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RECEIVED**MAR 22 1996****OSTI****SMALL-SCALE HIGH TEMPERATURE MELTER-1 (SSHTM-1) DATA PACKAGE****APPENDIX B - Alternate HTM Flowsheet 2 (Glycolic Acid Addition)****DISCLAIMER**

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QA LEVEL

This work was performed to satisfy PNL FY 1995 milestone number PVTB-C95-02.02Y and was conducted in accordance with Impact Level II quality assurance requirements as defined by the PVTB QA Plan ETC-001 and the PNL QA Manual PNL-MA-70.

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FIGURES	bii
TABLES	biii
LOGS	bv
1.0 INTRODUCTION	B1
2.0 OUTLINE	B1
3.0 LABORATORY MELTER FEED PREPARATION DATA	B2
3.1 Flowsheet Outline, Activity Data Log, and Gram Waste Oxide Tracking Sheet	B2
3.2 Melter Feed and Condensate Composition	B12
3.3 Off-Gas Data	B14
3.4 Rheology and Gram Waste Oxide Loading Data for Melter Feed . .	B18
3.4.1 Rheology of Preliminary Laboratory-Scale Feed Preparation and Aging Samples	B19
3.4.2 Rheology of Laboratory-Scale Modified Feed Preparation Samples	B33
3.5 Standard Crucible Redox Test Data	B39
4.0 SMALL-SCALE MELTER FEED PREPARATION DATA	B41
4.1 Flowsheet Outline and Activity Data Log	B41
4.2 Melter Feed and Condensate Composition	B61
4.3 Off-Gas Data	B64
4.4 Rheology and Gram Waste Oxide Loading Data for Melter Feed . .	B68
4.4.1 Rheology of Small-Scale Feed Preparation and Aging Samples	B69
4.4.2 Rheology of Small-Scale Feed Samples Aged in the Laboratory at 50°C	B131
4.4.3 Rheology of Small-Scale Feed Samples Aged in the Laboratory at 25°C	B149
4.5 Standard Crucible Redox Test Data	B167
5.0 REFERENCES	B168

FIGURES

Figure B3.3.1	Process Profile for Laboratory-Scale Alternate HTM Flowsheet 2, Test T95-SSHTMGLYC-1, for the First 300 Minutes Measured from the Start of Glycolic Acid Addition	B16
Figure B3.3.2	Process Profile for Laboratory-Scale Alternate HTM Flowsheet 2, Test T95-SSHTMGLYC-1, for 0 to 600 Minutes Measured from the Start of Glycolic Acid Addition	B17
Figure B4.3.1	Process Profile for Small-Scale Alternate HTM Flowsheet 2 for the First 300 Minutes Measured from the Start of Glycolic Acid Addition	B66
Figure B4.3.2	Process Profile for Small-Scale Alternate HTM Flowsheet 2 for 0 to 600 Minutes Measured from the Start of Glycolic Acid Addition	B67

TABLES

Table B3.1	Laboratory-Scale Melter Feed Gram Waste Oxide (gWO) Tracking Sheet for Test T95-SSHTMGLYC-1	B11
Table B3.2	Summary of Analytical Data for the Laboratory-Scale, Alternate HTM Flowsheet 2 Test, T95-SSHTMGLYC-1 .	B13
Table B3.3	Peak Off-Gas Generation Rates and Total Off-Gas Generated for Laboratory-Scale Alternate HTM Flowsheet 2 Tests	B15
Table B3.4.1	Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Preliminary Laboratory-Scale Melter Feed Preparation and Aging Samples	B20
Table B3.4.2	Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Modified Laboratory-Scale Melter Feed Preparation Samples	B34
Table B3.5	Standard Crucible Redox Test Data for Laboratory-Scale Melter Feed Alternate HTM Flowsheet 2 Testing	B40
Table B4.1	Small-Scale Melter Feed Preparation Sample Matrix Which Includes: Sample Numbers, Dates of Sampling, Analytical Request Form Numbers, and Dates of Analytical Report Issue	B59
Table B4.2.1	Small-Scale Melter Feed Preparation Condensate Sample Analysis Results for Alternate HTM Flowsheet 2 Processing	B62
Table B4.2.2	Small-Scale Melter Feed Preparation Slurry Sample Analysis Results for Alternate HTM Flowsheet 2 Processing	B63
Table B4.3	Peak Off-Gas Generation Rates and Total Off-Gas Generated for the Alternate HTM Flowsheet 2 Small-Scale Test	B65
Table B4.4.1	Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Small-Scale Melter Feed Preparation, Aging, Day Tank Samples	B71

TABLES (cont.)

Table B4.4.2	Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Laboratory Aged Small-Scale Melter Feed Preparation Samples (50°C)	B132
Table B4.4.3	Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Laboratory Aged Small-Scale Melter Feed Preparation Samples (25°C)	B150
Table B4.5	Standard Crucible Redox Test Data for Small-Scale Melter Feed Alternate HTM Flowsheet 2 Testing	B168

LOGS

Log B3.1	Laboratory-Scale Melter Feed Preparation Activity Log, Alternate HTM Flowsheet 2; Glycolic Acid Addition, Test T95-SSHTMGLYC-1	B4
Log B4.1	Small-Scale Melter Feed Preparation Activity Log, Alternate HTM Flowsheet 2; Glycolic Acid Addition	B43

1.0 INTRODUCTION

This appendix provides the data for Alternate HTM Flowsheet 2 (Glycolic Acid) melter feed preparation activities in both the laboratory- and small-scale testing. The first section provides an outline of this appendix. The melter feed preparation data are presented in the next two main sections, laboratory melter feed preparation data and small-scale melter feed preparation data. Section 3.0 provides the laboratory data which is discussed in the main body of the Small-Scale High Temperature-1 (SSHTM-1) Data Package, milestone C95-02.02Y. Section 3.1 gives the flowsheet in outline form as used in the laboratory-scale tests. This section also includes the "Laboratory Melter Feed Preparation Activity Log" which gives a chronological account of the test in terms of time, temperature, slurry pH, and specific observations about slurry appearance, acid addition rates, and samples taken. The "Laboratory Melter Feed Preparation Activity Log" provides a road map to the reader by which all the activity and data from the laboratory can be easily accessed. A summary of analytical data is presented next, section 3.2, which covers starting materials and progresses to the analysis of the melter feed. The next section, 3.3, characterizes the off-gas generation that occurs during the slurry processing. The following section, 3.4, provides the rheology data gathered including gram waste oxide loading information for the various slurries tested. The final section, 3.5, includes data from standard crucible redox testing. Section 4.0 provides the small-scale data in parallel form to section 3.0. Section 5.0 concludes with the references for this appendix. A preliminary comparison of the data is given in the Small-Scale High Temperature-1 (SSHTM-1) Data Package, milestone C95-02.02Y, main body. This data will provide the basis for a letter report, T3C-95-129.

2.0 OUTLINE

Melter Feed Preparation

- A. Laboratory Melter Feed Preparation Data
  - 1. Melter feed preparation activity data log
  - 2. Melter feed and condensate composition
  - 3. Melter feed preparation off-gas
  - 4. Rheology and gram waste oxide loading data
  - 5. Standard crucible redox test data
- B. Small-Scale Melter Feed Preparation Data
  - 1. Melter feed preparation activity data log
  - 2. Melter feed and condensate composition
  - 3. Melter feed preparation off-gas
  - 4. Rheology and gram waste oxide loading data
    - A. Aging data
    - B. Day tank samples from flowsheet 2 preparation
  - 5. Standard crucible redox test data

3.0 LABORATORY MELTER FEED PREPARATION DATA

3.1 Flowsheet Outline, Activity Data Log, and Gram Waste Oxide Tracking Sheet

The following outline covers the major stages of waste slurry simulant processing to make melter feed. The details resulting from implementing these steps are recorded in the "Laboratory Melter Feed Preparation Activity Log" included in this section. An accounting sheet for the gram waste oxide is also provided.

Laboratory Flowsheet

- Step 1: Obtain pre-concentrated slurry simulant from the small-scale slurry processing tank prior to processing (~179 gWO/L). Place 1.5 L of that slurry in reaction vessel. Set stirrer at 6 or higher and establish argon sweep gas flow.
- Take slurry and condensate samples.
- Step 2: Heat slurry to 93°C. Titrate with glycolic acid to a pH of 4.8 ± 0.1 . Add 70.6 wt % acid solution at a rate of 1.6 grams per minute.
- Take slurry and condensate samples.
- Step 3: Concentrate slurry to 240 gWO/L by boiling during digestion period of 7 hours. (The longer digestion/concentration period more accurately duplicates the small-scale processing run.)
- Take slurry and condensate samples.
- Step 4: Add NaNO_3 and SiO_2 to complete melter feed preparation. (12 g Na_2O per 100 gWO as 32.0 g NaNO_3 per 100 gWO, 88 g SiO_2 as sand (F-110) per 100 gWO). Final target HTM feed concentration is 433 gTO/L.
- Take melter feed samples.
- Step 5: Melter feed rheology evaluated.

Laboratory Melter Feed Preparation Activity Log

The "Laboratory Melter Feed Preparation Activity Log" is a transcription of the actual log kept during the test run. Though it does not contain all of the entries (redundant entries were eliminated) it contains all of the information of the original log. Its main purpose is to provide a record of observations made during the test that were not being recorded by other means and that would correlate with other data such as the off-gas data. The basis for correlation with other data is "24 hour" military time. The observations

recorded relate principally to the slurry condition: color, foaming, other phases present but also includes a record of condenser temperatures (laboratory tests) and other activities carried out during the test run along with comments about the operation of the apparatus itself.

The Date/Time column gives the day the run activity was occurring and the "24 hour" time that the data was recorded or an activity commenced. The cumulative time log, provided in minutes, provides the reader information that can be used to cross reference the off-gas data presented in graphical form in section 3.3 of this appendix. Zero time is defined as the start of acid addition and the final log entry is entered at the time off-gas data collection stopped. The pH was a manually recorded value from the pH probe (temperature compensated for laboratory tests but not for the small-scale system). The Activity/Observations column attempts to succinctly express relevant activities or actions at the time indicated in the first column. Activities may include:

- Heating or cooling the slurry.
- Observations about the slurry such as slurry color or degree of foaming.
- Condensate or slurry sample collection.
- Unusual off-gas activity.
- Start of acid addition and the rate of addition.

The Laboratory Melter Feed Preparation Activity Log B3.1 for T95-SSHTMGLYC-1 follows.

Log B3.1

**Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition, Test-T95-SSHTMGLYC-1**

<u>Date/ Time</u>	<u>Cumulative Time (min) (1)</u>	<u>pH</u>	<u>Temperature Slurry (C)</u>	<u>Activity/Observations</u>
<u>11/8/94</u> 10:44	-	-	-	Set up vessel for run tomorrow. Removed 32.1 g Initial slurry, T95-SSHTMGLYC-1-1-1 from slurry carboy.
<u>11/9/94</u> 6:30	-	-	-	Turned on Input Output Plexer, NO/NOx Analyzer. Gas Chromatograph temperature on to bake out at (180 to 150 degrees (C)). Stirrer set at 6. Water chiller on. Set vessel temperature at 73 degrees (C).
7:40	-	11.0	83	pH probe removed.
8:40	-	-	74	Increased temperature to 93 degrees (C).
9:01	-	9.1	97	Started collection of initial condensate.
9:10	-	9.4	97	Connecting off-gas system.
9:20	-	9.2	93	Preparing for acid addition.
9:36	-	10.1	91	Added 0.24 g Dow Corning 544 anti foam compound with 10 g deionized water.
9:45	0	10.0	92	Acid addition started. No initial condensate collected.
9:50	5	9.6	92	No foaming.

Log B3.1 (Cont.)

**Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition, Test-T95-SSHTMGLYC-1**

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
<u>11/9/94</u>				
9:53	8	9.3	92	Addition rate 1.94 g/min. over 5 min. Decreased addition rate.
9:55	10	9.1	92	Addition rate 1.80 g/min. over 1 min.
10:00	15	-	-	Addition rate 1.60 g/min. over 6 min.
10:11	26	7.3	92	Some slight foaming.
10:12	27	7.3	92	Foam spread across whole surface.
10:15	30	7.1	91	Addition rate 1.60 g/min. Heavy foaming.
10:20	35	7.0	92	Still heavy foam.
10:25	40	6.9	93	No change.
10:35	50	6.5	93	No change.
10:45	60	5.9	26	Foam breaking up. Partial surface coverage with some gray scum.
10:57	72	5.3	92	Removed 36.7 g condensate, T95-SSHTMGLYC-1-2-2.
11:00	75	5.3	92	An island of gray surface scum floating on surface.
11:03	78	5.0	92	No change.

Log B3.1 (Cont.)

**Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition, Test-T95-SSHTMGLYC-1**

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
<u>11/9/94</u>				
11:14	89	4.8	92	Acid addition complete. A total of 145.7 g added. Removed 21.3 g condensate, T95-SSHTMGLYC-1-2-2.
11:21	96	-	-	Removed 5.1 g secondary condensate, T95-SSHTMGLYC-1-2-2s.
11:27	102	5.1	92	Removed 16.9 g acid addition slurry, T95-SSHTMGLYC-1-1-2.
11:32	107	5.2	-	Increased temperature to 100 degrees (C) for start of digestion. Removed 16.3 g condensate, T95-SSHTMGLYC-1-2-2.
11:48	123	5.2	100	Increased temperature to 103 degrees (C).
11:50	125	5.3	100	Slight foaming.
11:52	127	5.3	100	Boiling.
11:55	130	5.3	101	Removed 31.3 g condensate, T95-SSHTMGLYC-1-2-3.
12:00	135	5.3	101	Slight foaming.
12:10	145	5.4	101	No change.
12:15	150	5.4	100	Removed 31.4 g condensate, T95-SSHTMGLYC-1-2-3.
12:27	162	5.5	101	Slight foaming.

Log B3.1 (Cont.)

**Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition, Test-T95-SSHTMGLYC-1**

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
<u>11/9/94</u>				
12:37	172	5.6	101	Removed 30.9 g condensate, T95-SSHTMGLYC-1-2-3.
13:14	209	-	-	Removed 37.8 g condensate, T95-SSHTMGLYC-1-2-3.
13:15	210	5.8	101	Slight foaming.
13:22	217	5.8	101	Slight foaming.
13:37	232	5.5	101	Slight foaming.
13:55	250	-	102	Removed 36.4 g condensate, T95-SSHTMGLYC-1-2-3. pH meter reading E-20 out of range. Changing probes.
14:15	270	-	102	No probe in for pH.
14:27	282	-	102	Still foaming.
14:32	287	7.1	-	pH questionable, replaced probe 22b with 23b.
14:35	290	7.1	102	Removed 34.2 g condensate, T95-SSHTMGLYC-1-2-3.
14:37	292	-	102	pH questionable, removed probe.
15:07	322	-	102	Removed 31.9 g condensate, T95-SSHTMGLYC-1-2-3.

Log B3.1 (Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition, Test-T95-SSHTMGLYC-1

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
<u>11/9/94</u>				
15:24	339	-	102	Slight foam coverage on surface.
15:30	345	-	102	Removed 16.8 g secondary condensate, T95-SSHTMGLYC-1-2-3s.
15:44	359	-	102	Removed 39 g condensate, T95-SSHTMGLYC-1-2-3.
16:06	381	-	102	Slight foam.
16:15	390	-	102	Removed 40.9 g condensate, T95-SSHTMGLYC-1-2-3.
16:37	412	-	102	Removed 35.7 g condensate, T95-SSHTMGLYC-1-2-3.
17:04	439	-	102	Removed 40.4g condensate, T95-SSHTMGLYC-1-2-3.
17:28	463	-	102	Removed 38.9 g condensate, T95-SSHTMGLYC-1-2-3.
17:32	467	-	102	Removed 8.6 g secondary condensate, T95-SSHTMGLYC-1-2-3s.
17:52	487	-	103	Removed 39.2 g condensate, T95-SSHTMGLYC-1-2-3.
17:57	492	-	-	Removed 1.7 g secondary condensate, T95-SSHTMGLYC-1-2-3s. Started refluxing. Gram waste oxide 243.
18:35	530	-	103	
18:58	553	-	-	Set temperature to 0.

Log B3.1 (Cont.)

**Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition, Test-T95-SSHTMGLYC-1**

<u>Date/ Time</u>	<u>Cumulative Time (min) (1)</u>	<u>pH</u>	<u>Temperature Slurry (C)</u>	<u>Activity/Observations</u>
<u>11/9/94</u> 19:10	565	-	-	Off-gas data collection stopped at this time, therefore, the cumulative time log is discontinued. Stirrer set on 3 overnight.
<u>11/10/94</u> 6:30	-	-	33	Added 160 g deionized water to bring slurry level up to level measured by dipstick last night. Slurry appears almost homogenous with some lumping. Final weight (1360.5 g). Removed 15.7 g digestion slurry, T95-SSHTMGLYC-1-1-3.
N/A	-	-	-	Final slurry product submitted to Eve Tracey for rheology measurements. Samples listed were taken from final slurry product and can be found on Table B3.4.2. Sample Day0glyc and Day0glyb are samples taken before silica and sodium addition. Samples SilGly0a and SilGly0b are after silica and sodium addition.

1. Time after initiation of acid addition (min.).

Gram Waste Oxide (gWO) Tracking Sheet

The gram waste oxide (gWO) tracking sheet (laboratory tests only) is an accounting sheet for estimating the volume (and weight) of slurry and waste oxide content at various stages of the test. The tracking methodology takes into account the affects of taking condensate and slurry samples during the test and allows adjustments to be made for slurry losses during upsets or for maintaining/achieving a given waste oxide loading. It also provides a way of estimating the amount of processed slurry resulting from the run.

The kind of sample taken or material added to the initial slurry material is indicated in the first column, Sample/Action, of the tracking sheet. In this column the sample number indicates the test period during which a sample was taken: initial, forming, or digestion. "Condensate" means that primary (and possibly secondary) condensate samples were taken. "Slurry" means that one or more slurry samples were taken. "Add" means that material, e.g., water, was added to the slurry at that stage of the test run. Under the column heading Volume(ml)/Wt.(g) // Sample - Slurry, the volume and weight of both the sample taken or material added and the resulting slurry are given. The sample weight is an actual weight but the sample volume is a calculated value obtained by dividing the sample weight by the slurry density (last column). The slurry volume and weight are computed by adding or subtracting the sample volume and weight to the corresponding values from the preceding line. Condensate samples were assumed to be essentially water. The initial slurry weight is a measured value and the initial slurry volume is obtained by dividing the weight by the initial slurry density which was previously estimated by weighing a known volume. Gram Waste Oxide per Liter (gWO/L) is obtained by dividing the Σ gWO (total grams Waste Oxide) by the slurry volume. The initial values were determined by calcining a weighed sample at 1050°C. Density is the slurry density where the initial value was determined as stated above, but all subsequent values are obtained by dividing the previously calculated weight by the previously calculated volume on the same line. The Gram waste oxide (gWO) tracking sheet, Table B3.1, for laboratory-scale test T95-SSHTMGLYC-1, follows.

**Table B3.1 Laboratory-Scale Melter Feed Gram Waste Oxide (gWO) Tracking
Sheet for Test T95-SSHTMGLYC-1.**

Sample/Action	Volume (mL)/Weight (g)		gWO/L	Total gWO	Density
	Sample	Slurry			
Initial Slurry	-	1549/1766	175	271	1.14
- Acid addition, condensate T95-SSHTMGLYC-1-2-2	58/58	1491/1708	182	271	1.15
Acid addition, secondary condensate T95-SSHTMGLYC-1-2-2s	5.1/5.1	1486/1703	182	271	1.15
Acid addition, slurry T95-SSHTMGLYC-1-1-2	14.9/16.9	1471/1686	182	268	1.15
Acid addition, condensate T95-SSHTMGLYC-1-2-2	16.3/16.3	1455/1670	184	268	1.15
Digestion, condensate T95-SSHTMGLYC-1-2-3	468/468	987/1202	272	268	1.22
Digestion, secondary condensate T95-SSHTMGLYC-1-2-3s	27/27	960/1175	279	268	1.22
Digestion, slurry T95-SSHTMGLYC-1-1-3	13/15.7	947/1159	279	264	1.22
Recorded volume on metered dip stick. Added ~160g deionized water to compensate for overnight volume loss.	-	-	-	-	-
Weight at end of test.	202/202	1149/1361	230	264	1.19

- This and following densities are calculated from measured density of the initial slurry.

3.2 Melter Feed and Condensate Composition

Table B3.2 summarizes the results of the analyses of condensate and slurry samples taken during the slurry simulant processing into melter feed on the laboratory-scale. The time and date that the sample was taken is given by the activity log. The gWO/L and slurry density values allow the samples to be compared on the basis of a common gWO/L value. The anions and the total organic carbon (TOC) values are weight percent values based on the "as received" slurry simulant or melter feed material. On the other hand, the oxide weight percent values are based on the total oxide content of the slurry simulant or melter feed material which have been normalized to 100%. When the sample is melter feed, the percent oxide values indicate the glass composition to be expected from melting this feed assuming that none of the oxides are lost by volatilization or decomposition other than the nitrates or carbonates. In these tables, zero values indicate that the element was not detected. The analytical data was obtained using the following procedures:

gWO/L and density-

Density was determined from the weight of a known volume of slurry under ambient conditions and the gWO/L was determined by firing the pre-weighed slurry to oxides in a furnace at 1050°C for two hours. The accuracy of these measurements is on the order of 2-3%.

pH-

The pH measurements were made at ambient conditions using calibrated pH sensing electrodes "Orion" (PNL-ALO-501, Rev. 1). The accuracy of these measurements is better than 0.1 pH unit.

Total Organic Carbon-

The total organic carbon was determined using the hot persulfate method (test procedure PNL-ALO-381, Rev.0). The accuracy of this method estimated on the basis of its reproducibility is about 4% when the organic carbon makes up 1-2 % of the system by weight. An ion chromatographic determination of the glycolate ion was made on the laboratory samples processed with glycolic acid (PNL-ALO-212, Rev.1).

Anions-

The anions were determined by ion chromatography using PNL-ALO-212, Rev.1 for the laboratory samples. The estimated accuracy is ± 5 to 10%.

Ammonia-

Ammonia is measured as the NH_4^+ ion with a standard ion electrode using procedure PNL-ALO-226, Rev.0. The accuracy is $\pm 15\%$ for concentrations < 1 ppm. For concentrations > 1 ppm the accuracy improves to $\pm 5\%$.

Elements, Oxides-

The elemental or oxide analyses were made by ICP-AES using procedure APSL-14. The estimated accuracy for major elements using this technique is $\pm 10\%$.

Table B3.2 Summary of Analytical Data for the Laboratory-Scale, Alternate HTM Flowsheet 2 Test, T95-SSHTMGLYC-1. The slurry samples were taken from the initial slurry material, after acid addition (-2) and after the digestion/concentration period (-3). Condensate samples were collected during acid addition (-2) and digestion/concentration period (-3).

	<u>gWO/L</u>	<u>Density (g/ml)</u>	<u>pH^a</u>	<u>Total Organic Carbon (wt%)</u>	<u>Nitrite (ppm)</u>	<u>Nitrate (ppm)</u>	<u>Glycolate (ppm)</u>	<u>Ammonia (ppm)</u>
<u>Alternate HTM Flowsheet 2^b</u>								
<u>T95-SSHTMGLYC-1</u>								
Slurry Sample:								
GLYC-1-1-1		1.148	11.74		23000	11000	4100	0.21
GLYC-1-1-2		1.18	5.74		20000	12000	44000	< 0.08
GLYC-1-1-3		1.3	8.03		15000	15000	51000	0.28
Condensate Sample:								
GLYC-1-2-2	N/A ^c	1.0	2.06		2.1	670	10	0.72
GLYC-1-2-3	N/A ^c	1.0	2.53		2.3	200	5	< 0.1

(a) pH measurements were taken at room temperature, approximately 22°C.

(b) Alternate HTM Flowsheet 2 is the alternate reductant, glycolic acid processed flowsheet.

(c) N/A: Not Applicable; no waste slurry solids make it past the condenser system.

3.3 Off-Gas Data

Laboratory-scale off-gas data is provided for the glycolic acid process flowsheet. The T95-SSHTMGLYC-1 laboratory-scale run provided the data. The off-gas record is organized in the following way:

1- Table B3.3 indicates the maximum generation rates for CO_2 , N_2O , NO_x , and H_2 and the cumulative amount of CO_2 , N_2O , NO_x , and H_2 measured.

2- Figures B3.3.1 and B3.3.2 present off-gas process profiles for laboratory-scale test T95-SSHTMGLYC-1. The off-gas generation rate of CO_2 , N_2O , NO_x , and H_2 is plotted as a function of time. Zero time is taken as the start of acid addition to the slurry simulant. The off-gas record continues through the digestion period of slurry processing. The slurry pH and temperature are also plotted.

The laboratory apparatus used to prepare melter feed and collect off-gas is diagrammed in Figure 3.1.1. It consists of a two liter pyrex reaction vessel with an agitator, temperature and pH indicators, sampling and chemical addition capabilities, and an off-gas system. The reaction vessel is held in a heating mantle capable of heating the system to boiling. The off-gas system sweeps the headspace of the reaction vessel with helium doped argon at a flow rate of 1.95 L/min. and analyses the off-gas quantitatively for CO_2 , N_2O , NO_x , and H_2 using an NO_x analyzer, a GC, and an FTIR which also screens the off-gas for unknown gas compounds and NH_3 . The gases were measured in the off-gas stream using a gas chromatograph to measure CO_2 , N_2O , and H_2 and a chemiluminescent NO/NO_x analyzer to measure NO_x . The overall accuracy of the measurements is estimated at $\pm 10\%$, including uncertainties from mass flow controllers and intermittent small leaks around the reaction vessel sampling and chemical addition ports.

Table B3.3 Peak Off-Gas Generation Rates and Total Off-Gas Generated for Laboratory-Scale Alternate HTM Flowsheet 2 Tests.

		Off-Gas Generated During Processing				
		CO ₂	H ₂ O	H ₂	NO _x	NH ₃ ^a
Alternate HTM Flowsheet 2 ^b						
T95-SSHTMGLYC-1:	Peak Generation Rate (mmoles/min)	7.9	0.06	0.003	0.95	c
	Total Off-Gas Generated (mmoles)	331	8.29	0.47	179.9	< 0.19

(a) Measured amounts in the condensate + maximum amount in the slurry.

(b) Alternate HTM Flowsheet 2 is the alternate reductant, glycolic acid process flowsheet.

(c) Not measured but usually equal to or less than the H₂ generation rate.

Figure B3.3.1 Process Profile for Laboratory-Scale Alternate HTM Flowsheet 2, Test T95-SSHTMGLYC-1, for the First 300 Minutes Measured from the Start of Glycolic Acid Addition.

Figure B3.3.2 Process Profile for Laboratory-Scale Alternate HTM Flowsheet 2, Test T95-SSHTMGLYC-1, for 0 to 600 Minutes Measured from the Start of Glycolic Acid Addition.

3.4 Rheology and Gram Waste Oxide Loading Data for Melter Feed

For Alternate HTM Flowsheet 2, a preliminary melter feed was prepared in the laboratory (500 ml) to predict the off-gases, rheology, and redox potential of the melter feed. These laboratory tests were presented in the SSHTM-1 Test Plan Addendum and on the basis of these laboratory tests, the basic test procedures given in section 3.1 were developed. Next, the melter feed was prepared in a small-scale system (1514 liters). Due to the newness of the system and some related engineering difficulties, the processing test parameters deviated from the original processing test parameters. A third feed was prepared in the laboratory in which the processing test parameters performed during SSHTM-1 testing were duplicated. This third feed preparation procedure was completed so that laboratory- and small-scale data could be more easily compared and in this section is called the "modified feed procedure." The rheology of the preliminary and modified feeds are discussed below. The data for the preliminary laboratory feed preparation and aging is contained in sections 3.4.1 and the data for the modified laboratory feed preparation is contained in section 3.4.2.

Experimental

After slurries were prepared, but before sodium and silica were added, (after step 3 of the laboratory flowsheet described in section 3.1), the physical properties of the waste simulants were evaluated. Density, pH (22°C), wt % solids, and gTO/L were measured for the preliminary and modified feeds prepared in the laboratory using HWVP technical procedure WTC-006-4. Rheological characterization was performed at 25°C for the preliminary melter feed and at 50°C for the modified feed. Next, the sodium and silica were added to the feeds (step 4 of the laboratory flowsheet described in section 3.1) and the rheological and physical properties were reevaluated. To meet the rheological specifications of 25 mPa·s for plastic viscosity and 15 Pa for yield stress, the preliminary melter feed was diluted after silica addition (step 4) and the modified feed was not diluted.

