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WSRC-TR-2004-00082
Revision 0

RHEOLOGICAL MODIFIER TESTING WITH DWPF PROCESS SLURRIES

M. E. Stone
A. R. Marinik
D. M. Marsh

February 2004

Immoblization Technology Section
Savannah River Technology Center
Aiken, SC 29808

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EXECUTIVE SUMMARY

Rheological modification agents were tested on simulated SRAT and SME products to determine if a suitable agent could be found for the DWPF process slurries. The agents tested were dispersants that lower the rheological properties of slurries by preventing agglomerization. Dolapix CE64, an ethylene glycol, and Disperse-Ayd W28, a polyacrylate, were the most effective dispersants tested.

Further evaluation and testing should be performed on Dolapix CE64 and Disperse-Ayd W28 to determine if implementation is possible in DWPF. The initial phase of future work will include optimization of the rheology modifier by the Illinois Institute of Technology (IIT) and development of a maximum concentration limit for the rheology modifiers. IIT has been commissioned to evaluate the properties of these chemicals to determine if the chemical makeup can be optimized to enhance the properties of these modifiers. An initial concentration limit based upon the DWPF flammability limit and other constraints should be calculated to determine the potential downstream impacts.

Once an optimized rheology modifier has been recommended by IIT, the second phase of future work concerning rheology modifiers should focus on the following areas:

- Effective pH range of dispersants
- Effect of boiling on dispersants
- Effect of aging on dispersants
- Impact of dispersants on foaming
- Impact of dispersants on hydrogen generation
- Impact of dispersants on glass redox
- Impact of dispersant components (such as ammonia) on process

It is anticipated that the second phase of this program will start in FY05.

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LIST OF ACRONYMS

DWPF	Defense Waste Processing Facility
HLW	High Level Waste
IIT	Illinois Institute of Technology
MFT	Melter Feed Tank
SME	Slurry Mix Evaporator
SRAT	Sludge Receipt Adjustment Tank

1.0 INTRODUCTION

The Defense Waste Processing Facility (DWPF) at the Savannah River Site (SRS) vitrifies high level radioactive waste (HLW) sludge currently stored at the site. DWPF receives washed HLW sludge in the Sludge Receipt Adjustment Tank (SRAT) on top of the heel from the previous batch. The sludge is then acidified by adding nitric and formic acid. Mercury is then removed from the sludge by reflux boiling through a decanter, and then the sludge is concentrated by evaporation. The concentrated sludge is transferred to the Slurry Mix Evaporator (SME). In the SME, the sludge is combined with the glass formers which are transferred to the vessel as a frit slurry. The mixture is then evaporated to remove water and decrease the heat load on the melter. SME product is transferred to the Melter Feed Tank (MFT) which slowly feeds the slurry to the melter.

Process upsets have occurred in the DWPF as a result of changes in the rheological properties of the process slurries. Reducing the yield stress of the process slurries is expected to minimize process upsets and to potentially allow higher solids loading in the SME product. Higher solids loading would decrease the amount of water the melter would be required to evaporate and could increase the glass production rate.

In commercial industries, dispersion agents are commonly used to decrease the yield stress of slurries. The dispersion agents prevent agglomerization of the particles in the slurry either by affecting the surface charge of the particles or by blocking the interaction between the particles through steric effects. Commercially available dispersants are usually effective in a narrow pH range and can act as flocculating agents outside the effective range. Many dispersion agents are based on acrylic polymers with variations in polymer chain length and the addition of other, often proprietary, species to the polymer.

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2.0 DISCUSSION

Scoping studies conducted during the summer of 2001[1] indicated that addition of a rheological modifier could reduce the yield stress and consistency of simulated waste sludges. Based on this preliminary study, additives were tested with DWPF process slurries to determine if the yield stress and consistency could be reduced. A batch of simulated SRAT product produced during FY02 melt rate testing was utilized to perform the tests. SME product was prepared from this SRAT product by addition of dry frit to the SRAT product. A Haake RS600 research grade rheometer was utilized to measure the rheological properties of the samples using the concentric cylinder geometry. Yield stress and consistency were determined from the data using the Bingham plastic model as described by Koopman.[2]

The additives selected for testing are shown in Table 1, along with additive type and manufacturer. Each additive was tested in SRAT and SME product at various concentrations, as shown in Appendix A.

