	250
D	0.125	67.44	542
E	0.125	43.63	349
F	0.130	8.94	69
G	0.130	20.00	154
H	0.130	36.00	277
I	0.130	60.00	461
J	0.130	100.00	769

TABLE A-2

SLURRIES TESTED IN THE VARIOUS TUBES

<u>Slurry</u>	<u>Concentration</u> <u>g Th/kg H₂O</u>	<u>Temp</u> <u>°C</u>	<u>Ref.</u> <u>No.</u>	<u>Slurry</u>	<u>Concentration</u> <u>g Th/kg H₂O</u>	<u>Temp</u> <u>°C</u>	<u>Ref.</u> <u>No.</u>
<u>Tube A</u> (0.097 in I.D., 60.09 in. long)				<u>Tube B</u> (0.097 in. I.D., 36 in. long)			
T-48	596	20	A106	S-52	1152	25	A15
D-13	1330	25	A134		1135	28	A54
	687	25	A138				
	1535	27	A143	S-54	1072	25	A36
	848	26	A148		1072	25	A47
	490	27	A153		864	25	A70
	1150	25	A158		715	25	A74
	965	24	A163		610	25	A78
D-13	820	53	A228		400	25	A82
	860	100	A231				
	824	26	A234	T-48	596	20	A115
	890	145	A237		596	23	A119
	850	75	A241	T-48 and	596	23	A123
	875	125	A244	Oxalic Acid			
	855	25	A247				
	783	25	A252	S-55	1132	25	A41
	856	25	A255		1145	25	A44
	856	100	A258				
D-25	1095	25	A214	<u>Tube C</u> (0.097 in. I.D., 24.25 in. long)			
	820	26	A218	T-48	596	25	A110
	1498	26	A223				
				<u>Tube D</u> (0.125 in. I.D., 67.44 in. long)			
S-59	452	23	A169	200A-1	670	25	A262
	800	23	A174		1075	25	A266
	667	25	A179		830	25	A275
	877	27	A186		493	25	A278
	667	25	A189		308	25	A282
					920	25	A286
200A-1	1060	25	A271		892	45	B24

TABLE A-2 (continued)

<u>Slurry</u>	<u>Concentration</u> <u>g Th/kg H₂O</u>	<u>Temp</u> <u>°C</u>	<u>Ref.</u> <u>No.</u>	<u>Slurry</u>	<u>Concentration</u> <u>g Th/kg H₂O</u>	<u>Temp</u> <u>°C</u>	<u>Ref.</u> <u>No.</u>
<u>Tube D</u> (0.125 in. I.D., 67.44 in long)				<u>Tube F</u> (0.130 in. I. D., 8.94 in. long)			
	1030	45	B27	200A-1	770	25	C214
	1175	45	B30				
	545	45	B33	<u>Tube G</u> (0.130 in. I.D., 20 in. long)			
	1038	45	B36	200A-1	766	25	C206
	393	45	B39				
	782	45	842	<u>Tube H</u> (0.130 in. I.D., 36 in. long)			
	1060	55	B45	200A-1	772	25	C217
	782	55	B48				
	423	55	B51	<u>Tube I</u> (0.130 in. I.D., 60 in. long)			
	700	55	B54	200A-1	775	25	C203
	867	55	B55				
	575	55	B59		772	25	C220
200A-1	696	27	C64	<u>Tube J</u> (0.130 in. I.D., 100 in. long)			
				200A-1	766	25	C210

Fig. 1. Pseudo - Shear Diagrams for Pumped Thorium Oxide Slurries at 25°C.

Fig. 2. Influence of Concentration on Pseudo-Shear Diagrams
(For S-59 Slurry at 25°C)

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Fig. 3. Influence of Concentration on Pseudo-Shear Diagrams
(For S-54 Slurry at 25°)

Fig. 4. Influence of Concentration on Psuedo-Shear Diagrams.
(For 200A-1 Slurry at 25°C)

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Fig. 5. Influence of Concentration on Pseudo-Shear Diagrams.
(For D-13 Slurry at 25°C)

Fig. 6. Influence of Concentration on Pseudo-Shear Diagrams.
(For D-25 Slurry at 25°C)

Fig. 7. Influence of Concentration on Pseudo-Shear Diagram,
(For 200A-1 Slurry at 45°C)

Fig. 8. Influence of Concentration on Pseudo-Shear Diagram.
(For 200A-1 Slurry at 55°C)

Fig. 9. Influence of Concentration on Pseudo-Shear Diagram
(For 200 A-1 Slurry at 75°C)

Fig. 10. Influence of Temperature on Pseudo-Shear Diagrams
(For D-13 Slurry)

Fig. 41. Influence of Concentration on Pseudo-Shear Diagrams.
(For D-13 Slurry at 25°C)

Fig. 12 Influence of Tube Length on Pseudo-Shear Diagrams
(For T-48 Slurry at 25°C)

Fig.13. Influence of Tube Length on Pseudo-Shear Diagram.
(For 200 A-1 Slurry at 25°C)