The rheological values were measured using the Haake M5 measuring system with the MV2 sensor. Rheograms were generated by increasing the applied shear rate from 0 to 451 s⁻¹ in a 2-minute interval. (Two rheograms were generated for each sample and the samples were not reloaded into the rheometer between the two measurements.) The increasing shear rheology data were fit with the Bingham equation:

$$\tau = \tau_0 + \eta \dot{\gamma}$$

where

τ = shear stress,
 τ_0 = yield stress,
 $\dot{\gamma}$ = shear strain rate, and
 η = plastic viscosity.

After the preliminary melter feed was prepared in the laboratory, the time dependence of the rheological properties was studied. Aging was not performed on the modified melter feed prepared in the laboratory. To study aging in the laboratory, the melter feed was transferred to a mixing vessel. The mixing vessel was a closed system which was monitored for water losses by measuring changes in slurry weight as a function of time. Deionized water was added to the slurry if any water losses were measured. Samples were aged under constant agitation. During the aging process, samples were taken at times: 0 days, 1 day, 7 days, 14 days, and 28 days. These samples were subjected to the following tests:

- rheological characterization (at 25°C)
- weight percent solids of dry melter feed
- pH measurement (at 22°C).

Results

3.4.1 Rheology of Preliminary Laboratory-Scale Feed Preparation and Aging Samples

The preliminary waste simulant prepared in the laboratory, at an oxide loading of 277 gTO/L, had a density of 1.29 g/ml, a pH of 8.0, and a solids loading of 37 wt % before silica and sodium nitrate addition. At these conditions the plastic viscosity was 30.5 mPa•s and the yield stress was 6.7 Pa. After silica (F-110) and sodium nitrate addition and dilution, the resulting HTM feed had a solids loading of 45 wt %, an oxide loading of 464 gTO/L, a density of 1.41 g/ml, a pH of 8.0, a plastic viscosity of 27.3 mPa•s, and a yield stress of 6.4 Pa.

The yield stress and plastic viscosity increased with sample age. From day 0 to day 28, yield stress increased from 6.4 Pa to 50.7 Pa (25°C) and plastic viscosity increased from 27.3 mPa•s to 88.4 mPa•s (25°C). The Bingham model fit the rheological data with correlation coefficients ranging from 0.67 to 0.98. Changes in the rheological measurements may not be attributed to water losses in the system because the solids loading remained at a constant value of 45 ± 1 weight percent. The pH remained at 8.0 ± 0.2 over the twenty-eight day period. The summary Table B3.4.1 of all data from the requisite rheograms and each individual rheogram follows.

Table B3.4.1 Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Preliminary Laboratory-Scale Melter Feed Preparation and Aging Samples.

		g TO/L	pH (@ 22°C)	wt % solids	Density (g/ml)	Yield Stress (Pa ^a)	Plastic Viscosity (mPa·s ^b)	Correlation Coeff. (R ²) ^c
<u>Alternate HTM Flowsheet 2^d</u>								
<u>Preliminary Laboratory-Scale^e:</u>								
<u>Before silica add. and dilution:</u>								
File ID/Date:	test 1/6-21-94	277	8.0	37	1.29	6.7	30.5	0.99
<u>After silica add. & dilution^f:</u>								
Day 0:								
File ID/Date:	test 1/6-21-94	464	8.0	45	1.41	8.4	27.3	0.98
	test 2/6-21-94					7.2	25.9	0.98
Day 1:								
File ID/Date:	test a/6-23-94	--	8.2	45	--	9.6	24.1	0.95
	test b/6-23-94	--			--	9.8	20.6	0.99
Day 6:								
File ID/Date:	test 1, day 7/6-28-94	--	8.0	45	--	20.8	19.3	0.72
	test 2, day 7/6-28-94	--			--	20.8	18.2	0.95
	test 2a, day 7/6-28-94	--			--	19.8	21.5	0.73
Day 14:								
File ID/Date:	day 14, test a/7-6-94	--	7.9	46	--	45.8	40.1	0.67
	day 14, test b/7-6-94	--			--	42.1	38.6	0.68
Day 28:								
File ID/Date:	day 28, test a/7-19-94	--	8.0	46	--	50.7	68.4	0.61
	day 28, test b/7-19-94	--			--	46.1	62.5	0.96

- (a) Laboratory rheological screening data value for the yield stress is 15 Pa. The yield stress was calculated by fitting the shear rate sweep data to the Bingham model.
- (b) Laboratory rheological screening data value for the plastic viscosity is 25 mPa·s. The plastic viscosity was calculated by fitting the shear rate sweep data to the Bingham model.
- (c) Least squares correlation coefficient of shear rheology data fit to the Bingham equation.
- (d) Alternate HTM Flowsheet 2 is the glycolic acid process flowsheet.
- (e) Rheology measurements were taken at 25°C for the preliminary laboratory-scale feeds.
- (f) Laboratory-scale results were diluted after the silica addition was made and before the rheological measurements were taken.

Rheogram and Data for Test Number Test 1

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.99$	$\dot{\sigma}_0 = 6.666$	$\text{Eta} = 0.03049$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.90$	$a = 2.418$	$n = 0.3272$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.91$	$a = 1.516$	$n = 0.3815$
Casson	: $\sqrt{\dot{\sigma} - \dot{\sigma}_0} = \sqrt{a} * D$	$R^2 = 0.97$	$\dot{\sigma}_0 = 4.226$	$\text{Eta} = 0.01246$
Linear	: $Y = a + b * X$	$R^2 = 0.99$	$a = 6.666$	$b = 0.03049$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.02$	$a = 1.735$	$b = -0.005563$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.39$	$a = 6.484$	$b = 0.002894$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.11$	$a = 13.75$	$b = -11.37$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.79$	$a = -4.128$	$b = 3.460$

Optimum :
Bingham : $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$ $R^2 = 0.99$ $\dot{\sigma}_0 = 6.666$ $\text{Eta} = 0.03049$

Rheogram and Data for Test Number Test 1

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R = 0.98$	$\dot{\sigma}_0 = 6.398$	$\text{Eta} = 0.02725$
Ostwald	: $\dot{\sigma} = a * D^n$	$R = 0.83$	$a = 1.629$	$n = 0.3881$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R = 0.95$	$a = 1.655$	$n = 0.3623$ $\dot{\sigma}_0 = 1.361$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R = 0.95$	$\dot{\sigma}_0 = 3.908$	$\text{Eta} = 0.01156$
Linear	: $Y = a + b * X$	$R = 0.98$	$a = 6.398$	$b = 0.02725$
Inv. linear	: $Y = 1 / (a + b * X)$	$R = 0.03$	$a = 0.2914$	$b = -732.4E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R = 0.57$	$a = 6.619$	$b = 0.002565$
Hyperbolic	: $Y = a + b / X$	$R = 0.10$	$a = 12.65$	$b = -4.814$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R = 0.83$	$a = -3.476$	$b = 3.139$

Optimum :

Bingham : $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$ $R = 0.98$ $\dot{\sigma}_0 = 6.398$ $\text{Eta} = 0.02725$

Rheogram and Data for Test Number Test 2

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.98$	$\dot{\sigma}_0 = 7.187$	$\text{Eta} = 0.02589$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.80$	$a = 2.229$	$n = 0.3363$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.91$	$a = 1.799$	$n = 0.3388$ $\dot{\sigma}_0 = 2.291$
Casson	: $\sqrt{\dot{\sigma}} = \sqrt{\dot{\sigma}_0} + \sqrt{\text{Eta} * D}$	$R^2 = 0.93$	$\dot{\sigma}_0 = 4.725$	$\text{Eta} = 0.009765$
Linear	: $Y = a + b * X$	$R^2 = 0.98$	$a = 7.187$	$b = 0.02589$
Inv. linear:	$Y = 1 / (a + b * X)$	$R^2 = 0.04$	$a = 0.2118$	$b = -474.5E-06$
Exponential:	$Y = a * \text{EXP}(b * X)$	$R^2 = 0.57$	$a = 7.390$	$b = 0.002292$
Hyperbolic:	$Y = a + b / X$	$R^2 = 0.11$	$a = 13.15$	$b = -5.404$
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R^2 = 0.81$	$a = -2.003$	$b = 2.945$

Optimum :
Bingham : $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$ $R^2 = 0.98$ $\dot{\sigma}_0 = 7.187$ $\text{Eta} = 0.02589$

Rheogram and Data for Test Number Test a

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.95$	$\dot{\sigma}_0 = 9.598$	$\eta = 0.02405$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.87$	$a = 3.755$	$n = 0.2655$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.95$	$a = 2.866$	$n = 0.2802$ $\dot{\sigma}_0 = 2.599$
Casson	: $\sqrt{\dot{\sigma}} = \sqrt{\dot{\sigma}_0} + \sqrt{\eta \cdot D}$	$R^2 = 0.91$	$\dot{\sigma}_0 = 6.892$	$\eta = 0.007482$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.95$	$a = 9.598$	$b = 0.02405$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.09$	$a = 0.1194$	$b = -195.3E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.61$	$a = 9.688$	$b = 0.001802$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.14$	$a = 15.13$	$b = -3.944$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.87$	$a = 0.5304$	$b = 2.841$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.95$	$a = 2.866$	$n = 0.2802$ $\dot{\sigma}_0 = 2.599$

Rheogram and Data for Test Number Test b

Bingham	: $\sigma = \sigma_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.99$	$\sigma_0 = 9.771$	$\eta = 0.02081$
Ostwald	: $\sigma = a \cdot \dot{\gamma}^n$	$R^2 = 0.86$	$a = 5.719$	$n = 0.1780$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.92$	$a = 0.5366$	$n = 0.4723$
Casson	: $\sqrt{\sigma - \sigma_0} = \sqrt{\eta} \cdot \sqrt{\dot{\gamma}}$	$R^2 = 0.98$	$\sigma_0 = 7.703$	$\eta = 0.005097$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.99$	$a = 9.771$	$b = 0.02081$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.18$	$a = 0.1077$	$b = -146.5E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.69$	$a = 9.784$	$b = 0.001609$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.12$	$a = 14.63$	$b = -9.634$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.78$	$a = 2.414$	$b = 2.359$

Optimum :
Bingham : $\sigma = \sigma_0 + \eta \cdot \dot{\gamma}$ $R^2 = 0.99$ $\sigma_0 = 9.771$ $\eta = 0.02081$

Rheogram and Data for Test Number Test 1, Day 7

Bingham	: $\sigma = \sigma_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.72$	$\sigma_0 = 20.78$	$\eta = 0.01929$
Ostwald	: $\sigma = a \cdot \dot{\gamma}^n$	$R^2 = 0.67$	$a = 10.74$	$n = 0.1642$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.98$	$a = 8.599$	$n = 0.1521$ $\sigma_0 = 6.300$
Casson	: $\sqrt{\sigma} = \sqrt{\sigma_0} + \sqrt{\eta} \cdot \sqrt{\dot{\gamma}}$	$R^2 = 0.65$	$\sigma_0 = 17.47$	$\eta = 0.003364$
Linear	: $\sigma = a + b \cdot \dot{\gamma}$	$R^2 = 0.72$	$a = 20.78$	$b = 0.01929$
Inv. linear	: $\sigma = 1 / (a + b \cdot \dot{\gamma})$	$R^2 = 0.06$	$a = 0.05662$	$b = -62.81E-06$
Exponential	: $\sigma = a \cdot \exp(b \cdot \dot{\gamma})$	$R^2 = 0.31$	$a = 20.23$	$b = 901.9E-06$
Hyperbolic	: $\sigma = a + b / \dot{\gamma}$	$R^2 = 0.40$	$a = 25.34$	$b = -7.123$
Logarithmic	: $\sigma = a + b \cdot \ln(\dot{\gamma})$	$R^2 = 0.96$	$a = 11.07$	$b = 2.757$

Optimum :

H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.98$	$a = 8.599$	$n = 0.1521$	$\sigma_0 = 6.300$

Rheogram and Data for Test Number Test 2, Day 7

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R = 0.95$	$\dot{\sigma}_0 = 20.77$	$\eta = 0.01819$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R = 0.86$	$a = 15.41$	$n = 0.09284$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R = 0.88$	$a = 0.9843$	$n = 0.3757$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R = 0.96$	$\dot{\sigma}_0 = 18.46$	$\eta = 0.002345$
Linear	: $Y = a + b \cdot X$	$R = 0.95$	$a = 20.77$	$b = 0.01819$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R = 0.10$	$a = 0.05279$	$b = -48.29E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R = 0.42$	$a = 20.15$	$b = 871.1E-06$
Hyperbolic	: $Y = a + b / X$	$R = 0.17$	$a = 25.05$	$b = -10.22$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R = 0.84$	$a = 13.75$	$b = 2.178$

Optimum :
Casson : $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$ $R = 0.96$ $\dot{\sigma}_0 = 18.46$ $\eta = 0.002345$

Rheogram and Data for Test Number Test 2a, Day 7, Reload

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R=0.73	$\dot{\sigma}_0 = 19.79$	Eta = 0.02146
Ostwald	: $\dot{\sigma} = a * D^n$	R=0.64	a = 8.521	n = 0.2045
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=0.98	a = 8.502	n = 0.1662 $\dot{\sigma}_0 = 4.572$
Casson	: $\sqrt{\dot{\sigma} - \dot{\sigma}_0} = \sqrt{a * D}$	R=0.66	$\dot{\sigma}_0 = 16.09$	Eta = 0.004371
Linear	: $Y = a + b * X$	R=0.73	a = 19.79	b = 0.02146
Inv. linear	: $Y = 1 / (a + b * X)$	R=0.02	a = 0.7098	b = -0.002238
Exponential	: $Y = a * \text{EXP}(b * X)$	R=0.13	a = 16.74	b = 0.001489
Hyperbolic	: $Y = a + b / X$	R=0.35	a = 24.84	b = -6.978
Logarithmic	: $Y = a + b * \text{LN}(X)$	R=0.97	a = 9.052	b = 3.055

Optimum :

H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ R=0.98 a=8.502 n=0.1662 $\dot{\sigma}_0 = 4.572$

Rheogram and Data for Test Number Day 14, Test a

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.67$	$\dot{\sigma}_0 = 45.75$	$\eta = 0.04014$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.71$	$a = 23.70$	$n = 0.1619$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.99$	$a = 17.82$	$n = 0.1579$ $\dot{\sigma}_0 = 14.56$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.64$	$\dot{\sigma}_0 = 38.44$	$\eta = 0.007013$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.67$	$a = 45.75$	$b = 0.04014$
Inv. linear:	$Y = 1 / (a + b \cdot X)$	$R^2 = 0.02$	$a = 0.1216$	$b = -347.6E-06$
Exponential:	$Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.13$	$a = 40.05$	$b = 0.001218$
Hyperbolic:	$Y = a + b / X$	$R^2 = 0.38$	$a = 55.20$	$b = -12.69$
Logarithmic:	$Y = a + b \cdot \ln(X)$	$R^2 = 0.98$	$a = 24.33$	$b = 5.976$

Optimum :
H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$ $R^2 = 0.99$ $a = 17.82$ $n = 0.1579$ $\dot{\sigma}_0 = 14.56$

Rheogram and Data for Test Number Day 14, Test b

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.88$	$\dot{\sigma}_0 = 42.12$	$\text{Eta} = 0.03859$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.87$	$a = 29.69$	$n = 0.1042$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.85$	$a = 4.731$	$n = 0.2735$ $\dot{\sigma}_0 = 31.00$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.90$	$\dot{\sigma}_0 = 36.86$	$\text{Eta} = 0.005478$
Linear	: $Y = a + b * X$	$R^2 = 0.88$	$a = 42.12$	$b = 0.03859$
Inv. linear:	$Y = 1 / (a + b * X)$	$R^2 = 0.13$	$a = 0.02602$	$b = -24.36E-06$
Exponential:	$Y = a * \text{EXP}(b * X)$	$R^2 = 0.43$	$a = 40.78$	$b = 910.4E-06$
Hyperbolic:	$Y = a + b / X$	$R^2 = 0.20$	$a = 51.08$	$b = -9.764$
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R^2 = 0.86$	$a = 26.72$	$b = 4.725$

Optimum :
Casson : $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$ $R^2 = 0.90$ $\dot{\sigma}_0 = 36.86$ $\text{Eta} = 0.005478$

Rheogram and Data for Test Number Day 28, Test a

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.81$	$\dot{\sigma}_0 = 50.68$	$\text{Eta} = 0.08841$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.81$	$a = 17.90$	$n = 0.2635$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.98$	$a = 16.28$	$n = 0.2534$ $\dot{\sigma}_0 = 9.717$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.81$	$\dot{\sigma}_0 = 37.07$	$\text{Eta} = 0.02568$
Linear	: $Y = a + b * X$	$R^2 = 0.81$	$a = 50.68$	$b = 0.08841$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.05$	$a = 0.02643$	$b = -44.49E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.43$	$a = 48.74$	$b = 0.001518$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.25$	$a = 71.48$	$b = -39.29$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.99$	$a = 8.060$	$b = 12.26$
 Optimum :				
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.99$	$a = 8.060$	$b = 12.26$

Rheogram and Data for Test Number Day 28, Test a

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.96$	$\dot{\sigma}_0 = 46.10$	$\text{Eta} = 0.08253$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.92$	$a = 26.53$	$n = 0.1716$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.93$	$a = 5.276$	$n = 0.3569$ $\dot{\sigma}_0 = 30.11$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.97$	$\dot{\sigma}_0 = 36.50$	$\text{Eta} = 0.01941$
Linear	: $Y = a + b * X$	$R^2 = 0.96$	$a = 46.10$	$b = 0.08253$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.13$	$a = 0.02427$	$b = -33.20E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.58$	$a = 45.04$	$b = 0.001488$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.12$	$a = 65.05$	$b = -13.12$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.85$	$a = 15.63$	$b = 9.621$

Optimum :
Casson : $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$ $R^2 = 0.97$ $\dot{\sigma}_0 = 36.50$ $\text{Eta} = 0.01941$

3.4.2 Rheology of Modified Laboratory-Scale Feed Preparation Samples

The feed prepared in the laboratory with the modified feed preparation procedure, before silica and sodium nitrate addition and at an oxide loading of 236 gWO/L, had a plastic viscosity of 19.6 mPa•s and a yield stress of 9.2 Pa. After silica and sodium nitrate addition to the modified feed, the density was 1.29 g/ml, the pH was 8.3 (25°C), the solids loading was 43.5%, and the oxide loading was 403 gTO/L. At these conditions, the plastic viscosity was 15.7 mPa•s and the yield stress was 7.3 Pa (50°C). The summary Table B3.4.2 of all data from the requisite rheograms and each individual rheogram follow.

Table B3.4.2 Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Modified Laboratory-Scale Melter Feed Preparation Samples.

		<u>g TO/L</u>	<u>pH (22°C)</u>	<u>wt % solids</u>	<u>Density (g/ml)</u>	<u>Yield Stress (Pa)^a</u>	<u>Plastic Viscosity (mPa·s)^b</u>	<u>Correlation Coeff. (R²)^c</u>
<u>Alternate HTM Flowsheet 2^d</u>								
<u>Modified Laboratory-Scale^e:</u>								
Before silica addition:								
File ID/Date:	day0glyc/11-10-94	236	8.3	29.2	1.22	9.2	19.6	0.92
	day0glyb/11-10-94					10.5	16.6	0.89
After silica addition:								
File ID/Date:	SI16ly0a/11-10-94	403	8.3	43.5	1.29	7.3	15.7	0.91
	SI16ly0b/11-10-94					7.9	13.4	0.92

- (a) Laboratory rheological screening data value for the yield stress is 15 Pa. The yield stress was calculated by fitting the shear rate sweep data to the Bingham model.
- (b) Laboratory rheological screening data value for the plastic viscosity is 25 mPa·s. The plastic viscosity was calculated by fitting the shear rate sweep data to the Bingham model.
- (c) Least squares correlation coefficient of shear rheology data fit to the Bingham equation.
- (d) Alternate HTM Flowsheet 2 is the glycolic acid process flowsheet.
- (e) Rheology measurements were taken at 50°C for the modified laboratory-scale feeds.

Rheogram and Data for Test Number day0glyc

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R=0.92	$\dot{\sigma}_0 = 9.211$	Eta = 0.01961
Ostwald	: $\dot{\sigma} = a * D^n$	R=0.87	a = 3.735	n = 0.2483
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=0.98	a = 2.886	n = 0.2616 $\dot{\sigma}_0 = 2.321$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R=0.90	$\dot{\sigma}_0 = 6.767$	Eta = 0.005712
Linear	: $Y = a + b * X$	R=0.92	a = 9.211	b = 0.01961
Inv. linear	: $Y = 1 / (a + b * X)$	R=0.02	a = 1.895	b = -0.006092
Exponential	: $Y = a * \text{EXP}(b * X)$	R=0.22	a = 8.057	b = 0.002066
Hyperbolic	: $Y = a + b / X$	R=0.20	a = 13.80	b = -8.159
Logarithmic	: $Y = a + b * \text{LN}(X)$	R=0.92	a = 0.9329	b = 2.488

Optimum :

H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ R=0.98 a=2.886 n=0.2616 $\dot{\sigma}_0 = 2.321$

Rheogram and Data for Test Number day0glyb

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.89$	$\dot{\sigma}_0 = 10.54$	$\eta = 0.01662$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.87$	$a = 5.173$	$n = 0.1956$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.97$	$a = 3.291$	$n = 0.2269$ $\dot{\sigma}_0 = 3.570$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.87$	$\dot{\sigma}_0 = 8.312$	$\eta = 0.003889$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.89$	$a = 10.54$	$b = 0.01662$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.05$	$a = 0.1453$	$b = -267.3E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.35$	$a = 9.852$	$b = 0.0015$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.22$	$a = 14.41$	$b = -4.401$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.93$	$a = 3.540$	$b = 2.107$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.97$	$a = 3.291$	$n = 0.2269$	$\dot{\sigma}_0 = 3.570$

Rheogram and Data for Test Number SilGly0a

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.91$	$\dot{\sigma}_0 = 7.328$	$\text{Eta} = 0.01517$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.92$	$a = 3.126$	$n = 0.2371$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.96$	$a = 2.120$	$n = 0.2746$ $\dot{\sigma}_0 = 1.852$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.91$	$\dot{\sigma}_0 = 5.396$	$\text{Eta} = 0.004389$
Linear	: $Y = a + b * X$	$R^2 = 0.91$	$a = 7.328$	$b = 0.01517$
Inv. linear:	$Y = 1 / (a + b * X)$	$R^2 = 0.23$	$a = 0.1441$	$b = -195.3E-06$
Exponential:	$Y = a * \text{EXP}(b * X)$	$R^2 = 0.66$	$a = 7.328$	$b = 0.001586$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.12$	$a = 10.74$	$b = -0.08011$
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R^2 = 0.89$	$a = 2.257$	$b = 1.669$

Optimum :
H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ $R^2 = 0.96$ $a = 2.120$ $n = 0.2746$ $\dot{\sigma}_0 = 1.852$

Rheogram and Data for Test Number SilGly0b

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R=0.92	$\dot{\sigma}_0 = 7.930$	Eta = 0.01337
Ostwald	: $\dot{\sigma} = a * D^n$	R=0.94	a = 4.156	n = 0.1864
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=0.97	a = 1.885	n = 0.2660 $\dot{\sigma}_0 = 3.382$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R=0.94	$\dot{\sigma}_0 = 6.255$	Eta = 0.003154
Linear	: $Y = a + b * X$	R=0.92	a = 7.930	b = 0.01337
Inv. linear	: $Y = 1 / (a + b * X)$	R=0.13	a = 0.1440	b = -197.6E-06
Exponential	: $Y = a * \text{EXP}(b * X)$	R=0.54	a = 7.680	b = 0.001452
Hyperbolic	: $Y = a + b / X$	R=0.24	a = 11.10	b = -8.467
Logarithmic	: $Y = a + b * \text{LN}(X)$	R=0.92	a = 2.258	b = 1.701

Optimum :
H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ R=0.97 a=1.885 n=0.2660 $\dot{\sigma}_0 = 3.382$

3.5 Standard Crucible Redox Test Data

Introduction

This section addresses the measurement of a glass oxidation state indicator, the $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratio for the laboratory-scale feed preparation. One melter feed and two methods of glass preparation are discussed for Alternate HTM Flowsheet 2. Two melter feeds for each flowsheet, one prepared in the laboratory and one prepared in the small-scale feed preparation system were melted using a standard crucible method. The measured $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratios are given in Table B3.5.

Experimental

Based on gram total oxide (gTO) loading, enough melter feed was placed in a quartz crucible to produce 25 g glass. Samples were dried in air at 98°C for 24 hours. After drying, a ceramic lid with a wide lip was placed on top of the quartz crucible, and this assembly was placed in a furnace at room temperature. The crucible was heated from room temperature to 1400°C at a rate of 5°C per minute or 15°C per minute. After soaking 30 minutes at 1400°C, the crucible was removed from the furnace and allowed to air cool.

The iron redox ratio of the resulting glass was measured by colorimetric titration of the glass¹. The glass is ground to a powder and dissolved under non-oxidizing conditions using a mixture of sulfuric and hydrofluoric acids. Boric acid is added to complex the excess fluoride ion. The ferrous ion in the solution is chelated by three molecules of phenanthroline, forming an orange-red complex. This colored solution obeys Beer's law. The total iron in the sample is determined by reducing the ferric ion to a ferrous ion with hydroquinone at room temperature. A 4% error has been estimated for this method when used to determine the redox state of typical simulated nuclear waste glasses².

Results

The relative quantities of oxidants and reductants are given in Table B3.5. Relative to the laboratory-scale melter feed, a slightly higher ratio of reductant to oxidant was measured for the small-scale; however, the sensitivity of the $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratio to differences of this order of magnitude is unknown. The $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratio of glasses prepared from the laboratory-scale melter feeds, using the standard crucible method, was 0.061 (mean value). Further analyses of the cold cap and high temperature redox chemistries will be addressed in the SSHTM-1 letter report, milestone T3C-95-129.

Table B3.5 Standard Crucible Redox Test Data for Laboratory-Scale Melter Feed Alternate HTM Flowsheet 2 Testing.

	Final pH (22°C)	Moles Oxidant ^a (gTO/L)	Moles Reductant ^b		Glass: Fe ⁺² /2Fe	Crucible	Melter
			NO ₃ ⁻	NO ₂ ⁻ (glycolate)			
Alternate HTM Flowsheet 2^c							
laboratory melter feed ^d	8.3	403	0.64	0.23	0.48	N/A	N/A
dried melter feed/glass	N/A	N/A	----	----	----	0.0610	N/A
	N/A	N/A	----	----	----	0.0599	N/A

- (a) Reported moles of NO₃⁻ are after addition of NaNO₃. The NaNO₃ contributed 0.47 moles of nitrate per 125 gWO.
- (b) Reductant used are moles of C₂H₃O₃⁻ per 125 gWO.
- (c) Alternate HTM Flowsheet 2 is the alternate reductant, glycolic acid process flowsheet; laboratory data from test T95-SSHTMGLYC-1.
- (d) Laboratory melter feed made from the T95-SSHTMGLYC-1 test slurry on Nov. 10, 1994.

4.0 SMALL-SCALE MELTER FEED PREPARATION DATA

4.1 Flowsheet Outline and Activity Data Log

Small Scale Flowsheet

- Step 1: Load mixing tank with slurry simulant components and concentrate to ~179 gWO/L. Use air as a cover gas and maintain slurry agitation. Set up off-gas analysis system and begin measurements of off-gas composition.
- Step 2: Bring slurry simulant temperature to 93°C and start acid addition. Add 70.6 wt% glycolic acid solution via a peristaltic pump to the simulant at successively greater rates as done during the small-scale feed preparation. Glycolic acid is added to obtain a pH of ~5 (95°C). The approach to pH ~5 was handled as follows: A) Add acid at the prescribed rate to a pH of 4.8 ± 0.1 as read via a pH probe. B) Stop acid addition and continue stirring for 5 minutes. C) If the pH is ≤ 5.0 , then add no more acid, if pH > 5.0 then return to step 1, part A.
- Take samples (feed and condensate)
- Step 3: Concentrate the treated slurry by boiling to an oxide loading of 240 gWO/L (or 337.5 kg WO in the tank for small-scale feed preparation).
- Take samples (feed and condensate)
- Step 4: Bag in 12 g Na_2O per 100 gWO as 32.0 g NaNO_3 per 100 gWO (or 111.05 kg NaNO_3 (40.5 kg Na_2O) for small-scale feed preparation).
- Take samples (feed and condensate)
- Step 5: Vacuum in 88 g SiO_2 as sand (F-110) per 100 gWO (or 297.0 kg SiO_2 for small-scale feed preparation). Final target HTM feed concentrations are 433 gTO/L. Note: this is the calculated loading based on a slurry simulant loading of 240 gWO/L. Final estimated volume is 1559 L or 412.3 gallons.
- Take samples (feed and condensate)

Small-Scale Melter Feed Preparation Activity Log

The "Small-Scale Melter Feed Preparation Activity Log" is a transcription of the actual log kept during the test run. Though it does not contain all of the entries (redundant entries were eliminated) it contains all of the information of the original log. Its main purpose is to provide a record of observations made during the test that were not being recorded by

other means and that would correlate with other data such as the off-gas data. The basis for correlation with other data is "24 hour" military time. The observations recorded relate principally to the slurry condition: color, foaming, other phases present and other activities carried out during the test run along with comments about the operation of the small-scale feed preparation system itself.