Table 1. Rheological Modifiers Tested

Additive	Type	Manufacturer
Sodium Meta-Silicate	Crystalized silicate	Various
Sodium Polyphosphate	Phosphate polymer	Various
Darvan 7	Polymethylacrylate, anionic	Vanderbilt Co. Inc.
Duramax 3005	Ammonium Polyacrylate	Rohm and Haas
Dolapix CE64	Proprietary Ethylene Glycol	Zschimmer and Schwartz
Disperse-Ayd W22	Proprietary Polyacrylate	Elementis Specialties
Disperse-Ayd W28	Proprietary Polyacrylate	Elementis Specialties
Disperse-Ayd W30	Proprietary Polyacrylate	Elementis Specialties
Disperse-Ayd W39	Proprietary Polyacrylate	Elementis Specialties
Alcosperse 149	Sodium polyacrylate	Alco Chemical
Alcosperse 240	Proprietary Polyacrylate	Alco Chemical
Alcosperse 408	Proprietary Polyacrylate	Alco Chemical
Alcosperse 725	Proprietary Polyacrylate	Alco Chemical
EDA Plan 470	Proprietary Polyacrylate	Ultra Additives
EDA Plan 472	Proprietary Polyacrylate	Ultra Additives
Pomosperse AL36	Proprietary Polyacrylate	Piedmont Chemical Co.
Cyanamer P-35	Proprietary Polyacrylate	Cytec
Cyanamer P-70	Proprietary Polyacrylate	Cytec
Sugar	Glucose	Various

[1] E. D. Kay, et al. "Rheological Modifiers for Radioactive Waste Slurries", WSRC-MS-2003-00136, July 2003.
[2] D. C. Koopman and D. H. Miller, Production of Simulated Sludge Batch 2 Melter Feed Containing Frit 320 in the 1/240th Glass Feed Preparation System, WSRC-TR-2002-00186, Revision 0, April 22, 2002.

The majority of the additives tested led to increased yield stress and consistency, but several led to significant improvements. The results from the two most effective dispersants are shown in Table 2. The variation in the amount of improvement at a given concentration may be the result of differences in pH in the slurries tested since the same SRAT and SME products were not utilized for all tests. pH was not measured during these tests, but will be added to the test protocol for any future testing.

Table 2. Data from Dolapix CE64 and Disperse-Ayd W28 Tests

Additive	Sample Type	Concentration of Additive in Sample	Yield Stress	Consistency	% Improvement in Yield Stress from Baseline
		PPM	Pa	cP	%
Dolapix CE64 Proprietary Ethylene Glycol	SRAT	747	10.8	13.3	8.6
	SRAT	1492	10.2	13.3	13
	SME	1621	16.4	20.1	-4.1
	SME	3234	13.8	18.8	13
	SRAT	3234	2.5	8.9	35
	SRAT	6436	2.3	8.6	41
	SRAT	9606	2.1	8.6	46
	SRAT	12745	1.7	8.3	56
	SRAT	649	10.4	14.8	-1.3
	SRAT	1621	9.2	14.3	10
	SRAT	3234	8.0	14.6	22
	SME	649	9.70	0.023	7.6
	SME	1621	9.66	0.023	8.0
	SME	3234	8.24	0.019	22
Disperse-Ayd W28 Proprietary Polyacrylate	SRAT	2289	1.8	9.0	52
	SRAT	4554	1.5	5.2	62
	SRAT	6798	0.82	7.5	79
	SRAT	9020	0.60	7.6	85
	SRAT	460	9.5	12.4	6.8
	SRAT	1147	9.6	10.2	6.7
	SRAT	2289	10.4	8.1	-1.4
	SME	460	10.8	20	-3.1
	SME	1147	8.6	17	18
	SME	2289	6.1	15	42

The impact of water additions on the SME products was tested to compare to the improvement in yield stress provided by the Dolapix CE64 and Disperse-Ayd W28. Water additions reduced the yield stress, but required much larger addition amounts than the dispersant. For a reduction of 20% in yield stress, the amount of dispersant was less than 1/10th the amount of water that would be required. The impact of water is shown in Figure 1.

