PNNL--10993-App.A

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SMALL-SCALE HIGH TEMPERATURE MELTER-1 (SSHTM-1) DATA PACKAGE

APPENDIX A - Alternate HTM Flowsheet 1 (No Reductant Addition, Nitric Acid)

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QA LEVEL

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

FIGURES

TABLES

TABLES (cont.)

LOGS

1.0 INTRODUCTION

This appendix provides the data for Alternate HTM Flowsheet 1 (No Reductant Addition, Nitric 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 nitric run: glycerine & boric acid add.'s
   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 nitric acid to a pH of 4.8 ± 0.1. Add 70 wt % acid solution: Period #1 - 1.04 g/min. for 14 minutes; Period #2 - 1.53 g/min. for 10 minutes; Period #3 - 1.94 g/min. for the remainder of the test. (Incremental rates duplicate the small-scale processing run more precisely.)

- Take slurry and condensate samples.

Step 3: Concentrate slurry to 240 gWO/L by boiling during digestion period of 8 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.

Laboratory Melter Feed Preparation Activity Logs A3.1.1, A3.1.2, and A3.1.3 for laboratory-scale tests T95-SSHTMHNO3-1, T95-SSHTMHNO3-2, and T95-SSHTMHNO3-3 follow, respectively.

Log A3.1.1

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-1

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
11/11/94				
6:20	-	11.6	27	Chiller on. Stirrer set at 6.
7:30	-	-	95	Temperature set at 93 degrees (C).
7:40	-	10.3	97	Changed pH meter and probe.
8:15	-	-	-	Added 0.24 g Dow Corning 544 anti foam compound with 10 g deionized water.
8:25	-	10.2	91	Initial weight of Nitric acid 890.0g.
8:40	0	-	-	Started HNO3 addition. Target addition rate period #1 - 1.04 g/min. for 14 minutes. Period #2 - 1.53 g/min. for 10 minutes. Period #3 - 1.94 g/min. until pH of < 4.9 is met.
8:41	1	-	-	Addition rate 1.7 g/min.
8:42	2	-	-	Addition rate 1.2 g/min.
8:45	5	10.0	92	Addition rate 0.8 g/min.
8:46	6	10.0	92	Addition rate 0.7 g/min.

Log A3.1.1
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-1

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
11/11/94				
8:47	7	9.8	92	Addition rate 0.6 g/min.
8:48	8	9.9	92	Addition rate 0.8 g/min.
8:53	13	9.5	92	Addition rate 0.9 g/min.
8:55	15	-	-	Increased pump rate for period #2 of acid addition . Target rate of 1.53 g/min for ~10 minutes.
8:56	16	9.2	91	Addition rate 1.7 g/min.
8:57	17	9.2	91	Addition rate 1 g/min.
8:58	18	9.1	92	Addition rate 1.5 g/min.
9:03	23	8.3	99	Obvious temperature increase.
9:05	25	7.9	100	Increase acid addition for period #3. Target addition rate 1.95 g/min until pH of <4.9 obtained. Still obvious temperature increase. Thick layer of foam.
9:06	26	7.7	91	Obvious temperature decrease.
9:07	27	7.6	90	Addition rate 1.6 g/min.

Log A3.1.1
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-1

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
11/11/94				
9:09	29	7.5	87	Addition rate 1.8 g/min.
9:10	30	7.4	84	Still foaming.
9:11	31	7.2	99	Noticeable temperature increase.
9:12	32	7.1	100	Foaming.
9:13	33	7.1	97	Temperature decreasing.
9:16	36	6.8	91	Thick foam over surface.
9:17	37	6.7	91	Still thick foam.
9:20	40	6.6	93	No change.
9:24	44	6.1	97	Added more anti foam to reduce foaming. Same amount as previous addition.
9:27	47	5.9	94	Foam slightly decreasing.
9:30	50	5.6	90	Foaming.
9:31	51	5.4	91	Still foaming.

Log A3.1.1
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-1

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
11/11/94				
9:37	57	5.0	94	Still foaming. Slurry appears to be thickening.
9:38	58	4.9	94	No change.
9:40	60	4.8	93	Acid addition complete. Added 85.7 g acid.
9:42	62	-	-	Removed 36.4 g of condensate, T95-SSHTMHNO3-1-2-2.
-				Removed 2.4 g of secondary condensate, T95-SSHTNHNO3-1-2-2s.
-				Removed 15.3 g of post acid addition slurry,
9:46	66	-	-	T95-SSHTMHNO3-1-1-2.
9:47	67	-	90	Increased temperature to 102 degrees (C) to begin digestion period.
9:50	70	5.0	90	Thick foaming. Possible jellying.
9:51	71	5.1	102	Boiling.
10:03	83	5.2	102	Still foaming.
10:25	105	5.3	101	No change.
10:35	115	5.3	102	No change.
10:52	132	5.3	103	Possible jellying.

Log A3.1.1
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-1

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
11/11/94				
10:54	134	-	-	Removed 37.2 g condensate, T95-SSHTMHNO3-1-2-3.
11:00	140	5.3	103	
11:24	164	5.4	102	Still appears to be jellying.
12:05	205	5.4	103	Removed 38.5 g condensate, T95-SSHTMHNO3-1-2-3. Slurry appears to be jellying.
12:45	245	5.4	103	No change.
13:03	263	-	-	Removed 34.1 g condensate, T95-SSHTMHNO3-1-2-3.
13:39	299	5.4	104	Condensate collection slow.
13:56	316	5.4	103	Decreased temperature to 98 degrees (C) to see if there is a difference in condensate collection.
13:59	319	-	-	Removed 29.6 g condensate, T95-SSHTMHNO3-1-2-3.
14:05	325	5.4	101	No change.
14:20	340	5.5	94	Increased stirrer setting to 8. Increasing temperature to 103 degrees (C).

Log A3.1.1
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-1

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
11/11/94				
14:36	356	5.3	104	Condensate appears to be coming off at a faster rate.
14:45	365	5.3	103	Surface not moving again even with stirrer set at 10. Decreased temperature to 100 degrees (C).
14:50	370	5.4	98	Stirring again. Removed 38.7 g condensate, T95-SSHTMHNO3-1-2-3.
14:53	373	5.4	96	Increased temperature to 102 degrees (C).
15:05	385	5.3	104	Surface stopped stirring. Decreased temperature to 100 degrees (C).
15:11	391	5.4	102	Temperature dropped to 99 degrees (C) then shot up to 103. Slight surface movement.
15:14	394	5.4	97	Mixing appears to be very fluid.
15:16	396	-	-	Removed 34.3 g condensate, T95-SSHTMHNO3-1-2-3.
15:18	398	5.4	96	Increased temperature to 101 degrees (C). Good surface movement.
15:33	413	5.4	102	A jellied appearance. Decrease to 100 degrees (C).
15:42	422	-	-	Removed 35.6 g condensate, T95-SSHTMHNO3-1-2-3.

Log A3.1.1
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-1

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
11/11/94				
16:12	452	5.5	96	Removed 39.2 g condensate, T95-SSHTMHNO3-1-2-3.
16:26	466	5.5	99	Removed 25.1 g condensate, T95-SSHTMHNO3-1-2-3.
17:18	518	5.6	83	No explanation of temperature changes.
17:34	534	5.5	104	Removed 36.6 g condensate, T95-SSHTMHNO3-1-2-3. Decreased temperature setting to 0.
18:00	560	-	-	Off-gas data collection stopped at this time, therefore, the cumulative time log is discontinued. Test completed at this time. Removed 16.1 g secondary condensate, T95-SSHTMHNO3-1-2-3s. Removed 9.8 g condensate, T95-SSHTMHNO3-1-2-3. Shut system down. Removed vessel to check slurry weight. Removed 15.4 g post digest slurry, T95-SSHTMHNO3-1-1-3. *Note: Removed 13.0 g initial slurry, T95-SSHTMHNO3-1-1-1 from carboy of slurry used for this test. The carboy is identified as SSHTM-1 dated 8-13-94
18:00				Final slurry product submitted to Eve Tracey for rheology measurements. Sample sshno1, sshno2, sshno3, sshno4, sshno5, and sshno6 are all samples taken from final slurry product. The rheology sample data can be found on Table A3.4.2.

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

Log A3.1.2

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
9:45	-	11.6	25.6	Stirrer on 6. Set point temperature to 93 degrees (C). Argon on.
9:53	-	11.6	25.5	Condensate collection started.
9:57	-	11.3	33	Off-gas equipment on.
10:03	-	11.3	32	Added 0.24 g Dow Corning 544 anti foam compound with 10 g deionized water.
10:14	-	10.7	60	Preparing for Nitric acid addition. Calibrating acid addition pump.
10:25	-	10.1	92	Temperature stabilizing. No condensate collected for intial sample. Off-gas detects a small air leak. Nitrogen reading 2500-3000 ppm Carbon Dioxide reading 10-40 ppm.
10:32	-	9.8	97	No change.
10:36	-	9.7	100	No change.
10:42	-	9.6	100	Waiting for temperature to stabilize.
10:59	-	9.5	100	Decreased temperature set point to 89 degrees (C) to help stabilize temperature.

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
11:07	-	9.5	99	Decreased temperature set point to 87 degrees (C).
11:11	-	9.5	99	Decreased temperature set point to 85 degrees (C) to stabilize temperature.
11:24	-	9.6	97	Slurry becoming thick with slight stirring movement. Increased stirrer speed to 7.
11:29	-	9.8	90	Slurry more fluid with increased stirrer movement. Temperature stabilization may be due to stir speed. Increased temperature set point to 93 degrees (C).
11:33	-	9.9	89	Waiting for temperature stabilization.
11:37	-	9.9	89	No change.
11:41	-	9.9	91	No change.
11:44	-	9.9	92	Removed 6.6 g of T95-SSHTMHNO3-2-2-1 Initial condensate.
11:51	0	9.9	92	Started Nitric acid addition. Initial acid weight 1236.7 g. Target addition rate, period #1 - 1.04 g/min. for 14 minutes. Period #2 - 1.53 g/min. for 10 minutes. Period #3 - 1.94 g/min. until pH of < 4.9 is met. Pump setting started on 2.4 for period #1.

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
11:53	2	9.8	93	Addition rate 1.04 g/min.
11:55	4	9.7	93	Addition rate 1.5 g/min. Decreased pump setting to 1.6.
11:58	7	9.5	93	Addition rate 0.8 g/min. Increased pump setting to 2.
12:00	9	9.4	93	Addition rate 1.2g/min. Decreased pump setting to 1.8.
12:02	11	9.3	92	Addition rate 1.0 g/min.
12:05	14	9.3	92	Increased pump rate to 2.4 for period #2 of acid addition . Target rate of 1.53 g/min for ~10 minutes.
12:07	16	8.9	92	Addition rate 1.5 g/min.
12:11	20	8.7	93	No change.
12:16	25	8.2	93	Increase pump rate to 5.0 for period #3. Target addition rate 1.95 g/min until pH of <4.9 obtained.
12:18	27	7.9	94	Addition rate 2.3 g/min. Decreased pump rate to 4.6. Slight foaming.
12:22	31	7.4	93	Addition rate 2.0 g/min. Decreased pump rate to 4.0. Increase in foaming.

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
12:24	33	7.1	93	Addition rate 1.8 g/min.
12:28	37	6.7	92	Still foaming.
12:33	42	6.2	93	No change.
12:37	46	5.9	94	Foam thickening.
12:41	50	5.5	93	Stirrer speed increased to 8 due to temperature fluctuations.
12:47	56	5.0	95	Still foaming.
12:50	59	4.8	94	Acid addition completed. Final weight of acid 1140.4 g. Total of 96.3 g of Nitric acid added.
12:58	67	-	-	Removed 27.3 g of T95-SSHTMHNO3-2-2-2 condensate.
13:01	70	4.9	91	No more foam. Removed 12.1 g of T95-SSHTMHNO3-2-1-2 slurry.
13:04	73	5.0	91	Increased temperature set point to 96 degrees (C).
13:10	79	5.0	94	No change.
13:15	84	5.0	96	Increased temperature set point to 97degrees (C). Some foaming.

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
13:17	86	5.0	97	Slurry thickening, surface movement reduced. Increased stirrer speed to 9. Slurry looks slightly jelled. Some foaming. Increased temperature set point to 98 degrees (C).
13:21	90	5.0	98	Increased set point to 99. Still foaming.
13:23	92	-	99	Slurry thickening, reducing surface movement. Increased stirrer speed to 10.
13:26	95	5.0	99	Slurry thick with minimal surface movement. Still foaming.
13:30	99	5.0	99	No change.
13:34	103	5.1	100	Still foaming. Slurry thickening agian and reducing surface movement.
13:40	109	5.2	99	Good stirring action.
13:45	114	5.2	97	Stirrer set back to 8. Slurry appears to be fluid with good stirring action.
13:52	121	5.2	98	Removed 36.4 g of T95-SSHTMHNO3-2-2-3 condensate. Stirring action still good.
13:55	124	5.2	98	Increased temperature set point to 100 degrees (C).

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
13:57	126	5.2	99	Slurry thickening with little surface movement. Increased stir speed to 10. Some stirring movement with foam.
14:00	129	5.2	100	Increased set point to 102 degrees (C). Very little surface movement due to slurry thickening. Some stirring movement with foam.
14:05	134	5.2	101	Foam to vessel lid interface.
14:15	144	5.3	102	Heavy foaming.
14:24	153	5.4	102	No change.
14:27	156	5.4	102	Removed 28.8 g of T95-SSHTMHNO3-2-2-3 condensate.
14:33	162	5.4	102	Still foaming.
14:38	167	5.4	101	A little foam covering surface.
14:46	175	5.5	102	Heavy foaming.
14:53	182	5.5	102	No change.
14:54	183	5.5	102	Removed 30.2 g of T95-SSHTMHNO3-2-2-3 condensate.
15:01	190	5.5	102	Still foaming.

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
15:11	200	5.6	102	No change.
15:15	204	5.6	102	No change.
15:21	210	5.6	102	Small amount of foam.
15:35	224	5.9	102	Foaming.
15:41	230	6.2	102	No change.
15:46	235	6.4	102	No change.
15:50	239	6.4	102	Heavy foaming.
16:01	250	6.5	102	No change.
16:09	258	6.7	102	No change.
16:16	265	6.8	102	Slow condensate collection.
16:22	271	6.8	102	Heavy foam.
16:26	275	6.8	102	No change.
16:30	279	6.7	102	No change.

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
16:39	288	6.8	102	No change.
16:44	293	6.9	102	No change.
16:48	297	6.9	102	No change.
16:56	305	7.0	102	Heavy Foam.
17:01	310	7.0	102	No change.
17:11	320	7.2	102	No change.
17:16	325	7.1	102	Removed 31.4 g of T95-SSHTMHNO3-2-2-3 condensate. (slow collection)
17:30	339	7.2	102	Foam to just below vessel flange.
17:35	344	7.2	102	Still foaming.
17:40	349	7.2	102	No change.
17:45	354	7.2	102	Foam to bottom of vessel flange.
17:50	359	7.3	102	No change.

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
18:05	374	7.3	102	Foam decreasing to ~ 1/4" from bottom of vessel flange. (rim)
18:10	379	7.3	102	No change.
18:16	385	7.2	102	Still foaming.
18:20	389	7.2	102	No change.
18:26	395	7.2	102	No change.
18:34	403	7.4	102	No change.
18:39	408	7.4	102	Decreased temperature set point to 101 degrees (C).
18:42	411	7.5	102	Foaming decreased to top of heating mantle.
18:46	415	7.6	102	Foam still decreasing.
18:53	422	7.7	102	No change.
19:02	431	7.6	102	Small amount of foam on surface. Removed 21.4 g of T95-SSHTMHNO3-2-2-3 condensate.
19:11	440	7.8	102	Small amount of foam covering surface.

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
19:21	450	7.9	102	No change.
19:29	458	7.9	102	Slow condensate collection. Slurry thickening with very little surface movement.
19:36	465	7.9	102	No change.
19:47	476	8.0	102	Thick bubbles. No stirring action. Stirrer set at 10. Condensate collection slow only ~3 ml of condensate removed since 19:02. Decreased temperature set point to 98 degrees (C) to regain stirring action.
20:15	504	8.1	102	No change.
20:21	510	7.9	99	Good stirring action.
20:25	514	7.9	99	Good stirring action. Increased temperature set point to 100 degrees (C).
20:30	519	7.9	99	Still good stirring action.
20:44	533	7.9	102	Slurry thickening, little stirring action. Decreased temperature set point to 99 degrees (C).
20:50	539	7.7	102	No stirring action. Condensate collected since 19:02 ~5 ml.

Log A3.1.2
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-2

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/1/94				
20:51	540	7.9	100	Good stirring action.
20:56	545	7.9	99	Stirring good.
21:10	559	7.8	95	Discovered a cracked inlet Argon gas line to vessel. Replaced tubing.
21:14	563	8.1	98	Stirring action good. Increased temperature set point to 101 degrees (C).
21:19	568	8.1	98	No change.
21:23	572	8.0	99	Stirring action good. Condensate removal rate faster.
21:30	579	8.0	100	Removed 40.4 g of T95-SSHTMHNO3-2-2-3 condensate.
21:32	581	-	-	Removed 5.1 g of T95-SSHTMHNO3-2-2-3s secondary condensate.
21:40	-	8.0	100	Decreased temperature set point to 0. Off-gas data collection stopped at this time, therefore, the cumulative time log is discontinued. Test completed at this time.
21:45	-	8.0	98	Removed 13.1 g of T95-SSHTMHNO3-2-1-3 slurry. Argon off. Stirrer left on 6.

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

Log A3.1.3

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
7:35	-	11.3	24	Stirrer on 6. Set point temperature to 93 degrees (C). Argon on.
8:09	-	10.4	92	No change.
8:31	-	9.0	100	Set point to 90 degrees (C).
9:05	-	9.2	88	Set point to 92 degrees (C).
9:29	-	9.2	91	Set Point to 93 degrees (C). Connecting off-gas system. Preparing for acid addition.
9:36	-	9.2	91	Added 0.24 g Dow Corning 544 anti foam compound with 10 g deionized water.
9:56	-	9.2	91	Waiting for off-gas system to start.
10:10	-	9.3	92	Detected small air leak from acid addition line. Sealed leak.
10:34	-	9.3	93	Possible air leak in off-gas system. No leak found.
10:36	-	9.3	92	Removed 18 g of T95-SSHTMHNO3-3-2-1 condensate.

Log A3.1.3
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
10:38	0	9.3	93	Started HNO3 addition. Initial acid weight 1111.2 g. Target addition rate, period #1 - 1.04 g/min. for 14 minutes. Period #2 - 1.53 g/min. for 10 minutes. Period #3 - 1.94 g/min. until pH of < 4.9 is met.
10:40	2	9.2	93	Addition rate 1.0 g/min. Pump rate set at 1.6.
10:45	7	8.9	93	No change.
10:47	9	8.8	92	Addition rate 1.1 g/min. Pump rate decreased to 1.4.
10:49	11	8.8	93	Addition rate 0.9 g/min.
10:51	13	8.7	93	Increased pump rate to 2.4 for period #2 of acid addition . Target rate of 1.53 g/min for ~10 minutes.
10:54	16	8.5	92	Addition rate 1.2 g/min. Increased pump rate to 2.8.
10:55	17	8.4	92	Addition rate 1.5 g/min.
10:59	21	8.1	92	Increased stir speed to 8 per phone call H.D. Smith and Test Instructions.
11:01	23	7.9	92	Increase pump rate to 4 for period #3. Target addition rate 1.95 g/min until pH of <4.9 obtained.

Log A3.1.3
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
11:04	26	7.5	92	Addition rate 1.5 g/min. Increased pump rate to 4.4.
11:07	29	6.9	92	Decrease pump rate to 4.2. Addition rate 1.9 g/min.
11:11	33	6.3	93	Slight foaming.
11:12	34	6.2	93	Stirrer increased to 9.
11:14	36	6.0	93	Foam covering surface.
11:18	40	5.8	94	Heavy foaming.
11:21	43	5.7	93	Still foaming.
11:24	46	5.5	93	Increased stirrer speed to 10.
11:25	47	5.3	93	Foaming.
11:28	50	5.1	93	Foaming.
11:30	52	4.9	93	Foaming.
11:33	55	4.7	93	Acid addition completed. Final weight 1031.2 g a total of 80 g acid added.

Log A3.1.3
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No ReJuctant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
11:36	58	4.6	92	Removed 33.3 g of T95-SSHTMHNO3-3-2-2 condensate.
11:38	60	-	-	Removed 4.0 g of T95-SSHTMHNO3-3-2-2s secondary condensate.
11:41	63	4.7	92	Good surface movement. No foam.
11:42	64	4.7	92	Increased temperature set point to 95 degrees (C) for beginning of digestion heat up phase.
11:45	67	4.7	92	Removed 14.4 g of T95-SSHTMHO3-3-1-2 slurry.
11:49	71	4.7	93	No change.
11:54	76	4.7	94	No change.
12:00	82	4.8	95	Increased temperature set point to 96 degrees (C).
12:05	87	4.8	95	No change.
12:06	88	4.8	95	Removed 33.0 g of T95-SSHTMHNO3-3-2-1 condensate.
12:11	93	4.8	95	Good surface movement.
12:14	96	4.8	95	Increased temperature set point to 97 degrees (C).

Log A3.1.3
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
12:19	101	4.8	96	Good surface movement.
12:24	106	4.8	96	No change. Increased temperature set point to 98 degrees (C).
12:28	110	4.8	97	Removed 27.6 g of T95-SSHTMHNO3-3-2-3 condensate. Increased temperature set point to 99 degrees (C).
12:34	116	4.8	98	No change.
12:39	121	4.8	99	Increased temperature set point to 102 degrees (C).
12:42	124	4.8	100	Thick surface foam not much action.
12:45	127	4.8	101	Boiling.
12:50	132	4.9	102	Small amount of foam, thick slurry with not much stirring action.
12:55	137	4.9	102	Thick boiling action.
13:00	142	4.9	102	Removed 37.5 g of T95-SSHTMHNO3-2-3 condensate.
13:05	147	4.9	102	Slurry thick, boiling with little stirring action.
13:15	157	4.9	102	No change.

Log A3.1.3
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
13:25	167	5.0	102	Removed 33.1 g of T95-SSHTMHNO3-2-3 condensate. 13:20 temperature dropped to 97 degrees (C) then back up to 102.
13:30	172	5.0	102	Temperature erratic.
13:32	174	5.0	100	Fluke thermometer reads a constant 102.2 degrees (C) on slurry temperature.
13:37	179	5.0	102	Temperature appears to be holding.
13:41	183	5.0	102	Removed 30.3 g of T95-SSHTMHNO3-3-2-3 condensate.
13:48	190	5.0	102	Very little surface movement.
13:50	192	5.0	102	Temperature bouncing between 102.7-103.1 degrees (C).
13:55	197	5.0	102	Surface appearance suggests slurry has elevated viscosity (i.e. thick).
14:03	205	5.0	102	No change.
14:09	211	5.0	102	Removed 37 g of T95-SSHTMHNO3-2-3 condensate. No noticable motion on surface with the exception of stationary surface bubbles.
14:21	223	5.0	102	No change.

Log A3.1.3
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
14:26	228	5.0	101	No change.
14:30	232	5.0	100	Slight temperature decrease. Removed 29.2 g of T95-SSHTMHNO3-3-2-3 condensate.
14:36	238	5.0	101	Temperature bouncing between 101-100 degrees (C).
14:41	243	5.0	103	No surface movement.
14:47	249	5.0	103	No change.
14:55	257	5.0	103	Removed 39.4 g of T95-SSHTMHNO3-3-2-3 condensate.
15:00	262	5.0	102	No surface movement.
15:06	268	5.0	103	No change.
15:09	271	5.0	103	Hard to see surface movement due to slurry spatter on reaction vessel lid and sides.
15:17	279	5.0	103	Removed 32.6 g of T95-SSHTMHNO3-3-2-3 condensate.
15:25	287	5.0	103	Still unable to see slurry movement in reaction vessel.
15:32	294	5.0	103	No change.

Log A3.1.3
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
15:43	305	5.0	103	Removed 32.1 g of T95-SSHTMHNO3-3-2-1 condensate.
15:47	309	5.0	103	No change.
15:52	314	5.0	103	No change.
16:01	323	5.0	104	Increased temperature set point to 101 degrees (C).
16:10	332	5.0	103	No change.
16:24	346	5.0	103	Removed 35.3 g of T95-SSHTMHNO3-3-2-3 condensate.
16:30	352	5.0	103	Removed 12.6 g of T95-SSHTMHNO3-3-2-3 secondary condensate.
16:42	364	5.0	102	No change.
16:48	370	5.0	103	Temperature erratic and fluctuated between 100-105 in 3 minutes.
16:55	377	5.0	103	No change.
17:12	394	5.0	101	No change.
17:19	401	5.0	102	Removed 29.9 g of T95-SSHTMHNO3-3-2-3 condensate.
17:24	406	5.0	102	No change.

Log A3.1.3
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
17:29	411	5.0	102	No change.
17:33	415	5.0	102	No change.
17:40	422	5.0	102	No change.
17:46	428	5.0	102	Unable to view slurry surface.
17:54	436	5.0	102	No change.
18:00	442	5.0	102	Removed 28.9 g of T95-SSHTMHNO3-3-2-3 condensate. Removed 2.9 g of T95-SSHTMHNO3-3-2-3s secondary condensate.
18:06	448	5.0	101	No change.
18:16	458	4.9	101	No change.
18:20	462	-	-	Removed 12.7 g of T95-SSHTMHNO3-3-2-3 condensate.
18:28	470	4.9	101	Refluxing started.
18:34	476	4.9	100	Increased temperature set point to 102 degrees (C).
18:43	485	4.9	101	No change.

Log A3.1.3
(Cont.)

Laboratory-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition, Test-T95-SSHTMHNO3-3

Date/ Time	Cumulative Time (min) (1)	pH	Temperature Slurry (C)	Activity/Observations
12/6/94				
18:50	492	4.9	101	No change.
19:00	502	4.9	103	No change.
19:07	509	4.9	103	No change.
19:22	524	4.9	101	Slurry appears to be thick with no surface movement. Little visibility.
19:28	530	4.9	103	No change.
19:35	537	4.9	103	No change.
19:45	547	4.9	101	Decreased temperature set point to 0.
19:59	561	4.9	98	Off-gas data collection stopped at this time, therefore, the cumulative time log is discontinued. Test completed at this time.
20:09	-	4.8	86	Argon off. Stirrer left on.
12/7/94				
9:12	-	41	-	Removed 21.9 g of T95-SSHTMHNO3-3-1-3 slurry.

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, formating, 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 ΣgWO (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. Gram waste oxide (gWO) tracking sheets, Tables A3.1.1, A3.1.2, and A3.1.3, for laboratory-scale tests T95-SSHTMHNO3-1, T95-SSHTMHNO3-2, and T95-SSHTMHNO3-3 follow, respectively.

Table A3.1.1 Laboratory-Scale Melter Feed Gram Waste Oxide (gWO) Tracking Sheet for Test T95-SSHTMHNO3-1.

Sample/Action	Volume (mL)/Weight (g) Sample	Slurry	gWO/L	Total gWO	Density
Initial Slurry	-	1534/1780	173	265	1.16
Acid addition, condensate T95-SSHTMHNO3-1-2-2	36.4/36.4	1498/1744	177	265	1.16*
Acid addition, secondary condensate T95-SSHTMHNO3-1-2-2s	2.4/2.4	1495/1741	177	265	1.16
Acid addition, slurry T95-SSHTMHNO3-1-1-2	13.2/15.3	1482/1726	177	263	1.16
Digestion, condensate T95-SSHTMHNO3-1-2-3	359/359	1123/1367	234	263	1.21
Digestion, secondary condensate T95-SSHTMHNO3-1-2-3s	16.1/16.1	1107/1351	238	263	1.22
Weight of slurry after completion of digestion.	26/26	1081/1377	243	263	1.27
Post digestion, slurry T95-SSHTMHNO3-1-1-3	15.2/18.2	1066/1359	243	259	1.27

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

Table A3.1.2 Laboratory-Scale Melter Feed Gram Waste Oxide (gWO) Tracking Sheet for Test T95-SSHTMHNO3-2.

	Volume (mL)/Weight (g)			Total	
Sample/Action	Sample	Slurry	gWO/L	gWO	Density
Initial Slurry	-	1523/1761	173	264	1.16
Initial condensate T95-SSHTMHNO3-2-2-1	6.6/6.6	1516/1760	174	264	1.16*
Acid addition, condensate T95-SSHTMHNO3-2-2-2	27.3/27.3	1489/1733	177	264	1.16
Acid addition, slurry T95-SSHTMHNO3-2-1-2	10/12.1	1479/1721	177	262	1.16
Digestion, condensate T95-SSHTMHNO3-2-2-3	208.7/208.7	1270/1512	206	262	1.19
Digestion, secondary condensate T95-SSHTMHNO3-2-2-3s	5.1/5.1	1265/1507	207	262	1.19
Post digestion, slurry T95-SSHTMHNO3-2-1-3	11/13.1	1254/1494	207	260	1.19

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

Table A3.1.3 Laboratory-Scale Melter Feed Gram Waste Oxide (gWO) Tracking Sheet for Test T95-SSHTMHNO3-3.

Sample/Action	Volume (mL)/Weight (g) Sample	Slurry	gWO/L	Total gWO	Density
Initial Slurry	-	1518/1761	173	263	1.16
Initial condensate T95-SSHTMHNO3-3-2-1	18/18	1500/1743	175	263	1.16*
Acid addition, condensate T95-SSHTMHNO3-3-2-2	33.3/33.3	1467/1710	179	263	1.17
Acid addition, secondary condensate T95-SSHTMHNO3-3-2-2s	4/4	1463/1706	180	263	1.17
Acid addition, slurry T95-SSHTMHNO3-3-1-2	12.3/14.4	1450/1692	180	261	1.17
Digestion, condensate T95-SSHTMHNO3-3-2-3	437.7/437.7	1013/1254	258	261	1.24
Digestion, secondary condensate T95-SSHTMHNO3-3-2-3s	15.5/15.5	997/1238	262	261	1.24
Post digestion, slurry T95-SSHTMHNO3-3-1-3	18/21.9	979/1217	262	256	1.24
Final weight at end of test.	65.7/65.7	914/1151	280	256	1.26

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

3.2 Melter Feed and Condensate Composition

Table A3.2 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:

<u>gWO/L and density-</u> 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%.

<u>pH-</u> 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.

<u>Total Organic Carbon-</u> 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).

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

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

<u>Elements, Oxides-</u> The elemental or oxide analyses were made by ICP-AES using procedure APSL-14. The estimated accuracy for major elements using this technique is ±10%.