The Date/Time column gives the day the run activity was occurring and the "24 hour" time that the data was recorded or an activity commenced. The cumulative time log, provided in minutes, provides the reader information that can be used to cross reference the off-gas data presented in graphical form in section 4.3 of this appendix. Zero time is defined as the start of acid addition and the final log entry is entered at the time off-gas data collection stopped. Slurry temperature ($^{\circ}\text{C}$) was recorded from a temporary readout on the third floor of the high bay near the off-gas cart. The pH was a manually recorded value from the pH probe (temperature compensated for laboratory tests but not for the small-scale system). The millivolt readings were taken from the pH meter at the same time the pH value was read as a check of the uncompensated pH value. The "Glycolic Addition" column provides the weight of glycolic acid added in the time period between each noted entry. The value was calculated from readings taken from the drum scale on which the nitric acid barrel was placed during acid addition. The Notes/Observations column attempts to succinctly express relevant activities or actions at the time indicated in the first column. Activities may include:

- Heating or cooling the slurry.
- Observations about the slurry such as slurry color or degree of foaming.
- Condensate or slurry sample collection.
- Unusual off-gas activity.
- Start of acid addition and the rate of addition.

The "Small-Scale Melter Feed Preparation Activity Log," Log B4.1, for the small-scale glycolic acid processed melter feed follows.

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
8/8/94						
11:00	-	-	-	-	-	Started SSHTM-1 feed preparation test run per run plan ETC-001-FPTS-RP01, Rev. 0. Stage ten 208 L drums of Optima slurry. Drum numbers: 6314, 6308, 6313, 6312, 6339, 6338, 6337, 6336, 6580, and 6341. Eight of these drums were full and two were approximately 2/3 full.
17:00	-	-	-	-	-	Stirred and pumped slurry from each drum into HB-32 on 3rd floor highbay. Each drum rinsed with water and rinse added to HB-32. Slurry allowed to mix for ~2 hours with agitator speed set at 117 rpm.
19:00	-	-	-	-	SSHTM1-6.2.6-1	Removed 250 ml sample for analysis by dipping from top of tank HB-32.
21:00	-	-	-	-	-	Added 19.20 kg of NaNO ₃ and 81.83 kg NaNO ₂ to HB-32. Slurry is moving smoothly in a radial motion near coil support. Some vortex motion near coil support. Agitator left at 117 rpm.
8/9/94						
8:30	-	-	-	-	SSHTM1-6.3.8-1	Removed 250 ml sample for analysis by dipping from top of tank HB-32.
12:16	-	-	-	-	-	Opened steam to 10% output.
12:21	-	-	-	-	-	Increased steam to 30%.
12:28	-	-	-	-	-	Increased steam to 40% output..
13:49	-	-	-	-	-	Reduced steam to 30% output.
13:54	-	-	-	-	-	Increased steam to 35% output.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/9/94</u>						
13:55	-	-	-	-	-	HB-32 boiling.
15:00	-	102.8	-	-	-	Temperature recorded on data log from a computer hook up to thermocouple TI-2049 slot 1 channel 3 on HB-32. Thermocouple reading at ~10.4 cm below weld crease on bottom portion of tank.
16:20	-	-	-	-	-	Added fourth drum of slurry to HB-32. Four Optima slurry drums added to replace condensate loss through initial concentration stage. The drums used were 6315, 6311, 6310, and 6309.
21:45	-	-	-	-	-	Turned cooling water on to HB-32.
22:10	-	-	-	-	-	Slurry thick and not mixing well. Turned agitator speed up to 155 rpm. Mixing improved even flow and no stagnant areas.
23:12	-	-	-	-	SSHTM1-6.4.12-1	Removed seven samples : Three 250 ml slurry samples for analysis.
-	-	-	-	-	SSHTM1-6.4.12-2	
-	-	-	-	-	SSHTM1-6.4.12-3	
-	-	-	-	-	SSHTM1-6.4.12-4	Removed four 500 ml slurry samples. Sample 4 and 5 archived.
-	-	-	-	-	SSHTM1-6.4.12-5	
-	-	-	-	-	SSHTM1-6.4.12-6	Sample 6 and 7 submitted for preacid addition test and rheology.
23:24	-	-	-	-	SSHTM1-6.4.12-7	
<u>8/10/94</u>						
10:50	-	-	-	-	-	Slurry mixing well. Some noticeable slow mixing near tank walls. Some caking/buildup noticed on coil support.
11:05	-	-	-	-	-	Moved one drum of 70% Glycolic acid to 3rd floor highbay.

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/10/94</u>						
12:05	-	64.7	-	-	-	Opened steam to 20% output for beginning of tank heating for acid addition.
12:18	-	-	-	-	-	Increased steam to 30% output.
13:17	-	93.9	-	-	-	Reduced steam to 20% output.
13:19	-	-	-	-	-	Closed steam control valve.
13:51	-	96.7	-	-	-	HB-32 temperature appears to have leveled out.
15:00	-				SSHTMGLYC 8/10/94 test1-a test-1a test-1b test-1c test-1d	Removed 5 L slurry sample for lab. Submitted to Ray Bell. Rheology samples renamed from initial sample SSHTMGLYC-8/10/94.
15:13	0	93.1	10.4	0.00kg	-	Start of Glycolic acid addition. NOx observed with onset of acid addition. *Note: Hydrogen was measured on the gas chromatograph with a Below Detection Limit of 15 ppm. Carbon Dioxide was measured on an Infrared CO ₂ Analyzer with a BDL of <0.05%. NOx is measured on a Chemiluminescent NO/NOx Analyzer in ppm with a BDL of <5 ppm.
15:27	14	94.1	9.7	-	-	Steam off. Small amount of H ₂ observed with CO ₂ concentration reading ~1.01%. Appears to be mixing vigorously. No foaming.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/10/94</u>						
15:30	17	93.8	-	-	-	Removed two 100 ml initial concentration condensate samples from condensate drum #266. Sample number 8 archived.
-					SSHTM1-6.4.12-8	Sample number 9 submitted for analysis.
-					SSHTM1-6.4.12-9	
-					SSHTM1-8.0.10-1	Removed 100 ml condensate sample from first drum #247 for analysis.
15:40	27					
15:33	20	-	-	36kg	-	Acid addition rate 1.8 kg/min.
15:40	27	-	8.5	52kg	-	Acid addition rate 2.28 kg/min.
15:44	31	-	8.0	60kg	-	Acid addition rate 2 kg/min.
15:48	35	-	7.5	68kg	-	Off-gas reading CO2 (1.5%), NOx (1074 ppm), and H2 (7 ppm). Acid addition rate 2 kg/min.
15:52	39	-	7.0	77kg	-	Off-gas reading CO2 (3.4%), NOx (1069 ppm), and H2 (10 ppm). Foaming. Acid addition rate 2.25 kg/min.
15:55	42	-	-	-	-	Opened steam to 10% output.
15:57	44	-	6.8	-	-	Off-gas reading CO2 (4.1%), NOx (842 ppm), and H2 (<d.l. (more gas dilution)).
15:58	45	-	-	90kg	-	Acid addition rate 2.16 kg/min.
15:59	46	-	6.7	-	-	Steam on 10% from main steam line downstairs. Line leading from main steam line to tank HB-32 open full.
16:00	47	-	6.6	94kg	-	Steam line to tank HB-32 open full. Foam grew to a thick layer and then stabilized. Acid addition rate 2 kg/min.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/10/94</u>						
16:01	48	90.7	6.6	-	-	Low temperature observed 90.03 degree C. Surface foam approximately 3-4 cm thick.
16:05	52	-	-	-	-	Off-gas reading CO2 (4.6%), NOx (845 ppm), and H2 (<d.l.).
16:07	54	-	6.3	108kg	-	Acid addition rate 2 kg/min.
16:10	57	-	-	-	-	Foam thickness decreasing.
16:12	59	-	6.1	118kg	-	Off-gas reading CO2 (4.2%), NOx (1128 ppm), and H2 (<d.l.). Acid addition rate 2 kg/min.
16:17	64	-	5.8	130kg	-	Acid addition rate 2.4 kg/min.
16:19	66	-	5.7	-	-	Off-gas reading CO2 (3.6%), NOx (1555 ppm), and H2 (<d.l.)
16:23	70	91.1	5.4	147kg	-	Surface foam possibly decreasing. Acid addition rate 2.83 kg/min.
16:26	73	-	5.2	148kg	-	Acid addition rate 0.33 kg/min.
16:29	76	91.6	5.0	156kg	-	Acid addition rate 2.66 kg/min.
16:30	77	-	-	-	-	Increased steam to 20% output.
16:31	78	-	4.9	161kg	-	Off-gas reading CO2 (2.39%), NOx (2329 ppm), and H2 (<d.l.) Acid addition rate 2.5 kg/min.
16:33	80	-	4.9	162kg	-	Acid addition completed.
16:35	82	-	5.0	-	-	Off-gas reading CO2 (0.91%), NOx (1782 ppm), and H2 (<d.l.)

Log B4.1 (Cont.)

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/10/94</u>						
16:38	85	-	5.1	-	-	Decision made not to add more acid with the exception of minor addition from line flush.
16:40	87	-	5.1	-	-	Off-gas reading CO2 (0.673%), NOx (1255 ppm), and H2 (3 ppm).
16:48	95	-	5.2	-	-	Off-gas reading CO2 (0.673%), NOx (2664 ppm), and H2 (4 ppm).
16:50	97	-	-	-	SSHTM1-8.0.10-2	Removed three 250ml post acid addition slurry samples for analysis. Removed two 1 L samples. Sample number 5 submitted to Rheology samples renamed from initial sample SSHTM1-8.0.10-5
.					SSHTM1-8.0.10-3	
.					SSHTM1-8.0.10-4	
.					SSHTM1-8.0.10-5	
.					test-1e	
.					test-1f	
.					test-1g	
.					test-1h	
17:02	109	96.7	5.3	-	SSHTM1-8.0.10-6	Ray Bell. Sample number 6 archived.
17:42	149	100.3	5.6	-	-	pH probe reading very erratic. Suspect probe failure. Installed backup probe. Original probe ok.
17:55	162	100.4	-	-	-	Adjusted steam control valve to 25% output. Steam flowmeter registering 126 pph.
17:58	165	100.1	5.9	-	-	Opening steam line from 20% to 22% output.
18:46	213	100.3	-	-	-	pH erratic again. Changing probes. Erratic behavior in probe is believed to be from boiling which would cause intermittent contact with the probe.
18:59	226	-	-	-	-	Condensate drum #247 weight is 149 kg.

Log B4.1 (Cont.)

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/10/94</u>						
19:05	232	100.2	-	-	-	Foaming in HB-32.
19:06	233	100.3	-	-	-	Opened steam valve to 30%.
19:09	237	-	-	-	-	Steam flow rate is 113 pph. Steam is at 30% output.
19:12	240	100.4	-	-	-	Heavy foaming. Reduced steam to 25%.
19:20	248	-	-	-	-	Adjusted water cooling supply to condenser on HB-32 this caused a low pressure in the header of cooling system. The main pump turned off. Restarted pump. Before adjustment of cooling system the off gas temperature had risen from ~31 degrees (C) to ~34 degrees (C).
19:40	268	-	-	-	-	Added ~0.24 L of Dow Corning Anti-Foam Compound lot #GA100322. Sample #SIPT2-9.5.4 taken for analysis. Analytical request form #M90534-13.
19:41	269	-	-	-	-	206 kg of condensate has been collected in drum #247.
20:00	287	-	-	-	-	First condensate drum #247 full starting second condensate collection drum #229.
20:25	312	100.7	-	-	-	Off gas reading H2. (19 ppm).
20:29	316	-	-	-	-	Condensate in drum #229 is 37 kg.
20:33	320	-	-	-	-	Slurry appears to have a thick pudding consistency with some foam. Added ~0.24 L anti-foam. Addition of anti-foam appears to have slurry moving freely.
20:46	333	-	7.2	-	-	Slurry appears fluid. No foam.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
8/10/94						
20:56	343	-	-	-	-	Increased steam output from 25% to 30% ~130 pph to 170 pph.
22:01	408	101.0	-	-	-	Condensate in drum #229 is 179 kg.
22:05	412	101.0	7.6	-	-	Slurry appears fluid.
22:29	436	-	-	-	SSHTM1-9.0.7-1	Second condensate drum #229 full. Total condensate weight of 207 kg. Removed 100 ml condensate sample from drum #229 for analysis. Started third drum #423.
22:30	437	101.1	-	-		
23:15	482	-	-	-	SSHTM1-9.0.7-2	Removed 100 ml condensate sample from drum #247 for analysis.
23:20	487	-	-	-	SSHTM1-9.0.7-3	Removed 100 ml condensate sample from drum #423 for analysis.
8/11/94						
00:15	542	101.8	7.5	-	-	
00:18	545	-	-	-	-	Decreased steam from 30% to 20% output.
00:22	549	-	-	-	-	Off-gas data collection stopped at this time, therefore, the cumulative time log entries are discontinued. Digestion complete. Refluxing condensate. Steam off. Slurry thick. Fluid is stagnant near tank wall. Near coil supports slurry motion stops, then will flow with mixing action then stop again.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
8/11/94 00:35	-	99.5	-	-	SSHTM1-9.0.7-4 SSHTM1-9.0.7-5 SSHTM1-9.0.7-6 SSHTM1-9.0.7-7 SSHTM1-9.0.7-8 SSHTM1-9.0.7-9 test-1l test-1j test-1k test-1l	Removed three 250 ml post acid addition concentration, slurry samples. Sample number 4 and 5 for analysis. Removed two 1 L post acid addition concentration, slurry samples. Sample number 7 submitted to Ray Bell. Sample number 8 archived. One 2 L sample removed for rheology. Rheology samples renamed from initial sample SSHTM1-9.0.7-9.
11:30	-	-	-	-	-	Slurry mixing poorly. Stagnant areas observed within 5 cm of tank periphery and ~5 cm around pipes and support brackets. Caking observed in these areas appears to be wet and have not dried out. Added water from hose. Caking around pipe and racket cleaned well and went back to liquid. Mixing improved. Top of coil just barely visible. Stagnant periphery.
13:45	-	-	-	-	SSHTMGLYCDIGEST test1-2a test1-2b test1-1-1 test1-1a test1-2	Removed slurry sample for rheology tests. Rheology sample renamed from initial sample SSHTMGLYCDIGEST.
15:00	-	-	-	-	SSHTMGLYCDIGEST/ REDOX	Mixing has improved. Still stagnant within ~5 to 8 cm of tank periphery. Redox sample taken.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
 Alternate HTM Flowsheet 2 (Alternate Reductant)
 Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (5)	Notes/Observations (5)
8/12/94						
9:00	-	-	-	-	-	Mixing deteriorated overnight. No movement within ~15 cm of tank periphery. Slow movement from outside the coil across the top of the coil. Caking and buildup observed within 2.5 cm of pipe and support bracket.
10:45	-	-	-	-	SSHTMW/GLYCOLIC AT 10:40 POST DIG. test-1o test-1p test-1m test-1n	Sample taken from HB-32 Post Glycolic for rheology. Rheology sample renamed from Initial sample SSHTMW/GLYCOLIC AT 10:45 POST DIG. Ready to transfer slurry from tank HB-32 to HB-33. Load cells on HB-33 reading 45 kg.
11:00	-	-	-	-	-	Transfer complete. Approximately 38 L of water used to flush HB-32. There were only slight slurry deposits on the piping and structures that penetrated the slurry surface. HB-33 load cells read 1352 kg. Mixing has improved and slurry appears to be more fluid.
13:30	-	-	-	-	SSHTMTANK33 AT 13:30 POST GLYC DIG Tst-1q test-1r test-1t test-1ta test-1s	Removed slurry sample from HB-33 for rheology tests. Rheology sample renamed from Initial sample SSHTMTANK33 AT 13:30 POST GLYC DIG.
8/13/94						
15:00	-	-	-	-	GLYCOLIC DIGESTION TANK 33 AT 15:00 test-u test-ua test-v test-va	Removed slurry sample for rheology tests from HB-33. Rheology sample renamed from Initial sample GLYCOLIC DIGESTION TANK 33 AT 15:00.

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/13/94</u>						
15:30	-	-	-	-	-	Slurry for flowsheet 2 in HB-33 mixing poorly around tank periphery. Increased agitator speed from 137 rpm to 155 rpm. Mixing improved, but still stagnant around periphery.
17:50	-	-	-	-	-	Mixing vigorously with no stagnant areas.
19:10	-	-	-	-	-	Mixing vigorously with no stagnant areas.
<u>8/14/94</u>						
13:55	-	-	-	-	FPHNO3 AT 13:48 test-1w test1-wa test-1z test-1za	Removed 250 ml slurry sample for rheology. Submitted to Ray Bell. Rheology sample renamed from Initial sample FPHNO3 AT 13:48.
14:20	-	-	-	-	-	Weighed out 120 kg of NaNO3 for addition to HB-33.
15:32	-	-	-	-	-	Completed adding 120 kg NaNO3 to HB-33 with vacuum conveyor system.
16:46	-	-	-	-	SSHTM1-11.1.7-1 1ab1 test1ab2 test1aa test1aa1 SSHTM1-11.1.7-2 SSHTM1-11.1.7-3 SSHTM1-11.1.7-4 SSHTM1-11.1.7-5	Collected samples from HB-33. Removed two 1 L post NaNO3 slurry samples. Sample number 1 for rheology. Sample number 2 archived. Removed three 250 ml slurry samples. Sample number 3 and 4 for analysis. Sample number 5 archived. Rheology sample renamed from Initial sample SSHTM1-11.1.7-1.
17:10	-	-	-	-	-	Began adding SiO2 to HB-33.

Log B4.1 (Cont.)

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/10/94</u>						
16:38	85	-	5.1	-	-	Decision made not to add more acid with the exception of minor addition from line flush.
16:40	87	-	5.1	-	-	Off-gas reading CO2 (0.673%), NOx (1255 ppm), and H2 (3 ppm).
16:48	95	-	5.2	-	-	Off-gas reading CO2 (0.673%), NOx (2664 ppm), and H2 (4 ppm).
16:50	97	-	-	-	SSHTM1-8.0.10-2	Removed three 250ml post acid addition slurry samples for analysis.
-					SSHTM1-8.0.10-3	
-					SSHTM1-8.0.10-4	
-					SSHTM1-8.0.10-5	
-					test-1e	
-					test-1f	Removed two 1 L samples. Sample number 5 submitted to Rheology samples renamed from initial sample SSHTM1-8.0.10-5
-					test-1g	
-					test-1h	
17:02	109	96.7	5.3	-	SSHTM1-8.0.10-6	Ray Bell. Sample number 6 archived.
17:42	149	100.3	5.6	-	-	pH probe reading very erratic. Suspect probe failure. Installed backup probe. Original probe ok.
17:55	162	100.4	-	-	-	Adjusted steam control valve to 25% output. Steam flowmeter registering 126 pph.
17:58	165	100.1	5.9	-	-	Opening steam line from 20% to 22% output.
18:46	213	100.3	-	-	-	pH erratic again. Changing probes. Erratic behavior in probe is believed to be from boiling which would cause intermittent contact with the probe.
18:59	226	-	-	-	-	Condensate drum #247 weight is 149 kg.

Log B4.1 (Cont.)

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/10/94</u>						
19:05	232	100.2	-	-	-	Foaming in HB-32.
19:06	233	100.3	-	-	-	Opened steam valve to 30%.
19:09	237	-	-	-	-	Steam flow rate is 113 pph. Steam is at 30% output.
19:12	240	100.4	-	-	-	Heavy foaming. Reduced steam to 25%.
19:20	248	-	-	-	-	Adjusted water cooling supply to condenser on HB-32 this caused a low pressure in the header of cooling system. The main pump turned off. Restarted pump. Before adjustment of cooling system the off gas temperature had risen from ~31 degrees (C) to ~34 degrees (C).
19:40	268	-	-	-	-	Added ~0.24 L of Dow Corning Anti-Foam Compound lot #GA100322. Sample #SIPT2-9.5.4 taken for analysis. Analytical request form #M90534-13.
19:41	269	-	-	-	-	206 kg of condensate has been collected in drum #247.
20:00	287	-	-	-	-	First condensate drum #247 full starting second condensate collection drum #229.
20:25	312	100.7	-	-	-	Off gas reading H2 (19 ppm).
20:29	316	-	-	-	-	Condensate in drum #229 is 37 kg.
20:33	320	-	-	-	-	Slurry appears to have a thick pudding consistency with some foam. Added ~0.24 L anti-foam. Addition of anti-foam appears to have slurry moving freely.
20:46	333	-	7.2	-	-	Slurry appears fluid. No foam.

Log B4.1 (Cont.)

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
8/10/94						
20:56	343	-	-	-	-	Increased steam output from 25% to 30% ~130 pph to 170 pph.
22:01	408	101.0	-	-	-	Condensate in drum #229 is 179 kg.
22:05	412	101.0	7.6	-	-	Slurry appears fluid.
22:29	436	-	-	-	SSHTM1-9.0.7-1	Second condensate drum #229 full. Total condensate weight of 207 kg. Removed 100 ml condensate sample from drum #229 for analysis. Started third drum #423.
22:30	437	101.1	-	-		
23:15	482	-	-	-	SSHTM1-9.0.7-2	Removed 100 ml condensate sample from drum #247 for analysis.
23:20	487	-	-	-	SSHTM1-9.0.7-3	Removed 100 ml condensate sample from drum #423 for analysis.
8/11/94						
00:15	542	101.8	7.5	-	-	
00:18	545	-	-	-	-	Decreased steam from 30% to 20% output.
00:22	549	-	-	-	-	Off-gas data collection stopped at this time, therefore, the cumulative time log entries are discontinued. Digestion complete. Refluxing condensate. Steam off. Slurry thick. Fluid is stagnant near tank wall. Near coil supports slurry motion stops, then will flow with mixing action then stop again.

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
8/11/94 00:35	-	99.5	-	-	SSHTM1-9.0.7-4 SSHTM1-9.0.7-5 SSHTM1-9.0.7-6 SSHTM1-9.0.7-7 SSHTM1-9.0.7-8 SSHTM1-9.0.7-9 test-1l test-1j test-1k test-1l	Removed three 250 ml post acid addition concentration, slurry samples. Sample number 4 and 5 for analysis. Removed two 1 L post acid addition concentration, slurry samples. Sample number 7 submitted to Ray Bell. Sample number 8 archived. One 2 L sample removed for rheology. Rheology samples renamed from initial sample SSHTM1-9.0.7-9.
11:30	-	-	-	-	-	Slurry mixing poorly. Stagnant areas observed within 5 cm of tank periphery and ~5 cm around pipes and support brackets. Caking observed in these areas appears to be wet and have not dried out. Added water from hose. Caking around pipe and racket cleaned well and went back to liquid. Mixing improved. Top of coil just barely visible. Stagnant periphery.
13:45	-	-	-	-	SSHTMGLYCDIGEST test1-2a test1-2b test1-1-1 test1-1a test1-2	Removed slurry sample for rheology tests. Rheology sample renamed from initial sample SSHTMGLYCDIGEST.
15:00	-	-	-	-	SSHTMGLYCDIGEST/ REDOX	Mixing has improved. Still stagnant within ~5 to 8 cm of tank periphery. Redox sample taken.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/12/94</u>						
9:00	-	-	-	-	-	Mixing deteriorated overnight. No movement within ~15 cm of tank periphery. Slow movement from outside the coil across the top of the coil. Caking and buildup observed within 2.5 cm of pipe and support bracket.
10:45	-	-	-	-	SSHTMW/GLYCOLIC AT 10:40 POST DIG. test-1o test-1p test-1m test-1n	Sample taken from HB-32 Post Glycolic for rheology. Rheology sample renamed from initial sample SSHTMW/GLYCOLIC AT 10:45 POST DIG. Ready to transfer slurry from tank HB-32 to HB-33. Load cells on HB-33 reading 45 kg.
11:00	-	-	-	-	-	Transfer complete. Approximately 38 L of water used to flush HB-32. There were only slight slurry deposits on the piping and structures that penetrated the slurry surface. HB-33 load cells read 1352 kg. Mixing has improved and slurry appears to be more fluid.
13:30	-	-	-	-	SSHTMTANK33 AT 13:30 POST GLYC DIG Tst-1q test-1r test-1t test-1ta test-1s	Removed slurry sample from HB-33 for rheology tests. Rheology sample renamed from initial sample SSHTM TANK33 AT 13:30 POST GLYC DIG.
<u>8/13/94</u>						
15:00	-	-	-	-	GLYCOLIC DIGESTION TANK 33 AT 15:00 test-u test-ua test-v test-va	Removed slurry sample for rheology tests from HB-33. Rheology sample renamed from initial sample GLYCOLIC DIGESTION TANK 33 AT 15:00.

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/13/94</u>						
15:30	-	-	-	-	-	Slurry for flowsheet 2 in HB-33 mixing poorly around tank periphery. Increased agitator speed from 137 rpm to 155 rpm. Mixing improved, but still stagnant around periphery.
17:50	-	-	-	-	-	Mixing vigorously with no stagnant areas.
19:10	-	-	-	-	-	Mixing vigorously with no stagnant areas.
<u>8/14/94</u>						
13:55	-	-	-	-	FPHNO3 AT 13:48 test-1w test1-wa test-1z test-1za	Removed 250 ml slurry sample for rheology. Submitted to Ray Bell. Rheology sample renamed from Initial sample FPHNO3 AT 13:48.
14:20	-	-	-	-	-	Weighed out 120 kg of NaNO3 for addition to HB-33.
15:32	-	-	-	-	-	Completed adding 120 kg NaNO3 to HB-33 with vacuum conveyor system.
16:46	-	-	-	-	SSHTM1-11.1.7-1 1ab1 test1ab2 test1aa test1aa1 SSHTM1-11.1.7-2 SSHTM1-11.1.7-3 SSHTM1-11.1.7-4 SSHTM1-11.1.7-5	Collected samples from HB-33. Removed two 1 L post NaNO3 slurry samples. Sample number 1 for rheology. Sample number 2 archived. Removed three 250 ml slurry samples. Sample number 3 and 4 for analysis. Sample number 5 archived. Rheology sample renamed from Initial sample SSHTM1-11.1.7-1.
17:10	-	-	-	-	-	Began adding SiO2 to HB-33.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
8/14/94						
18:05	-	-	-	-	-	Stopped adding SiO ₂ to HB-33. Total addition is 320 kg. Load cell reading 1658 kg including vacuum feed system. Ultrasonic level indicates 1609 L (LT2011). Agitator on.
19:14	-	-	-	-	SSHTM1-11.1.12-1 test-d01 test-d01a test-d0 test-d0a test-D01 test-D01A test-DO test-DOA	Removed 1 L slurry post SiO ₂ slurry sample for rheology. Rheology samples renamed from Initial sample SSHTM1-11.1.12-1. Lab-scale rheology aging study.
-	-	-	-	-	SSHTM1-11.1.12-2	Removed three 250 ml post SiO ₂ slurry samples for analysis.
-	-	-	-	-	SSHTM1-11.1.12-3	
-	-	-	-	-	SSHTM1-11.1.12-4	
-	-	-	-	-	SSHTM1-11.1.12-5	Removed two 1 L post SiO ₂ slurry samples for Ray Bell.
19:25	-	-	-	-	SSHTM1-11.1.12-6	
8/15/94						
10:00	-	-	-	-	-	HB-33 mixing well and much faster now than last night.
10:15	-	-	-	-	SSHTM1-15.2-1 test1ac1 test1ac test1ae test1ae1 testd1 testd1a testd15a testd15b	Removed 250 ml slurry sample for aging tests. Rheology samples renamed from Initial sample SSHTM1-15.2-1. Lab-scale rheology aging study.

Log B4.1 (Cont.)

**Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition**

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/15/94</u>						
14:17	-	-	-	-	-	Started HB-34 pump and Slurry Transfer Loop testing. Will test Glycolic melter feed through 1 1/2", 1", and 1/2" lines per SSHTM-1 run plan section 4.0. There is ~91 kg of feed in HB-34.
16:21	-	-	-	-	-	Completed Slurry Transfer Loop with the 1 1/2", 1", and 1/2" lines.
16:30	-	-	-	-	-	Flushed lines with air back to HB-34. Then flushed lines with water and air down to HB-15.
17:00	-	-	-	-	-	Pumped HB-34 Glycolic feed back to HB-33 melter feed tank.
13:33	-	-	-	-	-	Glycolic Melter feed run started.
<u>8/16/94</u>						
15:30	-	-	-	-	SSHTM1-15.2-2 testah testai testaf testag	Removed 250 ml aging slurry sample for rheology. Rheology samples renamed from initial sample SSHTM1-15.2-2.
<u>8/17/94</u>						
10:00	-	-	-	-	SSHTM1-15.2-3 testd3b testd3c testd3a testd3 day3a-52c day3b-52c testd3a test d3	Removed 250 ml aging slurry sample for rheology. Rheology samples renamed from initial sample SSHTM1-15.2-3. Lab-scale rheology aging study.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
<u>8/18/94</u> 11:00	-	-	-	-	SSHTM1-9.0.7-12 gly4a gly4b	Removed 250 ml slurry sample from HB-33 for rheology. Rheology samples renamed from initial sample SSHTM1-9.0.7-12.
<u>8/19/94</u> 10:01	-	-	-	-	SSHTM1GLYC10:01 day4-3b day4-3 day525ca day525cb day552ca day552cb	Rheology sample removed but not recorded in official sample log. Rheology samples renamed from initial sample SSHTM1GLYC10:01. Lab-scale rheology aging study.
<u>8/22/94</u> N/A	-	-	-	-	testd7 testd7a testd75 testd75a	Lab-scale rheology aging study.
<u>8/25/94</u> N/A	-	-	-	-	glyd10a glyd10b glyd10c glyd10d	Lab-scale rheology aging study.

Log B4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 2 (Alternate Reductant)
Glycolic Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	Glycolic (4) Addition	Sample Identification (6)	Notes/Observations (5)
8/29/94 N/A	.	.	.	.	glyd15b glyd15a gly15c glyd15d	Lab-scale rheology aging study.
9/7/94 N/A	.	.	.	.	t-25-31 t-25-31a t-52-31 t-52-31a	Lab-scale rheology aging study.

1. The time was taken from the off-gas computer.
2. Time after initiation of acid addition (min).
3. pH measurements are reported without a temperature compensation.
4. Totals representing acid addition is based on total acid added between each noted entry. A conversion from pounds to kilograms has been made.
5. Observations were made through the covered viewing window on the top of HB-32. A conversion from pounds to kilograms, inches to centimeters and gallons to liters have been made.
6. Lowercase sample names are aging rheology samples taken from tank and referred to on Table B4.4.1. Bold letter samples are from initial aging sample SSHTM1.11.1.12-1 used for lab-scale aging rheology samples from Table B4.4.2.

SSHTM-1 Sample Matrix

The following sample matrix, Table B4.1, was set up to identify samples to be taken during the Alternate HTM Flowsheet 1 feed preparation segment in the small-scale system. It has subsequently been augmented with sample numbers, dates of sampling, analytical request form numbers, and dates of analytical report issue. To correlate with the actual test activity, the sample numbers in this table have also been included in the activity log preceding this section. When samples were not taken either by test operator decision or oversight, "No sample taken" is indicated.

Table B4.1 Small-Scale Melter Feed Preparation Sample Matrix Which Includes: Sample Numbers, Dates of Sampling, Analytical Request Form Numbers, and Dates of Analytical Report Issue.

SSTHM 1, FLOWSHEET 2 (GLYCOLIC ACID)																11/18/94								
Item	General Information					Analyte Performed (after test activity)																		
	SSTHM-1 Test Activity	Approx. Tank Volume	# of Samples	Sample Size	Archive	wt% TS	wt% TO	pH Sp Gr	ICP	AA (Co)	IC	SIE (NH3)	Total C	Cres. Redox	Viscosity	Shear Stress	ON Gas	Sample Numbers	Date of Sample	Submitted for Analysis	ARF Number	Date Analysis Complete	Date Analytical Report Issued	
	LFCM-8 Feed Stock																							
1	Add Optima Pre-mixed Simulated Waste Slurry	503	1	250 mL	0	/	/	/	/		/						No	SSHTM1-8.2.8-1	8/8/94	8/8/94	K10185-1	8/8/94	8/8/94	
2	Add Chemicals and Shim as required	503	1	250 mL	0	/	/	/									No	SSHTM1-8.3.8-1	8/8/94	8/8/94	K10185-2	8/10/94	8/10/94	
3*	Concentrate	515	3	250 mL	0	1	1	1	1 (both)	1	1	1					No	SSHTM1-8.4.12-1	8/8/94	8/10/94	K10185-3	8/11/94	8/11/94	
																		SSHTM1-8.4.12-2	8/8/94	8/10/94	K10185-3	8/11/94	8/11/94	
																		SSHTM1-8.4.12-2	8/8/94	8/15/94	K10185-18	8/28/94	10/5/94	
																		SSHTM1-8.4.12-3	8/8/94	8/10/94	K10185-3	8/11/94	8/11/94	
																		SSHTM1-8.4.12-3	8/8/94	10/12/94	K27349-1	10/21/94	10/28/94	
3*	Concentrate	515	3	500 mL	2											1	1	No	SSHTM1-8.4.12-4, 5 & 6	8/8/94	8/10/94	NA		
	Add addition test sample	515	1	1.5 L	NA														SSHTM1-8.4.12-6, 7	8/8/94	8/10/94	NA		
		515	1	100 mL	1														SSHTM1-8.4.12-8	8/8/94		NA		
		515	1	100 mL	0														SSHTM1-8.4.12-9	8/8/94	8/12/94	K10185-8	8/00/94	8/8/94
4	Extra sample for Lab Tests	515	1	5 L	1												No							
5	Feed Preparation																No							
6*	pH Adjustment (Add Glycolic Acid); Measure Oil-Gas		3	250 mL		1	1	1	1 (both)	1	1	1					Yes	SSHTM1-8.0.10-2	8/10/94	8/15/94	K10185-18	8/28/94	10/5/94	
																		SSHTM1-8.0.10-3	8/10/94	8/11/94	K10185-5	8/00/94	8/8/94	
																		SSHTM1-8.0.10-2	8/10/94	10/12/94	K27349-1	10/21/94	10/28/94	
6*	pH Adjustment (Add Glycolic Acid); Measure Oil-Gas		2	1 L	1											1	1	Yes	SSHTM1-8.0.10-5 & 6	8/10/94		na		
6*	pH Adjustment (Add Glycolic Acid); Measure Oil-Gas		1 (Condensate)	100 mL				1			1	1	1					Yes	SSHTM1-8.0.10-1	8/10/94	8/12/94	K10185-7	8/30/94	8/30/94
7*	Concentration/Digestion		3	250 mL		1	1	1	1 (both)	1	1	1					Yes	SSHTM1-9.0.7-4	8/11/94	8/15/94	K10185-18	8/28/94	10/5/94	
																		SSHTM1-9.0.7-4	8/11/94	10/12/94	K27349-1	10/21/94	10/28/94	
																		SSHTM1-9.0.7-5	8/11/94	8/11/94	K10185-5	8/00/94	8/8/94	
																		SSHTM1-9.0.7-6	8/11/94					
7*	Concentration/Digestion		2	1 L	1										1	1	Yes	SSHTM1-9.0.7-7 & 8	8/11/94	8/11/94	NA			
																		SSHTM1-9.0.7-9	8/11/94	8/11/94	NA			
7*	Concentration/Digestion		1 (Condensate)	100 mL				1			1	1	1				Yes	SSHTM1-9.0.7-1	8/10/94	8/12/94	K10185-7	8/30/94	8/30/94	
																		SSHTM1-9.0.7-2	8/10/94	8/12/94	K10185-7	8/30/94	8/30/94	
																		SSHTM1-9.0.7-3	8/10/94	8/12/94	K10185-7	8/30/94	8/30/94	
8	Glass Former Addition																							
9*	Add Glass Formers (NaNO3)		3	250 mL	1	1	1	1	1 (both)	1	1	1					No	SSHTM1-11.1.7-3	8/14/94	8/15/94	K10185-11	8/00/94	8/20/94	
																		SSHTM1-11.1.7-4	8/14/94	8/15/94	K10185-18	8/28/94	10/5/94	
																		SSHTM1-11.1.7-5	8/14/94					

Table B4.1 (cont.) Small-Scale Melter Feed Preparation Sample Matrix Which Includes: Sample Numbers, Dates of Sampling, Analytical Request Form Numbers, and Dates of Analytical Report Issue.

Item	General Information					Analyte Performed (after test activity)																	
	SSHTM-1 Test Activity	Approx. Tank Volume	# of Samples	Sample Size	Analysis	wt% TS	wt% TO	pH Sp Gr	ICP	AA (Co)	IC	SE (NH3)	Total C	Cross. Redox	Viscosity	Shear Stress	OH Gas	Sample Numbers	Date of Sample	Submitted for Analysis	ARF Number	Date Analysis Complete	Date Analytical Report Issued
9*	Add Glass Formers (NaNO3)		2	1 L	2										1	1	No	SSHTM1-11.1.7-1,2	8/14/94				
9*	Add Glass Formers (NaNO3)		1 (Condensate)	100 mL				1			1	1	1				No						
10*	Add Glass Formers (SiO2)	1559 L	3	250 mL		1	1	1	1 (both)	1	1	1					No	SSHTM1-11.1.12-2	8/14/94	8/15/94	K10105-11	9/00/94	9/20/94
																		SSHTM1-11.1.12-3	8/14/94		K10105-19	9/29/94	10/5/94
																		SSHTM1-11.1.12-4	8/14/94	10/12/94	K27349-1	10/21/94	10/26/94
10*	Add Glass Formers (SiO2)	1559 L	3	1 L											1	1	No	SSHTM1-11.1.12-1, 5, 6	8/14/94				
10*	Add Glass Formers (SiO2)	1559 L	1	100 mL									1			No							
10*	Add Glass Formers (SiO2)	1559 L	1 (Condensate)	100 mL			1			1	1	1				No							
11	Concentrate/Dilute as Required	1559 L	3	30 mL		1	1	1									No						
11	Concentrate/Dilute as Required	1559 L	3	500 mL	2									1	1	No							
12	Slurry Test Loop Test	1559 L	NA	NA													No						
	Melter Feeding Support																						
13*	Melter Feeding (one sample every 10 hours)		4	250 mL		1	1	1	1 (Both)		1	1	1										
13*	Melter Feeding (one sample every 10 hours)		6	100 mL										1									
13*	Melter Feeding (one set of 3 samples every 10 hours)		4	250 mL	0										1	1		SSHTM1-15.2-1	8/15/94				
																		SSHTM1-15.2-2	8/16/94				
																		SSHTM1-15.2-3	8/17/94				
																		SSHTM1-9.0.7-12	8/18/94				
14	Extra sample for Lab Tests		1	5 L	1																		
	* Denotes samples required by test plan. Not all of the analyte will be performed, but the samples will be taken and available for analysis.																						

* Denotes samples required by test plan. Not all of the analysis will be performed, but the samples will be taken and available for analysis.

4.2 Melter Feed and Condensate Composition

Analytical Data Tables

Tables B4.2.1 and B4.2.2 summarize the results of the analyses of condensate and slurry samples taken during the slurry simulant processing into melter feed on the small-scale. The time and date that samples were taken is given by the activity log. The gWO/L and slurry density values allow the samples to be compared on the basis of a common gWO/L value. The anions and the total organic carbon (TOC) values are weight percent values based on the "as received" slurry simulant or melter feed material. On the other hand, the oxide weight percent values are based on the total oxide content of the slurry simulant or melter feed material which have been normalized to 100 %. When the sample is melter feed, the percent oxide values indicate the glass composition to be expected from melting this feed assuming that none of the oxides are lost by volatilization or decomposition other than the nitrates or carbonates. The data in the tables was obtained using the following procedures:

gWO/L and density-

Density was determined from the weight of a known volume of slurry under ambient conditions and the gWO/L was determined by reducing the pre-weighed slurry to oxides in a furnace at 1050°C for two hours. The accuracy of these measurements is on the order of 2-3%.

pH-

The pH measurements were made at ambient conditions using calibrated pH sensing electrodes "Orion" (PNL-ALO-501 Rev.1). The accuracy of these measurements is better than 0.1 pH unit.

Total Organic Carbon-

The total organic carbon was determined using the hot persulfate method (test procedure PNL-ALO-381, rev.0). The accuracy of this method estimated on the basis of its reproducibility is about 4% when the organic carbon makes up 1-2 % of the system by weight.

Anions-

The small-scale samples were analyzed by ion chromatography using a Dionex 300DX instrument using procedure APSL-12. The estimated accuracy is ± 5 to 10%.

Ammonia-

Ammonia is measured as the NH_4^+ ion with a standard ion electrode using procedure PNL-ALO-226 Rev.0. The accuracy is $\pm 15\%$ for concentrations < 1 ppm. For concentrations > 1 ppm the accuracy improves to $\pm 5\%$.

Elements, Oxides-

The elemental or oxide analyses were made by ICP-AES using procedure APSL-14. The estimated accuracy for major elements using this technique is $\pm 10\%$.

Table B4.2.1. Small-Scale Melter Feed Preparation Condensate Sample Analysis Results for Alternate Flowsheet 2 Processing. Note that pH values were measured under ambient conditions.

Processing stage	Sample Identification Number				
	SSHTM1- 6.4.12-9(2)	SSHTM1- 8.0.10-1(2)	SSHTM1- 9.0.7-2(2)	SSHTM1- 9.0.7-1(2)	SSHTM1- 9.0.7-3(2)
Initial Slurry concentration		Acid addition	Condensate barrel #1 Concentration digestion	Condensate barrel #2 Concentration digestion	Condensate barrel #3 Concentration digestion
pH	6.84	7.27	1.27	2.04	2.27
TOC (wt%)	N/A	N/A	N/A	N/A	N/A
Anions(ppm)					
fluoride	bd1 (a)	bd1 (a)	bd1 (a)	bd1 (a)	bd1 (a)
Chloride	bd1 (a)	bd1 (a)	bd1 (a)	bd1 (a)	bd1 (a)
nitrite	bd1 (a)	1.13	1436	bd1 (a)	bd1 (a)
nitrate	bd1 (a)	bd1 (a)	4797	776	391
phosphate	bd1 (a)	bd1 (a)	bd1 (a)	bd1 (a)	102
sulfate	bd1 (a)	bd1 (a)	bd1 (a)	bd1 (a)	bd1 (a)
Ammonia(ppm)	5.8	N/A	5.8	N/A	3.4
elements (ppm)					
Ag	0.00	0.00	0.00	N/A	N/A
Al	0.00	0.00	0.45	N/A	N/A
B	0.00	0.12	0.00	N/A	N/A
B	0.00	0.00	0.00	N/A	N/A
Be	0.00	0.00	0.00	N/A	N/A
Bi	0.00	0.00	0.00	N/A	N/A
Ca	0.11	0.17	0.42	N/A	N/A
Cd	0.00	0.00	0.00	N/A	N/A
Ce	0.00	0.00	0.00	N/A	N/A
Co	0.00	0.00	0.00	N/A	N/A
Cr	0.00	0.00	0.00	N/A	N/A
Cu	0.00	0.00	0.10	N/A	N/A
Dy	0.00	0.00	0.00	N/A	N/A
Eu	0.00	0.00	0.00	N/A	N/A
Fe	0.00	0.06	1.86	N/A	N/A
K	0.62	0.00	0.00	N/A	N/A
La	0.00	0.00	0.00	N/A	N/A
Li	0.00	0.00	0.00	N/A	N/A
Mg	0.00	0.00	0.00	N/A	N/A
Mn	0.00	0.00	0.16	N/A	N/A
Mo	0.00	0.00	0.00	N/A	N/A
Na	0.48	0.67	0.55	N/A	N/A
Nd	0.00	0.20	0.22	N/A	N/A
Ni	0.00	0.00	0.20	N/A	N/A
P	0.00	0.00	0.00	N/A	N/A
Pb	0.00	0.00	0.00	N/A	N/A
S	0.00	0.00	0.00	N/A	N/A
Si	0.00	0.00	0.68	N/A	N/A
Sn	0.00	0.00	0.00	N/A	N/A
Sr	0.00	0.00	0.00	N/A	N/A
Te	0.00	0.00	0.00	N/A	N/A
Ti	0.00	0.00	0.00	N/A	N/A
V	0.00	0.00	0.00	N/A	N/A
Y	0.00	0.00	0.00	N/A	N/A
Zn	0.02	3.97	4.05	N/A	N/A
Zr	0.00	0.00	0.00	N/A	N/A

(a) bd1 means below detection limit for the sample dilution. Concentrations of these anions should be similar to initial analysis adjusted for changes in concentration of slurry.

Table B4.2.2. Small-Scale Melter Feed Preparation Slurry Sample Analysis Results for Alternate HTM Flowsheet 2 Processing. Target composition of slurry in first column includes only elements actually analyzed for and found. Note that K₂O is also not included because ICP technique does not work well at dilution employed for analysis.

	Sample Identification Number						
	Target Composition	SSHTM1-6.2.6.1	SSHTM1-6.4.12-1,2,3	SSHTM1-8.0.10-3	SSHTM1-9.0.7-5	SSHTM1-11.1.7-3	SSHTM1-11.1.12-2
	NCAW slurry simulant	as received	NaNO ₂ , NaNO ₃ added	glycolic acid added	digest and concentrate	add NaNO ₃	add SiO ₂
gW/L	100	113	183	230	243	263	444
density	N/A	1.09	1.17	1.16	1.22	1.27	1.38
pH	N/A		11.74	5.25 ^(a)	7.5 ^(a)	8.6	8.6
TOC (wt%)(a)	0.0001	N/A	~0.01	1.52	2.08		1.67
Anions(ppm)							
Fluoride	90	bd1(b)	bd1(b)	0(c)	0(c)	0(c)	0(c)
Chloride	278	bd1(b)	bd1(b)	bd1(b)	bd1(b)	360	415
nitrite	14800	bd1(b)	24238	21900	22600	18781	21946
nitrate	5330	3317	10307	9940	13000	48653	58281
phosphate	72	bd1(b)	bd1(b)	bd1(b)	bd1(b)	bd1(b)	bd1(b)
sulfate	728	bd1(b)	bd1(b)	649	838	775	870
Ammonia(ppm)(a)	N/A	N/A	~0.2	0.66	0.9	0.7	0.58
Oxides(wt%)							
Ag2O	0.12	0.14	0.11	0.12	0.12	0.10	0.06
Al2O3	9.25	9.70	8.28	8.55	8.47	7.44	4.28
B2O3	0.01	0.08	0.09	0.09	0.09	0.24	0.16
BaO	0.18	0.19	0.16	0.16	0.17	0.14	0.10
CaO	0.81	0.46	0.39	0.39	0.39	0.39	0.22
CdO	3.08	3.44	2.88	2.62	2.87	2.46	1.44
CeO2	0.66	0.79	0.68	0.85	0.83	0.82	0.48
Cr2O3	0.27	0.31	0.27	0.27	0.26	0.24	0.13
CuO	0.25	0.30	0.24	0.25	0.26	0.24	0.13
Fe2O3	28.80	36.06	30.00	31.08	30.79	26.81	15.30
La2O3	0.67	0.73	0.61	0.63	0.62	0.53	0.32
MgO	0.37	0.58	0.54	0.60	0.49	0.48	0.32
MnO	2.19	2.68	2.27	2.26	2.31	2.03	1.15
MoO3	0.57	0.37	0.42	0.46	0.46	0.39	0.22
Na2O	21.90	8.70	20.37	19.13	20.52	30.39	16.96
Nd2O3	3.56	4.69	3.98	4.11	4.07	3.53	2.01
NiO	2.35	2.87	2.41	2.45	2.52	2.13	1.25
P2O5	0.89	0.84	0.58	0.61	0.55	0.48	0.32
PbO	0.72	0.76	0.68	0.69	0.62	0.58	0.35
SO3	0.67	0.72	0.95	0.88	0.90	0.87	0.54
SiO2	4.11	5.93	5.66	5.17	6.01	4.88	45.37
SrO	0.12	0.15	0.14	0.12	0.14	0.10	0.06
TeO2	0.10	0.14	0.15	0.15	0.00	0.10	0.06
TiO2	0.67	0.81	0.68	0.72	0.70	0.63	0.38
Y2O3	0.08	0.09	0.08	0.07	0.08	0.05	0.03
ZnO	0.34	1.02	0.88	1.06	1.12	0.48	0.29
ZrO2	15.40	17.67	14.75	15.08	14.99	13.00	7.38

- (a) These analyses were performed on sibling samples or pH probe was not temperature compensated.
 (b) bd1 means below detection limit for the sample dilution. Concentrations of these anions should be similar to initial analysis adjusted for changes in concentration of slurry.
 (c) Fluoride could not be differentiated from glycolate in this set of analyses.

4.3 Off-Gas Data

The off-gas record is organized in the following way:

1- Table B4.3 indicates the maximum generation rates for CO_2 , N_2O , NO_x , and H_2 and the cumulative amount of CO_2 , N_2O , NO_x , and H_2 measured.

2- Figures B4.3.1 and B4.3.2 present off-gas process profiles for the small-scale nitric acid processed melter feed. The off-gas generation rate of CO_2 , N_2O , NO_x , and H_2 is plotted as a function of time. Zero time is taken as the start of acid addition to the slurry simulant. The off-gas record continues through the digestion period of slurry processing. The slurry pH and temperature are also plotted.

The small-scale off-gas system used for analyzing off-gases (CO_2 , N_2O , NO_x , and H_2) during concentration, acid addition, and digestion is diagrammed in Figure 3.2.2. A pH probe was introduced into the slurry through a feed-through in the tank lid (see Figure 3.2.1). The gases were measured in the off-gas stream using a gas chromatograph to measure H_2 , infrared absorbance detectors for CO_2 and N_2O , and a chemiluminescent NO/NO_x analyzer to measure NO_x . The accuracy of the instrumentation is estimated at $\pm 2\%$, $\pm 3\%$ and $\pm 7\%$ respectively.

Table B4.3 Peak Off-Gas Generation Rates and Total Off-Gas Generated for the Alternate HTM Flowsheet 2 Small-Scale Test.

	Off-Gas Generated During Processing				
	CO ₂	N ₂ O	H ₂	NO _x	NH ₃
<u>Alternate HTM Flowsheet 2^a</u>					
Peak Generation Rate (moles/min)	10.4	BDL ^b	0.005	0.76	c
Total Off-Gas Generated (moles)	486	BDL ^b	1.3	207	0.19

(a) Alternate HTM Flowsheet 2 is the alternate reductant, glycolic acid process flowsheet.

(b) BDL: Below Detection Limit

(c) Not measured but usually equal to or less than the H₂ generation rate.

Figure B4.3.1 Process Profile for Small-Scale Alternate HTM Flowsheet 2 for the First 300 Minutes Measured from the Start of Glycolic Acid Addition.

Figure B4.3.2 Process Profile for Small-Scale Alternate HTM Flowsheet 2 for 0 to 600 Minutes Measured from the Start of Glycolic Acid Addition.

4.4 Rheology and Gram Waste Oxide Loading Data for Melter Feed

Introduction

For Alternate HTM Flowsheet 2, a preliminary melter feed was prepared in the laboratory (500 ml) to predict the off-gases, rheology, and redox potential of the melter feed. These laboratory tests were presented in the SSHTM-1 Test Plan Addendum and on the basis of these laboratory tests, the basic test procedures given in section 3.1 were developed. Next, the melter feed was prepared in a small-scale system (1514 liters). Due to the newness of the system and some related engineering difficulties, the processing test parameters deviated from the original processing test parameters. A third feed was prepared in the laboratory in which the processing test parameters performed during SSHTM-1 testing were duplicated. This third feed preparation procedure was completed so that laboratory- and small-scale data could be more easily compared. The rheology of the feed prepared in the small-scale system is discussed below. The data for the small-scale feed aged in the small-scale system is contained in section 4.4.1 and the data for the small-scale feed aged in the laboratory is contained in sections 4.4.2 and 4.4.1.

Experimental

Density, pH (22°C and 50°C), wt % solids, and gTO/L were measured for the melter feed using HWVP technical procedure WTC-006-4 before and after silica addition.

Measurements of rheological properties for the feed prepared in the small-scale tanks were taken at 25 and 50°C. Along with rheological data collected before and after silica and sodium nitrate addition, rheological data were also collected during the feed preparation process. Samples were collected from the tanks by dipping a bottle attached to a wooden pole into the tank from the top. No laboratory-scale rheological data were collected to compare with the rheological data collected during the feed preparation process.

The rheological values were measured using the Haake M5 measuring system with the MV2 sensor. Rheograms were generated by increasing the applied shear rate from 0 to 451 s⁻¹ in a 2-minute interval. (Two rheograms were generated for each sample and the samples were not reloaded into the rheometer between the two measurements.) The increasing shear rheology data were fit with the Bingham equation:

$$\tau = \tau_0 + \eta \dot{\gamma}$$

where:

τ = shear stress,
 τ_o = yield stress,
 $\dot{\gamma}$ = shear strain rate, and
 η = plastic viscosity.

During the small-scale run, after silica was added to the feed, samples were collected every day for four days for rheological testing. The temperature of the tank during this period was approximately 50°C. The rheology of the melter feed was measured using the procedure described above. Measurements were made at temperatures of 25 and 50°C.

Also, after silica addition, a 500 ml sample was taken from the small-scale melter feed tank (HB-33) and aged in the laboratory at 25°C and at 50°C. These results were compared with the results of the preliminary melter feed prepared and aged (at 25°C) in the laboratory.

To study aging in the laboratory, the melter feed was transferred to a mixing vessel. The mixing vessel was a closed system which was monitored for water losses by measuring changes in slurry weight as a function of time. Deionized water was added to the slurry if any water losses were measured. Samples were aged under constant agitation. During the aging process, samples were taken at times: 0 day, 1 day, 3 days, 5 days, 8 days, 11 days, 15 days, and 24 days for the sample prepared with the small-scale system. These samples were subjected to the following tests:

- rheological characterization (at 25°C and 50°C)
- weight percent solids of dry melter feed
- pH measurement (at 22°C and 50°C).

Results

4.4.1 Rheology of Small-Scale Feed Preparation and Aging Samples

The waste simulant prepared in the small-scale system, before silica and sodium nitrate addition and at an oxide loading of 234 gWO/L, had a solids loading of 28.3 wt%. At these conditions the plastic viscosity was 19.5 mPa·s and the yield stress was 17.8 Pa (50°C). After silica and sodium nitrate addition to the small-scale feed, the solids loading was 43.2%, and the oxide loading was 422 gTO/L. At these conditions, the plastic viscosity was 19.3 mPa·s and the yield stress was 16.3 Pa (50°C) (see Table B.4.4.1):

For the HTM feed prepared in the small-scale system and aged in the small-scale tank, yield stress and plastic viscosity did not increase with sample age. From day 0 to day 4, yield stress varied between 16.3 Pa and 21.8 Pa (50°C) and the plastic viscosity varied between 19.3 mPa·s to 18.3 mPa·s (50°C). The solids loading varied with values of 42 ± 1 weight percent. The pH (50°C) remained at 8.2 ± 0.2 (except for day 2). Rheological properties and pH were also measured at 25°C (see Table B.4.4.1).

Rheological characterization was also performed at various steps during the feed preparation procedure. These steps include:

- initial slurry in HB-32
- After acid addition in HB-32
- during digestion of glycolic acid in HB-32
- after digestion and transfer to tank HB-33
- after sodium nitrate addition in HB-33

Before concentration and acid addition in tank HB-32, the waste simulant had a plastic viscosity of 7.5 mPa·s and a yield stress of 3.6 Pa (at 50°C). After glycolic acid addition, the plastic viscosity was to 13.8 mPa·s and the yield stress was to 6.5 Pa. After the glycolic acid was added and the simulant digested, the simulant was transferred from tank HB-32 to HB-33 where it was concentrated to 230 gWO/L. At these conditions, the plastic viscosity was 15.7 mPas and the yield stress was 17.8 Pa (see Table B.4.4.1).