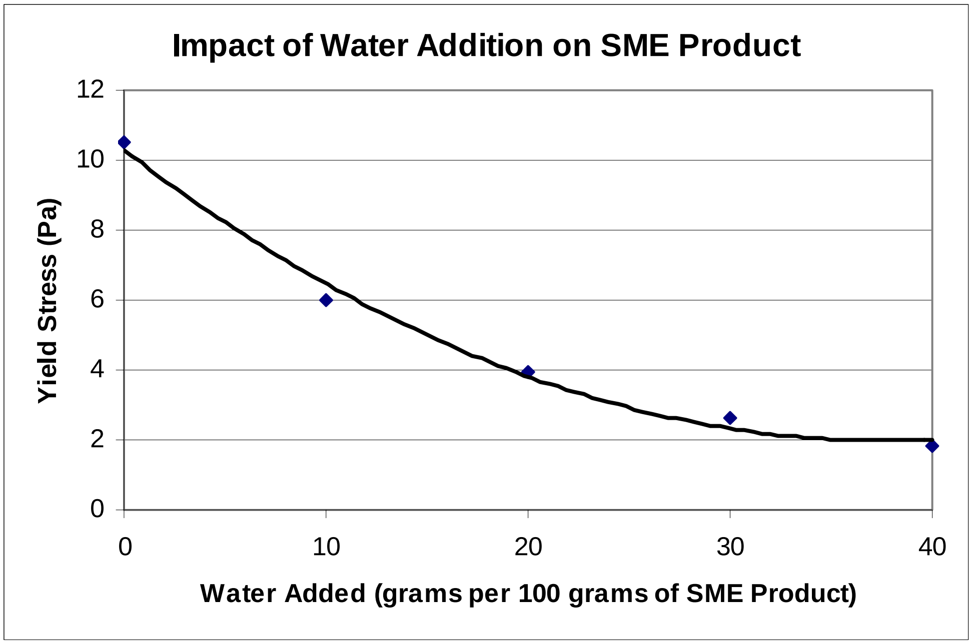

Figure 1. Impact of Water Additions on Yield Stress of SME Product

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3.0 CONCLUSIONS

Dolapix CE64 and Disperse-Ayd W28 were the most effective dispersants tested at reducing the yield stress of the SRAT and SME products tested. The effectiveness of the additives was likely impacted by the pH of the sample and varied considerably. The addition amount required by the dispersants was less than $1/10^{th}$ that of water to achieve similar reductions in yield stress for the SME product.

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4.0 RECOMMENDATIONS

Further evaluation and testing should be performed on Dolapix CE64 and Disperse-Ayd W28 to determine if implementation is possible in DWPF. The initial phase of future work will include optimization of the rheology modifier by IIT and development of a maximum concentration limit for the rheology modifiers. IIT has been commissioned to evaluate the properties of these chemicals to determine if the chemical makeup can be optimized to enhance the properties of these modifiers. An initial concentration limit based upon the DWPF flammability limit and other constraints should be calculated to determine the potential downstream impacts.

Once an optimized rheology modifier has been recommended by IIT, the second phase of future work concerning rheology modifiers should focus on the following areas:

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It is anticipated that the second phase of this program will start in FY05.

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APPENDIX A

Data From Rheological Modifier Testing

Sample ID	Sample Type	Additive	Additive Amount	Active Agent Concentration	Active Concentration in Sample	Yield Stress	Consistency	Yield Stress % of Baseline
			grams	wt %	ppm	Pascals	cP	
Rheo-12	SRAT	None	0	0	0	11.7	12.80	0.00
Rheo-13	SRAT	Sodium Polyphosphate	0.5	100	3444	17.0	14.90	(45.27)
Rheo-14	SRAT	Sodium Polyphosphate	1	100	6863	20.7	16.80	(76.81)
Rheo-15	SME	None	0	0	0	5.4	11.40	0.00
Rheo-16	SME	Sodium Polyphosphate	0.25	100	1719	6.3	13.00	(16.45)
Rheo-17	SME	Sodium Polyphosphate	0.5	100	3444	6.2	12.40	(13.86)
Rheo-18	SME	Sodium Polyphosphate	1	100	6863	6.7	12.50	(23.11)