Table A3.2 Summary of Analytical Data for the Laboratory-Scale, Alternate HTM Flowsheet 1 Tests, T95-SSHTMHNO3-1 and T95-SSHTMHNO3-3. 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).

		gWO/L	Density (g/ml)	pH[a]	Total Organic Carbon (wt%)	Nitrite (ppm)	Nitrate (ppm)	Ammonia (ppm)
Alternate HTM Flowsheet 1[b]								
T95-SSHTMHNO3-1								
Slurry Sample:								
	HNO3-1-1-1	172.5	1.154	11.64	0.05[c]	24000	11000	0.35
	HNO3-1-1-2	177	1.16	6.34		22000	45000	0.12
	HNO3-1-1-3	232[d]	1.3	7.17		27000	59000	< 0.25
Condensate Sample:								
	HNO3-1-2-2	N/A[e]	1.0	1.39		5	3750	2.2
	HNO3-1-2-3	N/A[e]	1.0	2.37		3.5	270	0.1
T95-SSHTMHNO3-3								
Slurry Sample:								
	HNO3-3-1-1	172.5	1.154	11.44	0.05[c]	24000	11000	1.3
	HNO3-3-1-2	180	1.16	6.60		18000	43000	< 0.43
	HNO3-3-1-3	267[d]	1.3	7.44		28000	66000	< 0.41
Condensate Sample:								
	HNO3-3-2-1	N/A[e]	1.0	3.85		2.7	22	4.6
	HNO3-3-2-2	N/A[e]	1.0	0.82		7.3	17500	2
	HNO3-3-2-3	N/A[e]	1.0	2.09		1.7	550	< 0.1

(a) pH measurements were taken at room temperature, approximately 22°C.
(b) Alternate HTM Flowsheet 1 is the no reductant, nitric acid process flowsheet.
(c) Same starting slurry was used for the laboratory- as for the small-scale processing tests.
(d) Note: these are measured values and differ slightly from the values calculated for the gram waste oxide tracking tables.
(e) 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 three nitric acid process flowsheets. The T95-SSHTMHNO3-3 laboratory-scale run provides the most reliable data due to known equipment malfunctions during the first and second runs. The off-gas record is organized in the following way:

1- Table A3.3 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 A3.3.1, A3.3.2, A3.3.3, A3.3.4, and A3.3.5 present off-gas process profiles for laboratory-scale tests T95-SSHTMHNO3-1, T95-SSHTMHNO3-2, and T95-SSHTMHNO3-3, respectively. 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 ±10%, including uncertainties from mass flow controllers and intermittent small leaks around the reaction vessel sampling and chemical addition ports.

Table A3.3 Peak Off-Gas Generation Rates and Total Off-Gas Generated for Laboratory-Scale Alternate HTM Flowsheet 1 Tests.

Alternate HTM Flowsheet 1[b]		Off-Gas Generated During Processing CO_2	N_2O	H_2	NO_x	NH_3[a]
T95-SSHTMHNO3-1[c]:	Peak Generation Rate (mmoles/min)	8.4	----[d]	BDL[e]	0.592	f
	Total Off-Gas Generated (mmoles)	250	0.07	BDL[e]	28	< 0.19
T95-SSHTMHNO3-2:	Peak Generation Rate (mmoles/min)	12.6	----[d]	BDL[e]	2.4	f
	Total Off-Gas Generated (mmoles)	N/A[g]	N/A[g]	N/A[g]	N/A[g]	N/A[g]
T95-SSHTMHNO3-3[h]:	Peak Generation Rate (mmoles/min)	7.0	----[d]	BDL[e]	2.51	f
	Total Off-Gas Generated (mmoles)	261	0.25	BDL[e]	78.5	< 0.17

(a) Measured amounts in the condensate + maximum amount in the slurry.
(b) Alternate HTM Flowsheet 1 is the no reductant, nitric acid process flowsheet.
(c) Major temperature swings and lack of adequate mixing experienced during T95-SSHTMHNO3-1.
(d) Isolated single data points not reliable. Peak generation rate not detectable; minimum detectable peak generation rate is approximately 0.01 mmoles/min.
(e) BDL: Below Detection Limit
(f) Not measured but usually equal to or less than the H_2 generation rate.
(g) N/A: Not Applicable; total off-gas data not available due to test interruption.
(h) The T95-SSHTMHNO3-3 run was most prototypic of the small-scale feed preparation process steps. These data are used for scaling comparisons.

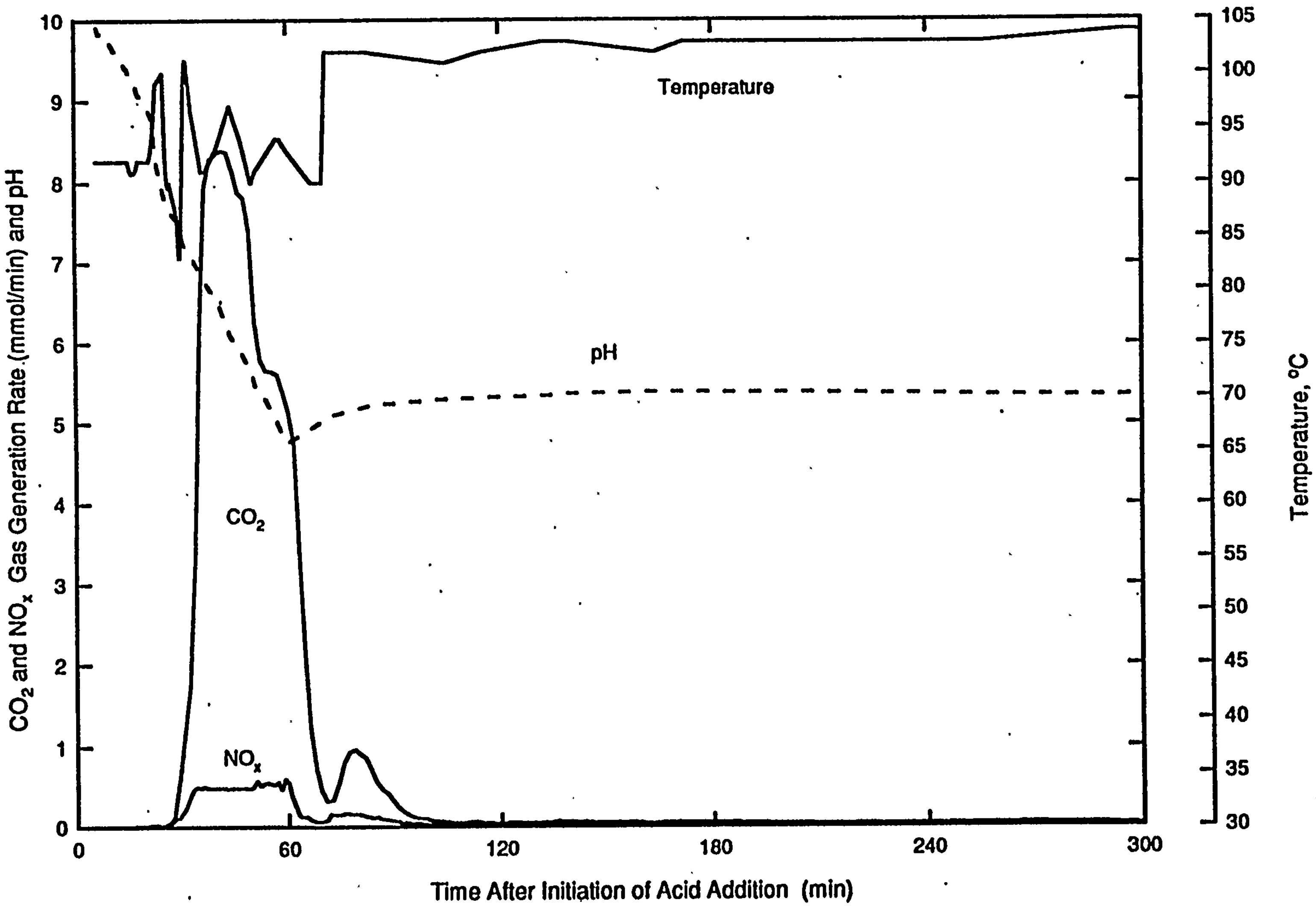


Figure A3.3.1 Process Profile for Laboratory-Scale Alternate HTM Flowsheet 1, Test T95-SSHTMHNO3-1, for the First 300 Minutes Measured from the Start of Nitric Acid Addition. The wide temperature swings during acid addition were due to insufficient stirring action in the slurry.

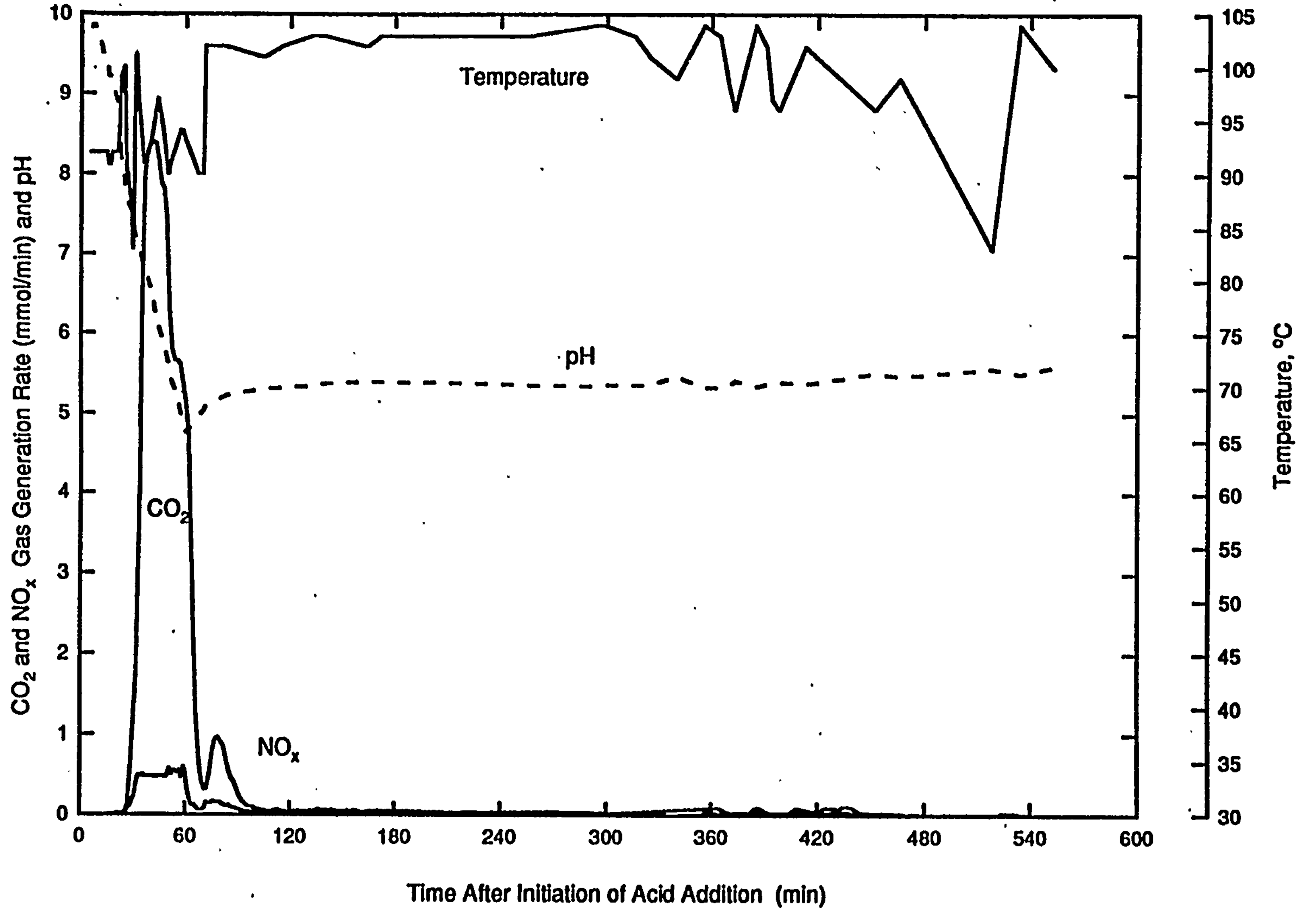


Figure A3.3.2 Process Profile for Laboratory-Scale Alternate HTM Flowsheet 1, Test T95-SSHTMHNO3-1, for 0 to 600 Minutes Measured from the Start of Nitric Acid Addition. The wide temperature swings during acid addition and later during the digestion/concentration period were due to insufficient stirring action in the slurry. The slurry exhibited a tendency to "gel" at temperatures of approximately 100°C when it be came more concentrated as it did toward the end of the digestion/concentration period.

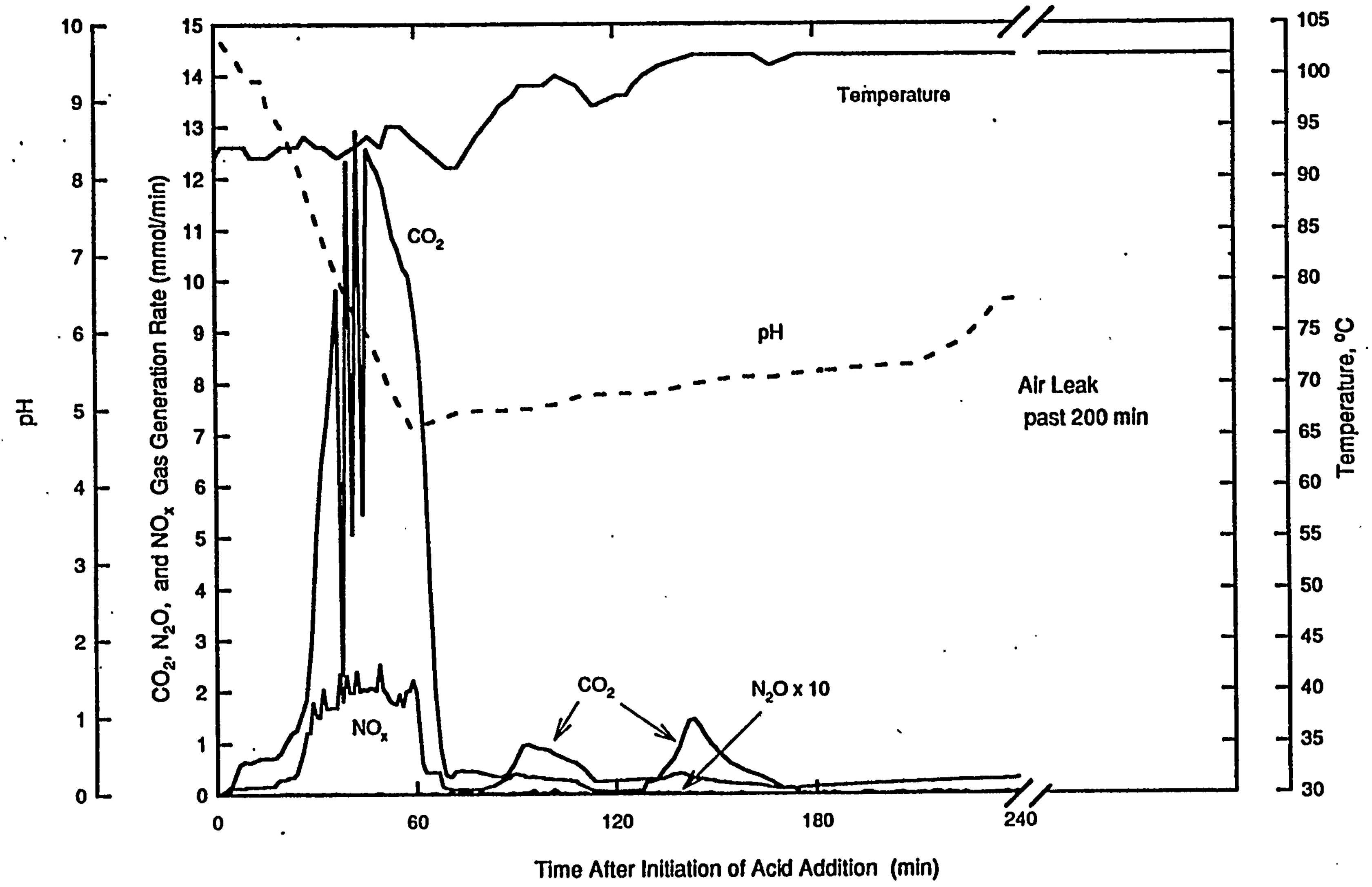


Figure A3.3.3 Process Profile for Laboratory-Scale Alternate HTM Flowsheet 1, Test T95-SSHTMHNO3-2, for the First 240 Minutes Measured from the Start of Nitric Acid Addition. The temperature control was improved by increased stirring action. Irregular CO_2 profile coincides with foaming activity. An air leak due to a broken gas line ruined the off-gas data beyond about 240 minutes.

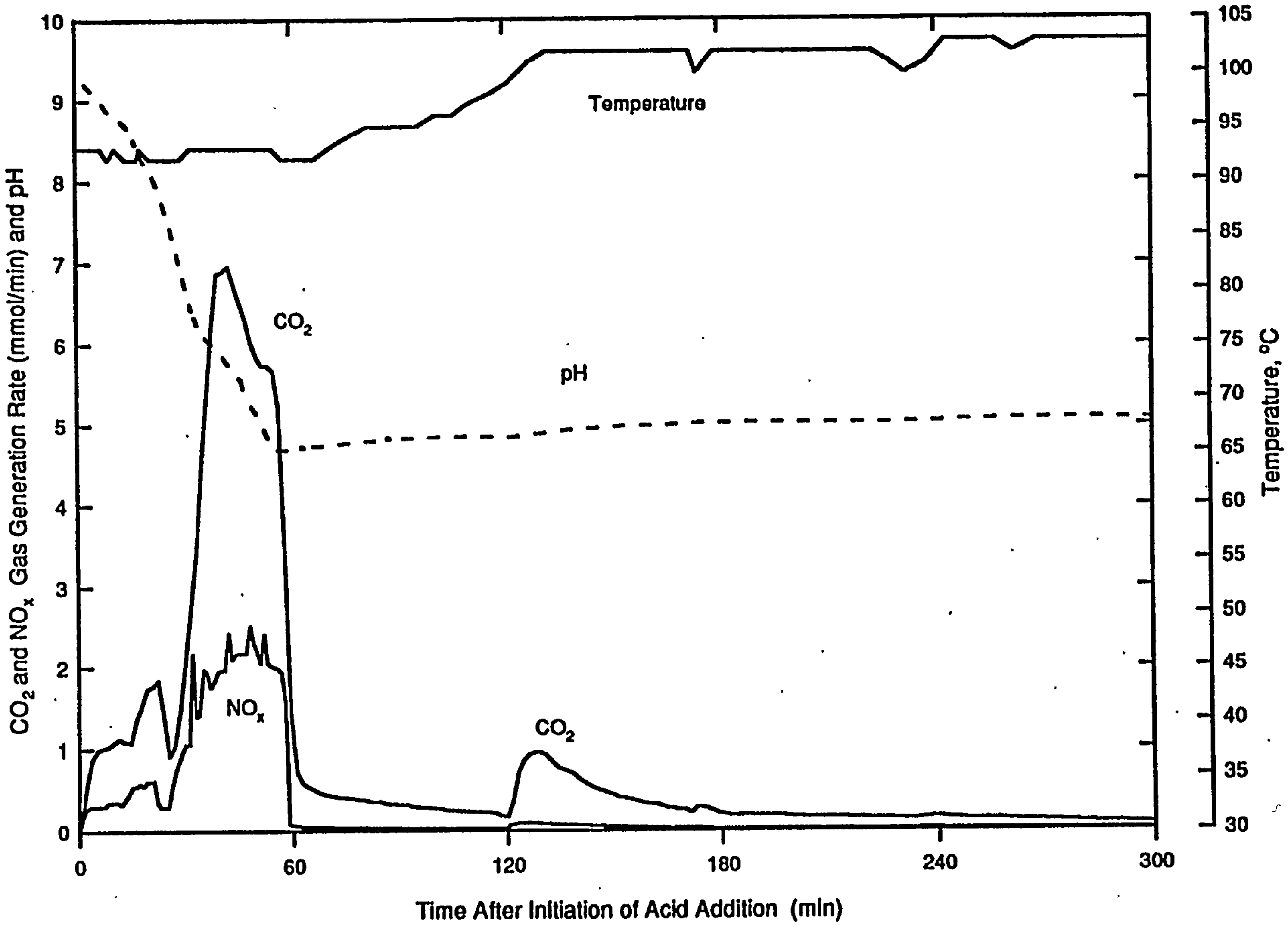


Figure A3.3.4 Process Profile for Laboratory-Scale Alternate HTM Flowsheet 1, Test T95-SSHTMHNO3-3, for the First 300 Minutes Measured from the Start of Nitric Acid Addition. Further improvement of the temperature control was achieved by increasing the stirring action, maximum stirrer setting used.

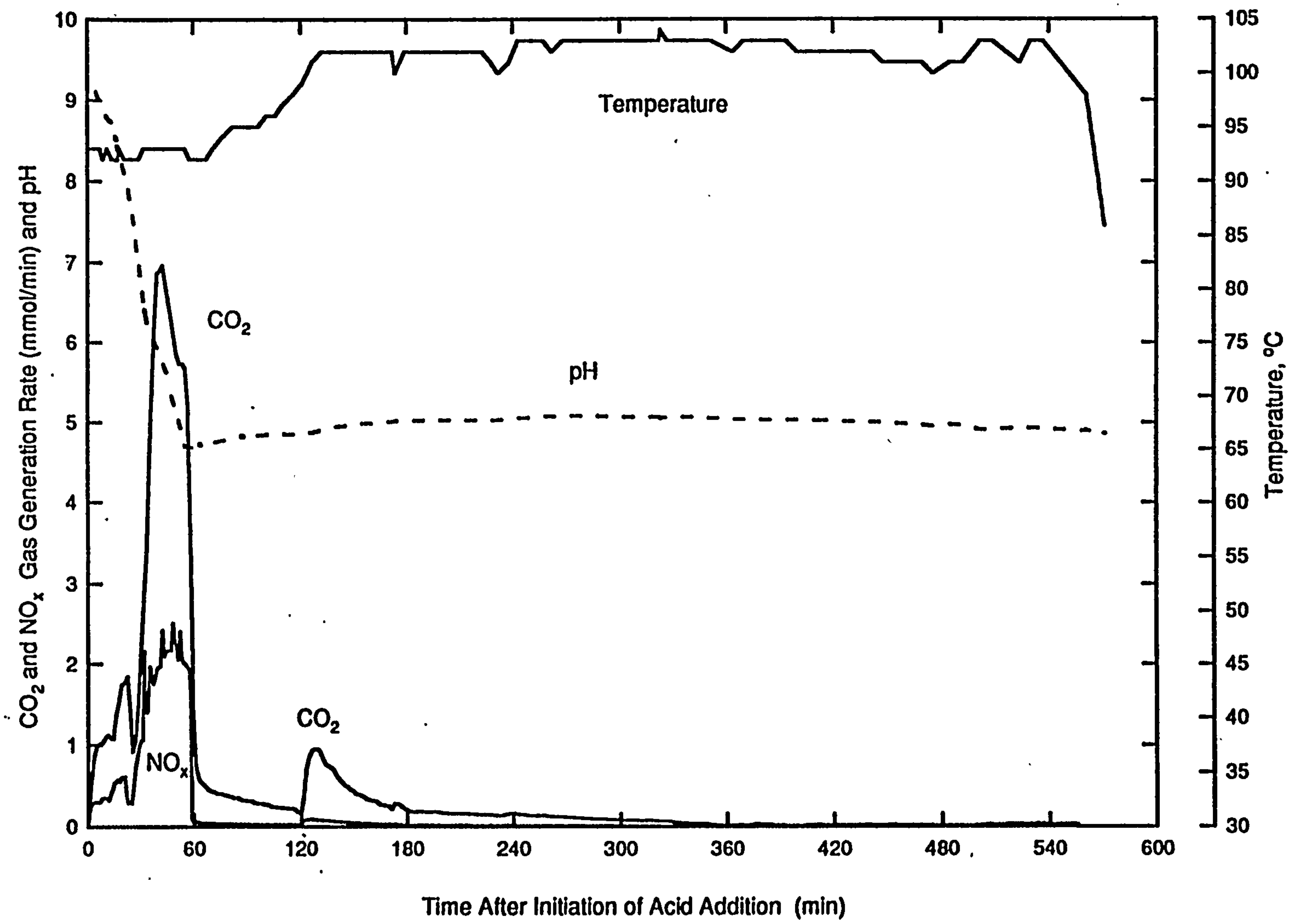


Figure A3.3.5 Process Profile for Laboratory-Scale Alternate HTM Flowsheet 1, Test T95-SSHTMHNO3-3, for 0 to 600 Minutes Measured from the Start of Nitric Acid Addition. Note the much improved temperature control near the end of the last 200 minutes of the test compared to Test T95-SSHTMHNO3-1 (Figure A3.3.2).

3.4 Rheology and Gram Waste Oxide Loading Data for Melter Feed

For Alternate Flowsheet 1, 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 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 and aging 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 diluted before silica addition.

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:

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

where

τ = shear stress,
τ_o = 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 Laboratory-Scale Feed Preparation and Aging Samples

The preliminary waste simulant prepared in the laboratory, at an oxide loading of 253 gTO/L, had a density of 1.22 g/ml, a pH of 7.2, and a solids loading of 36 wt % before silica and sodium nitrate addition. At these conditions the plastic viscosity was 28.1 mPa•s and the yield stress was 13.8 Pa. After silica (F-110) and sodium nitrate addition, the resulting HTM feed had a solids loading of 42 wt %, a density of 1.33 g/ml, a pH of 7.2, a plastic viscosity of 27.8 mPa•s, and a yield stress of 11.2 Pa.

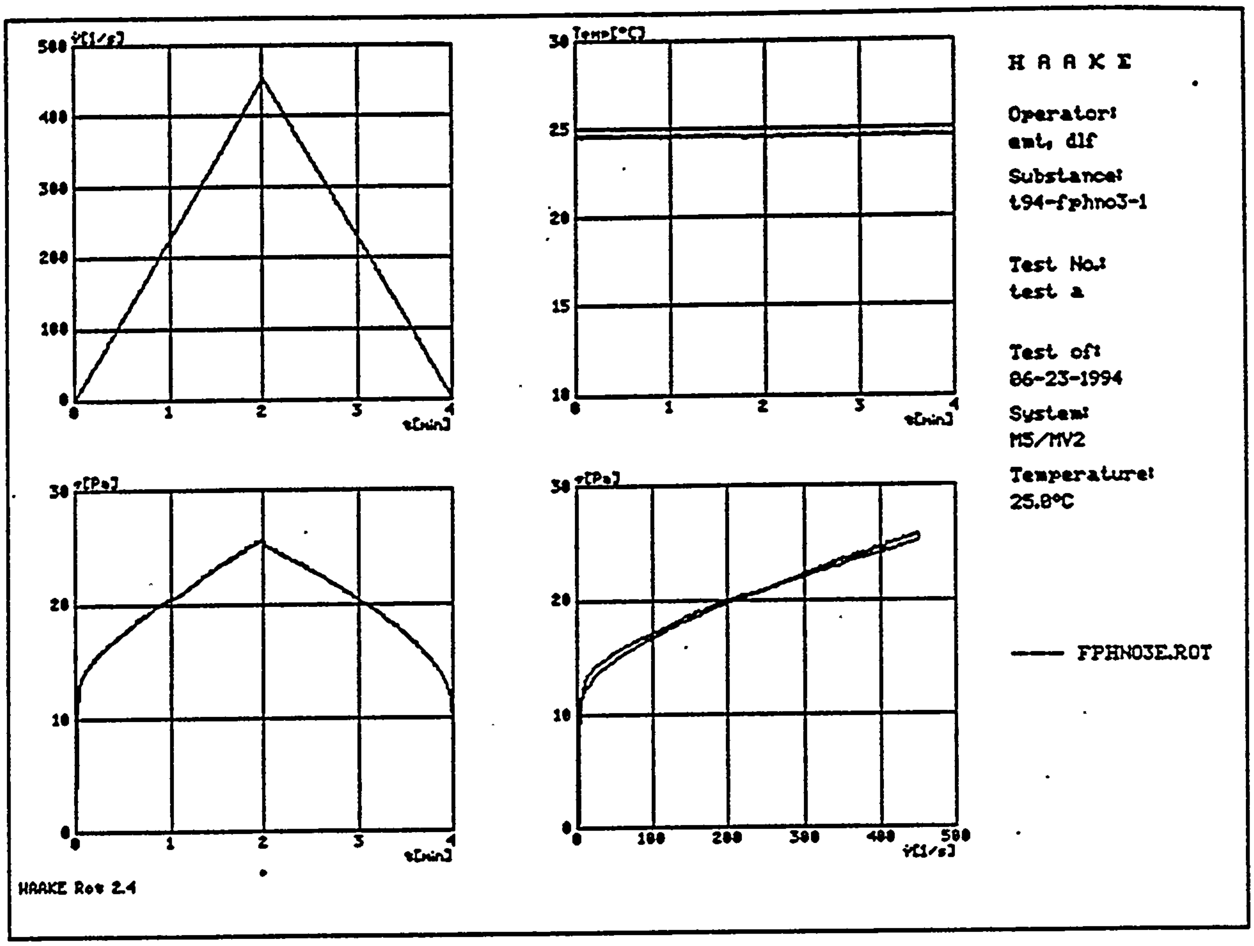
The yield stress increased with sample age and plastic viscosity did not significantly increase with sample age. From day 0 to day 28, yield stress increased from 11.2 Pa to 24.0 Pa (25°C) and plastic viscosity ranged from 27.8 mPa•s to 24.8 mPa•s (25°C). The Bingham model fit the rheological data with correlation coefficients ranging from 0.72 to 0.93. 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 43 ± 1 weight percent except for Day 1 in which the weight percent solids was much lower. The pH increased slightly from 7.2 to 7.7 over the twenty-eight day period. The melter feed prepared with the modified feed preparation procedure was not aged in the laboratory. The summary Table A3.4.1 of all data from the requisite rheograms and each individual rheogram follows.

Table A3.4.1 Physical Property and Rheology Data for Alternate HTM Flowsheet 1, Preliminary Laboratory-Scale Melter Feed Preparation and Aging Samples.