Table B4.4.1 Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Small-Scale Melter Feed Preparation, Aging.

		g TO/L	pH ($\approx 22^{\circ}\text{C}^{\text{a}}$)	Temperature ($^{\circ}\text{C}$)	wt % solids	Density (g/ml)	Yield Stress (Pa^{b})	Plastic Viscosity ($\text{mPa}\cdot\text{s}^{\text{c}}$)	Correlation Coeff. (R^2) ^d
<u>Alternate HTM Flowsheet 2^c</u>									
<u>Small-Scale Feed Prep. Samples:</u>									
gwo 178.8, initial:									
File ID/Date:	TEST-1/8-10-94	----	----	25	----	----	5.4	8.1	0.69
	TEST-1A/8-10-94	----	----	25	----	----	4.3	9.9	0.96
	TEST-1B/8-10-94	----	----	25	----	----	4.3	9.6	0.96
	TEST-1C/8-10-94	----	----	50	----	----	3.6	7.5	0.87
	TEST-1D/8-10-94	----	----	50	----	----	4.2	7.3	0.87
After acid addition:									
File ID/Date:	TEST-1G/8-10-94	----	----	25	----	----	9.2	20.9	0.72
	TEST-1H/8-10-94	----	----	25	----	----	6.8	26.2	0.97
	TEST-1E/8-10-94	----	----	50	----	----	6.5	13.8	0.82
	TEST-1F/8-10-94	----	----	50	----	----	5.9	15.1	0.93
At 00:35 hours, 8-11-94:									
File ID/Date:	TEST-1I/8-11-94	----	----	25	----	----	0.8	4.5	0.98
	TEST-1J/8-11-94	----	----	25	----	----	0.9	4.4	0.97
	TEST-1K/8-11-94	----	----	50	----	----	0.8	3.5	0.96
	TEST-1L/8-11-94	----	----	50	----	----	1.1	3.2	0.95
At 13:45, 8-11-94 Digest Glyc:									
File ID/Date:	TEST1-2/8-11-94	----	9.0	25	----	----	16.7	9.9	0.17
	TEST1-2A/8-11-94	----	----	25	----	----	12.5	23.9	0.89
	TEST1-2B/8-11-94	----	----	25	----	----	12.5	23.7	0.90
	TEST1-1/8-11-94	----	8.3	50	----	----	11.5	8.4	0.21
	TEST1-1A/8-11-94	----	----	50	----	----	10.2	11.6	0.73
Post Digest\glyc, Tank Transfer 32 to 33, at 13:30 hours, 8-11-94:									
File ID/Date:	TEST-1S/8-12-94	----	----	25	----	----	18.5	15.3	0.65
	TEST-1T/8-12-94	----	----	25	----	----	16.1	23.1	0.84
	TEST-1TA/8-12-94	----	----	25	----	----	15.4	23.8	0.83
	TEST-1Q/8-12-94	----	8.4	50	----	----	16.9	14.7	0.61
	TEST-1R/8-12-94	----	----	50	----	----	16.9	17.2	0.75

		<u>g TO/L</u>	<u>pH ($\approx 22^{\circ}\text{C}^a$)</u>	<u>Temperature ($^{\circ}\text{C}$)</u>	<u>wt % solids</u>	<u>Density (g/ml)</u>	<u>Yield Stress (Pa^b)</u>	<u>Plastic Viscosity ($\text{mPa}\cdot\text{s}^c$)</u>	<u>Correlation Coeff. (R^2)^d</u>
Post Digest\glyc at 10:40:									
File ID/Date:	TEST-10/8-12-94	234	8.8	25	28.3	1.2	15.0	25.4	0.81
	TEST-1P/8-12-94	----	----	25	----	----	15.8	21.6	0.89
	TEST-1M/8-12-94	----	8.3	50	----	----	17.8	19.5	0.75
	TEST-1N/8-12-94	----	----	50	----	----	18.7	17.8	0.72
Post Digest\glyc T33 time 15:00:									
File ID/Date:	TEST-V/8-13-94	----	----	25	----	----	20.3	16.6	0.57
	TEST-VA/8-13-94	----	----	25	----	----	17.0	23.2	0.83
	TEST-U/8-13-94	----	8.5	50	----	----	17.7	17.3	0.66
	TEST-Ua/8-13-94	----	----	50	----	----	17.5	18.3	0.73
Post Glyc T33, at 13:48 hours:									
File ID/Date:	TEST-1Z/8-14-94	----	8.6	25	----	----	18.3	21.7	0.68
	TEST-1ZA/8-14-94	----	----	25	----	----	17.0	22.2	0.88
	TEST-1W/8-14-94	----	8.5	50	----	----	17.8	15.7	0.59
	TEST-1WA/8-14-94	----	----	50	----	----	16.8	18.9	0.66
Post Digest with Glycolic and NaNO3 at 16:46 hrs, 8-14-94:									
File ID/Date:	TEST-1AA/8-14-94	----	8.5	25	----	----	17.3	14.3	0.60
	TEST1AA1/8-14-94	----	----	25	----	----	15.1	19.1	0.88
	TEST-1AB1/8-14-94	----	8.3	50	----	----	16.0	14.5	0.65
	TEST1AB2/8-14-94	----	----	50	----	----	12.2	13.2	0.70
SSHTM-1 with Glyc NaNO3, and F110, at 19:14 hrs, 8-11-94; Day 0:									
File ID/Date:	TEST-D01/8-14-94	422	8.4	25	43.2	1.3	17.7	17.0	0.72
	TEST-D01A/8-14-94	----	----	25	----	----	15.6	20.4	0.87
	TEST-D0/8-14-94	----	----	50	----	----	16.3	19.3	0.74
	TEST-D0A/8-14-94	----	----	50	----	----	17.6	19.1	0.75
SSHTM-1 with Glyc NaNO3, and F110, Day 1:									
File ID/Date:	TEST1AC/8-15-94	----	8.6	25	43.4	----	17.8	18.4	0.76
	TEST1AC1/8-15-94	----	----	25	----	----	16.3	21.3	0.83
	TEST1AE/8-15-94	----	8.1	50	----	----	16.8	18.6	0.75
	TEST1AE1/8-15-94	----	----	50	----	----	18.5	18.8	0.73

		g TO/L	pH ($\approx 22^{\circ}\text{C}^{\text{a}}$)	Temperature ($^{\circ}\text{C}$)	wt % solids	Density (g/ml)	Yield Stress (Pa^{b})	Plastic Viscosity ($\text{mPa}\cdot\text{s}^{\text{c}}$)	Correlation Coeff. (R^2) ^d
SSHTM-1 with Glyc NaNO ₃ , and F110, Day 2									
File ID/Date:	TEST1AH/8-16-94	----	----	25	43.4	----	24.5	15.6	0.47
	TEST1AI/8-16-94	----	----	25	----	----	19.2	26.6	0.80
	TEST1AF/8-16-94	----	7.7	50	----	----	23.1	19.8	0.57
	TEST1AG/8-16-94	----	----	50	----	----	22.0	23.1	0.74
SSHTM-1 with Glyc NaNO ₃ , and F110, Day 3									
File ID/Date:	TEST-D3B/8-17-94	----	8.5	25	40.7	----	20.6	18.9	0.67
	TEST-D3C/8-17-94	----	----	25	----	----	18.0	24.9	0.87
	TEST-D3/8-17-94	----	8.2	50	----	----	19.2	21.2	0.75
	TEST-D3a/8-17-94	----	----	50	----	----	19.9	22.9	0.75
SSHTM-1 with Glyc NaNO ₃ , and F110, Day 4, 11:34 am									
File ID/Date:	glyc4a/8-18-94	----	8.2	50	40.8	----	21.8	18.3	0.61
	gly4b/8-18-94	----	----	50	----	----	21.0	22.6	0.69

- (a) pH measured at 22°C unless the corresponding temperature value is listed as 50°C . If so, pH was measured at 50°C .
- (b) Laboratory rheological screening data value for the yield stress is 15 Pa. The yield stress was calculated by fitting the shear rate sweep data to the Bingham model. The plastic viscosity was calculated by fitting the shear rate sweep data to the Bingham model.
- (c) Laboratory rheological screening data value for the plastic viscosity is 25 mPa·s.
- (d) Least squares correlation coefficient of shear rheology data fit to the Bingham equation.
- (e) Alternate HTM Flowsheet 2 is the glycolic acid processed flowsheet.
- (f) File ID is the "name" of the rheogram and model fit data that follows this table.
- (g) Estimated value.

Rheogram and Data for Test Number TEST-1 @25c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.69	$\dot{\sigma}_0 = 5.385$	Eta = 0.008143
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.54	a = 1.311	n = 0.3267
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.79	a = 2.450	n = 0.1934 $\dot{\sigma}_0 = 0.5217$
Casson	: $\sqrt{\dot{\sigma}} = \sqrt{\dot{\sigma}_0} + \sqrt{\text{Eta} * D}$	R>=0.62	$\dot{\sigma}_0 = 4.083$	Eta = 0.002115
Linear	: $Y = a + b * X$	R>=0.69	a = 5.385	b = 0.008143
Inv. linear	: $Y = 1 / (a + b * X)$	R>=0.02	a = 1.493	b = -0.004545
Exponential	: $Y = a * \text{EXP}(b * X)$	R>=0.18	a = 4.879	b = 0.001556
Hyperbolic	: $Y = a + b / X$	R>=0.20	a = 7.269	b = -1.218
Logarithmic	: $Y = a + b * \text{LN}(X)$	R>=0.80	a = 1.779	b = 1.067
Optimum :				
Logarithmic	: $Y = a + b * \text{LN}(X)$	R>=0.80	a = 1.779	b = 1.067

Rheogram and Data for Test Number TEST-1A @25c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R=0.96	$\dot{\sigma}_0 = 4.319$	Eta = 0.009862
Ostwald	: $\dot{\sigma} = a * D^n$	R=0.96	a = 2.135	n = 0.2148
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=0.97	a = 0.6285	n = 0.3679 $\dot{\sigma}_0 = 2.184$
Casson	: $\dot{\sigma} - \dot{\sigma}_0 = \sqrt{\text{Eta} * D}$	R=0.98	$\dot{\sigma}_0 = 3.213$	Eta = 0.002818
Linear	: $Y = a + b * X$	R=0.96	a = 4.319	b = 0.009862
Inv. linear:	$Y = 1 / (a + b * X)$	R=0.50	a = 0.2333	b = -310.7E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R=0.79	a = 4.339	b = 0.001694
Hyperbolic	: $Y = a + b / X$	R=0.16	a = 6.625	b = -4.198
Logarithmic:	$Y = a + b * \text{LN}(X)$	R=0.88	a = 0.4515	b = 1.193

Optimum :

Casson : $\dot{\sigma} - \dot{\sigma}_0 = \sqrt{\text{Eta} * D}$ R=0.98 $\dot{\sigma}_0 = 3.213$ Eta = 0.002818

Rheogram and Data for Test Number TEST-1B @25c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	R>=0.96	$\dot{\sigma}_0=4.342$	$\eta=0.009622$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	R>=0.94	$a=2.233$	$n=0.2054$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	R>=0.96	$a=0.7086$	$n=0.3425$ $\dot{\sigma}_0=2.217$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	R>=0.98	$\dot{\sigma}_0=3.264$	$\eta=0.002673$
Linear	: $Y = a + b \cdot X$	R>=0.96	$a=4.342$	$b=0.009622$
Inv. linear:	$Y = 1 / (a + b \cdot X)$	R>=0.31	$a=0.2401$	$b=-330.4E-06$
Exponential:	$Y = a \cdot \exp(b \cdot X)$	R>=0.72	$a=4.321$	$b=0.001686$
Hyperbolic:	$Y = a + b/X$	R>=0.10	$a=6.546$	$b=-1.181$
Logarithmic:	$Y = a + b \cdot \ln(X)$	R>=0.86	$a=0.7626$	$b=1.127$

Optimum :

Casson : $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$ R>=0.98 $\dot{\sigma}_0=3.264$ $\eta=0.002673$

Rheogram and Data for Test Number TEST-1C @50c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R=0.87	$\dot{\sigma}_0 = 3.646$	Eta = 0.007457
Ostwald	: $\dot{\sigma} = a * D^n$	R=0.87	a = 1.332	n = 0.2659
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=0.93	a = 1.342	n = 0.2478 $\dot{\sigma}_0 = 0.4436$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R=0.84	$\dot{\sigma}_0 = 2.646$	Eta = 0.002222
Linear	: $Y = a + b * X$	R=0.87	a = 3.646	b = 0.007457
Inv. linear:	$Y = 1 / (a + b * X)$	R=0.03	a = 0.6713	b = -0.001662
Exponential:	$Y = a * \text{EXP}(b * X)$	R=0.33	a = 3.291	b = 0.001912
Hyperbolic	: $Y = a + b / X$	R=0.15	a = 5.361	b = -0.8749
Logarithmic:	$Y = a + b * \text{LN}(X)$	R=0.92	a = 0.5657	b = 0.9339

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=0.93	a = 1.342	n = 0.2478	$\dot{\sigma}_0 = 0.4436$

Rheogram and Data for Test Number TEST-1D @50c

Bingham : $\tau = \tau_0 + \eta \dot{\gamma}$ R²=0.87 τ_0 =4.206 η =0.007292
 Ostwald : $\tau = a \dot{\gamma}^n$ R²=0.96 a=1.983 n=0.2082
 H.-Bulkley : $\tau = \tau_0 + a \dot{\gamma}^n$ R²=0.95 a=1.228 n=0.2582 τ_0 =1.129
 Casson : $\sqrt{\tau} - \sqrt{\tau_0} = \sqrt{\eta} \sqrt{\dot{\gamma}}$ R²=0.89 τ_0 =3.215 η =0.001887
 Linear : $\tau = a + b \dot{\gamma}$ R²=0.87 a=4.206 b=0.007292
 Inv. linear: $\tau = 1/(a + b \dot{\gamma})$ R²=0.15 a=0.2785 b=-0.000401
 Exponential: $\tau = a \exp(b \dot{\gamma})$ R²=0.51 a=4.026 b=0.001523
 Hyperbolic : $\tau = a + b/\dot{\gamma}$ R²=0.17 a=5.892 b=-1.450
 Logarithmic: $\tau = a + b \ln(\dot{\gamma})$ R²=0.95 a=1.060 b=0.9391

Optimum :
 Ostwald : $\tau = a \dot{\gamma}^n$ R²=0.96 a=1.983 n=0.2082

Rheogram and Data for Test Number TEST-1G @25c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.72$	$\dot{\sigma}_0 = 9.230$	$\eta = 0.0209$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.67$	$a = 1.880$	$n = 0.3823$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.79$	$a = 3.115$	$n = 0.2774$ $\dot{\sigma}_0 = 0.6079$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.69$	$\dot{\sigma}_0 = 6.271$	$\eta = 0.007134$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.72$	$a = 9.230$	$b = 0.0209$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.03$	$a = 1.055$	$b = -0.003294$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.23$	$a = 7.553$	$b = 0.002359$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.13$	$a = 14.04$	$b = -2.762$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.76$	$a = 0.5386$	$b = 2.629$

Optimum :

H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$ $R^2 = 0.79$ $a = 3.115$ $n = 0.2774$ $\dot{\sigma}_0 = 0.6079$

Rheogram and Data for Test Number TEST-1H @25c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	R=0.97	$\dot{\sigma}_0 = 6.843$	$\eta = 0.0262$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	R=0.98	$a = 2.407$	$n = 0.3176$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	R=0.78	$a = 0.6977$	$n = 0.5188$ $\dot{\sigma}_0 = 1.964$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	R=0.98	$\dot{\sigma}_0 = 4.333$	$\eta = 0.01052$
Linear	: $\dot{\sigma} = a + b \cdot D$	R=0.97	$a = 6.843$	$b = 0.0262$
Inv. linear	: $\dot{\sigma} = 1 / (a + b \cdot D)$	R=0.30	$a = 0.1527$	$b = -273.8E-06$
Exponential	: $\dot{\sigma} = a \cdot \exp(b \cdot D)$	R=0.75	$a = 6.994$	$b = 0.00242$
Hyperbolic	: $\dot{\sigma} = a + b / D$	R=0.14	$a = 12.97$	$b = -11.32$
Logarithmic	: $\dot{\sigma} = a + b \cdot \ln(D)$	R=0.87	$a = -3.202$	$b = 3.125$

Optimum :

Casson : $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$ R=0.98 $\dot{\sigma}_0 = 4.333$ $\eta = 0.01052$

Rheogram and Data for Test Number TEST-1E @50c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R = 0.82$	$\dot{\sigma}_0 = 6.464$	$\text{Eta} = 0.0138$
Ostwald	: $\dot{\sigma} = a * D^n$	$R = 0.73$	$a = 1.701$	$n = 0.3306$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R = 0.88$	$a = 2.300$	$n = 0.2609$ $\dot{\sigma}_0 = 0.5915$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{(\text{Eta} * D)}$	$R = 0.78$	$\dot{\sigma}_0 = 4.522$	$\text{Eta} = 0.004457$
Linear	: $Y = a + b * X$	$R = 0.82$	$a = 6.464$	$b = 0.0138$
Inv. linear	: $Y = 1 / (a + b * X)$	$R = 0.02$	$a = 0.3849$	$b = -962.9E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R = 0.34$	$a = 6.070$	$b = 0.001833$
Hyperbolic	: $Y = a + b / X$	$R = 0.16$	$a = 9.654$	$b = -2.590$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R = 0.89$	$a = 0.5568$	$b = 1.768$

Optimum :

Logarithmic: $Y = a + b * \text{LN}(X)$ $R = 0.89$ $a = 0.5568$ $b = 1.768$

Rheogram and Data for Test Number TEST-1F @50c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.93$	$\dot{\sigma}_0 = 5.924$	$\text{Eta} = 0.01511$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.97$	$a = 2.361$	$n = 0.2632$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.98$	$a = 1.535$	$n = 0.3127$ $\dot{\sigma}_0 = 1.429$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.94$	$\dot{\sigma}_0 = 4.155$	$\text{Eta} = 0.004941$
Linear	: $Y = a + b * X$	$R^2 = 0.93$	$a = 5.924$	$b = 0.01511$
Inv. linear:	$Y = 1 / (a + b * X)$	$R^2 = 0.14$	$a = 0.2037$	$b = -353.4E-06$
Exponential:	$Y = a * \text{EXP}(b * X)$	$R^2 = 0.58$	$a = 5.734$	$b = 0.001969$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.12$	$a = 9.400$	$b = -2.499$
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R^2 = 0.91$	$a = -0.09575$	$b = 1.848$
Optimum :				
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.98$	$a = 1.535$	$n = 0.3127$ $\dot{\sigma}_0 = 1.429$

Rheogram and Data for Test Number TEST-11

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.98$	$\dot{\sigma}_0 = 0.8217$	$\text{Eta} = 0.004541$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.93$	$a = 0.1927$	$n = 0.4282$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.98$	$a = 0.1651$	$n = 0.4389$ $\dot{\sigma}_0 = 0.1588$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{(\text{Eta} * D)}$	$R^2 = 0.97$	$\dot{\sigma}_0 = 0.4397$	$\text{Eta} = 0.002267$
Linear	: $Y = a + b * X$	$R^2 = 0.98$	$a = 0.8217$	$b = 0.004541$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.05$	$a = 1.642$	$b = -0.00386$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.68$	$a = 0.8785$	$b = 0.002954$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.08$	$a = 1.861$	$b = -0.6256$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.83$	$a = -0.7974$	$b = 0.5181$
Optimum :				
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.98$	$a = 0.1651$	$n = 0.4389$ $\dot{\sigma}_0 = 0.1588$

Rheogram and Data for Test Number TEST-1J

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.97$	$\dot{\sigma}_0 = 0.8697$	$\eta = 0.004354$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.93$	$a = 0.2039$	$n = 0.4188$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.98$	$a = 0.1894$	$n = 0.4144$ $\dot{\sigma}_0 = 0.1591$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.96$	$\dot{\sigma}_0 = 0.4840$	$\eta = 0.002078$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.97$	$a = 0.8697$	$b = 0.004354$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.02$	$a = 8.086$	$b = -0.02534$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.42$	$a = 0.8141$	$b = 0.003203$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.06$	$a = 1.861$	$b = -0.2355$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.82$	$a = -0.6107$	$b = 0.4830$
Optimum :				
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.98$	$a = 0.1894$	$n = 0.4144$ $\dot{\sigma}_0 = 0.1591$

Rheogram and Data for Test Number TEST-1K @50C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.96$	$\dot{\sigma}_0 = 0.7894$	$\text{Eta} = 0.003456$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.92$	$a = 0.1952$	$n = 0.3963$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.98$	$a = 0.1770$	$n = 0.3944$ $\dot{\sigma}_0 = 0.1515$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.95$	$\dot{\sigma}_0 = 0.4596$	$\text{Eta} = 0.001556$
Linear	: $Y = a + b * X$	$R^2 = 0.96$	$a = 0.7894$	$b = 0.003456$
Inv. linear:	$Y = 1 / (a + b * X)$	$R^2 = 0.04$	$a = 1.949$	$b = -0.00459$
Exponential:	$Y = a * \text{EXP}(b * X)$	$R^2 = 0.61$	$a = 0.8117$	$b = 0.002638$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.08$	$a = 1.579$	$b = -0.3116$
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R^2 = 0.85$	$a = -0.4541$	$b = 0.3967$

Optimum :

H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ $R^2 = 0.98$ $a = 0.1770$ $n = 0.3944$ $\dot{\sigma}_0 = 0.1515$

Rheogram and Data for Test Number TEST-1L @50C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.95$	$\dot{\sigma}_0 = 1.059$	$\eta = 0.003248$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.96$	$a = 0.4471$	$n = 0.2652$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.98$	$a = 0.1100$	$n = 0.4595$ $\dot{\sigma}_0 = 0.5412$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.98$	$\dot{\sigma}_0 = 0.7222$	$\eta = 0.00114$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.95$	$a = 1.059$	$b = 0.003248$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.36$	$a = 0.9677$	$b = -0.001539$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.77$	$a = 1.070$	$b = 0.002096$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.14$	$a = 1.818$	$b = -1.407$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.87$	$a = -0.2054$	$b = 0.3912$

Optimum :

Casson : $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$ $R^2 = 0.98$ $\dot{\sigma}_0 = 0.7222$ $\eta = 0.00114$

Rheogram and Data for Test Number TEST1-2 @25C/8-11-94

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.17$	$\dot{\sigma}_0 = 16.68$	$\eta = 0.009857$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.51$	$a = 6.961$	$n = 0.1912$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.41$	$a = 8.698$	$n = 0.1378$ $\dot{\sigma}_0 = 1.053$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.25$	$\dot{\sigma}_0 = 13.90$	$\eta = 0.001797$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.17$	$a = 16.68$	$b = 0.009857$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.02$	$a = 0.2370$	$b = -618.9E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.10$	$a = 14.04$	$b = 0.001105$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.21$	$a = 19.04$	$b = -3.465$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.41$	$a = 9.342$	$b = 1.876$

Optimum :

Ostwald : $\dot{\sigma} = a \cdot D^n$ $R^2 = 0.51$ $a = 6.961$ $n = 0.1912$

Rheogram and Data for Test Number TEST1-2A @25C/8-11-94

Bingham : $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$ $R^2 = 0.89$ $\dot{\sigma}_0 = 12.48$ $\text{Eta} = 0.02386$
 Ostwald : $\dot{\sigma} = a * D^n$ $R^2 = 0.94$ $a = 5.544$ $n = 0.2247$
 H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ $R^2 = 0.67$ $a = 2.293$ $n = 0.3599$ $\dot{\sigma}_0 = 2.763$
 Casson : $\dot{\sigma} - \dot{\sigma}_0 = \sqrt{\text{Eta} * D}$ $R^2 = 0.91$ $\dot{\sigma}_0 = 9.381$ $\text{Eta} = 0.006515$
 Linear : $Y = a + b * X$ $R^2 = 0.89$ $a = 12.48$ $b = 0.02386$
 Inv. linear: $Y = 1 / (a + b * X)$ $R^2 = 0.18$ $a = 0.09162$ $b = -133.3E-06$
 Exponential: $Y = a * \text{EXP}(b * X)$ $R^2 = 0.56$ $a = 12.04$ $b = 0.001609$
 Hyperbolic : $Y = a + b / X$ $R^2 = 0.25$ $a = 18.15$ $b = -15.69$
 Logarithmic: $Y = a + b * \text{LN}(X)$ $R^2 = 0.95$ $a = 1.893$ $b = 3.127$

Optimum :
 Logarithmic: $Y = a + b * \text{LN}(X)$ $R^2 = 0.95$ $a = 1.893$ $b = 3.127$

Rheogram and Data for Test Number TEST1-2B @25C/8-11-94

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.90$	$\dot{\sigma}_0 = 12.52$	$\eta = 0.0237$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.95$	$a = 5.692$	$n = 0.2197$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.96$	$a = 3.849$	$n = 0.2583$ $\dot{\sigma}_0 = 3.051$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.91$	$\dot{\sigma}_0 = 9.461$	$\eta = 0.006373$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.90$	$a = 12.52$	$b = 0.0237$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.20$	$a = 0.08993$	$b = -127.6E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.57$	$a = 12.12$	$b = 0.001586$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.24$	$a = 18.14$	$b = -14.94$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.94$	$a = 2.082$	$b = 3.090$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.96$	$a = 3.849$	$n = 0.2583$ $\dot{\sigma}_0 = 3.051$

Rheogram and Data for Test Number TEST1-1-1 @50C/8-11-94

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.21$	$\dot{\sigma}_0 = 11.51$	$\eta = 0.008364$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.52$	$a = 3.956$	$n = 0.2332$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.52$	$a = 3.744$	$n = 0.2425$ $\dot{\sigma}_0 = 0.07712$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.30$	$\dot{\sigma}_0 = 9.247$	$\eta = 0.001791$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.21$	$a = 11.51$	$b = 0.008364$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.03$	$a = 0.1589$	$b = -290.3E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.14$	$a = 10.40$	$b = 986.5E-06$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.22$	$a = 13.52$	$b = -4.176$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.44$	$a = 5.730$	$b = 1.504$
Optimum :				
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.52$	$a = 3.956$	$n = 0.2332$

Rheogram and Data for Test Number TEST1-1A @50C/8-11-94

Bingham	: $g = g_0 + \text{Eta} * D$	R ² =0.73	$g_0=10.16$	Eta=0.0116
Ostwald	: $g = a * D^n$	R ² =0.82	$a=4.984$	$n=0.1915$
H.-Bulkley	: $g = g_0 + a * D^n$	R ² =0.85	$a=4.389$	$n=0.1791$ $g_0=1.677$
Casson	: $\sqrt{g - g_0} = \sqrt{\text{Eta} * D}$	R ² =0.72	$g_0=8.254$	Eta=0.002378
Linear	: $Y = a + b * X$	R ² =0.73	$a=10.16$	$b=0.0116$
Inv. linear	: $Y = 1 / (a + b * X)$	R ² =0.08	$a=0.1298$	$b=-0.000187$
Exponential	: $Y = a * \text{EXP}(b * X)$	R ² =0.31	$a=9.460$	$b=0.001211$
Hyperbolic	: $Y = a + b / X$	R ² =0.30	$a=12.89$	$b=-4.130$
Logarithmic	: $Y = a + b * \text{LN}(X)$	R ² =0.95	$a=4.364$	$b=1.649$

Optimum :
Logarithmic: $Y = a + b * \text{LN}(X)$ R²=0.95 a=4.364 b=1.649

Rheogram and Data for Test Number TEST-1S TANK TRANS. 32 to 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.65	$\dot{\sigma}_0=18.49$	Eta=0.01526
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.69	a=9.972	n=0.1522
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.87	a=8.701	n=0.1374 $\dot{\sigma}_0=4.267$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.60	$\dot{\sigma}_0=15.76$	Eta=0.002468
Linear	: $Y = a + b * X$	R>=0.65	a=18.49	b=0.01526
Inv. linear:	$Y = 1 / (a + b * X)$	R>=0.02	a=0.3615	b=-0.00106
Exponential:	$Y = a * \text{EXP}(b * X)$	R>=0.12	a=16.15	b=0.001184
Hyperbolic	: $Y = a + b / X$	R>=0.43	a=22.11	b=-6.319
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.93	a=10.36	b=2.270
 Optimum :				
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.93	a=10.36	b=2.270

Rheogram and Data for Test Number TEST-1T TANK TRANS. 32 to 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R=0.84	$\dot{\sigma}_0 = 16.12$	Eta = 0.02308
Ostwald	: $\dot{\sigma} = a * D^n$	R=0.89	a = 7.792	n = 0.1941
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=0.99	a = 5.189	n = 0.2227 $\dot{\sigma}_0 = 4.822$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R=0.84	$\dot{\sigma}_0 = 12.73$	Eta = 0.005327
Linear	: $Y = a + b * X$	R=0.84	a = 16.12	b = 0.02308
Inv. linear	: $Y = 1 / (a + b * X)$	R=0.07	a = 0.08356	b = -132.7E-06
Exponential	: $Y = a * \text{EXP}(b * X)$	R=0.37	a = 15.11	b = 0.001395
Hyperbolic	: $Y = a + b / X$	R=0.24	a = 21.51	b = -6.995
Logarithmic	: $Y = a + b * \text{LN}(X)$	R=0.97	a = 5.588	b = 3.086
Optimum :				
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=0.99	a = 5.189	n = 0.2227 $\dot{\sigma}_0 = 4.822$