Sample ID	Sample Type	Additive	Additive Amount	Active Agent Concentration	Active Concentration in Sample	Yield Stress	Consistency	Yield Stress % of Baseline
				wt %	ppm	Pascals	cP	
Rheo-45	SRAT	None	0.000	0	0	11.8	14.20	0.00
Rheo-46	SME	None	0.000	0	0	15.8	19.10	0.00
Rheo-47	SRAT	Darvan 7	0.115	25	287	11.8	14.50	(0.42)
Rheo-48	SRAT	Darvan 7	0.230	25	574	12.4	14.90	(5.61)
Rheo-49	SME	Darvan 7	0.250	25	623	17.7	20.70	(12.30)
Rheo-50	SME	Darvan 7	0.500	25	1244	17.8	20.90	(12.87)
Rheo-51	SRAT	Duramax 3005	0.115	35	402	13.5	14.80	(14.44)
Rheo-52	SRAT	Duramax 3005	0.230	35	803	13.9	14.80	(18.18)
Rheo-53	SME	Duramax 3005	0.250	35	873	17.4	19.40	(10.27)
Rheo-54	SME	Duramax 3005	0.500	35	1741	17.8	19.40	(12.56)
Rheo-55	SRAT	Sodium Metasilicate	0.115	100	1149	11.5	14.20	2.04
Rheo-56	SRAT	Sodium Metasilicate	0.230	100	2295	11.2	13.40	4.50
Rheo-57	SME	Sodium Metasilicate	0.250	100	2494	16.2	19.30	(2.73)
Rheo-58	SME	Sodium Metasilicate	0.500	100	4975	15.5	16.50	1.52
Rheo-59	SRAT	Dolapix CE64	0.115	65	747	10.8	13.30	8.58
Rheo-60	SRAT	Dolapix CE64	0.230	65	1492	10.2	13.30	13.25
Rheo-61	SME	Dolapix CE64	0.250	65	1621	16.4	20.10	(4.12)
Rheo-62	SME	Dolapix CE64	0.500	65	3234	13.8	18.80	12.68

Sample ID	Sample Type	Additive	Additive Amount	Active Agent Concentration	Active Concentration in Sample	Yield Stress	Consistency	Yield Stress % of Baseline
			wt. %	wt %	ppm	Pascals	cP	
Rheo-90	SRAT	None	0.0	0	0	3.9	9.20	0.00
Rheo-91	SRAT	Sodium Metasilicate	0.5	100	4975	3.9	8.40	(0.64)
Rheo-92	SRAT	Sodium Metasilicate	1.0	100	9901	5.7	10.10	(47.85)
Rheo-93	SRAT	Sodium Metasilicate	1.5	100	14778	5.3	10.30	(36.30)
Rheo-94	SRAT	Sodium Metasilicate	2.0	100	19608	3.9	8.40	0.03
Rheo-95	SRAT	Dolapix CE64	0.5	65	3234	2.5	8.90	34.73
Rheo-96	SRAT	Dolapix CE64	1.0	65	6436	2.3	8.60	40.83
Rheo-97	SRAT	Dolapix CE64	1.5	65	9606	2.1	8.60	45.60
Rheo-98	SRAT	Dolapix CE64	2.0	65	12745	1.7	8.30	56.41
Rheo-99	SRAT	Disperse-Ayd W22	0.5	30	1493	4.1	10.90	(4.30)
Rheo-100	SRAT	Disperse-Ayd W22	1.0	30	2970	3.7	8.10	5.33
Rheo-101	SRAT	Disperse-Ayd W22	1.5	30	4433	2.9	8.80	25.31
Rheo-102	SRAT	Disperse-Ayd W22	2.0	30	5882	2.8	10.70	27.39
Rheo-103	SRAT	Disperse-Ayd W28	0.5	46	2289	1.8	9.00	52.42
Rheo-104	SRAT	Disperse-Ayd W28	1.0	46	4554	1.5	5.20	62.36
Rheo-105	SRAT	Disperse-Ayd W28	1.5	46	6798	0.8	7.50	78.86
Rheo-106	SRAT	Disperse-Ayd W28	2.0	46	9020	0.6	7.60	84.55
Rheo-107	SRAT	Sugar	0.1	100	999	3.4	14.50	11.79
Rheo-108	SRAT	Sugar	0.2	100	1996	3.4	19.70	13.21
Rheo-109	SRAT	Sugar	0.3	100	2991	3.7	9.40	4.62
Rheo-110	SRAT	Sugar	0.5	100	4975	3.7	8.90	3.58

Sample ID	Sample Type	Additive	Additive Amount	Active Agent Concentration	Active Concentration in Sample	Yield Stress	Consistency	Yield Stress % of Baseline
			grams	wt %	ppm	Pascals	cP	
Rheo-111	SRAT	None	0	0	0	10.2	14.20	0.00
Rheo-112	SRAT	Dolapix CE64	0.1	65	649	10.4	14.84	(1.27)
Rheo-113	SRAT	Dolapix CE64	0.25	65	1621	9.2	14.30	9.96
Rheo-114	SRAT	Dolapix CE64	0.5	65	3234	8.0	14.59	21.91
Rheo-115	SRAT	Disperse-Ayd W28	0.1	46	460	9.5	12.42	6.80