Alternate HTM Flowsheet 1[d]	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]
Preliminary Laboratory-Scale[e]:							
Before silica addition:							
File ID/Date: test a/6-23-94	253	7.2	35.9	1.22	13.8	28.1	0.91
test b/6-23-94					14.0	28.4	0.94
After silica add. & before dilution							
File ID/Date: day 0, test a/6-23-94	----	----	----	----	16.7	37.0	0.91
After silica add. & dilution[f]:							
Day 0:							
File ID/Date: day 0, test b/6-23-94	425	7.2	42.2	1.33	11.2	27.8	0.93
day 0, test c/6-23-94					12.5	25.5	0.93
Day 1:							
File ID/Date: day 1, test a/6-24-94	----	----	39.4	----	13.9	30.9	0.93
day 1, test b/6-24-94	----	----		----	16.6	29.1	0.93
Day 7:							
File ID/Date: day 7, test a/6-30-94	----	7.5	42.7	----	27.4	33.0	0.85
day 7, test b/6-30-94	----			----	30.5	33.8	0.81
Day 14:							
File ID/Date: day 14, test a/7-7-94	----	7.6	42.5	----	23.9	26.8	0.77
day 14, test b/7-7-94	----			----	27.1	25.4	0.76
Day 28:							
File ID/Date: FPHN028A/7-21-94	----	7.7	43.0	----	24.0	24.8	0.72
FPHN028B/7-21-94	----			----	25.2	23.4	0.74

(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 1 is the no reductant, nitric 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.

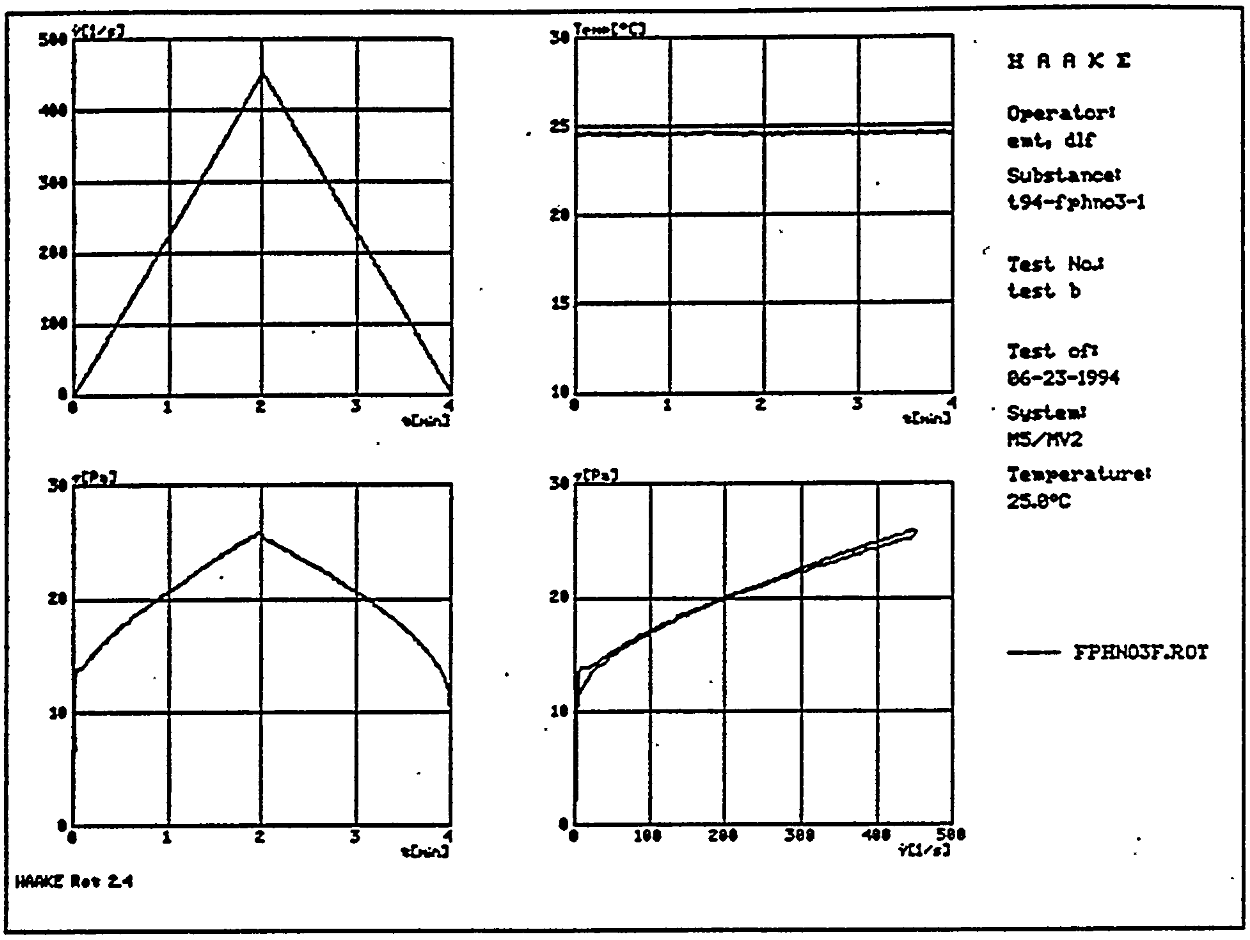
Rheogram and Data for Test Number test a



Bingham : б= бo+Eta*D R»=0.91 бo=13.83 Eta=0.02808
Ostwald : б= a*D■ R»=0.80 a=5.273 n=0.2573
H.-Bulkley : б= бo+a*D■ R»=0.95 a=5.132 n=0.2355 бo=2.656
Casson : «б-«бo = «(Eta*D) R»=0.86 бo=10.26 Eta=0.007947
Linear : Y= a+b*X R»=0.91 a=13.83 b=0.02808
Inv. linear: Y= 1/(a+b*X) R»=0.02 a=1.774 b=-0.005762
Exponential: Y= a*EXP(b*X) R»=0.20 a=11.86 b=0.002073
Hyperbolic : Y= a+b/X R»=0.18 a=20.32 b=-5.907
Logarithmic: Y= a+b*LN(X) R»=0.91 a=2.494 b=3.463

Optimum :
H.-Bulkley : б= бo+a*D■ R»=0.95 a=5.132 n=0.2355 бo=2.656

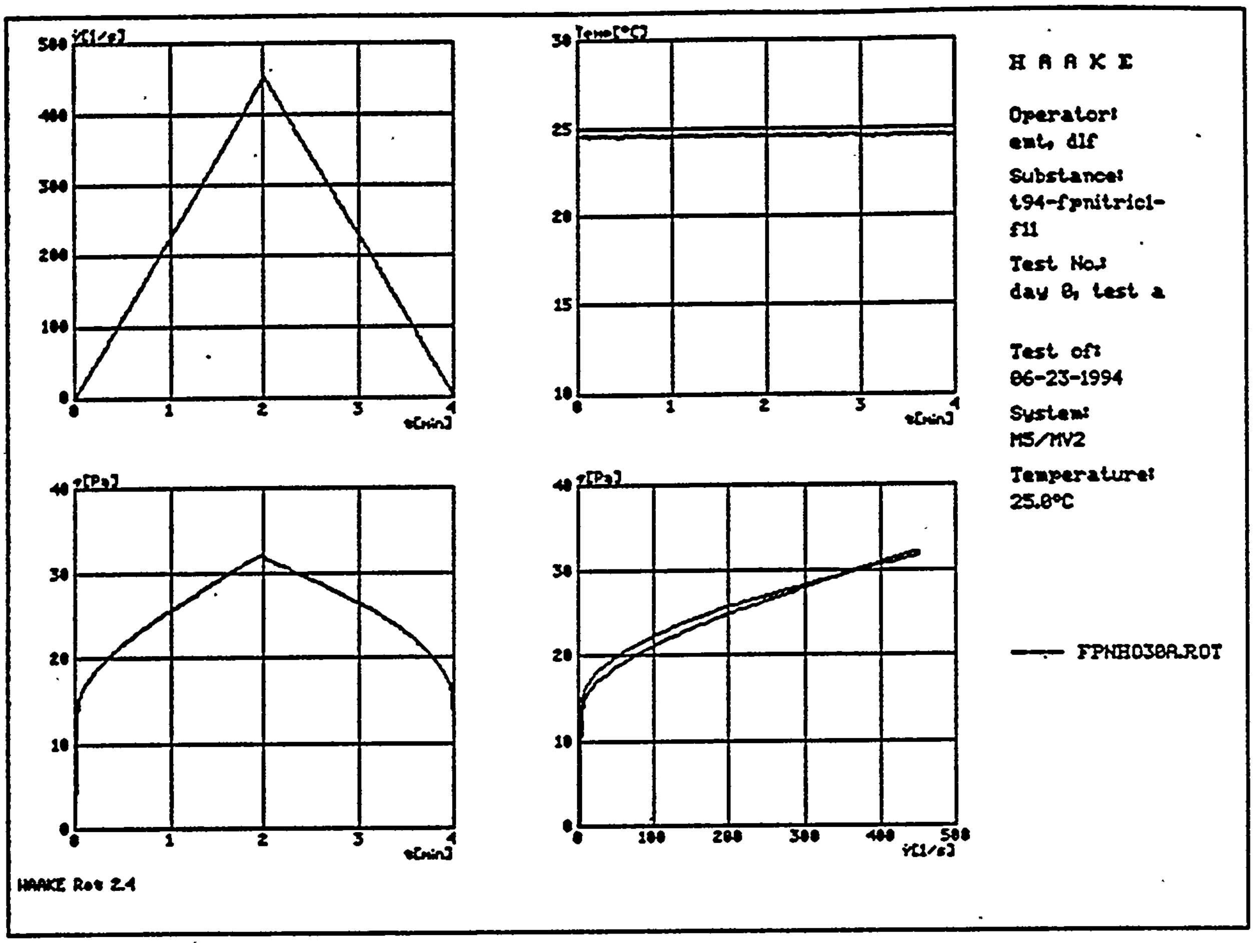
Rheogram and Data for Test Number test b




```
Bingham     : ó= óo+Eta*D      R»=0.94  óo=13.96   Eta=0.02836
Ostwald     : ó= a*D■          R»=0.91  a=6.909   n=0.2076
H.-Bulkley  : ó= óo+a*D■       R»=0.94  a=3.512   n=0.2739   óo=5.628
Casson      : «ó-«óo = «(Eta*D)  R»=0.93  óo=10.66   Eta=0.007474
Linear      : Y= a+b*X         R»=0.94  a=13.96   b=0.02836
Inv. linear: Y= 1/(a+b*X)      R»=0.12  a=0.08488   b=-0.000132
Exponential: Y= a*EXP(b*X)     R»=0.54  a=13.54   b=0.001661
Hyperbolic : Y= a+b/X          R»=0.15  a=20.50   b=-5.267
Logarithmic: Y= a+b*LN(X)      R»=0.87  a=3.224   b=3.358

Optimum :
Bingham     : ó= óo+Eta*D      R»=0.94  óo=13.96   Eta=0.02836
```

Rheogram and Data for Test Number day 0, test a

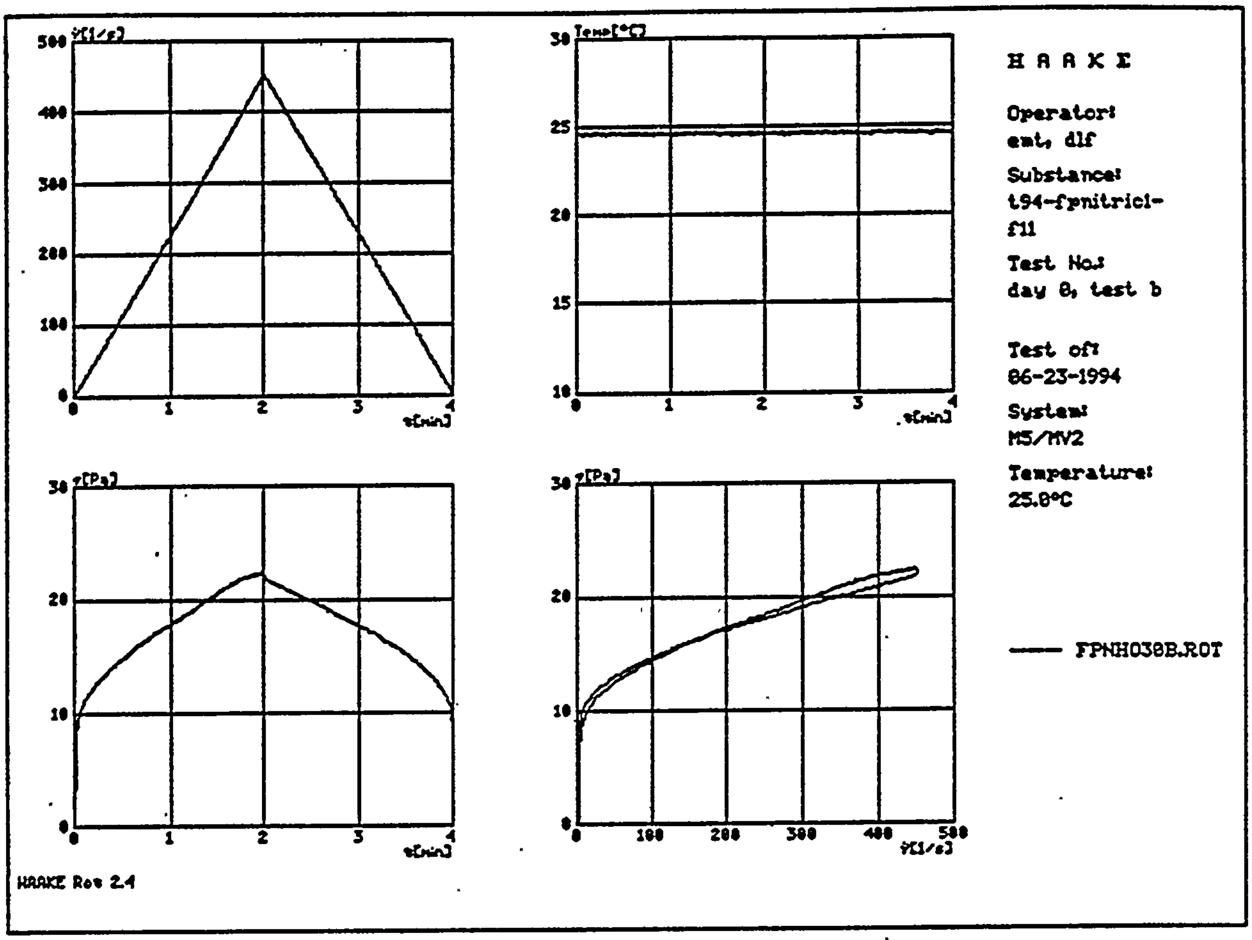


Bingham :	ó= óo+Eta*D	R»=0.91	óo=16.74	Eta=0.03695	
Ostwald :	ó= a*D■	R»=0.77	a=5.642	n=0.2858	
H.-Bulkley :	ó= óo+a*D■	R»=0.96	a=6.195	n=0.2473	óo=2.579
Casson :	«ó-«óo = «(Eta*D)	R»=0.86	óo=12.05	Eta=0.01128	
Linear :	Y= a+b*X	R»=0.91	a=16.74	b=0.03695	
Inv. linear:	Y= 1/(a+b*X)	R»=0.02	a=0.9774	b=-0.003136	
Exponential:	Y= a*EXP(b*X)	R»=0.23	a=14.37	b=0.002176	
Hyperbolic :	Y= a+b/X	R»=0.18	a=25.31	b=-9.273	
Logarithmic:	Y= a+b*LN(X)	R»=0.92	a=1.630	b=4.595	

Optimum :

H.-Bulkley :	ó= óo+a*D■	R»=0.96	a=6.195	n=0.2473	óo=2.579

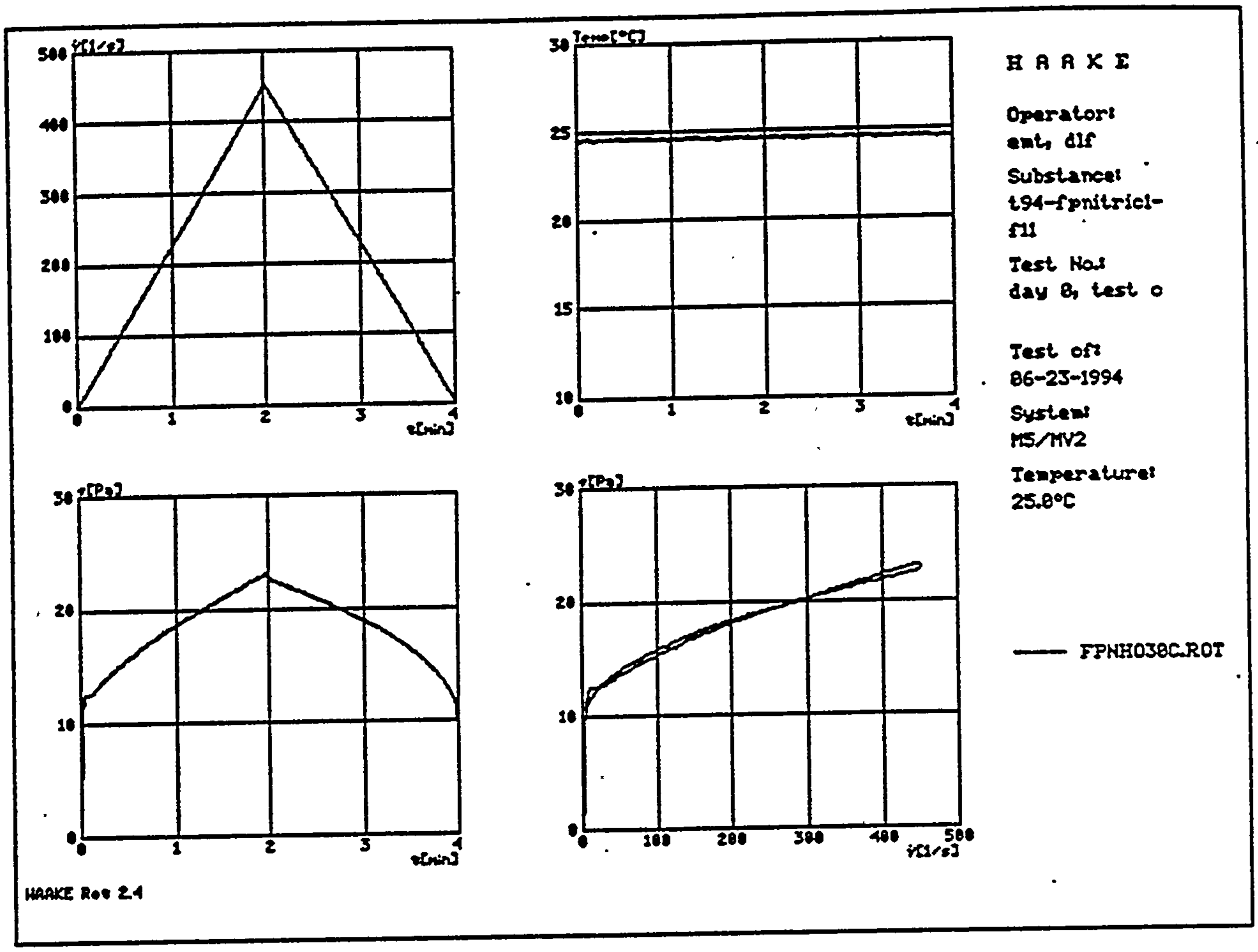
Rheogram and Data for Test Number day 0, test b




```
Bingham      : ő= őo+Eta*D      R»=0.93  őo=11.15  Eta=0.0278
Ostwald      : ő= a*D■          R»=0.85  a=3.726   n=0.2953
H.-Bulkley   : ő= őo+a*D■       R»=0.98  a=3.433   n=0.2832  őo=2.320
Casson       : «ő-«őo = «(Eta*D)  R»=0.90  őo=7.772  Eta=0.009169
Linear       : Y= a+b*X         R»=0.93  a=11.15   b=0.0278
Inv. linear: Y= 1/(a+b*X)       R»=0.05  a=0.1236  b=-237.3E-06
Exponential: Y= a*EXP(b*X)      R»=0.52  a=11.01   b=0.001872
Hyperbolic   : Y= a+b/X         R»=0.14  a=17.55   b=-4.570
Logarithmic: Y= a+b*LN(X)       R»=0.91  a=0.1184  b=3.392

Optimum :
H.-Bulkley   : ő= őo+a*D■       R»=0.98  a=3.433   n=0.2832  őo=2.320
```

Rheogram and Data for Test Number day 0, test c

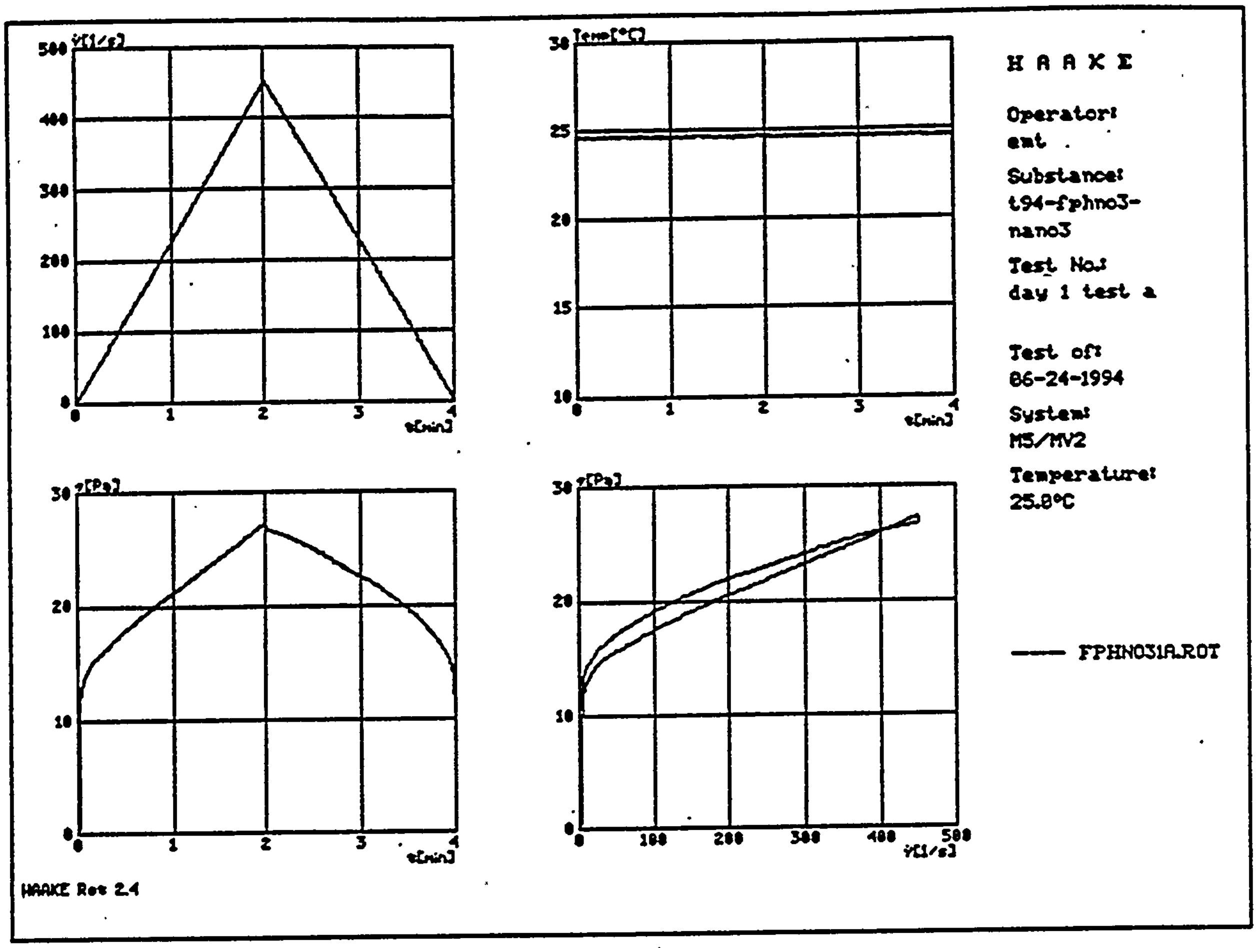


Bingham :	ó= óo+Eta*D	R»=0.93	óo=12.46	Eta=0.02551	
Ostwald :	ó= a*D■	R»=0.91	a=5.864	n=0.2177	
H.-Bulkley :	ó= óo+a*D■	R»=0.94	a=3.591	n=0.2616	óo=4.120
Casson :	«ó-«óo = «(Eta*D)	R»=0.92	óo=9.419	Eta=0.006913	
Linear :	Y= a+b*X	R»=0.93	a=12.46	b=0.02551	
Inv. linear:	Y= 1/(a+b*X)	R»=0.08	a=0.1029	b=-174.6E-06	
Exponential:	Y= a*EXP(b*X)	R»=0.49	a=11.95	b=0.00171	
Hyperbolic :	Y= a+b/X	R»=0.15	a=18.34	b=-4.878	
Logarithmic:	Y= a+b*LN(X)	R»=0.88	a=2.582	b=3.064	

Optimum :

H.-Bulkley :	ó= óo+a*D■	R»=0.94	a=3.591	n=0.2616	óo=4.120

Rheogram and Data for Test Number day 1 test a

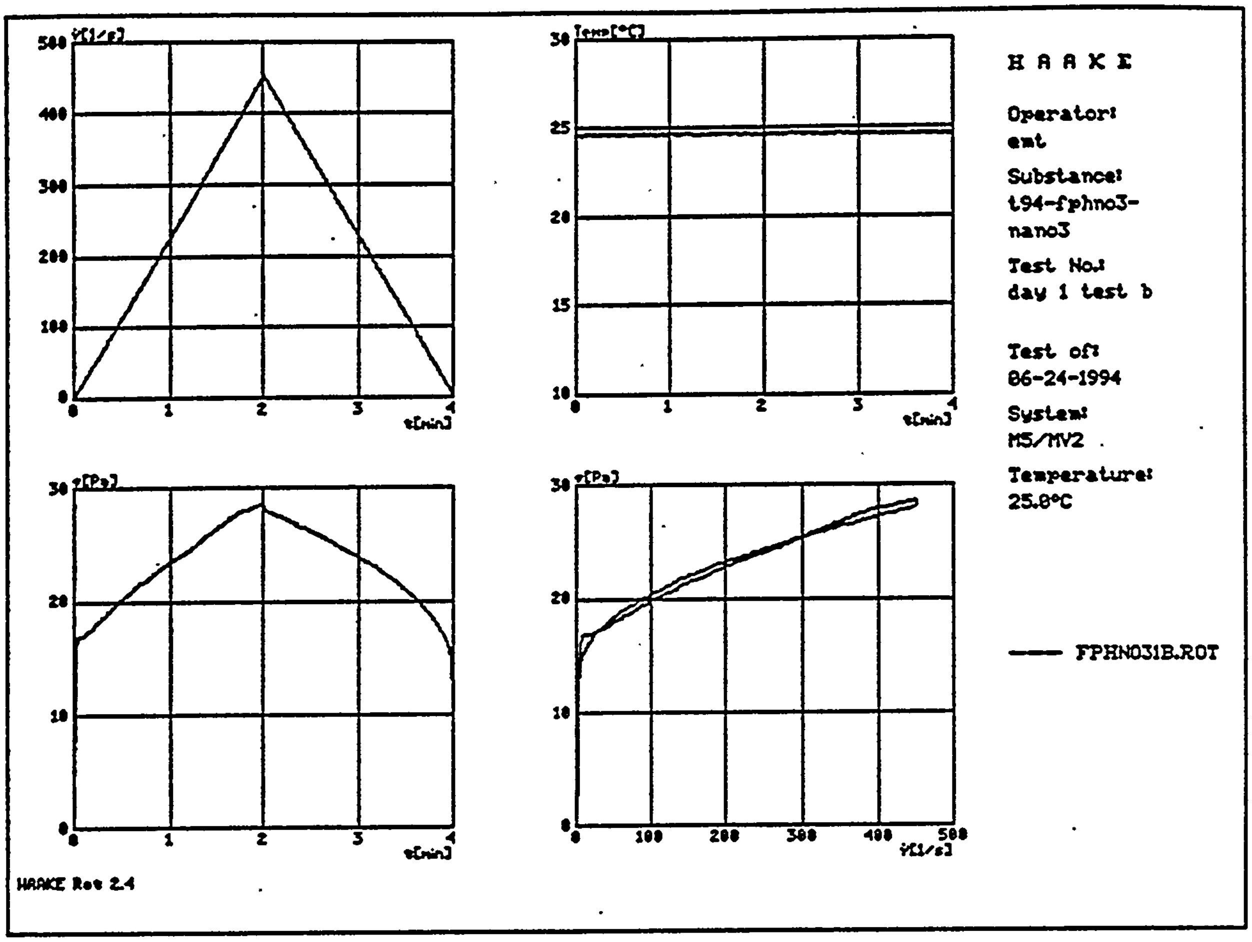


Bingham	:	ó= óo+Eta*D	R»=0.93	óo=13.92	Eta=0.03094	
Ostwald	:	ó= a*D■	R»=0.83	a=5.597	n=0.2525	
H.-Bulkley	:	ó= óo+a*D■	R»=0.95	a=4.580	n=0.2562	óo=3.440
Casson	:	«ó-«óo = «(Eta*D)	R»=0.89	óo=10.24	Eta=0.009016	
Linear	:	Y= a+b*X	R»=0.93	a=13.92	b=0.03094	
Inv. linear	:	Y= 1/(a+b*X)	R»=0.08	a=0.08383	b=-0.000133	
Exponential	:	Y= a*EXP(b*X)	R»=0.57	a=13.91	b=0.001674	
Hyperbolic	:	Y= a+b/X	R»=0.19	a=21.12	b=-9.736	
Logarithmic	:	Y= a+b*LN(X)	R»=0.89	a=1.660	b=3.770	

Optimum :