Rheogram and Data for Test Number TEST-1TA TANK TRANS. 32 to 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.83$	$\dot{\sigma}_0 = 15.36$	$\eta = 0.02375$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.96$	$a = 7.624$	$n = 0.1931$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.94$	$a = 3.890$	$n = 0.2636$ $\dot{\sigma}_0 = 5.386$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.85$	$\dot{\sigma}_0 = 11.94$	$\eta = 0.005817$
Linear	: $\dot{\sigma} = a + b \cdot X$	$R^2 = 0.83$	$a = 15.36$	$b = 0.02375$
Inv. linear:	$\dot{\sigma} = 1 / (a + b \cdot X)$	$R^2 = 0.17$	$a = 0.07088$	$b = -86.69E-06$
Exponential:	$\dot{\sigma} = a \cdot \exp(b \cdot X)$	$R^2 = 0.55$	$a = 15.12$	$b = 0.001308$
Hyperbolic	: $\dot{\sigma} = a + b / X$	$R^2 = 0.16$	$a = 20.78$	$b = -0.8587$
Logarithmic:	$\dot{\sigma} = a + b \cdot \ln(X)$	$R^2 = 0.95$	$a = 6.280$	$b = 2.843$
Optimum :				
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.96$	$a = 7.624$	$n = 0.1931$

Rheogram and Data for Test Number TEST-1Q TANK TRANS. 32 to 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.61$	$\dot{\sigma}_0 = 16.93$	$\text{Eta} = 0.01473$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.58$	$a = 7.389$	$n = 0.1942$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.85$	$a = 8.839$	$n = 0.1367$ $\dot{\sigma}_0 = 2.375$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.54$	$\dot{\sigma}_0 = 14.07$	$\text{Eta} = 0.002701$
Linear	: $Y = a + b * X$	$R^2 = 0.61$	$a = 16.93$	$b = 0.01473$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.03$	$a = 0.08743$	$b = -134.7E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.20$	$a = 16.12$	$b = 931.6E-06$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.43$	$a = 20.42$	$b = -5.938$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.94$	$a = 8.778$	$b = 2.250$

Optimum :

Logarithmic: $Y = a + b * \text{LN}(X)$ $R^2 = 0.94$ $a = 8.778$ $b = 2.250$

Rheogram and Data for Test Number TEST-1R TANK TRANS. 32 to 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R = 0.75$	$\dot{\sigma}_0 = 16.89$	$\eta = 0.0172$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R = 0.82$	$a = 9.197$	$n = 0.1573$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R = 0.94$	$a = 6.712$	$n = 0.1713$ $\dot{\sigma}_0 = 4.495$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R = 0.75$	$\dot{\sigma}_0 = 14.08$	$\eta = 0.003148$
Linear	: $Y = a + b \cdot X$	$R = 0.75$	$a = 16.89$	$b = 0.0172$
Inv. linear:	$Y = 1 / (a + b \cdot X)$	$R = 0.09$	$a = 0.07243$	$b = -89.63E-06$
Exponential:	$Y = a \cdot \exp(b \cdot X)$	$R = 0.33$	$a = 15.94$	$b = 0.001076$
Hyperbolic	: $Y = a + b / X$	$R = 0.41$	$a = 21.05$	$b = -14.20$
Logarithmic:	$Y = a + b \cdot \ln(X)$	$R = 0.97$	$a = 8.054$	$b = 2.492$

Optimum :

Logarithmic: $Y = a + b \cdot \ln(X)$ $R = 0.97$ $a = 8.054$ $b = 2.492$

Rheogram and Data for Test Number TEST-10 @1040 @25c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R ² =0.81	$\dot{\sigma}_0=14.95$	Eta=0.02539
Ostwald	: $\dot{\sigma} = a * D^n$	R ² =0.70	a=4.513	n=0.2921
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R ² =0.99	a=5.207	n=0.2380 $\dot{\sigma}_0=2.715$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R ² =0.78	$\dot{\sigma}_0=10.95$	Eta=0.007332
Linear	: $Y = a + b * X$	R ² =0.81	a=14.95	b=0.02539
Inv. linear	: $Y = 1 / (a + b * X)$	R ² =0.02	a=10.80	b=-0.03584
Exponential	: $Y = a * \text{EXP}(b * X)$	R ² =0.14	a=11.81	b=0.002143
Hyperbolic	: $Y = a + b / X$	R ² =0.22	a=20.84	b=-5.643
Logarithmic	: $Y = a + b * \text{LN}(X)$	R ² =0.98	a=3.090	b=3.448

Optimum :
H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ R²=0.99 a=5.207 n=0.2380 $\dot{\sigma}_0=2.715$

Rheogram and Data for Test Number TEST-1P @1040 @25c

Bingham	: $\sigma = \sigma_0 + \eta \cdot D$	R=0.89	$\sigma_0 = 15.81$	$\eta = 0.02158$
Ostwald	: $\sigma = a \cdot D^n$	R=0.99	$a = 8.941$	$n = 0.1618$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot D^n$	R=0.77	$a = 1.746$	$n = 0.3729$
Casson	: $\sqrt{\sigma} - \sqrt{\sigma_0} = \sqrt{\eta \cdot D}$	R=0.95	$\sigma_0 = 12.87$	$\eta = 0.004478$
Linear	: $Y = a + b \cdot X$	R=0.89	$a = 15.81$	$b = 0.02158$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	R=0.21	$a = 0.06841$	$b = -77.30E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	R=0.57	$a = 15.38$	$b = 0.001225$
Hyperbolic	: $Y = a + b / X$	R=0.36	$a = 21.17$	$b = -32.27$
Logarithmic	: $Y = a + b \cdot \ln(X)$	R=0.97	$a = 5.782$	$b = 2.916$

Optimum :

Ostwald : $\sigma = a \cdot D^n$

R=0.99 a=8.941 n=0.1618

Rheogram and Data for Test Number TEST-1M @1040

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.75$	$\dot{\sigma}_0 = 17.78$	$\eta = 0.01952$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.82$	$a = 8.947$	$n = 0.1753$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.94$	$a = 7.093$	$n = 0.1783$ $\dot{\sigma}_0 = 4.342$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.74$	$\dot{\sigma}_0 = 14.56$	$\eta = 0.003874$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.75$	$a = 17.78$	$b = 0.01952$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.06$	$a = 0.08049$	$b = -126.6E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.28$	$a = 16.46$	$b = 0.001199$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.31$	$a = 22.35$	$b = -5.544$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.98$	$a = 8.126$	$b = 2.757$

Optimum :

Logarithmic: $Y = a + b \cdot \ln(X)$ $R^2 = 0.98$ $a = 8.126$ $b = 2.757$

Rheogram and Data for Test Number TEST-1N @1040

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R ² =0.72	$\dot{\sigma}_0=18.67$	Eta=0.01777
Ostwald	: $\dot{\sigma} = a * D^n$	R ² =0.90	a=11.27	n=0.1358
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R ² =0.67	a=5.048	n=0.2209 $\dot{\sigma}_0=6.808$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R ² =0.74	$\dot{\sigma}_0=15.79$	Eta=0.003036
Linear	: $Y = a + b * X$	R ² =0.72	a=18.67	b=0.01777
Inv. linear	: $Y = 1 / (a + b * X)$	R ² =0.23	a=0.05629	b=-48.23E-06
Exponential	: $Y = a * \text{EXP}(b * X)$	R ² =0.48	a=18.40	b=878.9E-06
Hyperbolic	: $Y = a + b / X$	R ² =0.25	a=22.76	b=-1.282
Logarithmic	: $Y = a + b * \text{LN}(X)$	R ² =0.96	a=10.95	b=2.312

Optimum :

Logarithmic: $Y = a + b * \text{LN}(X)$ R²=0.96 a=10.95 b=2.312

Rheogram and Data for Test Number TEST-V SAMPLE TIME 15:00 @25C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.57	$\dot{\sigma}_0 = 20.34$	Eta=0.01658
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.52	a=8.483	n=0.2007
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.73	a=12.11	n=0.1220 $\dot{\sigma}_0 = 1.387$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.48	$\dot{\sigma}_0 = 17.01$	Eta=0.002942
Linear	: $Y = a + b * X$	R>=0.57	a=20.34	b=0.01658
Inv. linear:	$Y = 1 / (a + b * X)$	R>=0.02	a=0.08395	b=-147.7E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R>=0.17	a=19.18	b=919.5E-06
Hyperbolic	: $Y = a + b / X$	R>=0.45	a=24.29	b=-7.347
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.88	a=11.06	b=2.553
 Optimum :				
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.88	a=11.06	b=2.553

Rheogram and Data for Test Number TEST-VA SAMPLE TIME 15:00 @25C.

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * \dot{D}$	$R = 0.83$	$\dot{\sigma}_0 = 17.03$	$\text{Eta} = 0.02324$
Ostwald	: $\dot{\sigma} = a * \dot{D}^n$	$R = 0.90$	$a = 8.434$	$n = 0.1872$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * \dot{D}^n$	$R = 0.99$	$a = 5.398$	$n = 0.2205$ $\dot{\sigma}_0 = 5.305$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * \dot{D}}$	$R = 0.84$	$\dot{\sigma}_0 = 13.55$	$\text{Eta} = 0.005212$
Linear	: $Y = a + b * X$	$R = 0.83$	$a = 17.03$	$b = 0.02324$
Inv. linear	: $Y = 1 / (a + b * X)$	$R = 0.08$	$a = 0.07625$	$b = -114.3E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R = 0.38$	$a = 16.00$	$b = 0.00134$
Hyperbolic	: $Y = a + b / X$	$R = 0.24$	$a = 22.44$	$b = -5.858$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R = 0.98$	$a = 6.348$	$b = 3.122$
 Optimum :				
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * \dot{D}^n$	$R = 0.99$	$a = 5.398$	$n = 0.2205$ $\dot{\sigma}_0 = 5.305$

Rheogram and Data for Test Number TEST-U SAMPLE TIME 15:00 @50C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.66$	$\dot{\sigma}_0 = 17.67$	$\text{Eta} = 0.01726$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.62$	$a = 7.543$	$n = 0.2023$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.86$	$a = 8.879$	$n = 0.1495$ $\dot{\sigma}_0 = 2.366$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.59$	$\dot{\sigma}_0 = 14.47$	$\text{Eta} = 0.003392$
Linear	: $Y = a + b * X$	$R^2 = 0.66$	$a = 17.67$	$b = 0.01726$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.02$	$a = 0.3932$	$b = -0.001164$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.13$	$a = 15.11$	$b = 0.001373$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.38$	$a = 21.75$	$b = -5.977$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.95$	$a = 8.501$	$b = 2.562$
 Optimum :				
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.95$	$a = 8.501$	$b = 2.562$

Rheogram and Data for Test Number TEST-Ua SAMPLE TIME 15:00 @50C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.73	$\dot{\sigma}_0 = 17.49$	Eta = 0.01834
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.77	a = 8.713	n = 0.1754
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.91	a = 7.433	n = 0.1688 $\dot{\sigma}_0 = 3.839$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.70	$\dot{\sigma}_0 = 14.38$	Eta = 0.003559
Linear	: $Y = a + b * X$	R>=0.73	a = 17.49	b = 0.01834
Inv. linear	: $Y = 1 / (a + b * X)$	R>=0.05	a = 0.08793	b = -147.4E-06
Exponential	: $Y = a * \text{EXP}(b * X)$	R>=0.25	a = 16.06	b = 0.001188
Hyperbolic	: $Y = a + b / X$	R>=0.33	a = 21.80	b = -6.021
Logarithmic	: $Y = a + b * \text{LN}(X)$	R>=0.97	a = 8.237	b = 2.625
Optimum :				
Logarithmic	: $Y = a + b * \text{LN}(X)$	R>=0.97	a = 8.237	b = 2.625

Rheogram and Data for Test Number TEST-1Z @25C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.68$	$\dot{\sigma}_0 = 18.33$	$\eta = 0.02167$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.72$	$a = 7.308$	$n = 0.2224$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.91$	$a = 7.581$	$n = 0.1930$ $\dot{\sigma}_0 = 2.602$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.67$	$\dot{\sigma}_0 = 14.35$	$\eta = 0.005093$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.68$	$a = 18.33$	$b = 0.02167$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.02$	$a = 0.4387$	$b = -0.001328$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.16$	$a = 15.60$	$b = 0.001541$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.36$	$a = 23.50$	$b = -12.35$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.99$	$a = 6.508$	$b = 3.275$
Optimum :				
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.99$	$a = 6.508$	$b = 3.275$

Rheogram and Data for Test Number TEST-1ZA @25C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R = 0.88$	$\dot{\sigma}_0 = 17.02$	$\eta = 0.02218$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R = 0.99$	$a = 9.720$	$n = 0.1578$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R = 0.99$	$a = 3.065$	$n = 0.2820$ $\dot{\sigma}_0 = 8.651$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R = 0.93$	$\dot{\sigma}_0 = 13.91$	$\eta = 0.004502$
Linear	: $Y = a + b \cdot X$	$R = 0.88$	$a = 17.02$	$b = 0.02218$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R = 0.10$	$a = 0.06815$	$b = -85.31E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R = 0.46$	$a = 16.33$	$b = 0.001225$
Hyperbolic	: $Y = a + b / X$	$R = 0.29$	$a = 22.34$	$b = -18.68$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R = 0.97$	$a = 6.782$	$b = 2.984$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R = 0.99$	$a = 3.065$	$n = 0.2820$ $\dot{\sigma}_0 = 8.651$

Rheogram and Data for Test Number TEST-1W @50C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.59$	$\dot{\sigma}_0 = 17.78$	$\eta = 0.01569$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.51$	$a = 6.405$	$n = 0.2311$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.76$	$a = 10.02$	$n = 0.1352$ $\dot{\sigma}_0 = 1.204$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.50$	$\dot{\sigma}_0 = 14.58$	$\eta = 0.003061$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.59$	$a = 17.78$	$b = 0.01569$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.02$	$a = 0.4257$	$b = -0.00127$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.12$	$a = 14.83$	$b = 0.001382$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.42$	$a = 21.50$	$b = -5.846$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.92$	$a = 8.972$	$b = 2.421$

Optimum :
Logarithmic: $Y = a + b \cdot \ln(X)$ $R^2 = 0.92$ $a = 8.972$ $b = 2.421$

Rheogram and Data for Test Number TEST-1WA @50C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.66	$\dot{\sigma}_0 = 16.81$	Eta = 0.01886
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.84	a = 7.663	n = 0.1956
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.44	a = 3.767	n = 0.3016 $\dot{\sigma}_0 = 2.934$
Casson	: $\sqrt{\dot{\sigma} - \dot{\sigma}_0} = \sqrt{a} * D$	R>=0.65	$\dot{\sigma}_0 = 13.38$	Eta = 0.004187
Linear	: $Y = a + b * X$	R>=0.66	a = 16.81	b = 0.01886
Inv. linear	: $Y = 1 / (a + b * X)$	R>=0.08	a = 0.07384	b = -97.07E-06
Exponential	: $Y = a * \text{EXP}(b * X)$	R>=0.32	a = 16.05	b = 0.001114
Hyperbolic	: $Y = a + b / X$	R>=0.22	a = 21.13	b = -0.2951
Logarithmic	: $Y = a + b * \text{LN}(X)$	R>=0.97	a = 8.669	b = 2.445

Optimum :
Logarithmic: $Y = a + b * \text{LN}(X)$ R>=0.97 a=8.669 b=2.445

Rheogram and Data for Test Number TEST-1AA @25C @1655 HRS

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.60$	$\dot{\sigma}_0 = 17.33$	$\eta = 0.01433$
Ostwald	: $\dot{\sigma} = a \cdot \dot{\gamma}^n$	$R^2 = 0.45$	$a = 6.775$	$n = 0.2134$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.81$	$a = 9.874$	$n = 0.1213$ $\dot{\sigma}_0 = 2.136$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot \dot{\gamma}}$	$R^2 = 0.48$	$\dot{\sigma}_0 = 14.51$	$\eta = 0.002521$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.60$	$a = 17.33$	$b = 0.01433$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.02$	$a = 2.131$	$b = -0.006949$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.09$	$a = 14.01$	$b = 0.00144$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.48$	$a = 20.76$	$b = -7.382$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.89$	$a = 9.537$	$b = 2.161$

Optimum :
Logarithmic: $Y = a + b \cdot \ln(X)$ $R^2 = 0.89$ $a = 9.537$ $b = 2.161$

Rheogram and Data for Test Number TEST-1AA1 @25C @1655 HRS

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.88	$\dot{\sigma}_0=15.07$	Eta=0.01908
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.98	a=8.687	n=0.1548
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.66	a=1.898	n=0.3529 $\dot{\sigma}_0=7.294$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.93	$\dot{\sigma}_0=12.41$	Eta=0.003756
Linear	: $Y = a + b * X$	R>=0.88	a=15.07	b=0.01908
Inv. linear:	$Y = 1 / (a + b * X)$	R>=0.14	a=0.07393	b=-84.99E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R>=0.50	a=14.55	b=0.001178
Hyperbolic	: $Y = a + b / X$	R>=0.40	a=19.84	b=-30.67
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.97	a=6.071	b=2.603
Optimum :				
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.98	a=8.687	n=0.1548

Rheogram and Data for Test Number TEST-1AB1 @50C @1655HRS

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.65	$\dot{\sigma}_0 = 16.04$	Eta = 0.01445
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.58	a = 7.243	n = 0.1887
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.80	a = 8.870	n = 0.1309 $\dot{\sigma}_0 = 1.886$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.56	$\dot{\sigma}_0 = 13.39$	Eta = 0.002595
Linear	: $Y = a + b * X$	R>=0.65	a = 16.04	b = 0.01445
Inv. linear:	$Y = 1 / (a + b * X)$	R>=0.02	a = 0.8657	b = -0.002722
Exponential:	$Y = a * \text{EXP}(b * X)$	R>=0.11	a = 13.53	b = 0.001359
Hyperbolic	: $Y = a + b / X$	R>=0.42	a = 19.47	b = -5.956
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.91	a = 8.474	b = 2.122

Optimum :

Logarithmic: $Y = a + b * \text{LN}(X)$ R>=0.91 a=8.474 b=2.122

Rheogram and Data for Test Number TEST1AB2 @50C @1655HRS

Bingham : $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$ $R = 0.70$ $\dot{\sigma}_0 = 12.25$ $\text{Eta} = 0.01321$
Ostwald : $\dot{\sigma} = a * D^n$ $R = 0.63$ $a = 4.930$ $n = 0.2171$
H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ $R = 0.92$ $a = 5.788$ $n = 0.1625$ $\dot{\sigma}_0 = 1.832$
Casson : $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$ $R = 0.63$ $\dot{\sigma}_0 = 9.875$ $\text{Eta} = 0.002781$
Linear : $Y = a + b * X$ $R = 0.70$ $a = 12.25$ $b = 0.01321$
Inv. linear: $Y = 1 / (a + b * X)$ $R = 0.03$ $a = 0.1272$ $b = -217.3E-06$
Exponential: $Y = a * \text{EXP}(b * X)$ $R = 0.24$ $a = 11.65$ $b = 0.001094$
Hyperbolic : $Y = a + b / X$ $R = 0.36$ $a = 15.37$ $b = -4.821$
Logarithmic: $Y = a + b * \text{LN}(X)$ $R = 0.97$ $a = 5.377$ $b = 1.932$

Optimum :
Logarithmic: $Y = a + b * \text{LN}(X)$ $R = 0.97$ $a = 5.377$ $b = 1.932$

Rheogram and Data for Test Number TEST-D01 @25c @2200hr TANK 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.72$	$\dot{\sigma}_0 = 17.73$	$\eta = 0.01699$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.71$	$a = 9.422$	$n = 0.1597$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.90$	$a = 8.451$	$n = 0.1467$ $\dot{\sigma}_0 = 3.544$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.67$	$\dot{\sigma}_0 = 14.89$	$\eta = 0.002988$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.72$	$a = 17.73$	$b = 0.01699$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.03$	$a = 0.09612$	$b = -173.8E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.21$	$a = 16.22$	$b = 0.001132$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.45$	$a = 21.84$	$b = -13.81$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.94$	$a = 8.984$	$b = 2.464$
 Optimum :				
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.94$	$a = 8.984$	$b = 2.464$

Rheogram and Data for Test Number TEST-D01A @25c @2200hr TANK 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R = 0.87$	$\dot{\sigma}_0 = 15.64$	$\text{Eta} = 0.02041$
Ostwald	: $\dot{\sigma} = a * D^n$	$R = 0.98$	$a = 8.819$	$n = 0.1604$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R = 0.98$	$a = 3.330$	$n = 0.2619$ $\dot{\sigma}_0 = 7.192$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R = 0.92$	$\dot{\sigma}_0 = 12.76$	$\text{Eta} = 0.004174$
Linear	: $Y = a + b * X$	$R = 0.87$	$a = 15.64$	$b = 0.02041$
Inv. linear	: $Y = 1 / (a + b * X)$	$R = 0.11$	$a = 0.07366$	$b = -91.24E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R = 0.47$	$a = 15.01$	$b = 0.001226$
Hyperbolic	: $Y = a + b / X$	$R = 0.29$	$a = 20.52$	$b = -15.62$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R = 0.97$	$a = 6.201$	$b = 2.750$

Optimum :

H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ $R = 0.98$ $a = 3.330$ $n = 0.2619$ $\dot{\sigma}_0 = 7.192$

Rheogram and Data for Test Number TEST-D0 @2200hr TANK 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.74$	$\dot{\sigma}_0 = 16.26$	$\text{Eta} = 0.01927$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.76$	$a = 7.417$	$n = 0.1968$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.90$	$a = 7.524$	$n = 0.1747$ $\dot{\sigma}_0 = 2.026$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.72$	$\dot{\sigma}_0 = 13.03$	$\text{Eta} = 0.004175$
Linear	: $Y = a + b * X$	$R^2 = 0.74$	$a = 16.26$	$b = 0.01927$
Inv. linear:	$Y = 1 / (a + b * X)$	$R^2 = 0.03$	$a = 0.1508$	$b = -350.7E-06$
Exponential:	$Y = a * \text{EXP}(b * X)$	$R^2 = 0.22$	$a = 14.54$	$b = 0.001378$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.38$	$a = 20.89$	$b = -14.08$
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R^2 = 0.97$	$a = 6.286$	$b = 2.806$
 Optimum :				
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R^2 = 0.97$	$a = 6.286$	$b = 2.806$

Rheogram and Data for Test Number TEST-D0A @2200hr TANK 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.75	$\dot{\sigma}_0=17.55$	Eta=0.0191
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.80	a=8.916	n=0.1730
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.91	a=7.286	n=0.1732 $\dot{\sigma}_0=4.001$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{(\text{Eta} * D)}$	R>=0.73	$\dot{\sigma}_0=14.41$	Eta=0.003737
Linear	: $Y = a + b * X$	R>=0.75	a=17.55	b=0.0191
Inv. linear:	$Y = 1 / (a + b * X)$	R>=0.06	a=0.08086	b=-125.6E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R>=0.28	a=16.26	b=0.001188
Hyperbolic:	$Y = a + b / X$	R>=0.33	a=22.04	b=-7.308
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.97	a=8.072	b=2.702
 Optimum :				
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.97	a=8.072	b=2.702

Rheogram and Data for Test Number TEST1AC @25C @1030 T-33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R = 0.76$	$\dot{\sigma}_0 = 17.77$	$\text{Eta} = 0.01836$
Ostwald	: $\dot{\sigma} = a * D^n$	$R = 0.81$	$a = 9.611$	$n = 0.1591$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R = 0.92$	$a = 7.178$	$n = 0.1675$ $\dot{\sigma}_0 = 4.840$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R = 0.74$	$\dot{\sigma}_0 = 14.83$	$\text{Eta} = 0.003337$
Linear	: $Y = a + b * X$	$R = 0.76$	$a = 17.77$	$b = 0.01836$
Inv. linear:	$Y = 1 / (a + b * X)$	$R = 0.07$	$a = 0.07372$	$b = -101.8E-06$
Exponential:	$Y = a * \text{EXP}(b * X)$	$R = 0.29$	$a = 16.63$	$b = 0.001112$
Hyperbolic	: $Y = a + b / X$	$R = 0.32$	$a = 22.06$	$b = -5.105$
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R = 0.96$	$a = 8.947$	$b = 2.541$
Optimum :				
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R = 0.96$	$a = 8.947$	$b = 2.541$

Rheogram and Data for Test Number TEST1AC1 @25C @1030 T-33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R \gg 0.83$	$\dot{\sigma}_0 = 16.32$	$\text{Eta} = 0.02128$
Ostwald	: $\dot{\sigma} = a * D^n$	$R \gg 0.91$	$a = 8.485$	$n = 0.1760$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R \gg 0.99$	$a = 4.841$	$n = 0.2215$ $\dot{\sigma}_0 = 5.818$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R \gg 0.85$	$\dot{\sigma}_0 = 13.15$	$\text{Eta} = 0.004556$
Linear	: $Y = a + b * X$	$R \gg 0.83$	$a = 16.32$	$b = 0.02128$
Inv. linear:	$Y = 1 / (a + b * X)$	$R \gg 0.08$	$a = 0.07719$	$b = -109.5E-06$
Exponential:	$Y = a * \text{EXP}(b * X)$	$R \gg 0.38$	$a = 15.41$	$b = 0.001281$
Hyperbolic	: $Y = a + b / X$	$R \gg 0.25$	$a = 21.28$	$b = -6.064$
Logarithmic:	$Y = a + b * \text{LN}(X)$	$R \gg 0.98$	$a = 6.535$	$b = 2.859$
Optimum :				
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R \gg 0.99$	$a = 4.841$	$n = 0.2215$ $\dot{\sigma}_0 = 5.818$

Rheogram and Data for Test Number TEST1AE @50C @1030 T:33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.75	$\dot{\sigma}_0 = 16.75$	Eta=0.01858
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.78	a=8.426	n=0.1756
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.92	a=7.421	n=0.1692 $\dot{\sigma}_0 = 3.136$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.73	$\dot{\sigma}_0 = 13.72$	Eta=0.003687
Linear	: $Y = a + b * X$	R>=0.75	a=16.75	b=0.01858
Inv. linear:	$Y = 1 / (a + b * X)$	R>=0.04	a=0.09195	b=-156.4E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R>=0.26	a=15.43	b=0.001224
Hyperbolic	: $Y = a + b / X$	R>=0.40	a=21.23	b=-14.19
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.97	a=7.293	b=2.674

Optimum :

Logarithmic: $Y = a + b * \text{LN}(X)$ R>=0.97 a=7.293 b=2.674

Rheogram and Data for Test Number TEST1AE1 @50C @1030 T:33

Bingham	: $\sigma = \sigma_0 + \eta \cdot D$	R=0.73	$\sigma_0 = 18.49$	$\eta = 0.0188$
Ostwald	: $\sigma = a \cdot D^n$	R=0.77	$a = 9.631$	$n = 0.1658$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot D^n$	R=0.88	$a = 7.990$	$n = 0.1623$ $\sigma_0 = 4.245$
Casson	: $\sqrt{\sigma} - \sqrt{\sigma_0} = \sqrt{\eta \cdot D}$	R=0.70	$\sigma_0 = 15.37$	$\eta = 0.003481$
Linear	: $Y = a + b \cdot X$	R=0.73	$a = 18.49$	$b = 0.0188$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	R=0.05	$a = 0.07749$	$b = -119.5E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	R=0.26	$a = 17.12$	$b = 0.001138$
Hyperbolic	: $Y = a + b / X$	R=0.35	$a = 22.92$	$b = -6.579$
Logarithmic	: $Y = a + b \cdot \ln(X)$	R=0.96	$a = 9.155$	$b = 2.662$
Optimum :				
Logarithmic	: $Y = a + b \cdot \ln(X)$	R=0.96	$a = 9.155$	$b = 2.662$