Rheo-116	SRAT	Disperse-Ayd W28	0.25	46	1147	9.6	10.15	6.71
Rheo-117	SRAT	Disperse-Ayd W28	0.5	46	2289	10.4	8.08	(1.40)
Rheo-118	SRAT	Disperse-Ayd W30	0.1	33.5	335	10.2	13.64	0.25
Rheo-119	SRAT	Disperse-Ayd W30	0.25	33.5	835	9.7	13.10	4.99
Rheo-120	SRAT	Disperse-Ayd W30	0.5	33.5	1667	10.3	13.10	(0.10)
Rheo-121	SRAT	Disperse-Ayd W39	0.1	44	440	10.4	10.15	(1.72)
Rheo-122	SRAT	Disperse-Ayd W39	0.25	44	1097	8.6	9.60	15.92
Rheo-123	SRAT	Disperse-Ayd W39	0.5	44	2189	11.0	11.92	(7.49)
Rheo-124	SRAT	Alcosperse 149	0.1	40	400	11.0	1.17	(7.64)
Rheo-125	SRAT	Alcosperse 149	0.25	40	998	11.7	112.11	(14.28)
Rheo-126	SRAT	Alcosperse 149	0.5	40	1990	13.6	13.30	(32.49)
Rheo-127	SRAT	Alcosperse 240	0.1	44	440	13.0	10.20	(27.07)
Rheo-128	SRAT	Alcosperse 240	0.25	44	1097	12.4	11.05	(21.21)
Rheo-129	SRAT	Alcosperse 240	0.5	44	2189	12.1	12.60	(18.11)
Rheo-130	SRAT	Alcosperse 408	0.1	41	410	10.4	14.81	(1.94)
Rheo-131	SRAT	Alcosperse 408	0.25	41	1022	10.0	18.22	2.09
Rheo-132	SRAT	Alcosperse 408	0.5	41	2040	12.0	18.46	(17.09)
Rheo-133	SRAT	Alcosperse 725	0.1	35	350	11.9	13.52	(15.80)
Rheo-134	SRAT	Alcosperse 725	0.25	35	873	14.2	10.06	(38.65)
Rheo-135	SRAT	Alcosperse 725	0.5	35	1741	16.1	6.59	(57.59)
Rheo-136	SRAT	EDAPlan 470	0.1	50	500	11.1	13.99	(8.55)
Rheo-137	SRAT	EDAPlan 470	0.25	50	1247	10.9	14.27	(6.87)
Rheo-138	SRAT	EDAPlan 470	0.5	50	2488	10.9	14.40	(6.15)
Rheo-139	SRAT	EDAPlan 472	0.5	50	2488	10.4	13.13	(1.67)
Rheo-140	SRAT	EDAPlan 472	0.1	50	500	10.8	14.07	(5.67)
Rheo-141	SRAT	EDAPlan 472	0.25	50	1247	10.6	13.82	(3.99)
Rheo-142	SRAT	PomoSperse AL36	0.1	42	420	10.4	12.60	(1.27)
Rheo-143	SRAT	PomoSperse AL36	0.1	42	420	13.1	12.07	(28.25)
Rheo-144	SRAT	PomoSperse AL36	0.25	42	1047	16.7	10.88	(63.38)