H.-Bulkley	:	ó= óo+a*D■	R»=0.95	a=4.580	n=0.2562	óo=3.440

Rheogram and Data for Test Number day 1 test b

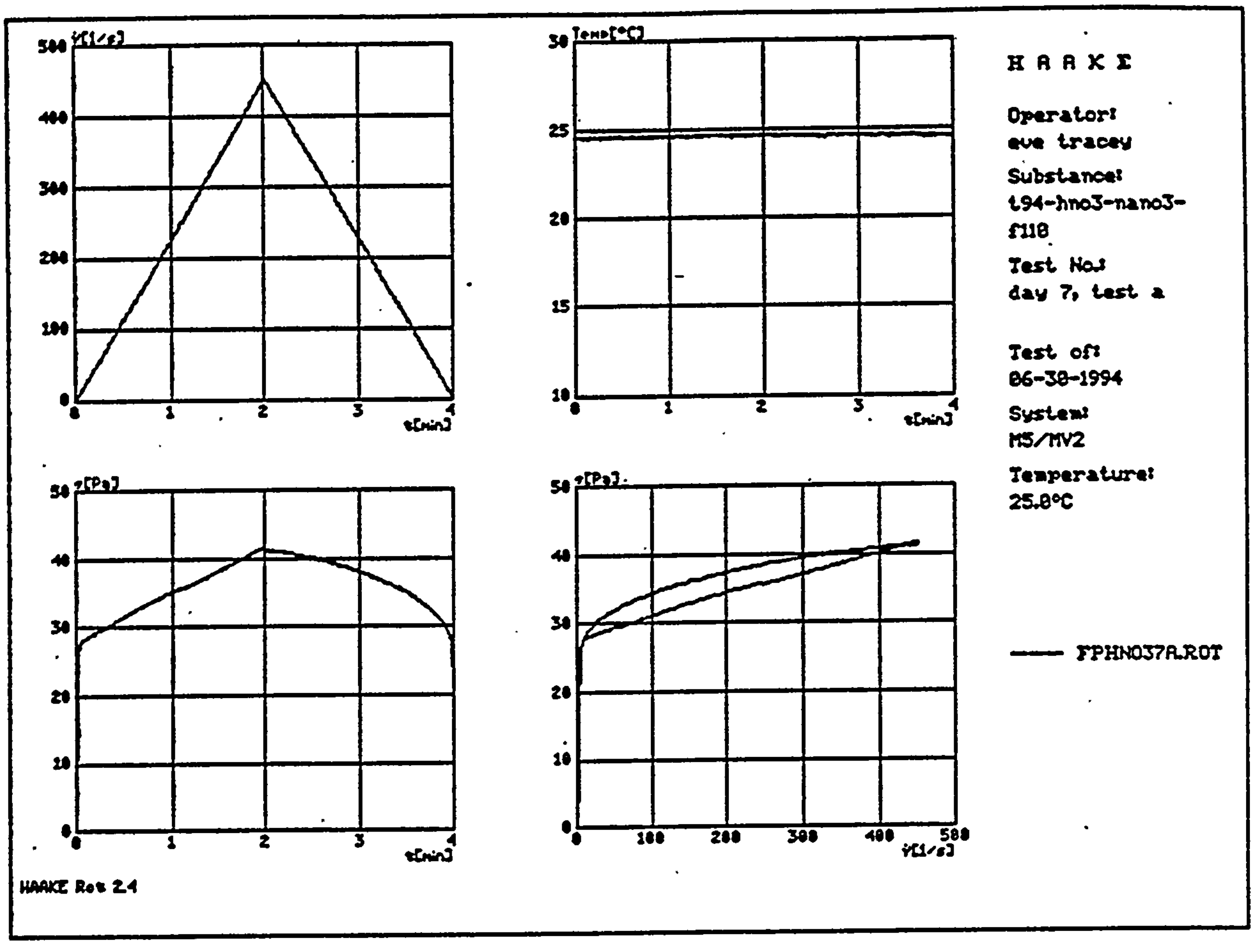


Bingham	: ó= óo+Eta*D	R»=0.93	óo=16.58	Eta=0.02909	
Ostwald	: ó= a*D■	R»=0.88	a=8.637	n=0.1896	
H.-Bulkley	: ó= óo+a*D■	R»=0.92	a=4.714	n=0.2420	óo=6.474
Casson	: «ó-«óo = «(Eta*D)	R»=0.90	óo=13.06	Eta=0.006916	
Linear	: Y= a+b*X	R»=0.93	a=16.58	b=0.02909	
Inv. linear	: Y= 1/(a+b*X)	R»=0.11	a=0.07202	b=-106.6E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.50	a=15.98	b=0.00151	
Hyperbolic	: Y= a+b/X	R»=0.18	a=23.31	b=-6.635	
Logarithmic	: Y= a+b*LN(X)	R»=0.87	a=5.338	b=3.490	

Optimum :

Bingham	: ó= óo+Eta*D	R»=0.93	óo=16.58	Eta=0.02909

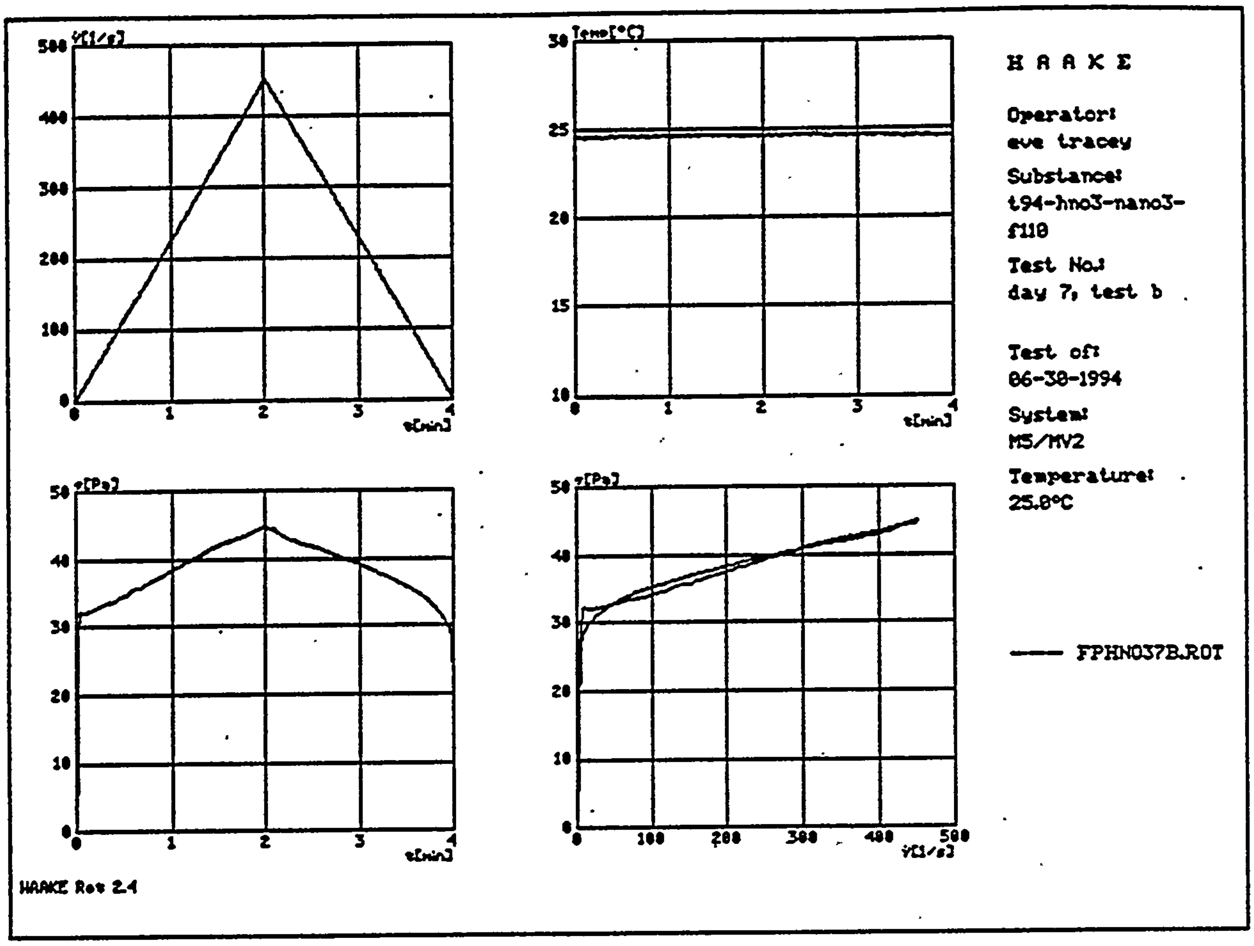
Rheogram and Data for Test Number day 7, test a




```
Bingham      : ó= óo+Eta*D        R»=0.85   óo=27.35   Eta=0.03296
Ostwald      : ó= a*D■            R»=0.80   a=15.39   n=0.1574
H.-Bulkley   : ó= óo+a*D■         R»=0.88   a=10.78   n=0.1726   óo=8.428
Casson       : «ó-«óo = «(Eta*D)  R»=0.79   óo=22.81   Eta=0.006106
Linear       : Y= a+b*X           R»=0.85   a=27.35   b=0.03296
Inv. linear: Y= 1/(a+b*X)         R»=0.09   a=0.04447   b=-58.03E-06
Exponential: Y= a*EXP(b*X)        R»=0.37   a=26.02   b=0.001183
Hyperbolic : Y= a+b/X             R»=0.27   a=35.04   b=-9.481
Logarithmic: Y= a+b*LN(X)         R»=0.89   a=13.46   b=4.181

Optimum :
Logarithmic: Y= a+b*LN(X)         R»=0.89   a=13.46   b=4.181
```

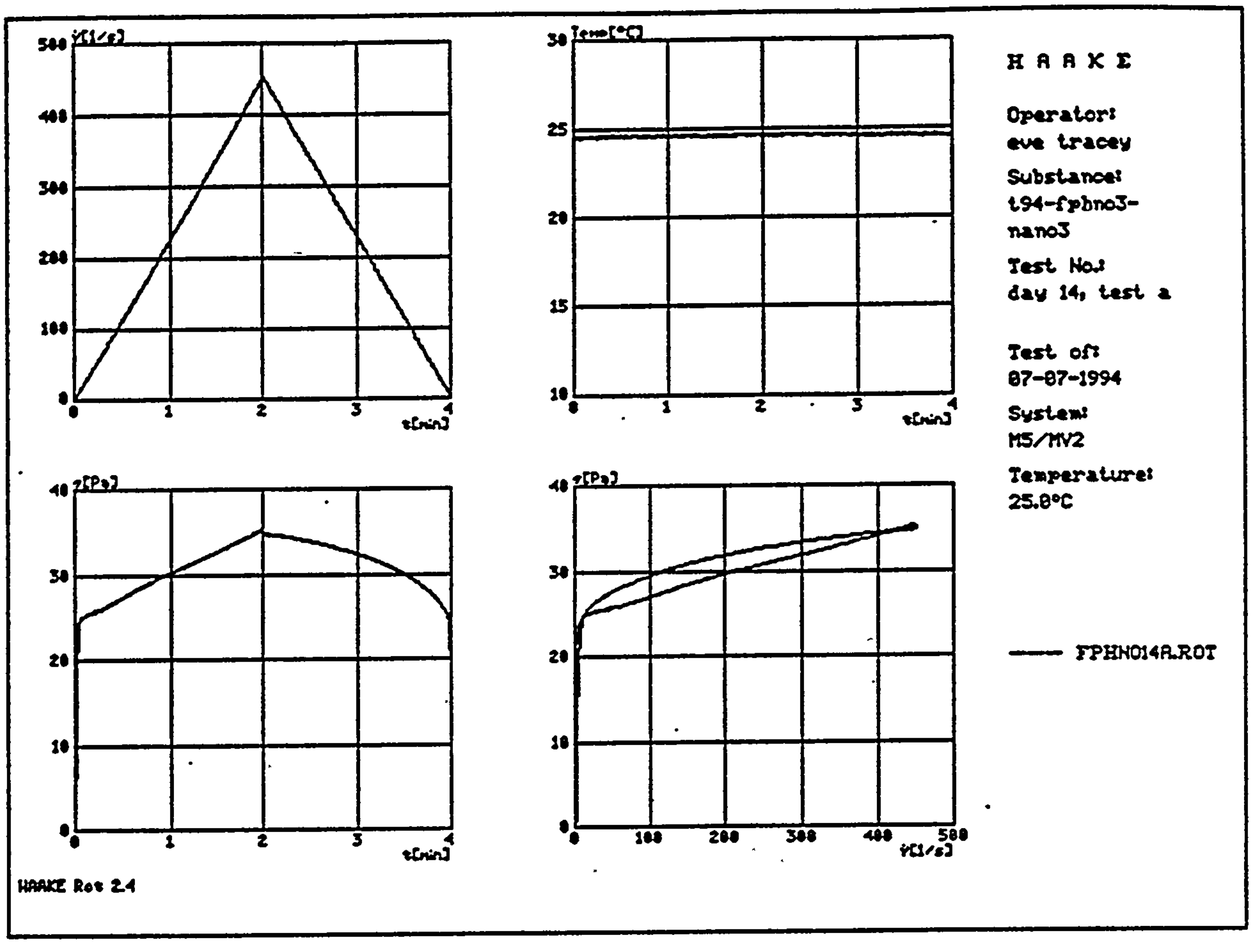
Rheogram and Data for Test Number day 7, test b



Bingham	: ó= óo+Eta*D	R»=0.81	óo=30.52	Eta=0.0338	
Ostwald	: ó= a*D■	R»=0.76	a=17.64	n=0.1488	
H.-Bulkley	: ó= óo+a*D■	R»=0.75	a=12.54	n=0.1674	óo=8.280
Casson	: «ó-«óo = «(Eta*D)	R»=0.75	óo=25.72	Eta=0.005915	
Linear	: Y= a+b*X	R»=0.81	a=30.52	b=0.0338	
Inv. linear	: Y= 1/(a+b*X)	R»=0.12	a=0.03799	b=-44.10E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.39	a=29.16	b=0.001097	
Hyperbolic	: Y= a+b/X	R»=0.38	a=38.64	b=-24.06	
Logarithmic	: Y= a+b*LN(X)	R»=0.86	a=15.73	b=4.391	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.86 a=15.73 b=4.391

Rheogram and Data for Test Number day 14, test a

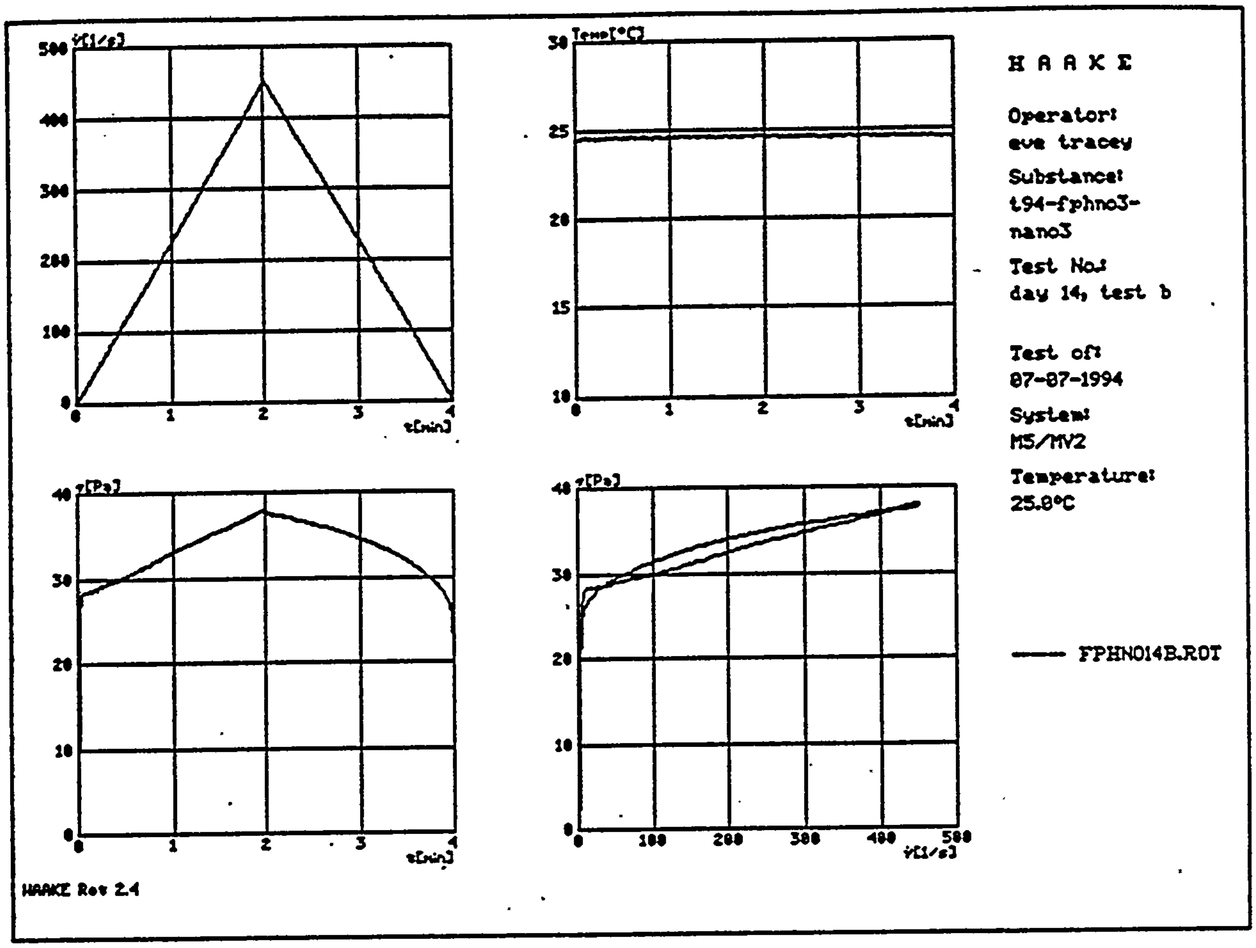


Bingham	: ó= óo+Eta*D	R»=0.77	óo=23.88	Eta=0.02676	
Ostwald	: ó= a*D■	R»=0.61	a=10.76	n=0.1968	
H.-Bulkley	: ó= óo+a*D■	R»=0.85	a=12.70	n=0.1429	óo=3.349
Casson	: «ó-«óo = «(Eta*D)	R»=0.65	óo=19.65	Eta=0.005181	
Linear	: Y= a+b*X	R»=0.77	a=23.88	b=0.02676	
Inv. linear	: Y= 1/(a+b*X)	R»=0.02	a=0.1634	b=-439.5E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.17	a=20.95	b=0.001392	
Hyperbolic	: Y= a+b/X	R»=0.34	a=30.17	b=-8.898	
Logarithmic	: Y= a+b*LN(X)	R»=0.90	a=11.62	b=3.586	

Optimum :

Logarithmic	: Y= a+b*LN(X)	R»=0.90	a=11.62	b=3.586

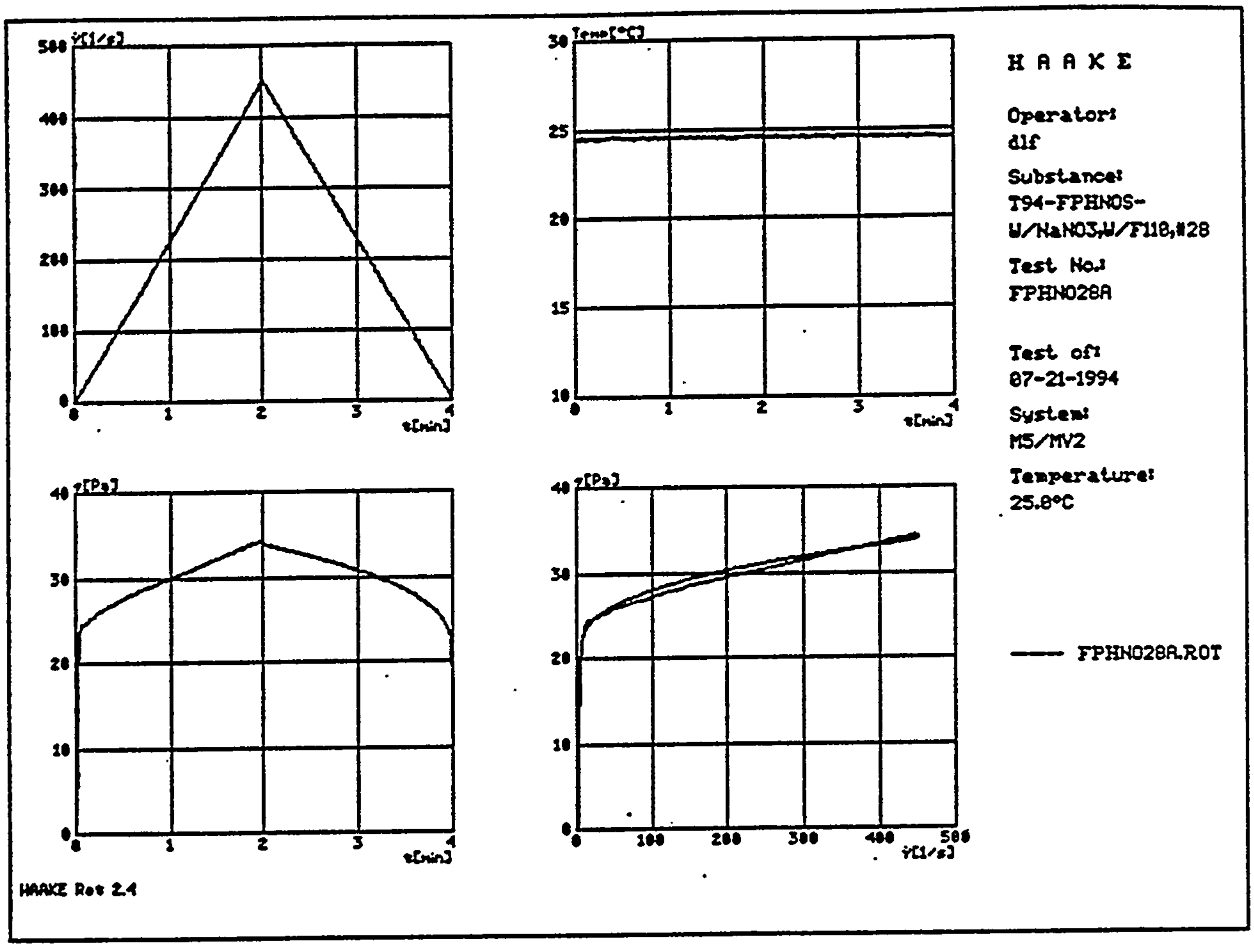
Rheogram and Data for Test Number day 14, test b




```
Bingham     : ό= όo+Eta*D       R»=0.76  όo=27.13  Eta=0.02543
Ostwald     : ό= a*D■           R»=0.67  a=15.35  n=0.1470
H.-Bulkley  : ό= όo+a*D■        R»=0.83  a=12.83  n=0.1328  όo=7.418
Casson      : «ό-«όo = «(Eta*D)  R»=0.65  όo=23.24  Eta=0.004033
Linear      : Y= a+b*X          R»=0.76  a=27.13  b=0.02543
Inv. linear: Y= 1/(a+b*X)       R»=0.05  a=0.05175  b=-76.09E-06
Exponential: Y= a*EXP(b*X)      R»=0.24  a=25.25  b=0.001057
Hyperbolic : Y= a+b/X           R»=0.36  a=33.09  b=-7.365
Logarithmic: Y= a+b*LN(X)       R»=0.87  a=15.75  b=3.355

Optimum :
Logarithmic: Y= a+b*LN(X)       R»=0.87  a=15.75  b=3.355
```

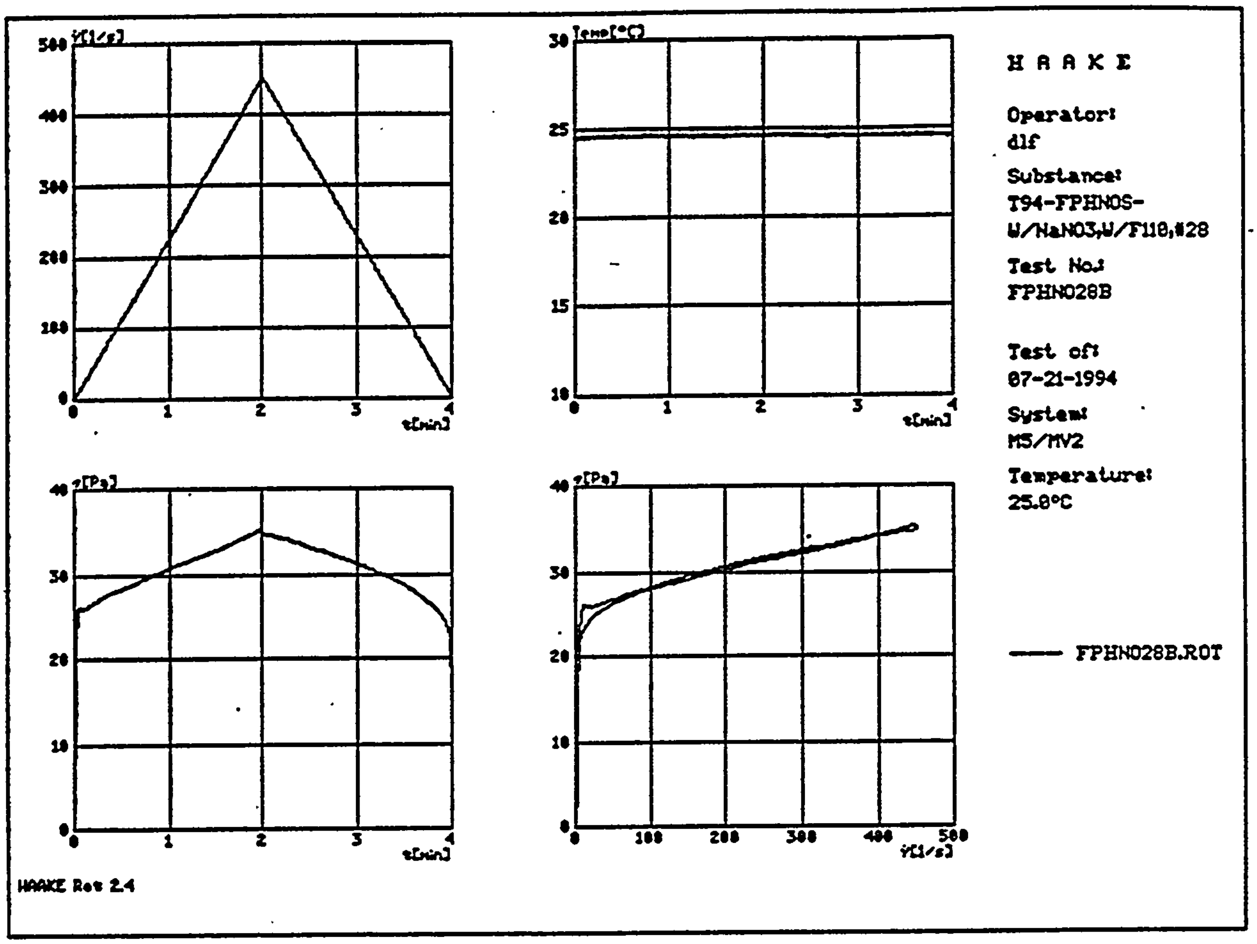
Rheogram and Data for Test Number FPHNO28A




```
Bingham      : ő= őo+Eta*D      R»=0.72   őo=24.01   Eta=0.02483
Ostwald      : ő= a*D■          R»=0.57   a=10.34    n=0.2025
H.-Bulkley   : ő= őo+a*D■       R»=0.84   a=13.01    n=0.1400   őo=2.816
Casson       : «ő-«őo = «(Eta*D)   R»=0.60   őo=19.75   Eta=0.004787
Linear       : Y= a+b*X         R»=0.72   a=24.01    b=0.02483
Inv. linear: Y= 1/(a+b*X)      R»=0.02   a=2.966    b=-0.009782
Exponential: Y= a*EXP(b*X)     R»=0.10   a=19.56    b=0.001576
Hyperbolic : Y= a+b/X          R»=0.39   a=29.88    b=-10.57
Logarithmic: Y= a+b*LN(X)      R»=0.92   a=11.81    b=3.489

Optimum :
Logarithmic: Y= a+b*LN(X)      R»=0.92   a=11.81    b=3.489
```

Rheogram and Data for Test Number FPHNO28B



Bingham	: ó= óo+Eta*D	R»=0.74	óo=25.21	Eta=0.0234	
Ostwald	: ó= a*D■	R»=0.68	a=14.12	n=0.1487	
H.-Bulkley	: ó= óo+a*D■	R»=0.84	a=11.92	n=0.1362	óo=6.420
Casson	: «ó-«óo = «(Eta*D)	R»=0.65	óo=21.52	Eta=0.003774	
Linear	: Y= a+b*X	R»=0.74	a=25.21	b=0.0234	
Inv. linear	: Y= 1/(a+b*X)	R»=0.05	a=0.05535	b=-80.55E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.24	a=23.45	b=0.001052	
Hyperbolic	: Y= a+b/X	R»=0.39	a=30.73	b=-8.840	
Logarithmic	: Y= a+b*LN(X)	R»=0.89	a=14.32	b=3.168	

Optimum :

Logarithmic	: Y= a+b*LN(X)	R»=0.89	a=14.32	b=3.168

3.4.2 Rheology of Laboratory-Scale Feed Preparation Samples

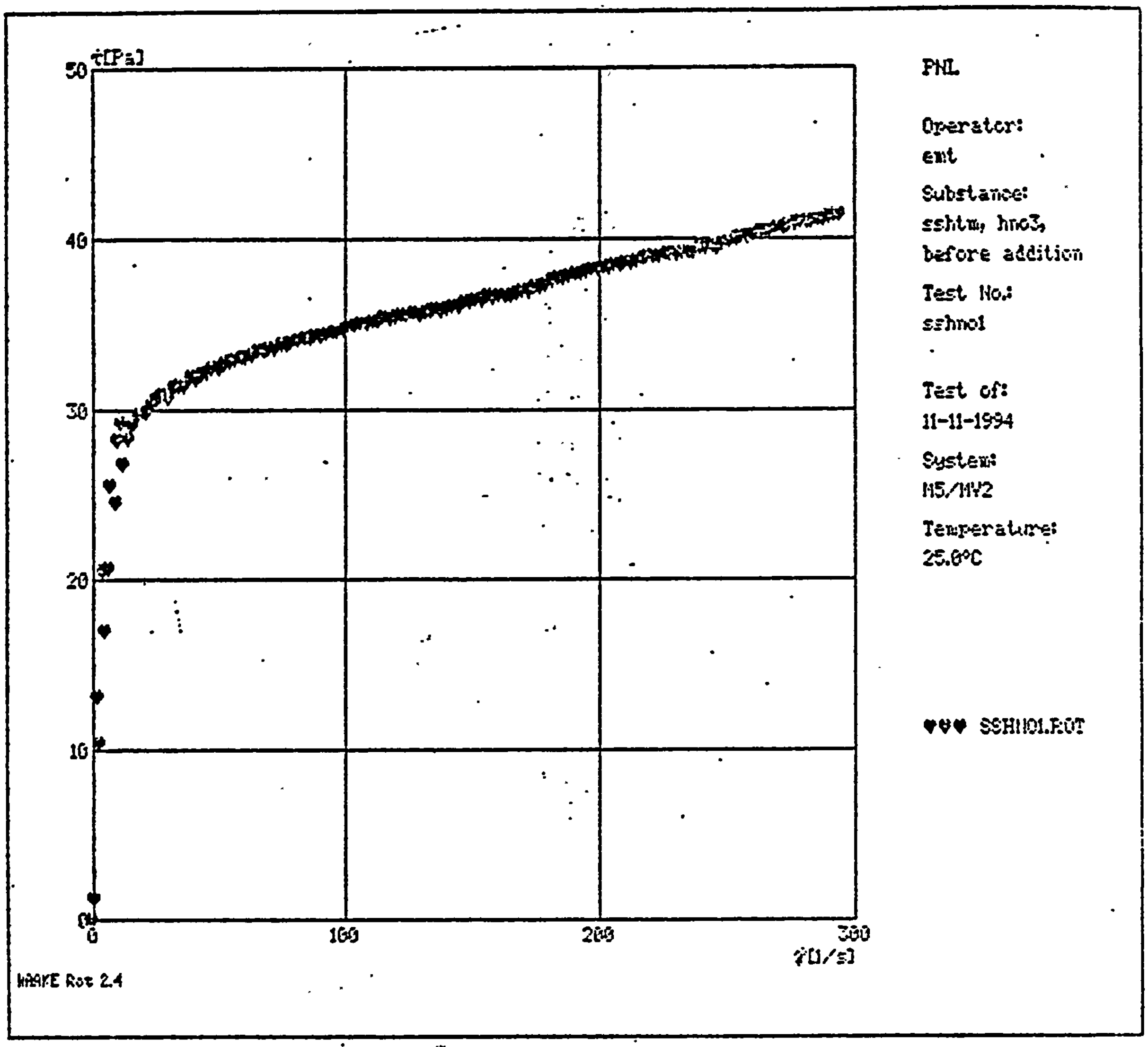
The feed prepared in the laboratory with the modified feed preparation procedure, before silica and sodium nitrate addition and at an oxide loading of 243 gWO/L, had a plastic viscosity of 44.9 mPa•s and a yield stress of 31.5 Pa. (During the small-scale run this is approximately the solids loading when the melter feed began accumulating on the mixer and the heating coils.) The melter feed was diluted to an oxide loading of 190 gWO/L. At this concentration, the wt % solids was 24.8, the pH was 7.4, and the density was 1.11 g/ml. After silica and sodium nitrate addition to the modified feed, the density was 1.23 g/ml, the pH was 7.4 (25°C), the solids loading was 36.6%, and the oxide loading was 339 gTO/L. At these conditions, the plastic viscosity was 17.1 mPa•s and the yield stress was 9.9 Pa (52°C). The summary Table A3.4.2 of all data from the requisite rheograms and each individual rheogram follow.