Rheogram and Data for Test Number TEST1AH @50C DAY 2 AGEING

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * \dot{\gamma}$	$R = 0.47$	$\dot{\sigma}_0 = 24.49$	$\text{Eta} = 0.01564$
Ostwald	: $\dot{\sigma} = a * \dot{\gamma}^n$	$R = 0.48$	$a = 10.81$	$n = 0.1834$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * \dot{\gamma}^n$	$R = 0.82$	$a = 13.24$	$n = 0.1143$ $\dot{\sigma}_0 = 4.214$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * \dot{\gamma}}$	$R = 0.42$	$\dot{\sigma}_0 = 20.87$	$\text{Eta} = 0.002525$
Linear	: $\dot{\sigma} = a + b * \dot{\gamma}$	$R = 0.47$	$a = 24.49$	$b = 0.01564$
Inv. linear	: $\dot{\sigma} = 1 / (a + b * \dot{\gamma})$	$R = 0.02$	$a = 0.5305$	$b = -0.001654$
Exponential	: $\dot{\sigma} = a * \exp(b * \dot{\gamma})$	$R = 0.08$	$a = 20.44$	$b = 0.001185$
Hyperbolic	: $\dot{\sigma} = a + b / \dot{\gamma}$	$R = 0.55$	$a = 28.27$	$b = -8.872$
Logarithmic	: $\dot{\sigma} = a + b * \ln(\dot{\gamma})$	$R = 0.89$	$a = 14.37$	$b = 2.676$

Optimum :
Logarithmic: $\dot{\sigma} = a + b * \ln(\dot{\gamma})$ $R = 0.89$ $a = 14.37$ $b = 2.676$

Rheogram and Data for Test Number TEST1AI @25C DAY 2 AGEING

Bingham	: $\sigma = \sigma_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.80$	$\sigma_0 = 19.17$	$\eta = 0.02656$
Ostwald	: $\sigma = a \cdot \dot{\gamma}^n$	$R^2 = 0.99$	$a = 11.07$	$n = 0.1591$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.99$	$a = 4.723$	$n = 0.2477$ $\sigma_0 = 8.063$
Casson	: $\sqrt{\sigma} - \sqrt{\sigma_0} = \sqrt{\eta \cdot \dot{\gamma}}$	$R^2 = 0.86$	$\sigma_0 = 15.18$	$\eta = 0.006092$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.80$	$a = 19.17$	$b = 0.02656$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.32$	$a = 0.05464$	$b = -58.47E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.60$	$a = 18.94$	$b = 0.001189$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.13$	$a = 25.21$	$b = -0.1476$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.93$	$a = 10.05$	$b = 2.983$

Optimum :

H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.99$	$a = 4.723$	$n = 0.2477$ $\sigma_0 = 8.063$

Rheogram and Data for Test Number TEST1AF @50C DAY 2 AGEING

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.57$	$\dot{\sigma}_0 = 23.09$	$\eta = 0.01975$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.52$	$a = 8.388$	$n = 0.2286$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.78$	$a = 12.66$	$n = 0.1373$ $\dot{\sigma}_0 = 1.847$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.50$	$\dot{\sigma}_0 = 18.90$	$\eta = 0.003889$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.57$	$a = 23.09$	$b = 0.01975$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.03$	$a = 0.2302$	$b = -0.000652$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.12$	$a = 19.47$	$b = 0.001325$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.42$	$a = 27.78$	$b = -7.775$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.94$	$a = 11.53$	$b = 3.141$

Optimum :

Logarithmic: $Y = a + b \cdot \ln(X)$ $R^2 = 0.94$ $a = 11.53$ $b = 3.141$

Rheogram and Data for Test Number TEST1AG @50C DAY 2 AGEING

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.74$	$\dot{\sigma}_0 = 21.98$	$\eta = 0.02306$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.78$	$a = 11.22$	$n = 0.1707$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.88$	$a = 9.327$	$n = 0.1669$ $\dot{\sigma}_0 = 5.068$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.71$	$\dot{\sigma}_0 = 18.13$	$\eta = 0.004424$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.74$	$a = 21.98$	$b = 0.02306$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.05$	$a = 0.06601$	$b = -104.2E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.26$	$a = 20.30$	$b = 0.00117$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.32$	$a = 27.38$	$b = -6.315$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.96$	$a = 10.65$	$b = 3.242$
Optimum :				
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.96$	$a = 10.65$	$b = 3.242$

Rheogram and Data for Test Number TEST-D3B @25C @10:00

Bingham	: $\sigma = \sigma_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.67$	$\sigma_0 = 20.64$	$\eta = 0.01894$
Ostwald	: $\sigma = a \cdot \dot{\gamma}^n$	$R^2 = 0.59$	$a = 9.206$	$n = 0.1918$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.95$	$a = 9.913$	$n = 0.1427$ $\sigma_0 = 4.232$
Casson	: $\sqrt{\sigma} - \sqrt{\sigma_0} = \sqrt{\eta \cdot \dot{\gamma}}$	$R^2 = 0.59$	$\sigma_0 = 17.13$	$\eta = 0.003507$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.67$	$a = 20.64$	$b = 0.01894$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.03$	$a = 0.06994$	$b = -106.1E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.22$	$a = 19.74$	$b = 952.7E-06$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.40$	$a = 25.11$	$b = -6.539$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.96$	$a = 10.61$	$b = 2.805$

Optimum :
Logarithmic: $Y = a + b \cdot \ln(X)$ $R^2 = 0.96$ $a = 10.61$ $b = 2.805$

Rheogram and Data for Test Number TEST-D3C @25C @10:00

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.87	$\dot{\sigma}_0 = 17.95$	Eta = 0.02491
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.98	a = 10.21	n = 0.1615
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.99	a = 2.555	n = 0.3166 $\dot{\sigma}_0 = 10.16$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.94	$\dot{\sigma}_0 = 14.48$	Eta = 0.005349
Linear	: $\dot{\sigma} = a + b * X$	R>=0.87	a = 17.95	b = 0.02491
Inv. linear:	$\dot{\sigma} = 1 / (a + b * X)$	R>=0.38	a = 0.05831	b = -62.27E-06
Exponential:	$\dot{\sigma} = a * \text{EXP}(b * X)$	R>=0.66	a = 17.58	b = 0.001217
Hyperbolic	: $\dot{\sigma} = a + b / X$	R>=0.21	a = 23.81	b = -11.80
Logarithmic:	$\dot{\sigma} = a + b * \text{LN}(X)$	R>=0.95	a = 6.920	b = 3.263
Optimum :				
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.99	a = 2.555	n = 0.3166 $\dot{\sigma}_0 = 10.16$

Rheogram and Data for Test Number TEST-D3 @50C @10:00

Bingham	: $\sigma = \sigma_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.75$	$\sigma_0 = 19.18$	$\eta = 0.02115$
Ostwald	: $\sigma = a \cdot \dot{\gamma}^n$	$R^2 = 0.70$	$a = 8.842$	$n = 0.1920$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.97$	$a = 8.354$	$n = 0.1665$ $\sigma_0 = 4.203$
Casson	: $\sqrt{\sigma} - \sqrt{\sigma_0} = \sqrt{\eta \cdot \dot{\gamma}}$	$R^2 = 0.70$	$\sigma_0 = 15.61$	$\eta = 0.004294$
Linear	: $y = a + b \cdot x$	$R^2 = 0.75$	$a = 19.18$	$b = 0.02115$
Inv. linear	: $y = 1 / (a + b \cdot x)$	$R^2 = 0.05$	$a = 0.06516$	$b = -85.93E-06$
Exponential	: $y = a \cdot \exp(b \cdot x)$	$R^2 = 0.32$	$a = 18.56$	$b = 0.001053$
Hyperbolic	: $y = a + b/x$	$R^2 = 0.34$	$a = 24.15$	$b = -7.482$
Logarithmic	: $y = a + b \cdot \ln(x)$	$R^2 = 0.98$	$a = 8.630$	$b = 3.003$

Optimum :

Logarithmic: $y = a + b \cdot \ln(x)$ $R^2 = 0.98$ $a = 8.630$ $b = 3.003$

Rheogram and Data for Test Number TEST-D3a @50C @10:00

Bingham	: $\sigma = \sigma_0 + \eta \cdot \dot{\gamma}$	R=0.75	$\sigma_0=19.86$	$\eta=0.02294$
Ostwald	: $\sigma = a \cdot \dot{\gamma}^n$	R=0.80	$a=9.962$	$n=0.1776$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	R=0.80	$a=8.086$	$n=0.1861$ $\sigma_0=3.817$
Casson	: $\sqrt{\sigma} = \sqrt{\sigma_0} + \sqrt{\eta \cdot \dot{\gamma}}$	R=0.74	$\sigma_0=16.13$	$\eta=0.004714$
Linear	: $Y = a + b \cdot X$	R=0.75	$a=19.86$	$b=0.02294$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	R=0.09	$a=0.06483$	$b=-90.76E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	R=0.33	$a=18.56$	$b=0.001209$
Hyperbolic	: $Y = a + b / X$	R=0.40	$a=25.38$	$b=-16.96$
Logarithmic	: $Y = a + b \cdot \ln(X)$	R=0.96	$a=8.444$	$b=3.251$

Optimum :
Logarithmic: $Y = a + b \cdot \ln(X)$ R=0.96 a=8.444 b=3.251

Rheogram and Data for Test Number glyc4a

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.61$	$\dot{\sigma}_0 = 21.77$	$\eta = 0.01826$
Ostwald	: $\dot{\sigma} = a \cdot \dot{\gamma}^n$	$R^2 = 0.61$	$a = 10.05$	$n = 0.1824$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.92$	$a = 10.34$	$n = 0.1412$ $\dot{\sigma}_0 = 4.484$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot \dot{\gamma}}$	$R^2 = 0.56$	$\dot{\sigma}_0 = 18.17$	$\eta = 0.003287$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.61$	$a = 21.77$	$b = 0.01826$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.03$	$a = 0.06214$	$b = -84.46E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.22$	$a = 20.85$	$b = 887.4E-06$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.43$	$a = 26.10$	$b = -6.783$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.96$	$a = 11.48$	$b = 2.826$

Optimum :
Logarithmic: $Y = a + b \cdot \ln(X)$ $R^2 = 0.96$ $a = 11.48$ $b = 2.826$

Rheogram and Data for Test Number gly4b

Bingham	: $\sigma = \sigma_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.69$	$\sigma_0 = 21.02$	$\eta = 0.02261$
Ostwald	: $\sigma = a \cdot \dot{\gamma}^n$	$R^2 = 0.90$	$a = 11.01$	$n = 0.1675$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.80$	$a = 8.091$	$n = 0.1884$ $\sigma_0 = 4.676$
Casson	: $\sqrt{\sigma} - \sqrt{\sigma_0} = \sqrt{\eta \cdot \dot{\gamma}}$	$R^2 = 0.70$	$\sigma_0 = 17.11$	$\eta = 0.004599$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.69$	$a = 21.02$	$b = 0.02261$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.11$	$a = 0.0539$	$b = -59.25E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.38$	$a = 20.41$	$b = 0.001024$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.22$	$a = 26.19$	$b = -0.1728$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.95$	$a = 11.75$	$b = 2.835$

Optimum :

Logarithmic: $Y = a + b \cdot \ln(X)$ $R^2 = 0.95$ $a = 11.75$ $b = 2.835$

4.4.2 Rheology of Small-Scale Feed Samples Aged in the Laboratory at 50°C

For the HTM feed prepared in the small-scale system but aged in the laboratory, yield stress and plastic viscosity changed with sample age. From day 0 to day 24, yield stress varied between 25.8 Pa and 12.1 Pa (50°C) and the plastic viscosity varied between 19.3 mPa•s to 32.7 mPa•s (50°C). The solids loading of the sample aged at 50°C was 44 ± 1 weight percent throughout the testing period except for days 5 and 8. The pH decreased from 8.2 to 7.4 (50°C) for the aging system at 50°C. Summary Table B4.4.2 contains all data from the requisite rheograms and each individual rheogram follows.

Table B4.4.2 Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Laboratory Aged (at 50°C) Small-Scale Melter Feed Preparation Samples.

		g TO/L	pH (@ ≈22°C)	wt % solids	Density (g/ml)	Yield Stress (Pa) ^a	Plastic Viscosity (mPa·s) ^b	Correlation Coeff. (R ²) ^c
Alternate HTM Flowsheet 2^d								
Aged Small-Scale Samples^e:								
After silica addition, Day 0:								
File ID/Date:	TEST-D0/8-14-94	422	NM	43.2	1.3	16.3	19.3	0.74
	TEST-DOA/8-14-94	----	----	----	----	17.5	19.1	0.75
Day 1 after silica addition:								
File ID/Date:	TESTD15A/8-15-94	----	8.2	NM	----	25.8	27.9	0.67
	TESTD15B/8-15-94	----	----	----	----	26.2	32.8	0.69
Day 3 after silica addition:								
File ID/Date:	DAY3A-52/8-17-94	----	8.1	45.3	----	25.8	32.7	0.78
	DAY3B-52/8-17-94	----	----	----	----	24.8	36.9	0.79
Day 5 after silica addition:								
File ID/Date:	day552ca/8-19-94	----	7.8	37.8	----	15.4	22.8	0.82
	day552cb/8-19-94	----	----	----	----	16.8	20.0	0.81
Day 8 after silica addition:								
File ID/Date:	TESTD75/8-22-94	----	7.8	46.9	----	20.2	27.0	0.80
	TESTD75A/8-22-94	----	----	----	----	21.0	24.3	0.80
Day 11 after silica addition:								
File ID/Date:	glyd10c/8-25-94	----	7.8	44.1	----	19.3	30.1	0.87
	glyd10d/8-25-94	----	----	----	----	20.4	27.7	0.85
Day 15 after silica addition:								
File ID/Date:	glyd15c/8-29-94	----	7.6	43.2	----	17.1	20.8	0.78
	glyd15d/8-29-94	----	----	----	----	17.9	20.9	0.86
Day 24 after silica addition:								
File ID/Date:	T-52-31/9-7-94	----	7.4	42.9	----	12.1	21.0	0.92
	T-52-31A/9-7-94	----	----	----	----	13.6	17.6	0.84

- (a) Laboratory rheological screening data value for the yield stress is 15 Pa. The yield stress was calculated by fitting the shear rate sweep data to the Bingham model.
- (b) Laboratory rheological screening data value for the plastic viscosity is 25 mPa·s. The plastic viscosity was calculated by fitting the shear rate sweep data to the Bingham model.
- (c) Least squares correlation coefficient of shear rheology data fit to the Bingham equation.
- (d) Alternate HTM Flowsheet 2 is the glycolic acid process flowsheet.
- (e) Rheology measurements were taken at 25°C for the aged small-scale feed samples.

Rheogram and Data for Test Number TEST-D0 @2200hr TANK 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.74$	$\dot{\sigma}_0 = 16.26$	$\eta = 0.01927$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.76$	$a = 7.417$	$n = 0.1968$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.90$	$a = 7.524$	$n = 0.1747$ $\dot{\sigma}_0 = 2.026$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.72$	$\dot{\sigma}_0 = 13.03$	$\eta = 0.004175$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.74$	$a = 16.26$	$b = 0.01927$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.03$	$a = 0.1508$	$b = -350.7E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.22$	$a = 14.54$	$b = 0.001378$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.38$	$a = 20.89$	$b = -14.08$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.97$	$a = 6.286$	$b = 2.806$
 Optimum :				
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.97$	$a = 6.286$	$b = 2.806$

Rheogram and Data for Test Number TEST-D0A @2200hr TANK 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.75$	$\dot{\sigma}_0 = 17.55$	$\eta = 0.0191$
Ostwald	: $\dot{\sigma} = a \cdot \dot{\gamma}^n$	$R^2 = 0.80$	$a = 8.916$	$n = 0.1730$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.91$	$a = 7.286$	$n = 0.1732$ $\dot{\sigma}_0 = 4.001$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot \dot{\gamma}}$	$R^2 = 0.73$	$\dot{\sigma}_0 = 14.41$	$\eta = 0.003737$
Linear	: $\dot{\sigma} = a + b \cdot \dot{\gamma}$	$R^2 = 0.75$	$a = 17.55$	$b = 0.0191$
Inv. linear	: $\dot{\sigma} = 1 / (a + b \cdot \dot{\gamma})$	$R^2 = 0.06$	$a = 0.08086$	$b = -125.6E-06$
Exponential	: $\dot{\sigma} = a \cdot \exp(b \cdot \dot{\gamma})$	$R^2 = 0.28$	$a = 16.26$	$b = 0.001188$
Hyperbolic	: $\dot{\sigma} = a + b / \dot{\gamma}$	$R^2 = 0.33$	$a = 22.04$	$b = -7.308$
Logarithmic	: $\dot{\sigma} = a + b \cdot \ln(\dot{\gamma})$	$R^2 = 0.97$	$a = 8.072$	$b = 2.702$
 Optimum :				
Logarithmic	: $\dot{\sigma} = a + b \cdot \ln(\dot{\gamma})$	$R^2 = 0.97$	$a = 8.072$	$b = 2.702$

Rheogram and Data for Test Number TESTD15A @52C AGEING

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.67	$\dot{\sigma}_0 = 25.78$	Eta = 0.02789
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.63	a = 9.954	n = 0.2251
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.94	a = 11.45	n = 0.1727 $\dot{\sigma}_0 = 4.096$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.64	$\dot{\sigma}_0 = 20.50$	Eta = 0.006191
Linear	: $\dot{\sigma} = a + b * X$	R>=0.67	a = 25.78	b = 0.02789
Inv. linear:	$\dot{\sigma} = 1 / (a + b * X)$	R>=0.03	a = 0.06304	b = -112.1E-06
Exponential:	$\dot{\sigma} = a * \text{EXP}(b * X)$	R>=0.24	a = 24.39	b = 0.001115
Hyperbolic	: $\dot{\sigma} = a + b / X$	R>=0.36	a = 32.37	b = -11.46
Logarithmic:	$\dot{\sigma} = a + b * \text{LN}(X)$	R>=0.99	a = 10.58	b = 4.212
 Optimum :				
Logarithmic:	$\dot{\sigma} = a + b * \text{LN}(X)$	R>=0.99	a = 10.58	b = 4.212

Rheogram and Data for Test Number TESTD15B @52C AGEING

Bingham	: $\sigma = \sigma_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.69$	$\sigma_0 = 26.15$	$\eta = 0.03279$
Ostwald	: $\sigma = a \cdot \dot{\gamma}^n$	$R^2 = 0.73$	$a = 10.03$	$n = 0.2322$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.96$	$a = 9.916$	$n = 0.2065$ $\sigma_0 = 4.615$
Casson	: $\sqrt{\sigma} - \sqrt{\sigma_0} = \sqrt{\eta \cdot \dot{\gamma}}$	$R^2 = 0.70$	$\sigma_0 = 20.16$	$\eta = 0.008141$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.69$	$a = 26.15$	$b = 0.03279$
Inv. linear:	$Y = 1 / (a + b \cdot X)$	$R^2 = 0.04$	$a = 0.05315$	$b = -84.03E-06$
Exponential:	$Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.32$	$a = 24.88$	$b = 0.001223$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.34$	$a = 33.96$	$b = -18.54$
Logarithmic:	$Y = a + b \cdot \ln(X)$	$R^2 = 1.00$	$a = 8.279$	$b = 4.952$
 Optimum :				
Logarithmic:	$Y = a + b \cdot \ln(X)$	$R^2 = 1.00$	$a = 8.279$	$b = 4.952$

Rheogram and Data for Test Number DAY3A-52

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.78	$\dot{\sigma}_0 = 25.79$	Eta = 0.03268
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.74	a = 11.37	n = 0.2062
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.99	a = 10.05	n = 0.1897 $\dot{\sigma}_0 = 6.308$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.75	$\dot{\sigma}_0 = 20.50$	Eta = 0.007303
Linear	: $Y = a + b * X$	R>=0.78	a = 25.79	b = 0.03268
Inv. linear:	$Y = 1 / (a + b * X)$	R>=0.05	a = 0.04868	b = -68.03E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R>=0.37	a = 24.99	b = 0.001169
Hyperbolic	: $Y = a + b / X$	R>=0.29	a = 33.44	b = -10.75
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.98	a = 10.03	b = 4.534

Optimum :
H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ R>=0.99 a=10.05 n=0.1897 $\dot{\sigma}_0 = 6.308$

Rheogram and Data for Test Number DAY3B-52

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R = 0.79$	$\dot{\sigma}_0 = 24.75$	$\eta = 0.03694$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R = 0.70$	$a = 9.061$	$n = 0.2488$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R = 0.99$	$a = 8.582$	$n = 0.2206$ $\dot{\sigma}_0 = 6.085$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R = 0.77$	$\dot{\sigma}_0 = 18.82$	$\eta = 0.009599$
Linear	: $Y = a + b \cdot X$	$R = 0.79$	$a = 24.75$	$b = 0.03694$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R = 0.03$	$a = 0.06377$	$b = -118.8E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R = 0.33$	$a = 23.64$	$b = 0.001374$
Hyperbolic	: $Y = a + b / X$	$R = 0.25$	$a = 33.38$	$b = -11.77$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R = 0.98$	$a = 7.044$	$b = 5.103$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R = 0.99$	$a = 8.582$	$n = 0.2206$ $\dot{\sigma}_0 = 6.085$

Rheogram and Data for Test Number day552ca

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	R $\gg$ =0.82	$\dot{\sigma}_0$ =15.43	η =0.0228
Ostwald	: $\dot{\sigma} = a \cdot D^n$	R $\gg$ =0.80	a=6.593	n=0.2190
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	R $\gg$ =0.99	a=5.277	n=0.2181 $\dot{\sigma}_0$ =4.193
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	R $\gg$ =0.81	$\dot{\sigma}_0$ =11.95	η =0.005601
Linear	: $Y = a + b \cdot X$	R $\gg$ =0.82	a=15.43	b=0.0228
Inv. linear:	$Y = 1 / (a + b \cdot X)$	R $\gg$ =0.02	a=0.8384	b=-0.002644
Exponential:	$Y = a \cdot \exp(b \cdot X)$	R $\gg$ =0.18	a=13.26	b=0.001715
Hyperbolic:	$Y = a + b / X$	R $\gg$ =0.24	a=20.73	b=-5.621
Logarithmic:	$Y = a + b \cdot \ln(X)$	R $\gg$ =0.97	a=4.983	b=3.056

Optimum :

H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$ R $\gg$ =0.99 a=5.277 n=0.2181 $\dot{\sigma}_0$ =4.193

Rheogram and Data for Test Number day552cb

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R = 0.81$	$\dot{\sigma}_0 = 16.79$	$\eta = 0.02003$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R = 0.77$	$a = 8.738$	$n = 0.1719$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R = 0.87$	$a = 6.825$	$n = 0.1742$ $\dot{\sigma}_0 = 4.483$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R = 0.76$	$\dot{\sigma}_0 = 13.79$	$\eta = 0.003945$
Linear	: $\dot{\sigma} = a + b \cdot D$	$R = 0.81$	$a = 16.79$	$b = 0.02003$
Inv. linear	: $\dot{\sigma} = 1 / (a + b \cdot D)$	$R = 0.05$	$a = 0.0872$	$b = -143.5E-06$
Exponential	: $\dot{\sigma} = a \cdot \exp(b \cdot D)$	$R = 0.29$	$a = 15.59$	$b = 0.001257$
Hyperbolic	: $\dot{\sigma} = a + b / D$	$R = 0.29$	$a = 21.47$	$b = -5.938$
Logarithmic	: $\dot{\sigma} = a + b \cdot \ln(D)$	$R = 0.92$	$a = 7.851$	$b = 2.638$
 Optimum :				
Logarithmic	: $\dot{\sigma} = a + b \cdot \ln(D)$	$R = 0.92$	$a = 7.851$	$b = 2.638$

Rheogram and Data for Test Number TESTD75 @52C AGEING

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.80$	$\dot{\sigma}_0 = 20.17$	$\eta = 0.02697$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.64$	$a = 7.786$	$n = 0.2336$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.95$	$a = 8.703$	$n = 0.1835$ $\dot{\sigma}_0 = 3.746$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.72$	$\dot{\sigma}_0 = 15.85$	$\eta = 0.006286$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.80$	$a = 20.17$	$b = 0.02697$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.02$	$a = 8.067$	$b = -0.02677$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.11$	$a = 16.16$	$b = 0.001851$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.30$	$a = 26.50$	$b = -9.556$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.96$	$a = 7.479$	$b = 3.680$

Optimum :

Logarithmic: $Y = a + b \cdot \ln(X)$ $R^2 = 0.96$ $a = 7.479$ $b = 3.680$

Rheogram and Data for Test Number TESTD75A @52C AGEING

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.80$	$\dot{\sigma}_0 = 21.01$	$\text{Eta} = 0.02433$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.77$	$a = 11.40$	$n = 0.1627$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.80$	$a = 9.525$	$n = 0.1606$ $\dot{\sigma}_0 = 4.619$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.74$	$\dot{\sigma}_0 = 17.45$	$\text{Eta} = 0.004566$
Linear	: $Y = a + b * X$	$R^2 = 0.80$	$a = 21.01$	$b = 0.02433$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.07$	$a = 0.06267$	$b = -90.36E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.32$	$a = 19.71$	$b = 0.001197$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.28$	$a = 26.69$	$b = -6.586$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.88$	$a = 10.46$	$b = 3.145$

Optimum :

Logarithmic: $Y = a + b * \text{LN}(X)$ $R^2 = 0.88$ $a = 10.46$ $b = 3.145$

Rheogram and Data for Test Number glyd10c

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot \dot{\gamma}$	$R^2 = 0.87$	$\dot{\sigma}_0 = 19.28$	$\eta = 0.03008$
Ostwald	: $\dot{\sigma} = a \cdot \dot{\gamma}^n$	$R^2 = 0.74$	$a = 8.046$	$n = 0.2260$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.98$	$a = 6.965$	$n = 0.2111$ $\dot{\sigma}_0 = 5.213$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot \dot{\gamma}}$	$R^2 = 0.80$	$\dot{\sigma}_0 = 14.98$	$\eta = 0.007338$
Linear	: $\dot{\sigma} = a + b \cdot \dot{\gamma}$	$R^2 = 0.87$	$a = 19.28$	$b = 0.03008$
Inv. linear	: $\dot{\sigma} = 1 / (a + b \cdot \dot{\gamma})$	$R^2 = 0.05$	$a = 0.06784$	$b = -107.2E-06$
Exponential	: $\dot{\sigma} = a \cdot \exp(b \cdot \dot{\gamma})$	$R^2 = 0.41$	$a = 18.80$	$b = 0.001349$
Hyperbolic	: $\dot{\sigma} = a + b / \dot{\gamma}$	$R^2 = 0.25$	$a = 26.30$	$b = -9.038$
Logarithmic	: $\dot{\sigma} = a + b \cdot \ln(\dot{\gamma})$	$R^2 = 0.94$	$a = 6.115$	$b = 3.910$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot \dot{\gamma}^n$	$R^2 = 0.98$	$a = 6.965$	$n = 0.2111$ $\dot{\sigma}_0 = 5.213$

Rheogram and Data for Test Number glyd10d

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.85	$\dot{\sigma}_0 = 20.37$	Eta = 0.02767
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.78	a = 11.31	n = 0.1648
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.79	a = 9.061	n = 0.1699 $\dot{\sigma}_0 = 4.727$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.79	$\dot{\sigma}_0 = 16.81$	Eta = 0.005419
Linear	: $Y = a + b * X$	R>=0.85	a = 20.37	b = 0.02767
Inv. linear	: $Y = 1 / (a + b * X)$	R>=0.09	a = 0.06063	b = -84.62E-06
Exponential	: $Y = a * \text{EXP}(b * X)$	R>=0.39	a = 19.39	b = 0.001286
Hyperbolic	: $Y = a + b / X$	R>=0.23	a = 26.82	b = -8.342
Logarithmic	: $Y = a + b * \text{LN}(X)$	R>=0.82	a = 9.412	b = 3.370

Optimum :

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.85	$\dot{\sigma}_0 = 20.37$	Eta = 0.02767

Rheogram and Data for Test Number glyd15c

Bingham	: $\sigma = \sigma_0 + \eta \cdot D$	$R = 0.78$	$\sigma_0 = 17.08$	$\eta = 0.02078$
Ostwald	: $\sigma = a \cdot D^n$	$R = 0.60$	$a = 7.664$	$n = 0.2007$
H.-Bulkley	: $\sigma = \sigma_0 + a \cdot D^n$	$R = 0.78$	$a = 9.754$	$n = 0.1403$ $\sigma_0 = 1.619$
Casson	: $\sqrt{\sigma} - \sqrt{\sigma_0} = \sqrt{\eta \cdot D}$	$R = 0.65$	$\sigma_0 = 13.99$	$\eta = 0.00412$
Linear	: $Y = a + b \cdot X$	$R = 0.78$	$a = 17.08$	$b = 0.02078$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R = 0.04$	$a = 0.08185$	$b = -128.6E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R = 0.30$	$a = 16.49$	$b = 0.001141$
Hyperbolic	: $Y = a + b / X$	$R = 0.31$	$a = 21.95$	$b = -6.883$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R = 0.84$	$a = 8.187$	$b = 2.661$