Sample ID	Sample Type	Additive	Additive Amount	Active Agent Concentration	Active Concentration in Sample	Yield Stress	Consistency	Yield Stress % of Baseline
			grams	wt %	ppm	Pascals	cP	
Rheo-145	SME	none	0	0	0	10.5	20.00	0.00
Rheo-146	SME	Dolapix CE64	0.1	65	649	9.7	23.00	7.63
Rheo-147	SME	Dolapix CE64	0.25	65	1621	9.7	23.00	8.00
Rheo-148	SME	Dolapix CE64	0.5	65	3234	8.2	19.00	21.57
Rheo-149	SME	Disperse-Ayd W28	0.1	46	460	10.8	20.00	(3.08)
Rheo-150	SME	Disperse-Ayd W28	0.25	46	1147	8.6	17.00	18.32
Rheo-151	SME	Disperse-Ayd W28	0.5	46	2289	6.1	15.00	42.12
Rheo-152	SME	Disperse-Ayd W30	0.1	33.5	335	11.2	23.00	(6.29)
Rheo-153	SME	Disperse-Ayd W30	0.25	33.5	835	10.7	19.00	(1.43)
Rheo-154	SME	Disperse-Ayd W30	0.5	33.5	1667	10.9	19.00	(3.33)
Rheo-155	SME	Disperse-Ayd W39	0.1	44	440	10.5	21.00	(0.10)
Rheo-156	SME	Disperse-Ayd W39	0.25	44	1097	9.7	19.00	7.52
Rheo-157	SME	Disperse-Ayd W39	0.5	44	2189	10.5	18.00	0.29
Rheo-158	SME	Alcosperse 149	0.1	40	400	12.7	24.15	(20.57)
Rheo-159	SME	Alcosperse 149	0.25	40	998	13.1	23.89	(24.67)
Rheo-160	SME	Alcosperse 149	0.5	40	1990	12.5	23.99	(18.57)
Rheo-161	SME	Alcosperse 240	0.1	44	440	13.2	21.00	(25.33)
Rheo-162	SME	Alcosperse 240	0.25	44	1097	17.8	15.00	(69.14)
Rheo-163	SME	Alcosperse 240	0.5	44	2189	18.4	14.00	(74.76)
Rheo-164	SME	Alcosperse 408	0.1	41	410	11.9	23.00	(13.62)
Rheo-165	SME	Alcosperse 408	0.25	41	1022	14.2	20.00	(35.62)
Rheo-166	SME	Alcosperse 408	0.5	41	2040	16.0	23.00	(52.38)
Rheo-167	SME	Alcosperse 725	0.1	35	350	12.2	230.00	(16.38)
Rheo-168	SME	Alcosperse 725	0.25	35	873	13.0	24.11	(23.52)
Rheo-169	SME	Alcosperse 725	0.5	35	1741	11.5	25.36	(9.81)
Rheo-170	SME	EDAPlan 470	0.1	50	500	11.7	24.00	(10.95)
Rheo-171	SME	EDAPlan 470	0.25	50	1247	11.4	21.00	(8.76)
Rheo-172	SME	EDAPlan 470	0.5	50	2488	12.6	23.00	(19.71)
Rheo-173	SME	EDAPlan 472	0.1	50	500	11.9	22.00	(13.24)
Rheo-174	SME	EDAPlan 472	0.25	50	1247	11.5	24.00	(9.71)

Rheo-175	SME	EDAPlan 472	0.5	50	2488	12.1	22.00	(14.86)
Rheo-176	SME	PomoSperse AL36	0.1	42	420	11.8	22.00	(12.48)
Rheo-177	SME	PomoSperse AL36	0.25	42	1047	15.3	20.00	(46.10)
Rheo-178	SME	PomoSperse AL36	0.5	42	2090	18.1	18.00	(72.29)
Rheo-179	SME	Water	10	100	90,909	6.0	17.00	43.05
Rheo-180	SME	Water	20	100	166,667	4.0	13.00	62.38
Rheo-181	SME	Water	30	100	230,769	2.6	11.00	75.05
Rheo-182	SME	Water	40	100	285,714	1.8	10.00	82.57

Sample ID	Sample Type	Additive	Additive Amount	Active Agent Concentration	Active Concentration in Sample	Yield Stress	Consistency	Yield Stress % of Baseline
			grams	wt %	ppm	Pascals	cP	
Rheo-183	SME	Cyanamer P-35	0.10	50	500	17.0	20.00	(62.29)
Rheo-184	SME	Cyanamer P-35	0.25	50	1247	27.9	3.89	(165.71)
Rheo-185	SME	Cyanamer P-35	0.50	50	2488	33.5	-4.70	(219.43)
Rheo-186	SME	Cyanamer P-70	0.10	49.8	498	13.4	24.48	(27.24)
Rheo-187	SME	Cyanamer P-70	0.25	49.8	1242	13.7	21.64	(30.48)
Rheo-188	SME	Cyanamer P-70	0.50	49.8	2478	11.4	20.62	(8.76)
Rheo-189	SRAT	Cyanamer P-35	0.10	50	500	12.8	14.51	(24.51)
Rheo-190	SRAT	Cyanamer P-35	0.25	50	1247	21.6	9.43	(110.45)
Rheo-191	SRAT	Cyanamer P-35	0.50	50	2488	37.8	-9.94	(269.43)
Rheo-192	SRAT	Cyanamer P-70	0.10	49.8	498	10.8	13.98	(5.37)
Rheo-193	SRAT	Cyanamer P-70	0.25	49.8	1242	11.3	14.03	(9.86)
Rheo-194	SRAT	Cyanamer P-70	0.50	49.8	2478	11.3	13.21	(10.25)