Table A3.4.2 Physical Property and Rheology Data for Alternate HTM Flowsheet 1, Modified Laboratory-Scale Melter Feed Preparation Samples.

Alternate HTM Flowsheet 1[d]		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]
Modified Laboratory-Scale[e]:								
Before silica addition & dilution:								
File ID/Date:	sshno1/11-11-94	243	----	----	1.27	24.1	70.1	0.53
	sshno2/11-11-94					31.5	44.9	0.71
Before silica addition:								
File ID/Date:	sshno3/11-11-94	190	7.4	24.8	1.11	11.1	15.4	0.82
	sshno4/11-11-94					11.4	15.0	0.88
After silica addition:								
File ID/Date:	sshno5/11-11-94	339	7.4	36.6	1.23	9.9	17.1	0.87
	sshno6/11-11-94					11.1	20.1	0.91

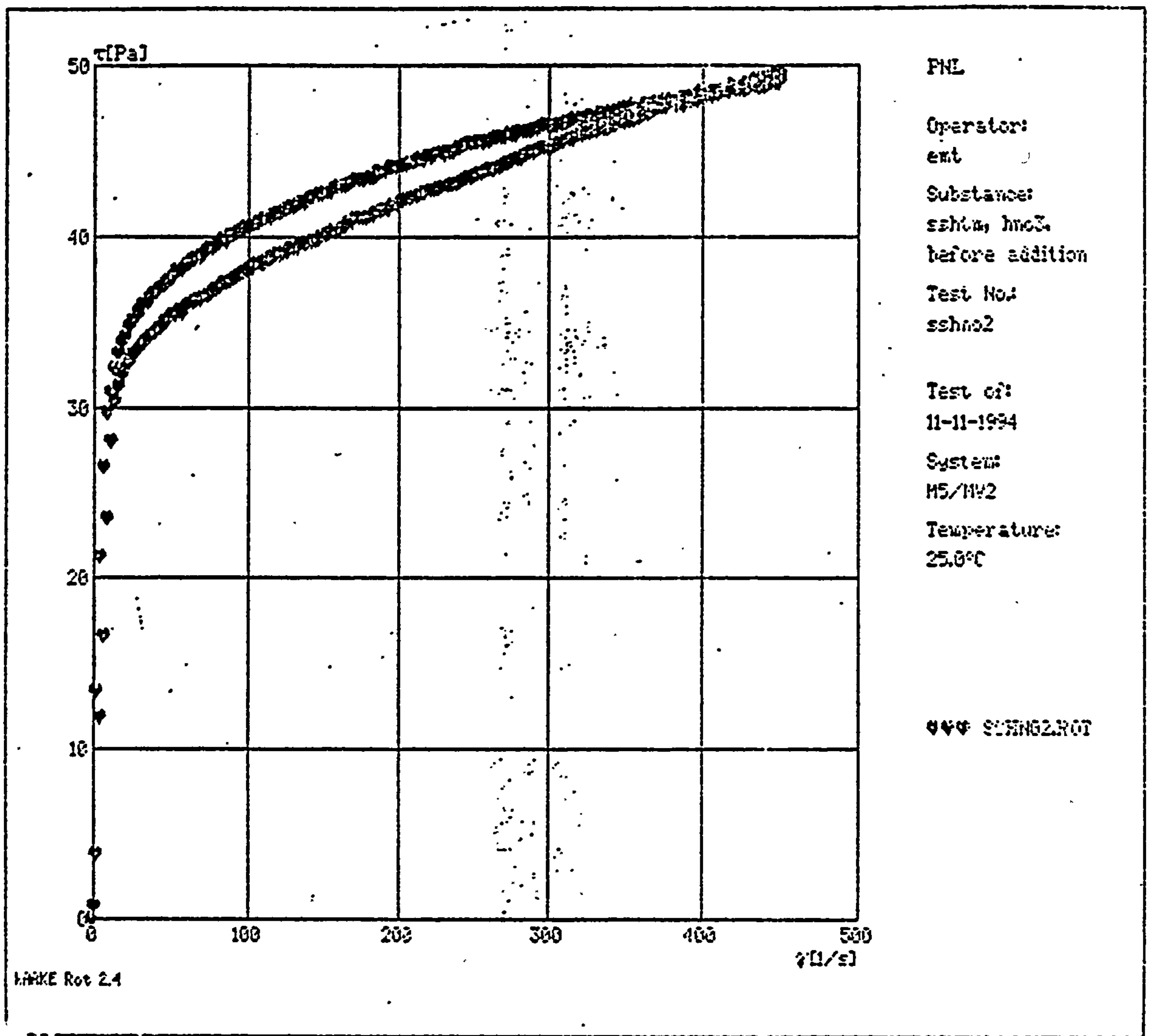
(a) Laboratory rheological screening data value for the yield stress is 15 Pa. The yield stress was calculated by fitting the shear rate sweep data to the Bingham model.
(b) Laboratory rheological screening data value for the plastic viscosity is 25 mPa·s. The plastic viscosity was calculated by fitting 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 1 is the no reductant, nitric acid process flowsheet.
(e) Rheology measurements were taken at 50°C for the modified laboratory-scale feeds.

Rheogram and Data for Test Number sshno1




```
Bingham     : g= go+Eta*D      R}=0.53  go=24.13  Eta=0.07019
Ostwald     : g= a*D/          R}=0.62  a=2.007  n=0.5829
H.-Bulkley  : g= go+a*D/       R}=0.83  a=14.68  n=0.1757  go=1.715
Casson      : {g-{go = {(Eta*D)  R}=0.52  go=17.18  Eta=0.02278
Linear      : Y= a+b*X         R}=0.53  a=24.13  b=0.07019
```

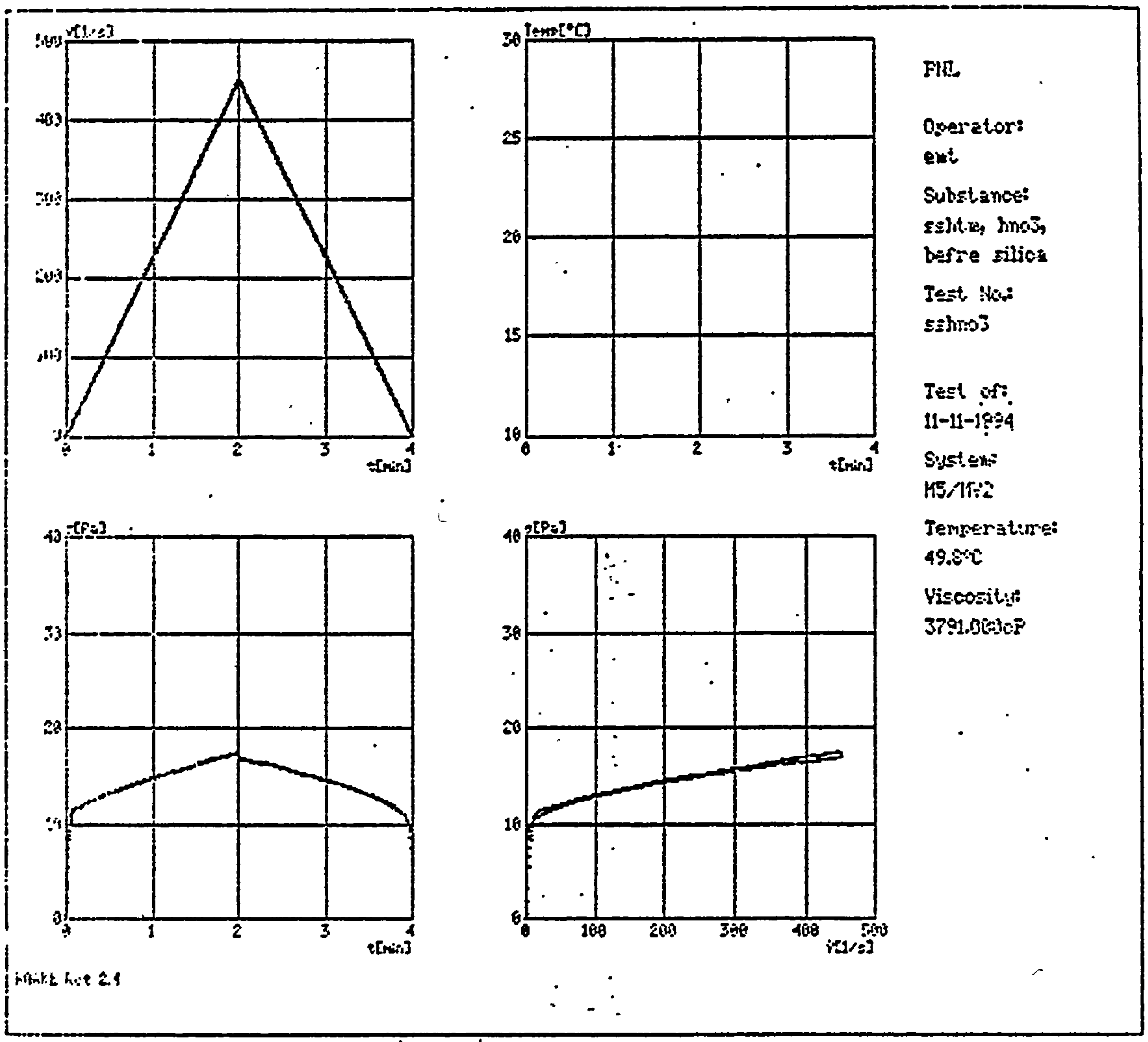
Rheogram and Data for Test Number sshno2




```
Bingham     : g= go+Eta*D      R}=0.71  go=31.54  Eta=0.04492
Ostwald     : g= a*D/          R}=0.64  a=9.415   n=0.2849
H.-Bulkley  : g= go+a*D/       R}=0.74  a=13.39   n=0.2296  go=-2.568
Casson      : {g-{go = {(Eta*D)  R}=0.64  go=23.54  Eta=0.01226
Linear      : Y= a+b*X         R}=0.71  a=31.54   b=0.04492
Inv. linear: Y= 1/(a+b*X)      R}=0.03  a=0.06058  b=-127.2E-06
Exponential: Y= a*EXP(b*X)     R}=0.26  a=29.10   b=0.001443
Hyperbolic  : Y= a+b/X         R}=0.42  a=42.48   b=-41.29
Logarithmic: Y= a+b*LN(X)      R}=0.95  a=8.098   b=6.579

Optimum :
Logarithmic: Y= a+b*LN(X)      R}=0.95  a=8.098   b=6.579
```

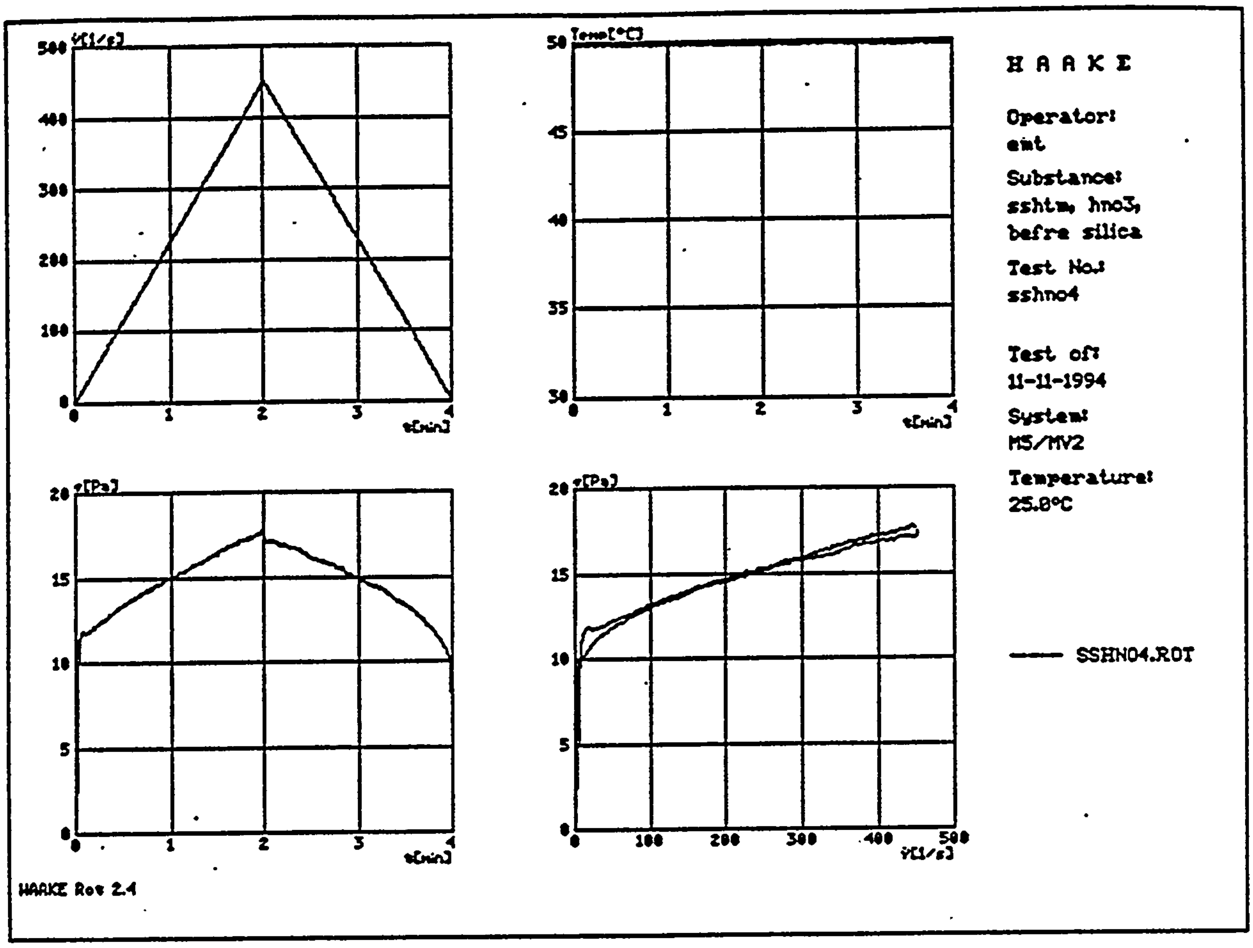
Rheogram and Data for Test Number sshno3




```
Bingham      : g= go+Eta*D       R²=0.82   go=11.05   Eta=0.01538
Ostwald      : g= a*D/           R²=0.71   a=4.572    n=0.2222
H.-Bulkley   : g= go+a*D/        R²=0.93   a=5.434    n=0.1736   go=1.164
Casson       : {g-{go = {(Eta*D)  R²=0.74   go=8.718   Eta=0.003541
Linear       : Y= a+b*X          R²=0.82   a=11.05    b=0.01538
Inv. linear  : Y= 1/(a+b*X)      R²=0.02   a=1.801    b=-0.005788
Exponential  : Y= a*EXP(b*X)     R²=0.15   a=9.356    b=0.001704
Hyperbolic   : Y= a+b/X          R²=0.27   a=14.64    b=-4.785
Logarithmic  : Y= a+b*LN(X)      R²=0.94   a=3.984    b=2.064

Optimum :
Logarithmic  : Y= a+b*LN(X)      R²=0.94   a=3.984    b=2.064
```

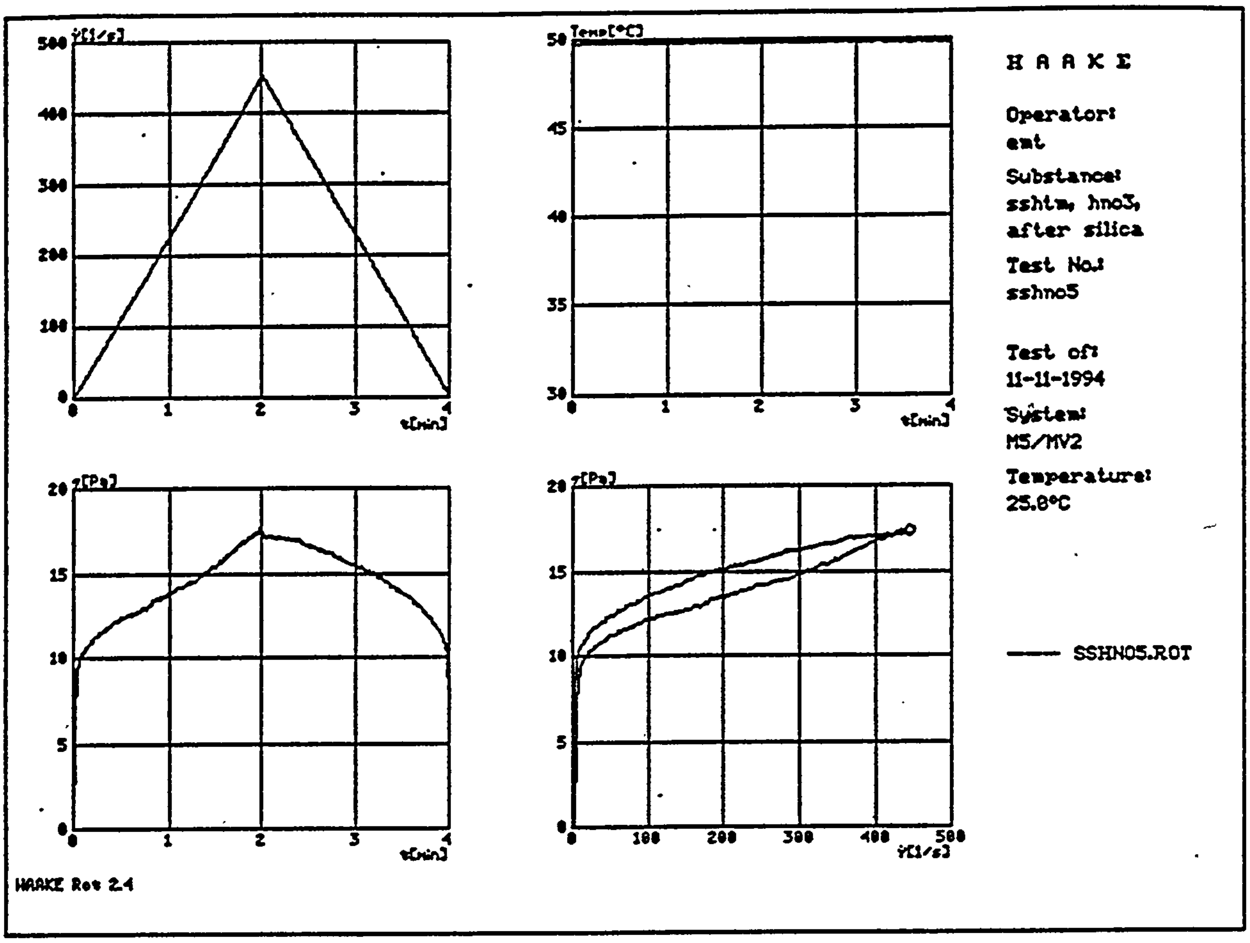
Rheogram and Data for Test Number sshno4




```
Bingham      : ó= óo+Eta*D      R»=0.88  óo=11.44  Eta=0.01503
Ostwald      : ó= a*D■          R»=0.87  a=6.449   n=0.1605
H.-Bulkley   : ó= óo+a*D■       R»=0.91  a=4.227   n=0.1910  óo=3.429
Casson       : «ó-«óo = «(Eta*D)  R»=0.86  óo=9.418  Eta=0.002962
Linear       : Y= a+b*X         R»=0.88  a=11.44   b=0.01503
Inv. linear: : Y= 1/(a+b*X)     R»=0.15  a=0.09825  b=-116.6E-06
Exponential: : Y= a*EXP(b*X)    R»=0.48  a=11.03   b=0.001213
Hyperbolic   : Y= a+b/X         R»=0.31  a=15.03   b=-11.38
Logarithmic: : Y= a+b*LN(X)     R»=0.91  a=4.856   b=1.952

Optimum :
H.-Bulkley   : ó= óo+a*D■       R»=0.91  a=4.227   n=0.1910  óo=3.429
```

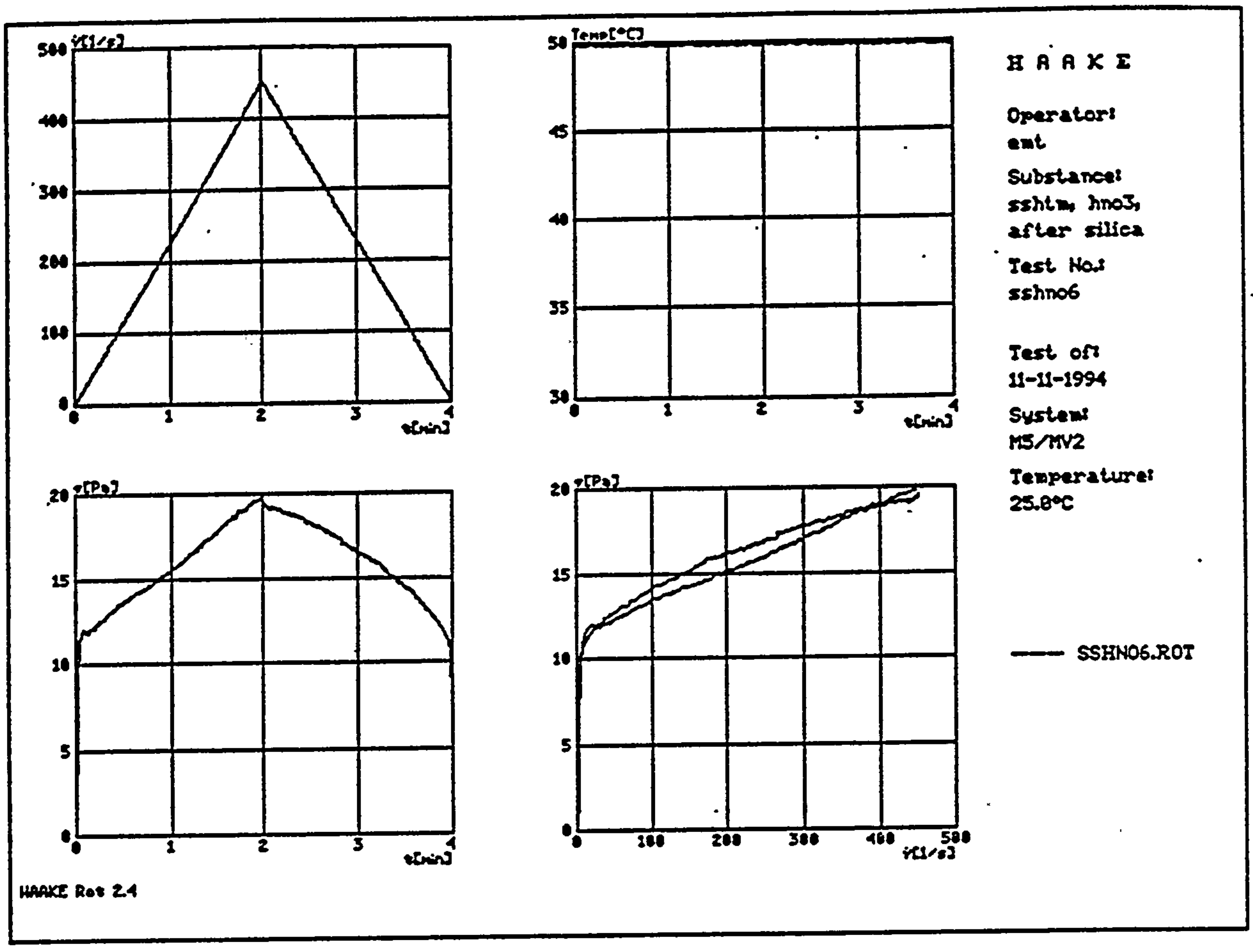
Rheogram and Data for Test Number sshno5




```
Bingham      : ó= óo+Eta*D       R»=0.87   óo=9.921   Eta=0.01713
Ostwald      : ó= a*D■           R»=0.51   a=2.503    n=0.3267
H.-Bulkley   : ó= óo+a*D■        R»=0.92   a=4.785    n=0.1897   óo=0.9747
Casson       : «ó-«óo = «(Eta*D)   R»=0.74   óo=7.532   Eta=0.004484
Linear       : Y= a+b*X          R»=0.87   a=9.921    b=0.01713
Inv. linear: Y= 1/(a+b*X)        R»=0.02   a=0.9083   b=-0.002802
Exponential: Y= a*EXP(b*X)       R»=0.20   a=9.157    b=0.001636
Hyperbolic : Y= a+b/X            R»=0.23   a=13.90    b=-4.590
Logarithmic: Y= a+b*LN(X)        R»=0.90   a=2.716    b=2.169

Optimum :
H.-Bulkley : ó= óo+a*D■          R»=0.92   a=4.785    n=0.1897   óo=0.9747
```

Rheogram and Data for Test Number sshno6




```
Bingham      : τ= τo+Eta*D      R»=0.91  τo=11.05  Eta=0.02009
Ostwald      : τ= a*D■          R»=0.83  a=5.312   n=0.2068
H.-Bulkley   : τ= τo+a*D■       R»=0.88  a=4.342   n=0.2121  τo=2.480
Casson       : «τ-«τo = «(Eta*D)  R»=0.86  τo=8.637  Eta=0.004898
Linear       : Y= a+b*X         R»=0.91  a=11.05   b=0.02009
Inv. linear: Y= 1/(a+b*X)       R»=0.06  a=0.1276  b=-227.1E-06
Exponential: Y= a*EXP(b*X)      R»=0.41  a=10.47   b=0.001605
Hyperbolic  : Y= a+b/X          R»=0.19  a=15.72   b=-5.466
Logarithmic: Y= a+b*LN(X)       R»=0.86  a=3.230   b=2.421

Optimum :
Bingham      : τ= τo+Eta*D      R»=0.91  τo=11.05  Eta=0.02009
```

3.5 Standard Crucible Redox Test Data

Introduction

This section addresses the measurement of a glass oxidation state indicator, the $Fe^{+2}/\Sigma Fe$ ratio for the laboratory-scale feed preparation. One melter feed and two methods of glass preparation are discussed for Alternate HTM Flowsheet 1. 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 $Fe^{+2}/\Sigma Fe$ ratios are given in Table A3.5.

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 glass[1]. 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 glasses[2].

Results

The relative quantities of oxidants are given in Table A3.5 (a reductant was not added during Alternate HTM Flowsheet 1 processing). The $Fe^{+2}/\Sigma Fe$ ratio of glasses prepared from the laboratory-scale melter feeds, using the standard crucible method, was 0.063 (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 A3.5 Standard Crucible Redox Test Data for Laboratory-Scale Melter Feed Alternate HTM Flowsheet 1 Testing.

	Final		Moles Oxidant[a]		Moles Reductant[b]	Glass: $Fe^{+2}/\Sigma Fe$	
	pH (@ ≈22°C)	(gTO/L)	NO_3^-	NO_2^-		Crucible	Melter
Alternate HTM Flowsheet 1[c]							
laboratory melter feed[d]	7.4	339	1.14	0.41	N/A	N/A	N/A
dried melter feed/glass	N/A	N/A	----	----	N/A	0.0633	N/A
	N/A	N/A	----	----	N/A	0.0627	N/A

(a) Reported moles of NO_3^- are after addition of $NaNO_3$. The $NaNO_3$ contributed 0.47 moles of nitrate per 125 gWO.
(b) No reductant added for Alternate HTM Flowsheet 1.
(c) Alternate HTM Flowsheet 1 is the no reductant, nitric acid process flowsheet; laboratory data from test T95-SSHTMHNO3-1.
(d) Laboratory melter feed made from the T95-SSHTMHNO3-1 test slurry on Nov. 11, 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 nitric acid (70 wt%) via a peristaltic pump to the simulant at successively greater rates as done during the small-scale feed preparation. Nitric 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 (°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 "HNO3 Addition" column provides the weight of nitric 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 A4.1, for the small-scale nitric acid processed melter feed follows.