Optimum :
Logarithmic: $Y = a + b \cdot \ln(X)$ $R = 0.84$ $a = 8.187$ $b = 2.661$

Rheogram and Data for Test Number glyd15d

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \dot{\gamma}$	$R^2 = 0.86$	$\dot{\sigma}_0 = 17.92$	$\eta = 0.0209$
Ostwald	: $\dot{\sigma} = a \dot{\gamma}^n$	$R^2 = 0.79$	$a = 11.17$	$n = 0.1362$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \dot{\gamma}^n$	$R^2 = 0.56$	$a = 4.476$	$n = 0.2510$ $\dot{\sigma}_0 = 6.014$
Casson	: $\sqrt{\dot{\sigma}} = \sqrt{\dot{\sigma}_0} + \sqrt{\eta \dot{\gamma}}$	$R^2 = 0.83$	$\dot{\sigma}_0 = 15.20$	$\eta = 0.003588$
Linear	: $Y = a + bX$	$R^2 = 0.86$	$a = 17.92$	$b = 0.0209$
Inv. linear	: $Y = 1/(a + bX)$	$R^2 = 0.10$	$a = 0.06414$	$b = -74.96E-06$
Exponential	: $Y = a \exp(bX)$	$R^2 = 0.42$	$a = 17.23$	$b = 0.00112$
Hyperbolic	: $Y = a + b/X$	$R^2 = 0.32$	$a = 22.97$	$b = -19.67$
Logarithmic	: $Y = a + b \ln(X)$	$R^2 = 0.82$	$a = 9.282$	$b = 2.614$

Optimum :

Bingham : $\dot{\sigma} = \dot{\sigma}_0 + \eta \dot{\gamma}$ $R^2 = 0.86$ $\dot{\sigma}_0 = 17.92$ $\eta = 0.0209$

Rheogram and Data for Test Number T-52-31

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.90$	$\dot{\sigma}_0 = 12.38$	$\eta = 0.02103$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.76$	$a = 5.886$	$n = 0.2052$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.86$	$a = 4.887$	$n = 0.2003$ $\dot{\sigma}_0 = 3.269$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.82$	$\dot{\sigma}_0 = 9.779$	$\eta = 0.004921$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.90$	$a = 12.38$	$b = 0.02103$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.02$	$a = 0.3284$	$b = -913.7E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.24$	$a = 11.16$	$b = 0.001696$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.20$	$a = 17.26$	$b = -4.836$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.84$	$a = 4.330$	$b = 2.508$

Optimum :

Bingham : $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$ $R^2 = 0.90$ $\dot{\sigma}_0 = 12.38$ $\eta = 0.02103$

Rheogram and Data for Test Number T-52-31A

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.84	$\dot{\sigma}_0 = 13.56$	Eta = 0.01762
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.89	a = 8.265	n = 0.1456
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.82	a = 4.925	n = 0.1901 $\dot{\sigma}_0 = 4.329$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.80	$\dot{\sigma}_0 = 11.22$	Eta = 0.003412
Linear	: $Y = a + b * X$	R>=0.84	a = 13.56	b = 0.01762
Inv. linear	: $Y = 1 / (a + b * X)$	R>=0.23	a = 0.07676	b = -77.46E-06
Exponential	: $Y = a * \text{EXP}(b * X)$	R>=0.58	a = 13.49	b = 0.001101
Hyperbolic	: $Y = a + b / X$	R>=0.17	a = 17.57	b = -0.1068
Logarithmic	: $Y = a + b * \text{LN}(X)$	R>=0.83	a = 8.022	b = 1.875

Optimum :

Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.89	a = 8.265	n = 0.1456

4.4.3 Rheology of Small-Scale Feed Samples Aged in the Laboratory at 25°C

For the HTM feed prepared in the small-scale system but aged in the laboratory, yield stress and plastic viscosity increased with sample age. From day 0 to day 24, yield stress increased from 17.7 Pa and 26.1 Pa (25°C) and the plastic viscosity varied between 17.0 mPa•s to 41.2 mPa•s (25°C). The solids loading of the sample aged at 25°C was 44 ± 1 weight percent throughout the testing period. The pH remained steady at 8.3 ± 0.3 (25°C) for the aging system at 25°C. Summary Table B4.4.3 contains all data from the requisite rheograms and each individual rheogram follows.

Table B4.4.3 Physical Property and Rheology Data for Alternate HTM Flowsheet 2, Laboratory Aged (at 25°C) Small-Scale Melter Feed Preparation Samples.

		<u>g TO/L</u>	<u>pH (at 22°C)</u>	<u>wt % solids</u>	<u>Density (g/ml)</u>	<u>Yield Stress (Pa)^a</u>	<u>Plastic Viscosity (mPa·s)^b</u>	<u>Correlation Coeff. (R²)^c</u>
<u>Alternate HTM Flowsheet 2^d</u>								
<u>Aged Small-Scale Samples^e:</u>								
After silica addition, Day 0:								
File ID/Date:	TEST-D01/8-14-94	422	8.4	43.2	1.3	17.7	17.0	0.72
	TEST-D01A/8-14-94	----	----	----	----	15.6	20.4	0.87
Day 1 after silica addition:								
File ID/Date:	TEST-D1/8-15-94	----	8.6	43.4	----	21.7	25.3	0.72
	TEST-D1A/8-15-94	----	----	----	----	21.3	23.0	0.87
Day 3 after silica addition:								
File ID/Date:	TESTD3/8-17-94	----	8.5	NM	----	26.1	30.1	0.74
	TESTD3A/8-17-94	----	----	----	----	25.6	29.5	0.86
Day 5 after silica addition:								
File ID/Date:	day525ca/8-19-94	----	8.4	43.3	----	22.7	41.2	0.84
	day525cb/8-19-94	----	----	----	----	25.0	33.1	0.90
Day 8 after silica addition:								
File ID/Date:	TEST-D7/8-22-94	----	8.3	42.9	----	25.4	38.8	0.79
	TEST-D7A/8-22-94	----	----	----	----	25.6	34.6	0.91
Day 11 after silica addition:								
File ID/Date:	glyd10a/8-25-94	----	8.3	44.7	----	22.8	36.0	0.87
	glyd10b/8-25-94	----	----	----	----	24.2	31.8	0.95
Day 15 after silica addition:								
File ID/Date:	glyd15a/8-29-94	----	8.2	44.4	----	25.7	35.5	0.89
	glyd15b/8-29-94	----	----	----	----	24.8	32.8	0.97
Day 24 after silica addition:								
File ID/Date:	T-25-31/9-7-94	----	8.2	44.5	----	25.7	26.4	0.80
	T-25-31A/9-7-94	----	----	----	----	22.3	30.0	0.98

- (a) Laboratory rheological screening data value for the yield stress is 15 Pa. The yield stress was calculated by fitting the shear rate sweep data to the Bingham model.
- (b) Laboratory rheological screening data value for the plastic viscosity is 25 mPa·s. The plastic viscosity was calculated by fitting the shear rate sweep data to the Bingham model.
- (c) Least squares correlation coefficient of shear rheology data fit to the Bingham equation.
- (d) Alternate HTM Flowsheet 2 is the glycolic acid process flowsheet.
- (e) Rheology measurements were taken at 25°C for the aged small-scale feed samples.

Rheogram and Data for Test Number TEST-D01 @25c @2200hr TANK 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.72	$\dot{\sigma}_0 = 17.73$	Eta = 0.01699
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.71	a = 9.422	n = 0.1597
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.90	a = 8.451	n = 0.1467 $\dot{\sigma}_0 = 3.544$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.67	$\dot{\sigma}_0 = 14.89$	Eta = 0.002988
Linear	: $Y = a + b * X$	R>=0.72	a = 17.73	b = 0.01699
Inv. linear:	$Y = 1 / (a + b * X)$	R>=0.03	a = 0.09612	b = -173.8E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R>=0.21	a = 16.22	b = 0.001132
Hyperbolic	: $Y = a + b / X$	R>=0.45	a = 21.84	b = -13.81
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.94	a = 8.984	b = 2.464
 Optimum :				
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.94	a = 8.984	b = 2.464

Rheogram and Data for Test Number TEST-D01A @25c @2200hr TANK 33

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.87$	$\dot{\sigma}_0 = 15.64$	$\text{Eta} = 0.02041$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.98$	$a = 8.819$	$n = 0.1604$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.98$	$a = 3.330$	$n = 0.2619$ $\dot{\sigma}_0 = 7.192$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{(\text{Eta} * D)}$	$R^2 = 0.92$	$\dot{\sigma}_0 = 12.76$	$\text{Eta} = 0.004174$
Linear	: $Y = a + b * X$	$R^2 = 0.87$	$a = 15.64$	$b = 0.02041$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.11$	$a = 0.07366$	$b = -91.24E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.47$	$a = 15.01$	$b = 0.001226$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.29$	$a = 20.52$	$b = -15.62$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.97$	$a = 6.201$	$b = 2.750$
 Optimum :				
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.98$	$a = 3.330$	$n = 0.2619$ $\dot{\sigma}_0 = 7.192$

Rheogram and Data for Test Number TEST-D1 W/NaNO3/F110 @1143hr

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.72$	$\dot{\sigma}_0 = 21.72$	$\eta = 0.02531$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.66$	$a = 8.499$	$n = 0.2255$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.94$	$a = 9.776$	$n = 0.1743$ $\dot{\sigma}_0 = 3.349$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.67$	$\dot{\sigma}_0 = 17.22$	$\eta = 0.005684$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.72$	$a = 21.72$	$b = 0.02531$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.03$	$a = 0.07167$	$b = -124.6E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.27$	$a = 20.65$	$b = 0.001159$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.31$	$a = 27.65$	$b = -7.212$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.99$	$a = 8.819$	$b = 3.649$
Optimum :				
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.99$	$a = 8.819$	$b = 3.649$

Rheogram and Data for Test Number TEST-D1A W/NaNO3/F110 @1143hr

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R=0.87	$\dot{\sigma}_0 = 21.31$	Eta = 0.02295
Ostwald	: $\dot{\sigma} = a * D^n$	R=0.98	a = 13.33	n = 0.1328
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=1.00	a = 3.401	n = 0.2702 $\dot{\sigma}_0 = 12.56$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R=0.93	$\dot{\sigma}_0 = 18.01$	Eta = 0.003973
Linear	: $Y = a + b * X$	R=0.87	a = 21.31	b = 0.02295
Inv. linear:	$Y = 1 / (a + b * X)$	R=0.14	a = 0.05192	b = -54.24E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R=0.47	a = 20.58	b = 0.001041
Hyperbolic	: $Y = a + b / X$	R=0.29	a = 26.81	b = -17.82
Logarithmic:	$Y = a + b * \text{LN}(X)$	R=0.97	a = 10.72	b = 3.089
 Optimum :				
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=1.00	a = 3.401	n = 0.2702 $\dot{\sigma}_0 = 12.56$

Rheogram and Data for Test Number TESTD3

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R = 0.74$	$\dot{\sigma}_0 = 26.09$	$\eta = 0.03007$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R = 0.76$	$a = 11.99$	$n = 0.1944$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R = 0.99$	$a = 9.640$	$n = 0.1887$ $\dot{\sigma}_0 = 7.265$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R = 0.73$	$\dot{\sigma}_0 = 20.96$	$\eta = 0.006445$
Linear	: $Y = a + b \cdot X$	$R = 0.74$	$a = 26.09$	$b = 0.03007$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R = 0.02$	$a = 0.1556$	$b = -423.2E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R = 0.18$	$a = 22.96$	$b = 0.001409$
Hyperbolic	: $Y = a + b/X$	$R = 0.30$	$a = 33.13$	$b = -8.762$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R = 0.99$	$a = 10.99$	$b = 4.292$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R = 0.99$	$a = 9.640$	$n = 0.1887$ $\dot{\sigma}_0 = 7.265$

Rheogram and Data for Test Number TESTD3A

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R»=0.86	$\dot{\sigma}_0 = 25.62$	Eta=0.02952
Ostwald	: $\dot{\sigma} = a * D^n$	R»=0.97	a=15.61	n=0.1404
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R»=0.99	a=3.956	n=0.2830 $\dot{\sigma}_0 = 14.93$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R»=0.93	$\dot{\sigma}_0 = 21.35$	Eta=0.005484
Linear	: $Y = a + b * X$	R»=0.86	a=25.62	b=0.02952
Inv. linear:	$Y = 1 / (a + b * X)$	R»=0.37	a=0.04078	b=-38.71E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R»=0.62	a=25.08	b=0.001052
Hyperbolic:	$Y = a + b / X$	R»=0.25	a=32.62	b=-16.67
Logarithmic:	$Y = a + b * \text{LN}(X)$	R»=0.96	a=12.21	b=3.933

Optimum :

H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ R»=0.99 a=3.956 n=0.2830 $\dot{\sigma}_0 = 14.93$

Rheogram and Data for Test Number day525ca

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.84$	$\dot{\sigma}_0 = 22.71$	$\text{Eta} = 0.04119$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.79$	$a = 7.900$	$n = 0.2685$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.99$	$a = 6.937$	$n = 0.2556$ $\dot{\sigma}_0 = 5.717$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.83$	$\dot{\sigma}_0 = 16.59$	$\text{Eta} = 0.01202$
Linear	: $Y = a + b * X$	$R^2 = 0.84$	$a = 22.71$	$b = 0.04119$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.04$	$a = 0.06542$	$b = -0.000122$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.41$	$a = 21.84$	$b = 0.001561$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.19$	$a = 32.25$	$b = -8.518$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.97$	$a = 4.145$	$b = 5.462$

Optimum :

H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ $R^2 = 0.99$ $a = 6.937$ $n = 0.2556$ $\dot{\sigma}_0 = 5.717$

Rheogram and Data for Test Number day525cb

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.90$	$\dot{\sigma}_0 = 24.99$	$\eta = 0.03306$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.96$	$a = 15.02$	$n = 0.1487$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.97$	$a = 3.219$	$n = 0.3159$ $\dot{\sigma}_0 = 15.58$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.95$	$\dot{\sigma}_0 = 20.53$	$\eta = 0.006604$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.90$	$a = 24.99$	$b = 0.03306$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.43$	$a = 0.04133$	$b = -41.55E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.69$	$a = 24.60$	$b = 0.001154$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.21$	$a = 32.78$	$b = -16.88$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.93$	$a = 10.86$	$b = 4.229$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.97$	$a = 3.219$	$n = 0.3159$	$\dot{\sigma}_0 = 15.58$

Rheogram and Data for Test Number TEST-D7 @25C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.79$	$\dot{\sigma}_0 = 25.41$	$\eta = 0.03875$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.76$	$a = 9.534$	$n = 0.2452$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 1.00$	$a = 8.636$	$n = 0.2277$ $\dot{\sigma}_0 = 5.946$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.78$	$\dot{\sigma}_0 = 19.22$	$\eta = 0.01024$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.79$	$a = 25.41$	$b = 0.03875$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.04$	$a = 0.05537$	$b = -93.83E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.37$	$a = 24.40$	$b = 0.001379$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.24$	$a = 34.45$	$b = -11.21$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.99$	$a = 6.789$	$b = 5.364$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 1.00$	$a = 8.636$	$n = 0.2277$ $\dot{\sigma}_0 = 5.946$

Rheogram and Data for Test Number TEST-D7A @25C

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R=0.91	$\dot{\sigma}_0 = 25.61$	Eta = 0.03458
Ostwald	: $\dot{\sigma} = a * D^n$	R=0.91	a = 15.47	n = 0.1486
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R=0.93	a = 4.512	n = 0.2727 $\dot{\sigma}_0 = 14.62$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R=0.93	$\dot{\sigma}_0 = 21.13$	Eta = 0.006811
Linear	: $Y = a + b * X$	R=0.91	a = 25.61	b = 0.03458
Inv. linear	: $Y = 1 / (a + b * X)$	R=0.32	a = 0.04089	b = -42.86E-06
Exponential	: $Y = a * \text{EXP}(b * X)$	R=0.64	a = 25.14	b = 0.001182
Hyperbolic	: $Y = a + b / X$	R=0.23	a = 33.77	b = -18.56
Logarithmic	: $Y = a + b * \text{LN}(X)$	R=0.89	a = 11.39	b = 4.313

Optimum :

Casson : $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$ R=0.93 $\dot{\sigma}_0 = 21.13$ Eta = 0.006811

Rheogram and Data for Test Number glyd10a

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R^2 = 0.87$	$\dot{\sigma}_0 = 22.79$	$\text{Eta} = 0.03596$
Ostwald	: $\dot{\sigma} = a * D^n$	$R^2 = 0.54$	$a = 7.502$	$n = 0.2714$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.93$	$a = 7.518$	$n = 0.2152$ $\dot{\sigma}_0 = 7.870$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R^2 = 0.75$	$\dot{\sigma}_0 = 17.59$	$\text{Eta} = 0.008925$
Linear	: $Y = a + b * X$	$R^2 = 0.87$	$a = 22.79$	$b = 0.03596$
Inv. linear	: $Y = 1 / (a + b * X)$	$R^2 = 0.02$	$a = 0.1397$	$b = -365.2E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R^2 = 0.24$	$a = 21.49$	$b = 0.00147$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.25$	$a = 31.17$	$b = -10.76$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R^2 = 0.90$	$a = 7.645$	$b = 4.557$

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R^2 = 0.93$	$a = 7.518$	$n = 0.2152$	$\dot{\sigma}_0 = 7.870$

Rheogram and Data for Test Number glyd10b

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	$R = 0.95$	$\dot{\sigma}_0 = 24.23$	$\text{Eta} = 0.03177$
Ostwald	: $\dot{\sigma} = a * D^n$	$R = 0.85$	$a = 16.25$	$n = 0.1271$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	$R = 0.86$	$a = 1.190$	$n = 0.4250$ $\dot{\sigma}_0 = 20.03$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	$R = 0.96$	$\dot{\sigma}_0 = 20.42$	$\text{Eta} = 0.005723$
Linear	: $Y = a + b * X$	$R = 0.95$	$a = 24.23$	$b = 0.03177$
Inv. linear	: $Y = 1 / (a + b * X)$	$R = 0.35$	$a = 0.0425$	$b = -42.04E-06$
Exponential	: $Y = a * \text{EXP}(b * X)$	$R = 0.69$	$a = 23.93$	$b = 0.001132$
Hyperbolic	: $Y = a + b / X$	$R = 0.11$	$a = 31.52$	$b = -5.441$
Logarithmic	: $Y = a + b * \text{LN}(X)$	$R = 0.81$	$a = 12.82$	$b = 3.640$

Optimum. :

Casson : $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$ $R = 0.96$ $\dot{\sigma}_0 = 20.42$ $\text{Eta} = 0.005723$

Rheogram and Data for Test Number glyd15a

Bingham : $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$ $R = 0.89$ $\dot{\sigma}_0 = 25.69$ $\text{Eta} = 0.03548$
 Ostwald : $\dot{\sigma} = a * D^n$ $R = 0.97$ $a = 14.77$ $n = 0.1592$
 H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ $R = 0.99$ $a = 4.301$ $n = 0.2918$ $\dot{\sigma}_0 = 13.92$
 Casson : $\sqrt{\dot{\sigma} - \dot{\sigma}_0} = \sqrt{\text{Eta} * D}$ $R = 0.94$ $\dot{\sigma}_0 = 20.87$ $\text{Eta} = 0.007424$
 Linear : $Y = a + b * X$ $R = 0.89$ $a = 25.69$ $b = 0.03548$
 Inv. linear: $Y = 1 / (a + b * X)$ $R = 0.11$ $a = 0.04495$ $b = -57.37E-06$
 Exponential: $Y = a * \text{EXP}(b * X)$ $R = 0.48$ $a = 24.71$ $b = 0.001272$
 Hyperbolic : $Y = a + b / X$ $R = 0.23$ $a = 34.07$ $b = -18.88$
 Logarithmic: $Y = a + b * \text{LN}(X)$ $R = 0.95$ $a = 10.23$ $b = 4.598$

Optimum :
 H.-Bulkley : $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$ $R = 0.99$ $a = 4.301$ $n = 0.2918$ $\dot{\sigma}_0 = 13.92$

Rheogram and Data for Test Number glyd15b

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.97$	$\dot{\sigma}_0 = 24.79$	$\eta = 0.03284$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.85$	$a = 16.68$	$n = 0.1269$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.88$	$a = 0.4173$	$n = 0.5894$
Casson	: $\dot{\sigma} - \dot{\sigma}_0 = \sqrt{\eta \cdot D}$	$R^2 = 0.98$	$\dot{\sigma}_0 = 20.89$	$\eta = 0.005934$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.97$	$a = 24.79$	$b = 0.03284$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.76$	$a = 0.04006$	$b = -36.41E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.88$	$a = 24.85$	$b = 0.001093$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.11$	$a = 32.42$	$b = -12.09$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.82$	$a = 12.77$	$b = 3.805$

Optimum :

Casson : $\dot{\sigma} - \dot{\sigma}_0 = \sqrt{\eta \cdot D}$ $R^2 = 0.98$ $\dot{\sigma}_0 = 20.89$ $\eta = 0.005934$

Rheogram and Data for Test Number T-25-31

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \text{Eta} * D$	R>=0.80	$\dot{\sigma}_0 = 25.73$	Eta = 0.02643
Ostwald	: $\dot{\sigma} = a * D^n$	R>=0.66	a = 14.29	n = 0.1536
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.93	a = 11.07	n = 0.1488 $\dot{\sigma}_0 = 7.841$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\text{Eta} * D}$	R>=0.71	$\dot{\sigma}_0 = 21.74$	Eta = 0.004539
Linear	: $Y = a + b * X$	R>=0.80	a = 25.73	b = 0.02643
Inv. linear:	$Y = 1 / (a + b * X)$	R>=0.08	a = 0.04309	b = -44.06E-06
Exponential:	$Y = a * \text{EXP}(b * X)$	R>=0.40	a = 25.32	b = 940.6E-06
Hyperbolic:	$Y = a + b / X$	R>=0.42	a = 32.11	b = -21.42
Logarithmic:	$Y = a + b * \text{LN}(X)$	R>=0.90	a = 13.53	b = 3.558

Optimum :

H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a * D^n$	R>=0.93	a = 11.07	n = 0.1488 $\dot{\sigma}_0 = 7.841$

Rheogram and Data for Test Number T-25-31A

Bingham	: $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$	$R^2 = 0.98$	$\dot{\sigma}_0 = 22.31$	$\eta = 0.03003$
Ostwald	: $\dot{\sigma} = a \cdot D^n$	$R^2 = 0.80$	$a = 15.43$	$n = 0.1223$
H.-Bulkley	: $\dot{\sigma} = \dot{\sigma}_0 + a \cdot D^n$	$R^2 = 0.70$	$a = 0.03627$	$n = 0.9696$ $\dot{\sigma}_0 = 21.91$
Casson	: $\sqrt{\dot{\sigma}} - \sqrt{\dot{\sigma}_0} = \sqrt{\eta \cdot D}$	$R^2 = 0.97$	$\dot{\sigma}_0 = 18.92$	$\eta = 0.005305$
Linear	: $Y = a + b \cdot X$	$R^2 = 0.98$	$a = 22.31$	$b = 0.03003$
Inv. linear	: $Y = 1 / (a + b \cdot X)$	$R^2 = 0.81$	$a = 0.0442$	$b = -39.85E-06$
Exponential	: $Y = a \cdot \exp(b \cdot X)$	$R^2 = 0.90$	$a = 22.46$	$b = 0.001093$
Hyperbolic	: $Y = a + b / X$	$R^2 = 0.09$	$a = 29.27$	$b = -10.08$
Logarithmic	: $Y = a + b \cdot \ln(X)$	$R^2 = 0.76$	$a = 11.97$	$b = 3.354$

Optimum :

Bingham : $\dot{\sigma} = \dot{\sigma}_0 + \eta \cdot D$ $R^2 = 0.98$ $\dot{\sigma}_0 = 22.31$ $\eta = 0.03003$

4.5 Standard Crucible and Melter Glass Redox Test Data

Introduction

This section addresses the measurement of a glass oxidation state indicator, the $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratio for small-scale feed preparations. The measured redox for two methods of glass preparation, the standard crucible melt and the small-scale melter, are discussed for Alternate HTM Flowsheet 2. The $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratios of some glass samples collected from the small-scale melter were also determined for this flowsheet. The measured $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratios are given in Table B4.5.

Experimental

Based on gram total oxide (gTO) loading, enough melter feed was placed in a quartz crucible to produce 25 g glass. Samples were dried in air at 98°C for 24 hours. After drying, a ceramic lid with a wide lip was placed on top of the quartz crucible, and this assembly was placed in a furnace at room temperature. The crucible was heated from room temperature to 1400°C at a rate of 5°C per minute or 15°C per minute. After soaking 30 minutes at 1400°C, the crucible was removed from the furnace and allowed to air cool.

The iron redox ratio of the resulting glass was measured by colorimetric titration of the glass¹. The glass is ground to a powder and dissolved under non-oxidizing conditions using a mixture of sulfuric and hydrofluoric acids. Boric acid is added to complex the excess fluoride ion. The ferrous ion in the solution is chelated by three molecules of phenanthroline, forming an orange-red complex. This colored solution obeys Beer's law. The total iron in the sample is determined by reducing the ferric ion to a ferrous ion with hydroquinone at room temperature. A 4% error has been estimated for this method when used to determine the redox state of typical simulated nuclear waste glasses².

Results

The relative quantities of oxidants and reductants are given in Table B4.5. Relative to the laboratory-scale melter feed, a slightly higher ratio of reductant to oxidant was measured for the small-scale. However, the sensitivity of the $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratio to differences of this order of magnitude is unknown. The $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratio of glasses prepared from the small-scale melter feed, using the standard crucible method, was 0.084 (mean value). The small-scale melter feed produced a slightly more reduced glass. The $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratio of glasses collected from the bottom drain of the SSHTM ranged from 0.061 to 0.175. Significant differences between the laboratory- and small-scale $\text{Fe}^{+2}/\Sigma\text{Fe}$ ratios are not evident from the current limited data base. Further analyses of the cold cap and high temperature redox chemistries will be addressed in the SSHTM-1 letter report, milestone T3C-95-129.

Table B4.5 Standard Crucible and Small-Scale Melter Glass Redox Test Data for Small-Scale Melter Feed Alternate HTM Flowsheet 2 Testing.

	<u>Final</u>	<u>Moles Oxidant^a</u>	<u>Moles Reductant^b</u>	<u>Glass: Fe⁺²/2Fe</u>			
	<u>pH (22°C)</u>	<u>(gTO/L)</u>	<u>NO₃⁻</u>	<u>NO₂⁻</u>	<u>(glycolate)</u>	<u>Crucible</u>	<u>Melter</u>
<u>Alternate HTM Flowsheet 2^c</u>							
small-scale melter feed ^d	8.4	422	0.60	0.31	0.54	N/A	N/A
dried melter feed/glass	N/A	N/A	----	----	----	0.0792	0.0687 ^e
	N/A	N/A	----	----	----	0.0882	0.0719 ^f
	N/A	N/A	----	----	----		0.0614 ^g
	N/A	N/A	----	----	----		0.1753 ^h

- (a) Reported moles of NO₃⁻ are after addition of NaNO₃. The NaNO₃ contributed 0.47 moles of nitrate per 125 gWO.
- (b) Reductant used are moles of C₂H₃O₃⁻ per 125 gWO.
- (c) Alternate HTM Flowsheet 2 is the alternate reductant, glycolic acid process flowsheet; laboratory data from test T95-SSHTMGLYC-1.
- (d) Small-scale melter feed sample corresponds to rheology sample SSHTM1-11.1.12-1 taken from the small-scale feed preparation system on Aug. 14, 1994.
- (e) Glass sample was retrieved from an SSHTM-1 canister and supplied by DA Lamar.
- (f) Sample number SSHTM1-24; sampled on Aug. 17, 1994 at 0300 hours. Glass sample is a bottom drain sample taken during melter operation.
- (g) Sample number SSHTM1-42; sampled on Aug. 18, 1994 at 0710 hours. Glass sample is a bottom drain sample taken during melter operation.
- (h) Sample number SSHTM1-50; sampled on Aug. 18, 1994 at 2009 hours. Glass sample is a bottom drain sample taken during melter operation.

5.0 REFERENCES

- 1 Technical Procedure APSL-02, "Iron II and Total Iron Ratio".
- 2 Goldman, D.S. and D.W. Brite. 1986. "Redox Characterization of Simulated Nuclear Waste Glass," J. Am. Cer. Soc. 69(5), 292-298.