Log A4.1

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/12/94							
15:30	-	-	-	-	-	-	Added 10 drums of Optima mixed hydroxide slurry to HB-32. Total of 2993 kg added. Tank volume ~2040 L. This is the start of flowsheet 1 of SSHTM1 Feed Preparation Test (second batch). The ten drums of Optima mixed hydroxide slurry are: 6307, 6304, 6305, 6306, 6326, 5325, 6327, 6324, 6478, and 6480.
15:40	-	-	-	-	-	-	Mixing well.
16:30	-	-	-	-	-	-	Added 82 kg of NaNO2 and 19 kg of NaNO3 to tank HB-32.
16:38	-	-	-	-	-	SSHTM1-6.2.6-1 SSHTM1-6.2.6-2	Removed two pre NaNO2 slurry samples. Sample amount 30 L submitted to Dave Lamar for analysis. Sample amount 250 ml submitted for analysis.
8/13/94							
8:10	-	-	-	-	-	SSHTM1-6.3.8-1	Removed one 250 ml post NaNO2 slurry sample for analysis.
8:15	-	-	-	-	-	-	Steam turned on to HB-32, PIC-2001 steam control valve open to 15% output.
8:40	-	-	-	-	-	-	Increased steam to 20% output.
9:50	-	-	-	-	-	-	Increased steam to 30% output.

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/13/94 1420	-	99.9	-	-	-	-	Adding a total of three barrels to HB-32 to replace condensate from initial slurry. Addition of first drum #6475 of mixed hydroxide slurry. This addition caused foaming within about ~8 cm of the viewing window. This resulted in some slurry being drawn into the condensate line. Reduced steam from 40% to 30% output. The control valve has been at 40% since 12:00.
14:20	-	-	-	-	-	-	Temperature recorded on data log from a computer hook up to thermocouple TI-2049 slot 1 channel 3 on HB-32. Thermocouple reading at ~10cm below weld crease on bottom portion of tank.
15:30	-	-	-	-	-	-	Still covered with foam, but stable.
15:35	-	-	-	-	-	-	Start addition of second drum #6479 mixed hydroxide slurry to HB-32.
16:00	-	-	-	-	-	-	Brown condensate coming through condensate line. Second drum of mixed hydroxide slurry added. Some foaming in HB-32. This could have caused a possible problem with slurry contaminating the condenser line and condensate, but remained stable.
16:15	-	-	-	-	-	-	Condensate almost clear.
17:50	-	-	-	-	-	-	Slurry mixing vigorously with no stagnant areas.
18:35	-	-	-	-	-	-	Finished taking condensate. Refluxing . A total of 880 L of condensate removed from HB-32.

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/13/94							
18:49	-	-	-	-	-	-	Steam to HB-32 shut off.
18:55	-	-	-	-	-	-	Added third drum #6481 of mixed hydroxide slurry into HB-32.
19:10	-	-	-	-	-	-	Completed concentration and addition of mixed hydroxide slurry.
20:15	-	-	-	-	-	SSHTM1-6.4.12-1	Two 100 ml condensate samples removed from last two
-						SSHTM1-6.4.12-2	condensate drums #13 and 9. Drums emptied into HB-15.
-							No samples taken from first two condensate Drums
-							#9 and 11. Drum #9 reused after first condensate
						SSHTM1-6.4.12-3	collection. Removed one 2 L slurry sample for
-						SSHTM1-6.4.12-4	HNO3 lab test. Removed one 10 L sample
-						SSHTM1-6.4.12-5	for Ray Bell.
-						SSHTM1-6.4.12-6	Removed three 250 ml slurry samples for analysis.
-						SSHTM1-6.4.12-7	
						SSHTM1-6.4.12-8	Removed one1 L and one 500 ml slurry
20:45	-	-	-	-	-	SSHTM1-6.4.12-9	samples for rheology.
21:00	-	-	-	-	-	-	HB-32 mixing slow, slurry thick but moving. Material buildup and caking observed on pipes and brackets above slurry level.

*Note:
Hydrogen was measured on the gas chromatoghraph with a Below Detection Level of 15 ppm. Carbon Dioxide measured on the Infrared CO2 Analyzer with a BDL of <0.05%. NOx is measured on the Chemiluminescent NO/NOx Analyzer in ppm with a BDL of <5 ppm.

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/14/94							
9:00	-	-	-	-	-	-	Increased steam to 20% to begin heat up for Nitric acid addition.
9:30	-	-	-	-	-	-	Increased steam to 30% output.
10:15	-	-	-	-	-	-	Reduced steam to 20% output.
10:40	-	93	-	-	-	-	Shut off steam. Tank HB-32 temperature at 93 degrees (C).
10:59	0	94	10.4	-179.0	0.00kg	-	Started Nitric addition. Added ~0.24 L Dow Corning Anti-Foam Compound lot #GA100322 with ~6 L water. HNO3 barrel starting weight 317 kg.
11:01	2	94	10.4	-177.4	-	-	Brown cloud almost immediately.
11:05	6	94	10.2	-164.6	6kg	-	Acid addition rate 1 kg/min.
11:07	8	93	10.0	-157.6	9kg	-	Acid addition rate 1.5 kg/min.
11:09	10	93	9.9	-151.2	12kg	-	Acid addition rate 1.5 kg/min.
11:11	12	93	9.8	-144.7	16kg	-	Acid addition rate 2 kg/min.
11:13	14	93	9.7	-137.5	18kg	-	Acid addition rate 1.5 kg/min.
11:15	16	92	9.6	-130.8	22kg	-	Acid addition rate 2 kg/min.
11:17	18	92	9.5	-123.3	26kg	-	Acid addition rate 2 kg/min.
11:19	20	92	9.3	-115.1	30kg	-	Increased acid addition rate 2 kg/min..

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/14/94							
11:21	22	92	9.1	-104.4	34kg	-	Acid addition rate2 kg//min.
11:23	24	91	8.9	-91.8	38kg	-	Acid addition rate 2 kg/min.
11:25	26	91	8.7	-77.8	42kg	-	Acid addition rate 2 kg/min.
11:25:30	26:30	91	-	-	-	-	Steam on, control valve actually off.
11:27	28	91	8.4	-59.5	48kg	-	Acid addition rate 3 kg/min.
11:29	30	91	8.0	-35.1	52kg	-	Acid addition rate 2 kg/min.
11:31	32	91	7.7	-11.9	58kg	-	Acid addition rate 3 kg/min. Opened steam to 20% output.
11:33	34	91	7.3	7.6	63kg	-	Acid addition rate 2.5 kg/min.
11:35	36	91	6.9	29.9	68kg	-	Acid addition rate 2.5 kg/min.
11:37	38	92	6.6	46.3	73kg	-	Acid addition rate 2.5 kg/min.
11:39	40	92	6.4	60.0	78kg	-	Acid addition rate 2.5 kg/min.
11:41	42	92	6.1	73.8	83kg	-	Acid addition rate 2.5 kg/min.
11:43	44	92	5.9	84.3	88kg	-	Acid addition rate 2.5 kg/min.
11:45	46	92	5.7	98.4	92kg	-	Acid addition rate 2 kg/min.
11:47	48	92	5.5	111.2	97kg	-	Acid addition rate 2.5 kg/min.

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/14/94							
11:49	50	93	5.2	124.8	102kg	-	Acid addition rate 2.5 kg/min.
11:51	52	93	5.0	137.2	107kg	-	Acid addition rate 2.5 kg/min.
11:53:30	54	93	4.8	-	110kg	-	Stopped addition of Nitric acid.
11:58	59	94	4.9	141.4	-	-	Purged acid line into tank HB-32.
12:15	76	-	-	-	-	SSHTM1-8.0.10-1	Removed two 1 L post acid addition slurry samples. First submitted to Ray Bell for rheology.
-						test-1y	Rheology samples renamed from initial sample SSHTM1-8.0.10-1.
-						test-1ya	
-						test-1x	
-						test-1xa	
-						SSHTM1-8.0.10-2	Second sample for archive.
-						SSHTM1-8.0.10-3	Removed three 250 ml post acid addition for analysis.
-						SSHTM1-8.0.10-4	
12:25	86	-	-	-	-	SSHTM1-8.0.10-5	
12:27	88	99	-	-	-	-	Added ~0.24 L Dow Corning Anti-Foam Compound with ~1 L water.
12:38	99	101	5.1	128.3	-	-	Boiling.
12:45	106	-	-	-	-	SSHTM1-8.0.10-6	Removed 100 ml condensate sample from drum #1.
12:50	111	101	5.1	127.1	-	-	Off-gas measurement H2-0 ppm, NOx (1135 ppm).
13:35	156	-	-	-	-	-	Increased steam to 25% output.
15:00	241	102	5.2	-	-	-	First drum #1 of condensate collected.

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/14/94							
17:05	366	-	-	-	-	SSHTM1-9.0.7-1	Removed 100 ml condensate sample from drum #11.
19:30	511	-	-	-	-	-	HB-32 mixing poorly. No motion except near coils and inside coils. Boiling. Fluid level at top of coils.
20:05	546	-	-	-	-	-	Turned steam off. Probing slurry with a stick showed that the slurry appears very thick for several inches. An ~8 cm buildup of slurry solids observed on top of coils. Concentration stopped approximately 95 L short of target.
20:12	553	100	5.4	-	-	-	Off-gas data collection stopped at this time, therefore, the cumulative time log entries are discontinued. Steam off. Caking on coils ~5 cm above top coil, not mixing at all.
20:30	-	-	-	-	-	SSHTM1-9.0.7-2	Removed 250 ml condensate sample from HB-32. Cooling water off.
20:35	-	-	-	-	-	SSHTM1-9.0.7-3 hno3out1	Removed two 250 ml slurry samples. First sample dipped from inside coil. Second sample dipped from outside coil. Samples submitted for rheology.
20:40	-	-	-	-	-	SSHTM1-9.0.7-4	First rheology sample renamed from initial sample SSHTM1-9.0.7-3.
20:50	-	-	-	-	-	-	Added ~19 to 38 L of water. Buildup on top of coils sprayed off. Slurry moving slowly across top of coil and circulating.
21:12	-	-	-	-	-	-	Added 19 L of water to HB-32. Slurry is flowing smoothly over coils and slow around edges of tank.

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/14/94 21:40	-	-	-	-	-	-	Added ~8 to 11 L water. Slurry flow around coils faster. Outside edges still slow.
8/15/94 10:00	-	-	-	-	-	- SSHTM1-9.0.7-5 test-1ad test-1ad1 SSHTM1-9.0.7-7 SSHTM1-9.0.7-8 SSHTM1-9.0.7-6	HB-32 mixing slightly better than last night. Motion is slow and slurry is rotating as well as moving in toward the center. Stagnant near tank wall. No buildup. Removed one 2 L slurry sample for rheology and renamed from initial sample SSHTM1-9.0.7-5. Removed three 500 ml slurry samples. Two archived.
11:00	-	-	-	-	-	SSHTM1-9.0.7-10 SSHTM1-9.0.7-11 SSHTM1-9.0.7-9	Removed three 250 ml slurry samples for analysis.
11:20	-	-	-	-	-	-	Add 38 L water to HB-32 to improve mixing, based on lab rheology tests from samples taken yesterday.
13:15	-	-	-	-	-	-	Mixing is fluid except for ~10 cm along tank wall, which is not moving.
8/17/94 14:40	-	-	-	-	-	-	Transferred HB-32 to HB-34 .
15:00	-	-	-	-	-	SSHTM1-15.2-4	Removed one 250 ml slurry sample from HB-34 for analysis.

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/18/94							
N/A	-	-	-	-	-	hno3t34	Samples removed for rheology. These samples
						hno3t4b	were not recorded in sample log.
						hno3t34c	
						hno3t34d	
8/19/94							
7:00	-	-	-	-	-	-	Added 106 kg of NaNO3 to HB-33. Mixed for ~2 hours.
						hno3sod1	Samples removed after sodium addition for rheology
						hno3sod2	measurements. Samples not entered on sample log.
						hno3sod3	
						hno3sod4	
						hno3sod5	
9:30	-	-	-	-	-	-	Added 284 kg of SiO2 to HB-33. Removed one
						SSHTM1-15.2-6	250 ml post SiO2 addition slurry sample for
						hno3d0a	rheology. Rheology samples renamed from
						hno3d0b	initial sample SSHTM1-15.2-6.
						hno3-5d0	*hno3d0a and hno30b samples used for lab-scale
						hno3d0a	rheology aging study.
15:00	-	-	-	-	-	-	Transferred slurry simulant to HB-33 to
							prepare for melter.
8/22/94							
N/A	-	-	-	-	-	testd3aa	Ageing samples removed for rheology
						testd3ab	measurement. Samples not listed in sample log.
						testd3a	
						testd3	
						HNO3-D1	Lab-scale rheology aging study.
						HNO3-D1A	

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/24/94 8:15	-	-	-	-	-	SSHTM1-15.2-5	Sample removed from HB-33 for rheology.
						day5a	Rheology samples renamed from initial sample
						day5b	SSHTM1-15.2-5.
						day5c	
						day5d	
9:30	-	-	-	-	-	SSHTM1-15.2-1	Removed three 250 ml slurry samples for analysis.
						SSHTM1-15.2-2	
						SSHTM1-15.2-3	
						HNO3-D5	Lab-scale rheology aging study.
						HNO3-D5A	
8/26/94 8:50	-	-	-	-	-	SSHTM1-15.2-7	Removed 250 ml post SiO2 addition slurry sample
						hnod7c	for rheology. Rheology samples renamed from
						hno7d	initial sample SSHTM1-15.2-7.
						hno7a	
						hno7b	
						HNOD7E	Lab-scale rheology aging study.
						HNOD7F	
8/27/94 9:00	-	-	-	-	-	SSHTM1-15.2-8	Removed 250 ml post SiO2 addition slurry sample for rheology.
8/29/94 N/A	-	-	-	-	-	hnod10a	Ageing samples removed for rheology measurement.
						hnod10b	Samples not listed in sample log.
						hnod10c	
						hnod10d	
						HNOD10E	Lab-scale rheology aging study.
						HNOD10F	

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)
8/31/94							
9:00	-	-	-	-	-	tglycer2 tglycer2a tglycer1 tglycer1a	Sample for rheology taken from daytank after glycerin addition.
14:23	-	-	-	-	-	SSHTM1-15.2-9 t-age-3a t-age-3b t-age-31 t-age-32	Removed 250 ml post SIO2 addition samples slurry sample for rheology. Rheology renamed from initial sample SSHTM1-15.2-9.
9/1/94							
14:00	-	-	-	-	-	SSHTM1-15.2-10	Removed 250 ml post SIO2 addition slurry sample for rheology.
						HNOD14A HNOD14B	Lab-scale rheology aging study.
9/2/94							
10:30	-	-	-	-	-	SSHTM1-15.2-11 t-age-9d t-age-9e t-age-9b t-age-9c t-age-9a	Removed 250 ml post SIO2 addition slurry sample for rheology. Rheology samples renamed from initial sample SSHTM1-15.2-11.
9/8/94							
-	-	-	-	-	-	HNOD28A HNOD28C	Lab-scale rheology aging study.

Log A4.1 (Cont.)

Small-Scale Melter Feed Preparation Activity Log
Alternate HTM Flowsheet 1 (No Reductant Addition)
Nitric Acid Addition

Date/ Time (1)	Cumulative Time (min) (2)	Temp. (C) Slurry	pH (3)	mV	HNO3 Addition (4)	Sample Identification (6)	Notes/Observations (5)

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 a covered viewing window at the top of tank HB-32. A conversion from pounds to kilograms, inches to centimeters and gallons to liters have been made.
6. Lowercase sample names are rheology samples from Table A4.4.1. Upper case samples starting with HNO are from lab scale ageing study Table A4.4.2

SSHTM-1 Sample Matrix

The following sample matrix, Table A4.1, 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 A4.1 Small-Scale Melter Feed Preparation Sample Matrix Which Includes: Sample Numbers, Dates of Sampling, Analytical Request Form Numbers, and Dates of Analytical Report Issue.

	Alternate HTM Flowsheet 1 (Nitric Acid)													1/4/95									
	General Information					Analysis Performed (after test activity)													Test Engineer:				
Item	SSHTM-1 Test Activity	Approx. Tank Volume	# of Samples	Sample Size	Archive	wt% TS	wt% TO	pH Sp Gr	ICP	AA (Cs)	IC	ISE (NH3)	Total C	Crus. Redox	Viscosity	Shear Stress	Off Gas	Sample Numbers	Date of Sample	Submitted for Analysis	ARF Number	Date Analysis Complete	Date Analytical Report Issued
	LFCM-9 Feed Stock																						
			1	30 L														SSHTM1-6.2.6-1	8/12/94				
1	Add Optima Pre-mixed Simulated Waste Slurry	503	1	250 mL	0	/	/	/	/		/						No	SSHTM1-6.2.6-2	8/12/94	8/12/94	K10165-8	8/30/94	9/7/94
2	Add Chemicals and Shim as required	*503	1	250 mL	0	/	/	/									No	SSHTM1-6.3.8-1	8/13/94	8/13/94	K10165-9	8/30/94	9/7/94
3*	Concentrate	515	3	250 mL	0	1	1	1	1 (both)		1	1	1				No	SSHTM1-6.4.12-5	8/13/94	8/15/94	K10165-10	8/30/94	9/8/94
																		SSHTM1-6.4.12-6	8/13/94	10/12/94	K27349-1	10/24/94	10/26/94
																		SSHTM1-6.4.12-7	8/13/94	9/15/94	K10165-20	9/29/94	10/5/94
3*	Concentrate	515	3	500 mL	2										1	1	No	SSHTM1-6.4.12-8, 9	8/13/94		NA		
	Acid addition test sample	515	1	2 L	NA													SSHTM1-6.4.12-3	8/13/94		NA	NA	NA
	Condensate samples	515	1	100 mL	1													SSHTM1-6.4.12-1	8/13/94		NA		
	Condensate samples	515	1	100 mL	0													SSHTM1-6.4.12-2	8/13/94		NA		
4	Extra sample for Lab Tests	515	1	5 L	1												No						
5	Deleted																No						
	Feed Preparation																No						
6*	pH Adjustment (Add Nitric Acid); Measure Off-Gas		3	250 mL		1	1	1	1 (both)		1	1	1				Yes	SSHTM1-8.0.10-3	8/14/94	8/15/94	K10165-11	8/30/94	9/20/94
																		SSHTM1-8.0.10-4	8/14/94	9/15/94	K10165-20	9/29/94	10/5/94
																		SSHTM1-8.0.10-5	8/14/94	10/12/94	K27349-1	10/24/94	10/26/94
																		SSHTM1-8.0.10-4	8/14/94				
6*	pH Adjustment (Add Nitric Acid); Measure Off-Gas		2	1 L	1										1	1	Yes	SSHTM1-8.0.10-1, 2	8/14/94		NA		
6*	pH Adjustment (Add Nitric Acid); Measure Off-Gas		1 (Condensate)	100 mL				1			1	1	1				Yes	SSHTM1-8.0.10-6	8/14/94				
7*	Concentration/Digestion		3	250 mL		1	1	1	1 (both)		1	1	1				Yes	SSHTM1-9.0.7-9	8/15/94	8/15/94	K10165-14	8/30/94	9/7/94
																		SSHTM1-9.0.7-10	8/15/94	10/12/94	K27349-1	10/24/94	10/26/94
																		SSHTM1-9.0.7-11	8/15/94	9/15/94	K10165-20	9/29/94	10/5/94
7*	Concentration/Digestion		3	500 mL	2										1	1	Yes	SSHTM1-9.0.7-6, 7, 8	8/15/94	NA	NA	NA	NA
			1	2 L														SSHTM1-9.0.7-5	8/15/94	NA	NA	NA	NA
7*	Concentration/Digestion		1	100 mL				1			1	1	1				Yes	SSHTM1-9.0.7-1	8/15/94				
			1	250 mL														SSHTM1-9.0.7-2	8/15/94				
			1	250 mL														SSHTM1-15.2-4	8/17/94	8/18/94	K10165-16	8/18/94	8/18/94
	Glass Former Addition																						
9*	Add Glass Formers (NaNO3)		3	250 mL	1	1	1	1	1 (both)		1	1	1				No	No samples taken					
9*	Add Glass Formers (NaNO3)		2	1 L	2										1	1	No	No samples taken					
9*	Add Glass Formers (NaNO3)		1 (Condensate)	100 mL				1			1	1	1				No	No samples taken					

Table A4.1 (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.

	General Information					Analysis Performed (after test activity)																	
Item	SSHTM-1 Test Activity	Approx. Tank Volume	# of Samples	Sample Size	Archive	wt% TS	wt% TO	pH Sp Gr	ICP	AA (Cs)	IC	SIE (NH3)	Total C	Crus. Redox	Viscosity	Shear Stress	Off Gas	Sample Numbers	Date of Sample	Submitted for Analysis	ARF Number	Date Analysis Complete	Date Analytical Report Issued
10*	Add Glass Formers (SiO2)	1559 L	3	250 mL		1	1	1	1 (both)		1	1	1				No	SSHTM1-15.2-1	8/24/94	8/24/94	K10165-17	8/27/94	9/7/94
																		SSHTM1-15.2-2	8/24/94	9/15/94	K10165-20	9/29/94	10/5/94
																		SSHTM1-15.2-3	8/24/94	10/12/94	K27349-1	10/24/94	10/26/94
10*	Add Glass Formers (SiO2)	1559 L	3	1 L											1	1	No	No samples taken					
10*	Add Glass Formers (SiO2)	1559 L	1	100 mL										1			No	No samples taken					
10*	Add Glass Formers (SiO2)	1559 L	1 (Condensate)	100 mL				1			1	1	1				No	No samples taken					
11	Concentrate/Dilute as Required	1559 L	3	30 mL		1	1	1									No	No samples taken					
11	Concentrate/Dilute as Required	1559 L	3	500 mL	2										1	1	No	No samples taken					
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							
14	Extra sample for Lab Tests		1	5 L	1																		
	* 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 A4.2.1 and A4.2.2 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 analytical data in the tables was obtained using the following procedures:

<u>gWO/L and density-</u> 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%.

<u>pH-</u> 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.

<u>Total Organic Carbon-</u> 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.

<u>Anions-</u> 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%.

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

<u>Elements, Oxides-</u> The elemental or oxide analyses were made by ICP-AES using procedure APSL-14. The estimated accuracy for major elements using this technique is ±10%

Table A4.2.1. Small-Scale Melter Feed Preparation Condensate Sample Analysis, Results for Alternate Flowsheet 1 Processing. Note that pH values were measured under ambient conditions.

	Sample Identification Number				
	SSHTM1-6.4.12-1	SSHTM1-6.4.12-2	SSHTM1-8.0.10-6	SSHTM1-9.0.7-1	SSHTM1-9.0.7-2
Processing stage	Initial slurry concentration	Initial slurry concentration	Acid addition	Concentration digestion	Concentration digestion
pH(ambient temp.)	7.32	9.1	0.38	2.78	2.92
TOC (wt%)	N/A	N/A	N/A	N/A	N/A
Anions(ppm)					
fluoride	bdl[a]	bdl[a]	bdl[a]	N/A	N/A
Chloride	bdl[b]	bdl[a]	bdl[a]	N/A	N/A
nitrite	10.6	34.9	bdl[a]	N/A	N/A
nitrate	7.7	19	28838	N/A	N/A
phosphate	bdl[a]	bdl[a]	bdl[a]	N/A	N/A
sulfate	bdl[a]	bdl[a]	bdl[a]	N/A	N/A
Ammonia(ppm)	2.6	5	<0.1	<0.1	<0.1

(a) bdl 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 A4.2.2. Small-Scale Melter Feed Preparation Slurry Sample Analysis Results for Alternate HTM Flowsheet 1 Processing. Target composition of slurry in first column includes only elements actually analyzed for and found. Note that K_2O is also not included because ICP technique does not work well at dilution employed for analysis.

	Target Composition	Sample Identification Number: SSHTM1-6.2.6.2	SSHTM1-6.4.12-5	SSHTM1-8.0.10-3	SSHTM1-9.0.7-9	SSHTM1-15.2-1
Processing stage	NCAW slurry simulant	as received	$NaNO_2$, $NaNO_3$ added	HNO3 added	digest and concentrate	add SiO_2, $NaNO_3$
gWO/L	100	108	174	171	208	346
density	N/A	1.08	1.16	1.16	1.21	1.28
pH(ambient temp.)	N/A	11.7	11.66	6.9	7.7	7.8
TOC (wt%)[a]	0.0001	N/A	0.05	0.03	0.03	0.07
Anions(ppm)						
fluoride	90	58	bdl[b]	bdl[b]	bdl[b]	bdl[b]
Chloride	278	bdl[b]	bdl[b]	bdl[b]	bdl[b]	bdl[b]
nitrite	14800	bdl[b]	25546	19901	21939	22863
nitrate	5330	3430	10244	43778	49296	49399
phosphate	72	201	bdl[b]	bdl[b]	bdl[b]	bdl[b]
sulfate	728	564	bdl[b]	bdl[b]	bdl[b]	bdl[b]
Ammonia(ppm)[a]	N/A	N/A	~0.2	~0.2	<~0.1	~0.2
Oxides(wt%)						
Ag2O	0.12	0.11	0.14	0.14	0.12	0.08
Al2O3	9.25	9.57	8.90	8.85	8.87	5.06
B2O3	0.01	0.11	0.07	0.07	0.06	0.13
BaO	0.18	0.22	0.21	0.20	0.18	0.08
CaO	0.81	0.54	0.41	0.41	0.42	0.25
CdO	3.08	3.33	3.08	3:04	3.04	1.72
CeO2	0.66	1.08	0.96	1.01	0.95	0.59
Cr2O3	0.27	0.33	0.27	0.27	0.30	0.17
CuO	0.25	0.33	0.27	0.27	0.24	0.17
Fe2O3	28.80	34.19	30.62	30.41	30.50	17.15
La2O3	0.67	0.75	0.68	0.68	0.65	0.38
MgO	0.37	0.65	0.55	0.54	0.54	0.38
MnO	2.19	2.47	2.19	2.16	2.14	1.21
MoO3	0.57	0.54	0.55	0.54	0.54	0.29
Na2O	21.90	11.72	21.99	22.30	22.20	19.16
Nd2O3	3.56	4.41	3.77	3.72	3.75	2.09
NiO	2.35	2.58	2.53	2.43	2.38	1.34
P2O5	0.89	0.65	0.62	0.61	0.60	0.38
PbO	0.72	0.75	0.68	0.68	0.66	0.42
SO3	0.67	1.08	0.96	1.01	0.95	0.63
SiO2	4.11	4.84	4.52	4.59	4.35	39.00
SrO	0.12	0.11	0.14	0.14	0.12	0.08
TeO2	0.10	0.22	0.14	0.14	0.12	0.08
TiO2	0.67	0.75	0.75	0.74	0.71	0.42
Y2O3	0.08	0.11	0.07	0.07	0.06	0.04
ZnO	0.34	0.86	0.68	0.54	0.60	0.38
ZrO2	15.40	17.74	14.38	14.66	14.70	8.24

(a) These analyses were performed on sibling samples.
(b) bdl 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.

4.3 Off-Gas Data

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

1- Table A4.3 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 A4.3.1 and A4.3.2 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 ±2%, ±3% and ±7% respectively.

Table A4.3 Peak Off-Gas Generation Rates and Total Off-Gas Generated for the Alternate HTM Flowsheet 1 Small-Scale Test.

	Off-Gas Generated During Processing				
	CO_2	N_2O	H_2	NO_x	NH_3
Alternate HTM Flowsheet 1[a]					
Peak Generation Rate (moles/min)	9.9	BDL[b]	BDL[b]	2.17	c
Total Off-Gas Generated (moles)	383	BDL[b]	BDL[b]	79.3	0.01

(a) Alternate HTM Flowsheet 1 is the no reductant, nitric acid process flowsheet.
(b) BDL: Below Detection Limit
(c) Not measured but usually equal to or less than the H_2 generation rate.

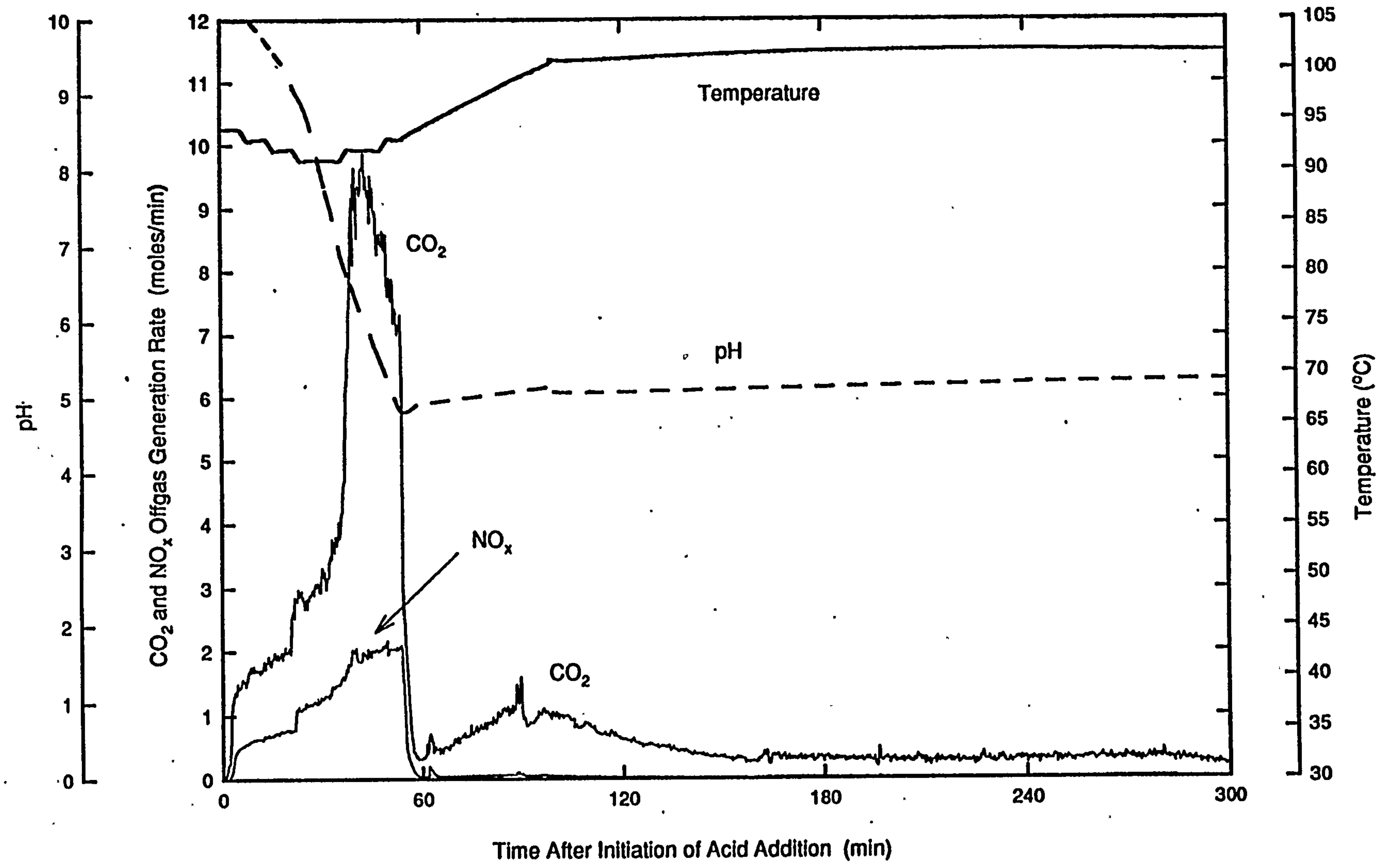


Figure A4.3.1 Process Profile for Small-Scale Alternate HTM Flowsheet 1 for the First 300 Minutes Measured from the Start of Nitric Acid Addition.

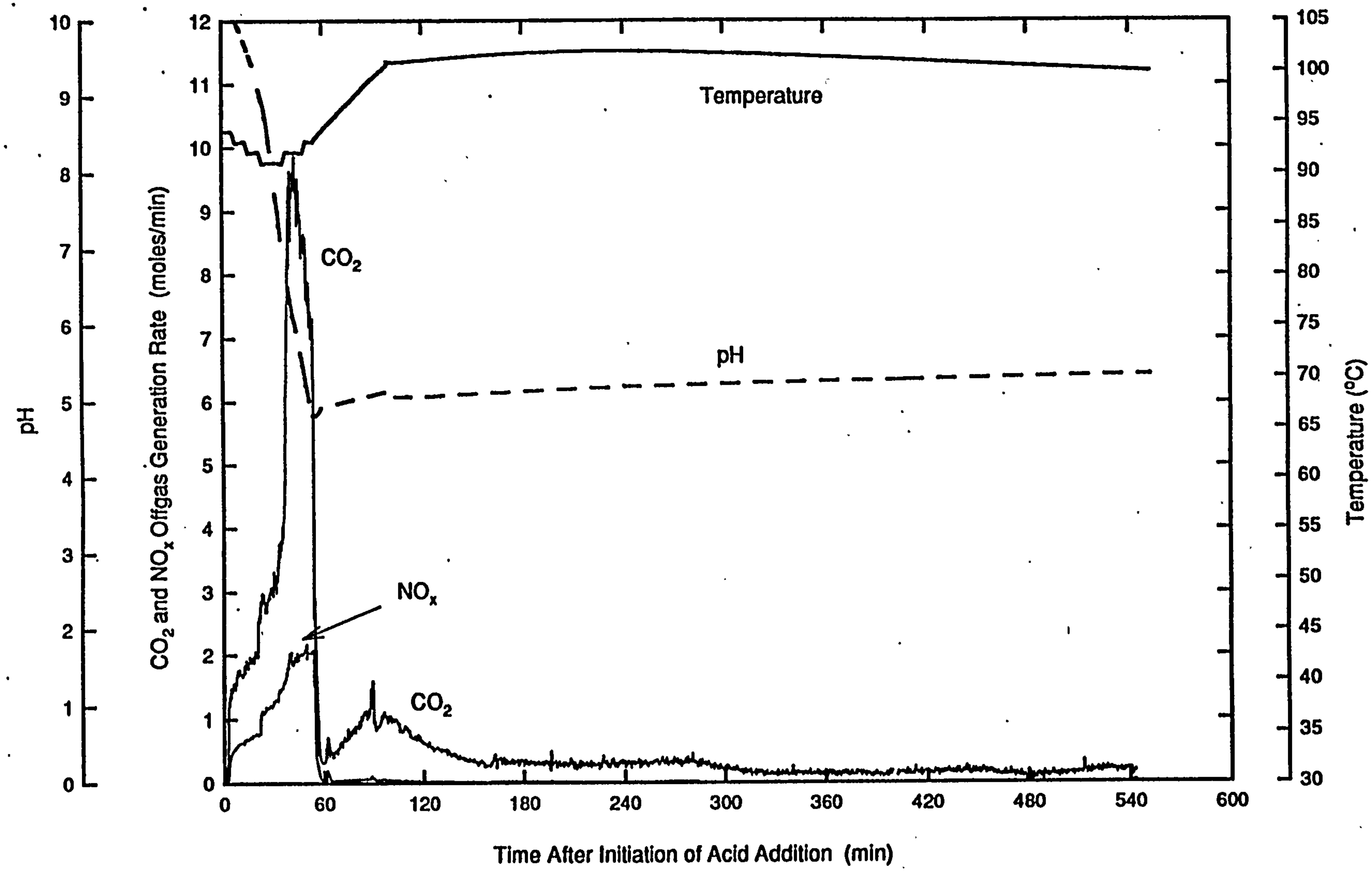


Figure A4.3.2 Process Profile for Small-Scale Alternate HTM Flowsheet 1 for 0 to 600 Minutes Measured from the Start of Nitric Acid Addition.

4.4 Rheology and Gram Waste Oxide Loading Data for Melter Feed

Introduction

For Alternate HTM Flowsheet 1, 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. In this section it is called the "modified feed procedure." 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 section 4.4.2.

Experimental

Density, pH (22°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 feed from the feed preparation tanks was fed into a day tank before feeding into the melter. Rheological measurements were also taken for samples pulled from the day tank.

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:

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

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 approximately every other day for twelve 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. These results were compared with the results of the melter feed prepared (following procedures developed in the lab) 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, 3 days, 5 days, 7 days, 10 days, 13 days, and 20 days for the sample prepared with the small-scale system. 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).

<u>Results</u>

<u>4.4.1 Rheology of Small-Scale Feed Preparation and Aging Samples</u>

An attempt was made to concentrate the feed to 240 gWO/L but at an oxide loading of 216 gWO/L the feed began to collect on the heating coils and the mixing became poor. Water was added to the feed, bringing the oxide loading down to 178 gWO/L, but this did not improve the mixing in the tank. The feed was then transferred from tank HB-32 to tank HB-34 in which the design allowed for better mixing.

The waste simulant prepared in the small-scale system, before silica and sodium nitrate addition and at an oxide loading of 191 gWO/L, had a solids loading of 25.3 wt%. At these conditions the plastic viscosity was 16.2 mPa•s and the yield stress was 11.7 Pa (50°C). After silica and sodium nitrate addition to the small-scale feed, the solids loading was 36.6%, and the oxide loading was 354 gTO/L. At these conditions, the plastic viscosity was 18.6 mPa•s and the yield stress was 15.1 Pa (50°C)

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 12, yield stress varied between 12.0 Pa and 16.5 Pa (50°C) and plastic viscosity varied between 14.2 mPa•s to 21.3 mPa•s (50°C). The Bingham model was fit to the data with correlation coefficients between 0.68 and 0.88. The solids loading varied with values of 38 ± 2 weight percent. The pH was not measured often enough to detect any changes. No pH changes were observed over the time period and with the frequency of measurement used. Rheological properties and pH were also measured at 25°C

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

- before concentration in tank HB-32
- during mixing problems in HB-32
- after dilution
- after transfer to HB-34
- after addition of sodium nitrate

Before concentration in tank HB-32, the waste simulant had a plastic viscosity of 12.3 mPa•s and a yield stress of 3.7 Pa (at 50°C). The simulant was concentrated to approximately 220 gWO/L. The plastic viscosity increased to 30.9 mPa•s and the yield stress increased to 20.0 Pa. At these rheological properties, tank HB-32 was not mixing properly, therefore the waste simulant was diluted to 178 gWO/L. The rheological properties at this concentration were a plastic viscosity of 18.4 mPa•s and a yield stress of 11.7 Pa. The simulant was then transferred to tank HB-34. After sodium nitrate addition, the plastic viscosity was 12.9 mPa•s and the yield stress was 14.3 Pa.

Rheological measurements were also taken of samples pulled from the day tank after glycerine addition and after boric acid addition. After glycerine addition, the plastic viscosity at 50°C was 13.1 mPa•s and the yield stress was 10.8 Pa. After boric acid addition, the plastic viscosity at 50°C was 16.2 mPa•s and the yield stress was 12.6 Pa. These values were compared with the day 12 rheological properties in which the plastic viscosity was 14.2 mPa•s and the yield stress was 12.7 Pa. The plastic viscosities and yield stresses can be found in summary Table 4.4.1 which follows. The individual rheograms are also included.

Table A4.4.1 Physical Property and Rheology Data for Alternate HTM Flowsheet 1, Small-Scale Melter Feed Preparation, Aging, and Day Tank Samples.

Alternate HTM Flowsheet 1[e]		g TO/L	pH (≈22°C[a])	Temperature (°C)	wt % solids	Density (g/ml)	Yield Stress (Pa[b])	Plastic Viscosity (mPa·s[c])	Correlation Coeff. (R^2)[d]
Small-Scale Feed Prep. Samples:									
Before concentration in HB-32:									
File ID/Date:	TEST-1Y/8-14-94	----	----	25	----		3.4	13.7	0.94
	TEST-1YA/8-14-94	----	6.8	25	----		3.1	14.0	0.95
	TEST-1X/8-14-94	----	----	50	----		3.7	12.3	0.92
	TEST-1XA/8-14-94	----	6.4	50	----		3.6	11.7	0.92
During problems with mixing:									
File ID[f]/Date:	HNO3OUT1/8-14-94	216	----	50	26.2	1.19	20.0	30.9	0.82
After dilution:									
File ID/Date:	TEST-1AD/8-15-94	178	----	50	----		11.7	18.4	0.84
	TEST1AD1/8-15-94	----	----	50	----		12.7	17.9	0.86
After transfer to HB-34:									
File ID/Date:	wellmixed/8-18-94	191	7.0	25	25.3	1.15	11.8	19.0	0.89
	hno3t34b/8-18-94	----	----	25	----	----	12.7	19.2	0.89
	hno3t34c/8-18-94	----	----	50	----	----	11.7	16.2	0.81
	hno3t34d/8-18-94	----	----	50	----	----	12.3	14.2	0.77
After sodium addition:									
File ID/Date:	HNO3sod1/8-19-94	----	7.5	25	----		10.1	13.9	0.81
	HNO3sod2/8-19-94	----	----	25	----		10.1	13.2	0.88
	HNO3sod3/8-19-94	----	----	50	----		14.3	12.9	0.68
	HNO3sod4/8-19-94	----	----	50	----		13.7	13.3	0.71
After silica addition:									
File ID/Date:	HNO3D0A/8-19-94	----	----	25	----	----	13.6	19.6	0.83
	HNO3D0B/8-19-94	----	----	25	----	----	15.1	22.4	0.88
	HNO3-5D0/8-19-94	354	----	50	36.6	1.29	15.1	18.6	0.75
	HNO35D0A/8-19-94	----	----	50	----	----	16.2	19.2	0.76

(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.
(c) 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.
(d) Least squares correlation coefficient of shear rheology data fit to the Bingham equation.
(e) Alternate HTM Flowsheet 1 is the no reductant, nitric acid processed flowsheet.
(f) File ID is the "name" of the rheogram and model fit data that follows this table.

Table A4.4.1 (cont.) Physical Property and Rheology Data for Alternate HTM Flowsheet 1, Small-Scale Melter Feed Preparation, Aging, and Day Tank Samples.

	g TO/L	pH (≈22°C[a])	Temperature (°C)	wt % solids	Density (g/ml)	Yield Stress (Pa[b])	Plastic Viscosity (mPa·s[c])	Correlation Coeff. (R^2)[d]
Alternate HTM Flowsheet 1[e]								
Small-Scale Feed Prep. Samples:								
Day 3 after silica addition:								
File ID/Date: TESTD3AA/8-22-94	----	7.8	25	35.9		14.6	20.0	0.81
TESTD3AB/8-22-94	----	----	25	----		15.5	19.0	0.86
TESTD3A/8-22-94	----	7.6	50	----		17.1	23.8	0.89
TESTD3/8-22-94	----	----	50	----		16.5	18.5	0.69
Day 5 after silica addition:								
File ID/Date: DAY5A/8-24-94	----	7.8	25	40.0		12.1	16.7	0.85
DAY5b/8-24-94	----	----	25	----		12.4	16.6	0.88
DAY5c/8-24-94	----	----	50	----		12.0	21.3	0.88
DAY5d/8-24-94	----	----	50	----		14.1	14.8	0.80
Day 7 after silica addition:								
File ID/Date: HNOD7C/8-26-94	----	----	25	----		12.5	19.0	0.89
HNOD7D/8-26-94	----	----	25	----		13.0	18.7	0.88
HNOD7A/8-26-94	----	8.0	50	35.7		14.2	17.7	0.70
HNOD7B/8-26-94	----	----	50	----		15.9	17.8	0.81
Day 10 after silica addition:								
File ID/Date: hnod10a/8-29-94	----	7.6	25	----		10.3	17.1	0.91
hnod10b/8-29-94	----	----	25	----		11.4	18.0	0.85
hnod10c/8-29-94	----	7.6	50	----		16.2	17.5	0.73
hnod10d/8-29-94	----	----	50	----		15.6	18.4	0.80
Day 12 after silica addition:								
File ID/Date: T-AGE-3A/8-31-94	----	----	25	----		10.5	15.8	0.91
T-AGE-3B/8-31-94	----	----	25	----		10.5	16.2	0.92
T-AGE-31/8-31-94	----	----	50	----		12.7	14.2	0.71
T-AGE-32/8-31-94	----	----	50	----		13.4	15.6	0.80

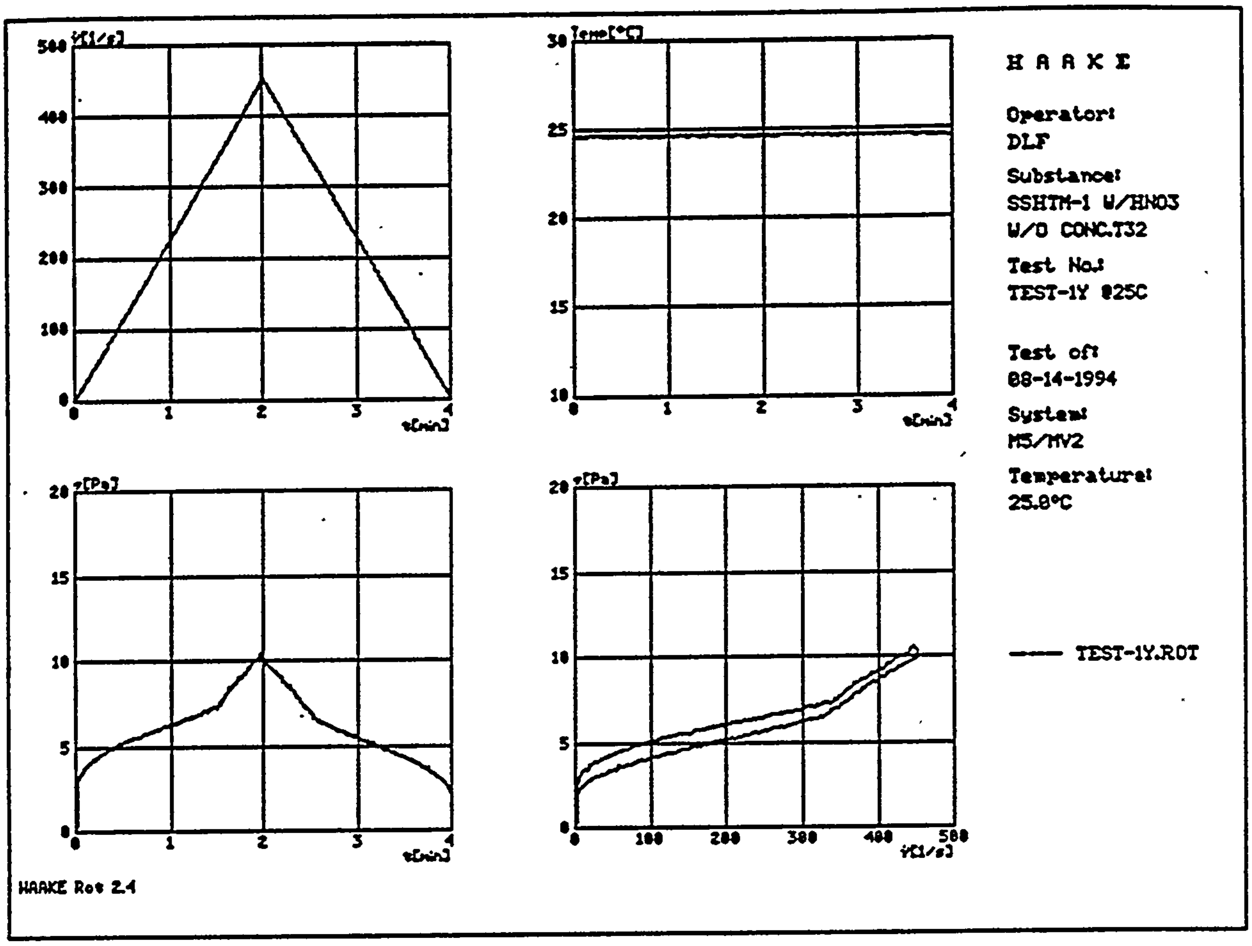
(a) pH measured at 22°C unless the corresponding temperature value is listed as 50°C. If so, pH was measured at 50°C.
(b) Laboratory rheological screening data value for the yield stress is 15 Pa. The yield stress was calculated by fitting the shear rate sweep data to the Bingham model.
(c) Laboratory 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.
(d) Least squares correlation coefficient of shear rheology data fit to the Bingham equation.
(e) Alternate HTM Flowsheet 1 is the no reductant, nitric acid processed flowsheet.

Table A4.4.1 (cont.) Physical Property and Rheology Data for Alternate HTM Flowsheet 1, Small-Scale Melter Feed Preparation, Aging, and Day Tank Samples.

Alternate HTM Flowsheet 1[e]		g TO/L	pH (≈22°C[a])	Temperature (°C)	wt % solids	Density (g/ml)	Yield Stress (Pa[b])	Plastic Viscosity (mPa·s[c])	Correlation Coeff. (R^2)[d]
Small-Scale Feed Prep. Samples:									
Day tank, after glycerine addition:									
File ID/Date:	TGLYCERA/8-31-94	----	----	25	----		9.4	14.6	0.85
	TGLYCERB/8-31-94	----	----	25	----		9.0	14.4	0.93
	TGLYCER1/8-31-94	----	----	50	----		10.8	13.1	0.76
	TGLYCER2/8-31-94	----	----	50	----		11.5	15.5	0.86
Day tank, after boric acid addition:									
File ID/Date:	T-AGE-9d/9-02-94	----	----	25	----		9.8	15.8	0.87
	T-AGE-9e/9-02-94	----	----	25	----		10.1	17.6	0.90
	T-AGE-9a/9-02-94	----	----	50	----		12.6	16.2	0.78
	T-AGE-9b/9-02-94	----	----	50	----		14.0	20.2	0.86
	T-AGE-9c/9-02-94	----	----	50	----		14.7	19.9	0.87

(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.
(c) 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.
(d) Least squares correlation coefficient of shear rheology data fit to the Bingham equation.
(e) Alternate HTM Flowsheet 1 is the no reductant, nitric acid processed flowsheet.

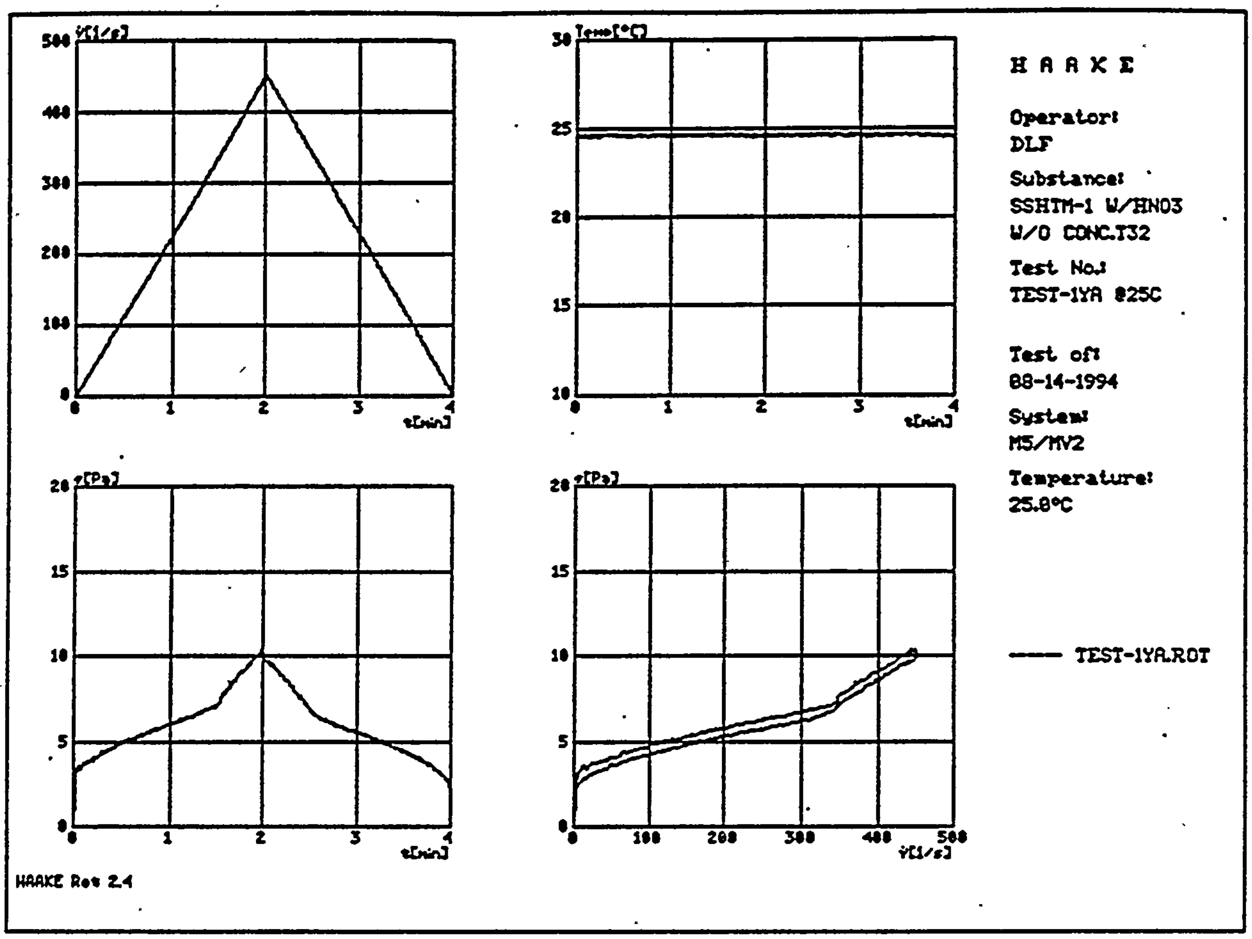
Rheogram and Data for Test Number TEST-1Y @25C



Bingham	: ó= óo+Eta*D	R»=0.94	óo=3.411	Eta=0.0137	
Ostwald	: ó= a*D■	R»=0.89	a=1.378	n=0.2951	
H.-Bulkley	: ó= óo+a*D■	R»=0.91	a=0.8119	n=0.3569	óo=1.148
Casson	: «ó-«óo = «(Eta*D)	R»=0.92	óo=2.315	Eta=0.005005	
Linear	: Y= a+b*X	R»=0.94	a=3.411	b=0.0137	
Inv. linear	: Y= 1/(a+b*X)	R»=0.02	a=0.9328	b=-0.002644	
Exponential	: Y= a*EXP(b*X)	R»=0.45	a=3.405	b=0.002561	
Hyperbolic	: Y= a+b/X	R»=0.10	a=6.571	b=-3.407	
Logarithmic	: Y= a+b*LN(X)	R»=0.74	a=-1.244	b=1.517	

Optimum :
Bingham : ó= óo+Eta*D R»=0.94 óo=3.411 Eta=0.0137

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

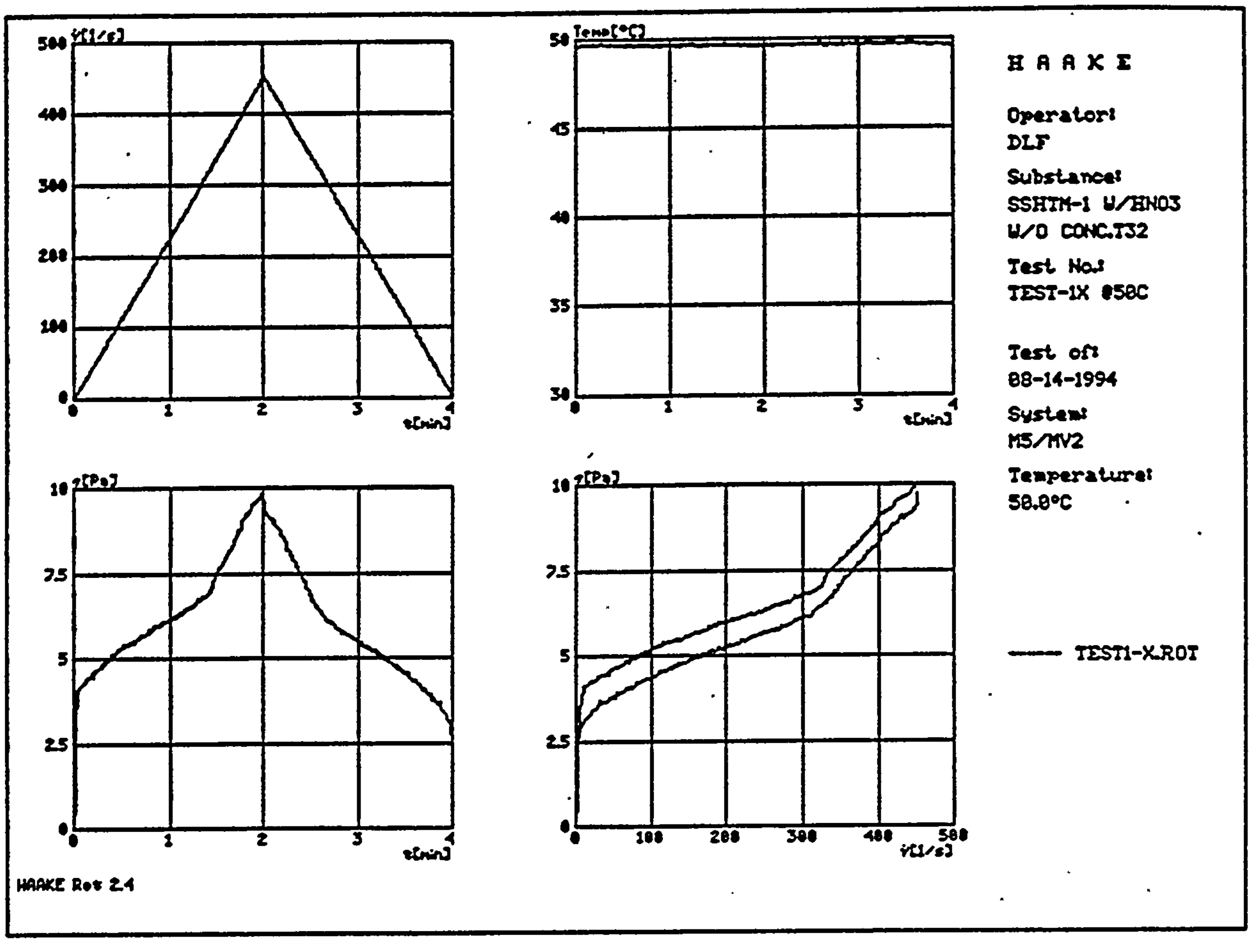


Bingham	:	ő= őo+Eta*D	R»=0.95	őo=3.112	Eta=0.01402	
Ostwald	:	ő= a*D▪	R»=0.86	a=1.442	n=0.2790	
H.-Bulkley	:	ő= őo+a*D▪	R»=0.90	a=0.4558	n=0.4323	őo=1.722
Casson	:	«ő-«őo = «(Eta*D)	R»=0.93	őo=2.135	Eta=0.005149	
Linear	:	Y= a+b*X	R»=0.95	a=3.112	b=0.01402	
Inv. linear	:	Y= 1/(a+b*X)	R»=0.23	a=0.3040	b=-530.7E-06	
Exponential	:	Y= a*EXP(b*X)	R»=0.80	a=3.422	b=0.002439	
Hyperbolic	:	Y= a+b/X	R»=0.09	a=6.361	b=-4.748	
Logarithmic	:	Y= a+b*LN(X)	R»=0.69	a=-1.411	b=1.505	

Optimum :

Bingham	:	ő= őo+Eta*D	R»=0.95	őo=3.112	Eta=0.01402

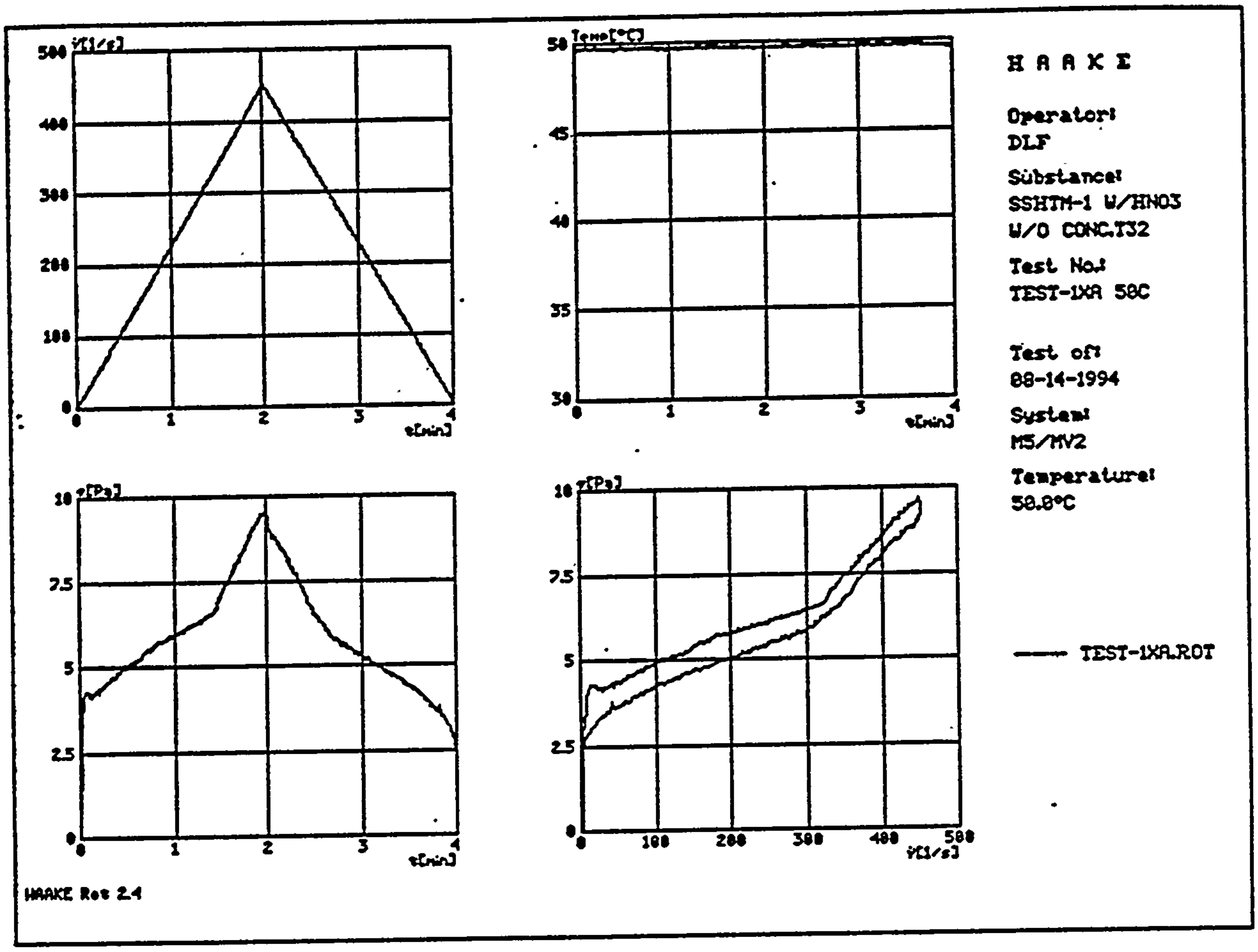
Rheogram and Data for Test Number TEST-1X @50C




```
Bingham     : ó= óo+Eta*D     R»=0.92   óo=3.692   Eta=0.01226
Ostwald     : ó= a*D■         R»=0.85   a=1.652   n=0.2598
H.-Bulkley  : ó= óo+a*D■      R»=0.84   a=1.116   n=0.3023   óo=0.9732
Casson      : «ó-«óo = «(Eta*D)  R»=0.89   óo=2.663   Eta=0.003927
Linear      : Y= a+b*X        R»=0.92   a=3.692   b=0.01226
Inv. linear: Y= 1/(a+b*X)     R»=0.03   a=0.5041   b=-0.001208
Exponential: Y= a*EXP(b*X)    R»=0.48   a=3.674   b=0.00225
Hyperbolic : Y= a+b/X         R»=0.13   a=6.546   b=-4.566
Logarithmic: Y= a+b*LN(X)     R»=0.72   a=-0.4473   b=1.352

Optimum :
Bingham     : ó= óo+Eta*D     R»=0.92   óo=3.692   Eta=0.01226
```

Rheogram and Data for Test Number TEST-1XA 50C

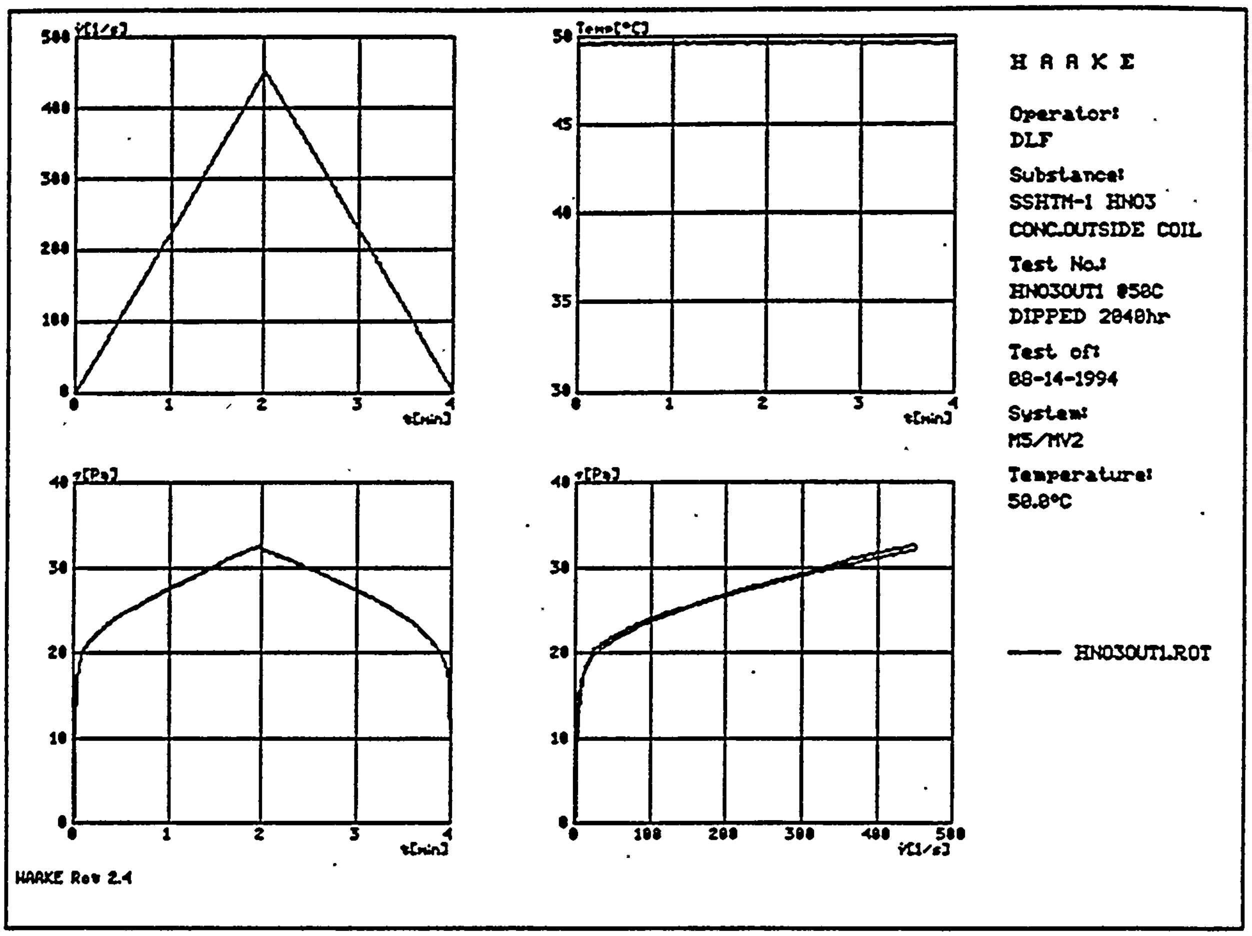


Bingham	: ó= óo+Eta*D	R»=0.92	óo=3.559	Eta=0.01168	
Ostwald	: ó= a*D■	R»=0.70	a=1.235	n=0.3072	
H.-Bulkley	: ó= óo+a*D■	R»=0.80	a=1.335	n=0.2650	óo=0.8125
Casson	: «ó-«óo = «(Eta*D)	R»=0.83	óo=2.551	Eta=0.003766	
Linear	: Y= a+b*X	R»=0.92	a=3.559	b=0.01168	
Inv. linear	: Y= 1/(a+b*X)	R»=0.03	a=0.4628	b=-0.00105	
Exponential	: Y= a*EXP(b*X)	R»=0.50	a=3.695	b=0.002088	
Hyperbolic	: Y= a+b/X	R»=0.10	a=6.243	b=-2.014	
Logarithmic	: Y= a+b*LN(X)	R»=0.69	a=-0.2262	b=1.258	

Optimum :

Bingham	: ó= óo+Eta*D	R»=0.92	óo=3.559	Eta=0.01168	

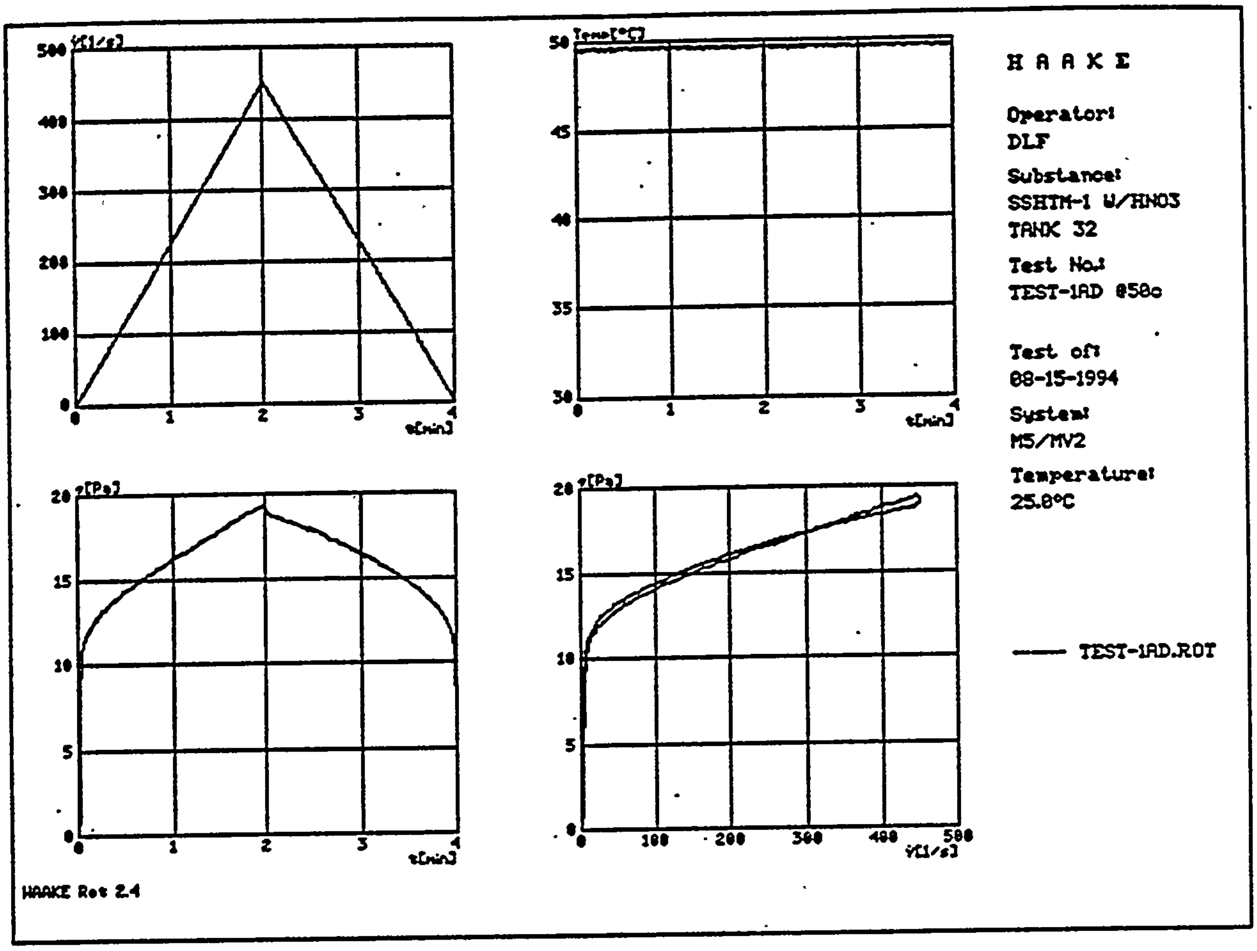
Rheogram and Data for Test Number HNO3OUT1 @50C DIPPED 2040hr




```
Bingham      : б= бo+Eta*D       R»=0.82   бo=19.96   Eta=0.0309
Ostwald      : б= a*D■           R»=0.75   a=7.703   n=0.2404
H.-Bulkley   : б= бo+a*D■        R»=0.90   a=8.929   n=0.2000   бo=1.715
Casson       : «б-«бo = «(Eta*D)   R»=0.76   бo=15.27   Eta=0.007891
Linear       : Y= a+b*X          R»=0.82   a=19.96   b=0.0309
Inv. linear: Y= 1/(a+b*X)        R»=0.05   a=0.06679   b=-107.5E-06
Exponential: Y= a*EXP(b*X)       R»=0.38   a=19.25   b=0.001377
Hyperbolic : Y= a+b/X            R»=0.30   a=27.26   b=-14.78
Logarithmic: Y= a+b*LN(X)        R»=0.96   a=5.520   b=4.195

Optimum :
Logarithmic: Y= a+b*LN(X)        R»=0.96   a=5.520   b=4.195
```

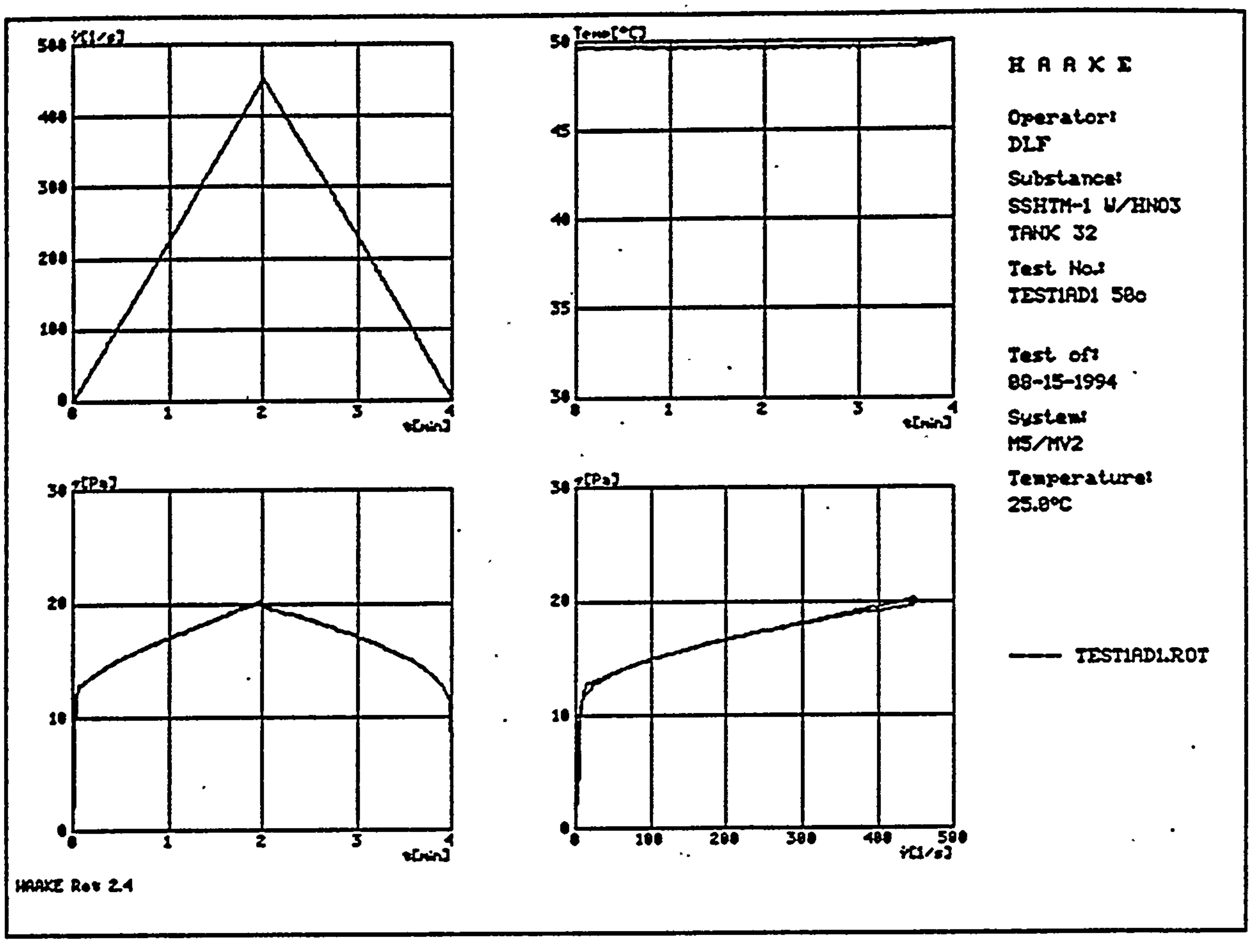
Rheogram and Data for Test Number TEST-1AD @50c



Bingham :	ó= óo+Eta*D	R»=0.84	óo=11.73	Eta=0.01838	
Ostwald :	ó= a*D■	R»=0.81	a=5.110	n=0.2179	
H.-Bulkley :	ó= óo+a*D■	R»=0.84	a=4.813	n=0.2104	óo=1.514
Casson :	«ó-«óo = «(Eta*D)	R»=0.80	óo=9.083	Eta=0.004534	
Linear :	Y= a+b*X	R»=0.84	a=11.73	b=0.01838	
Inv. linear:	Y= 1/(a+b*X)	R»=0.02	a=0.2381	b=-598.3E-06	
Exponential:	Y= a*EXP(b*X)	R»=0.26	a=10.54	b=0.001626	
Hyperbolic :	Y= a+b/X	R»=0.33	a=16.12	b=-12.34	
Logarithmic:	Y= a+b*LN(X)	R»=0.94	a=3.371	b=2.450	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.94 a=3.371 b=2.450

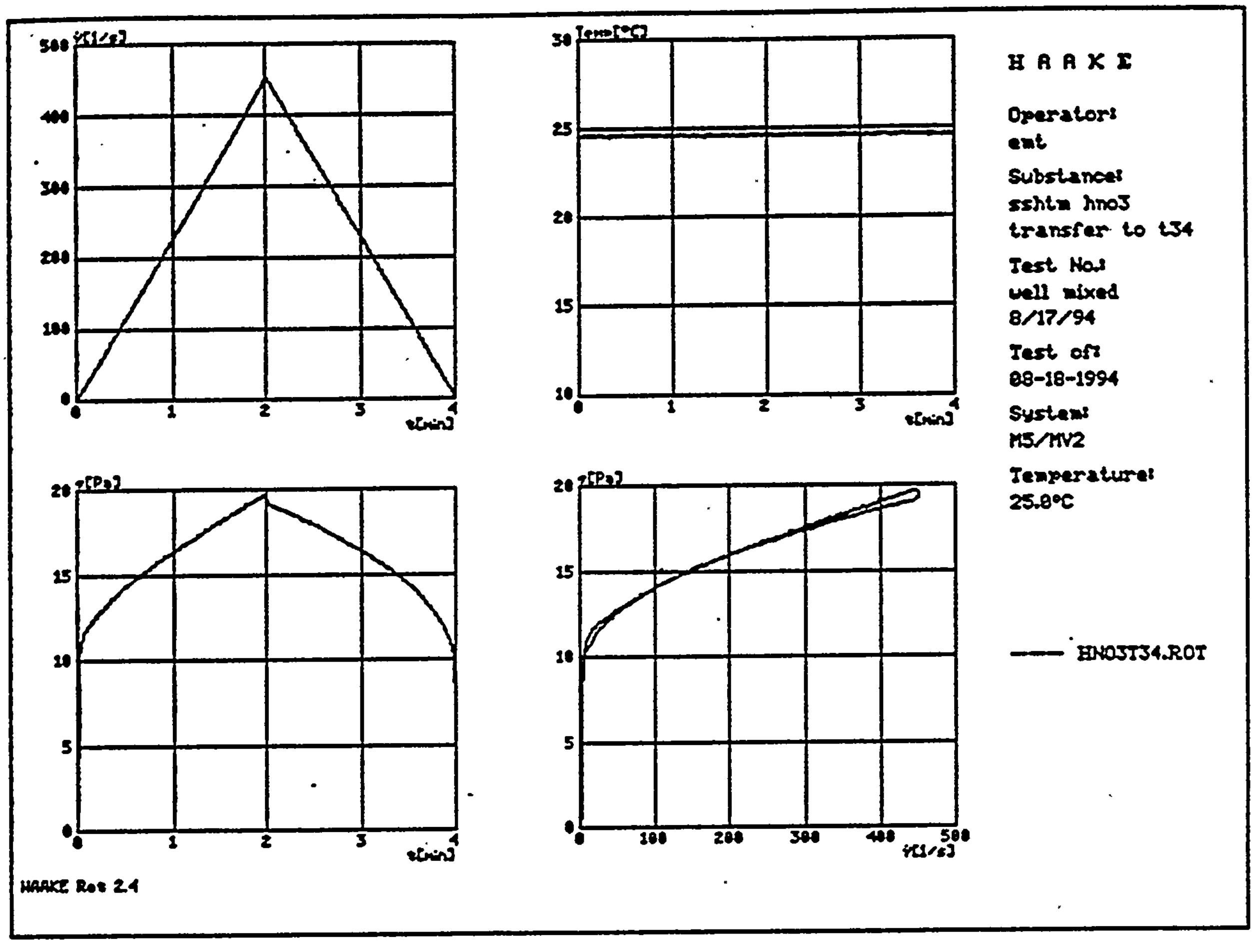
Rheogram and Data for Test Number TEST1AD1 50c




```
Bingham      : ő= őo+Eta*D       R»=0.86   őo=12.68   Eta=0.01793
Ostwald      : ő= a*D■           R»=0.87   a=6.526    n=0.1813
H.-Bulkley   : ő= őo+a*D■        R»=0.55   a=2.767    n=0.3103   őo=2.714
Casson       : «ő-«őo = «(Eta*D)   R»=0.84   őo=10.19   Eta=0.003889
Linear       : Y= a+b*X          R»=0.86   a=12.68    b=0.01793
Inv. linear: Y= 1/(a+b*X)        R»=0.13   a=0.09245  b=-121.9E-06
Exponential: Y= a*EXP(b*X)       R»=0.45   a=12.12    b=0.001314
Hyperbolic : Y= a+b/X            R»=0.30   a=16.95    b=-11.62
Logarithmic: Y= a+b*LN(X)        R»=0.93   a=4.614    b=2.372

Optimum :
Logarithmic: Y= a+b*LN(X)        R»=0.93   a=4.614    b=2.372
```

Rheogram and Data for Test Number well mixed 8/17/94

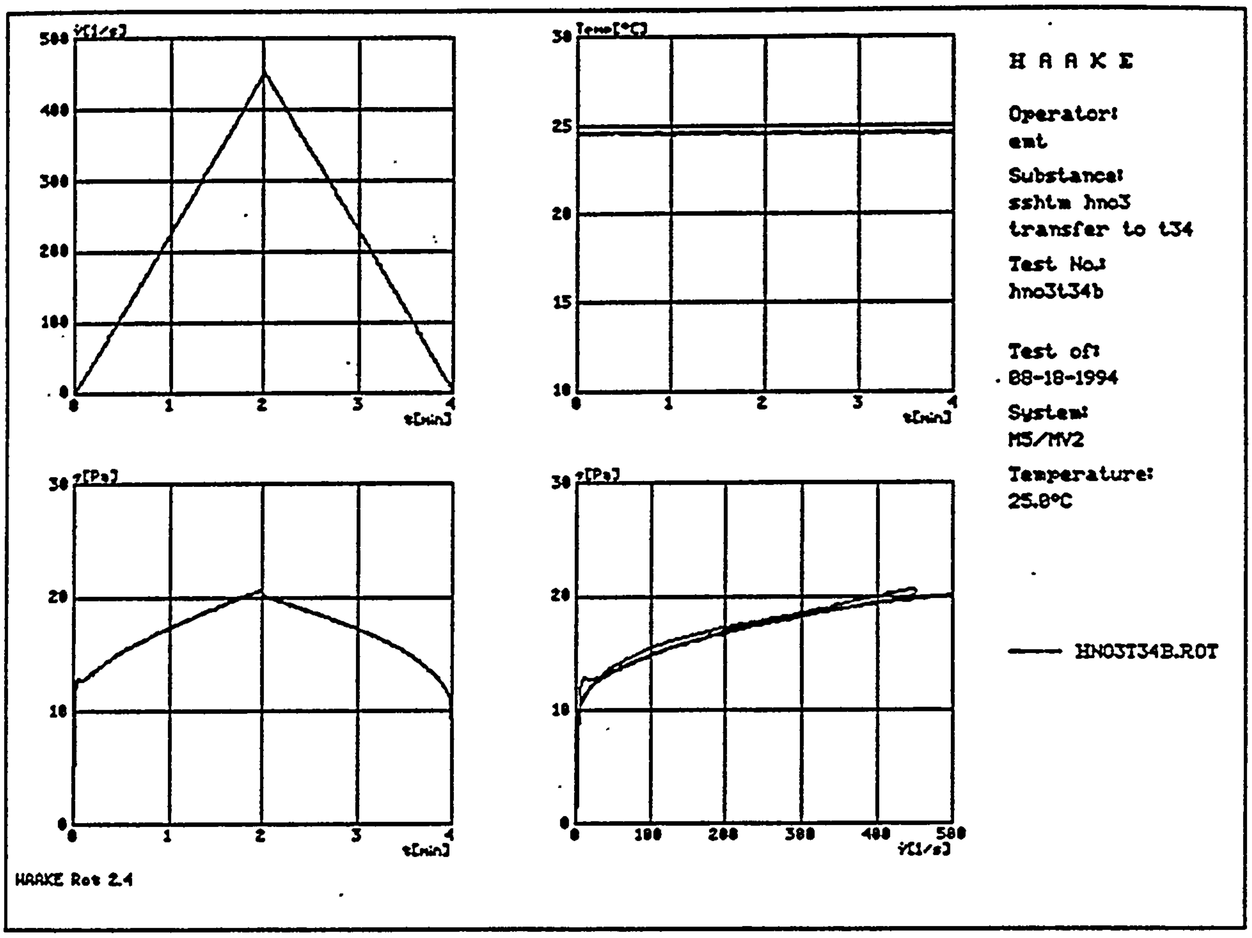


Bingham	: ó= óo+Eta*D	R»=0.89	óo=11.77	Eta=0.0190	
Ostwald	: ó= a*D▪	R»=0.81	a=5.384	n=0.2103	
H.-Bulkley	: ó= óo+a*D▪	R»=0.96	a=4.162	n=0.2141	óo=3.387
Casson	: «ó-«óo = «(Eta*D)	R»=0.84	óo=9.212	Eta=0.004555	
Linear	: Y= a+b*X	R»=0.89	a=11.77	b=0.0190	
Inv. linear	: Y= 1/(a+b*X)	R»=0.03	a=0.2025	b=-482.2E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.28	a=10.72	b=0.00161	
Hyperbolic	: Y= a+b/X	R»=0.21	a=16.17	b=-3.617	
Logarithmic	: Y= a+b*LN(X)	R»=0.92	a=3.958	b=2.373	

Optimum :

H.-Bulkley	: ó= óo+a*D▪	R»=0.96	a=4.162	n=0.2141	óo=3.387

Rheogram and Data for Test Number hno3t34b

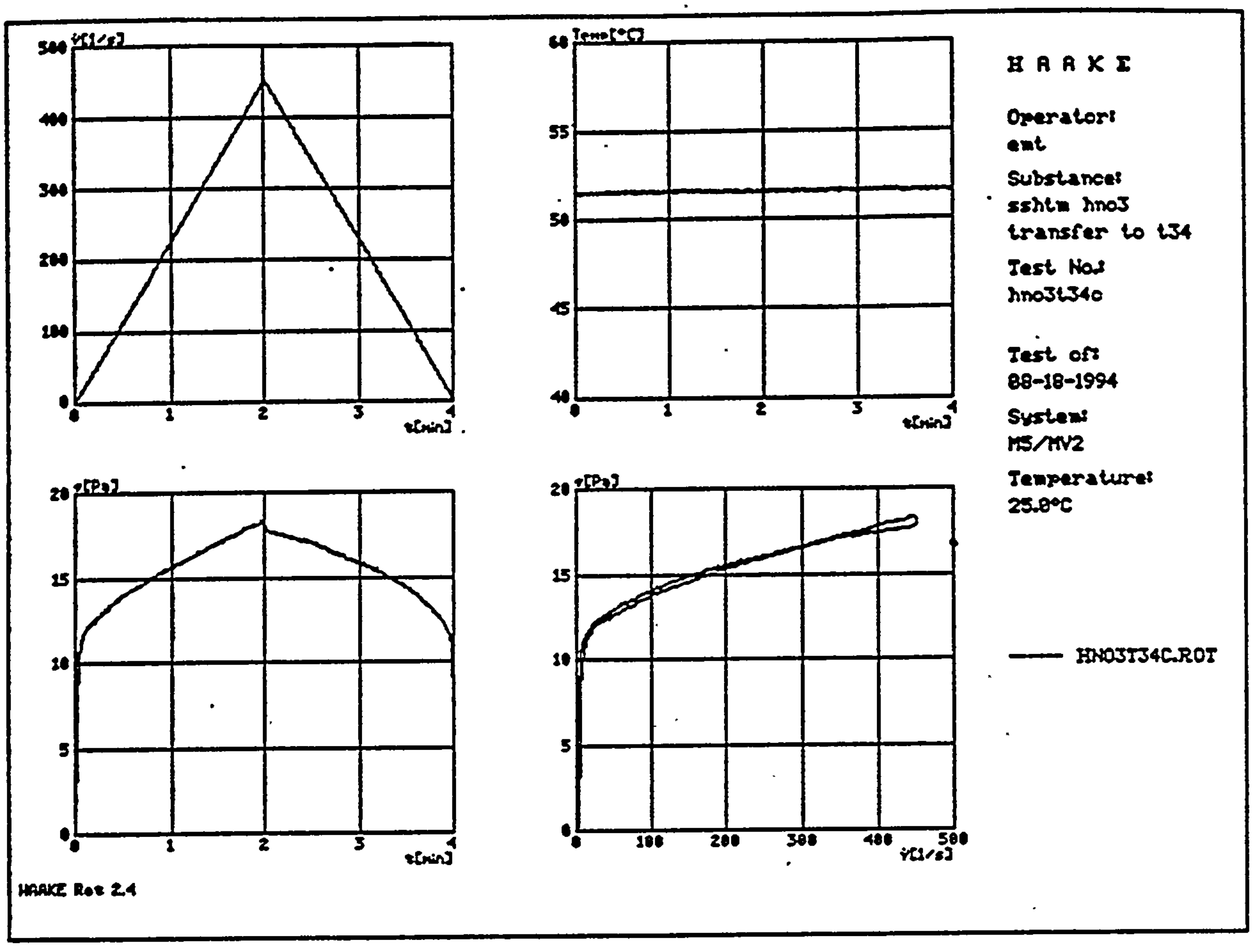


Bingham :	ó= óo+Eta*D	R»=0.89	óo=12.66	Eta=0.01918	
Ostwald :	ó= a*D■	R»=0.87	a=6.554	n=0.1834	
H.-Bulkley :	ó= óo+a*D■	R»=0.94	a=4.182	n=0.2143	óo=4.230
Casson :	«ó-«óo = «(Eta*D)	R»=0.86	óo=10.12	Eta=0.004275	
Linear :	Y= a+b*X	R»=0.89	a=12.66	b=0.01918	
Inv. linear:	Y= 1/(a+b*X)	R»=0.07	a=0.1024	b=-158.3E-06	
Exponential:	Y= a*EXP(b*X)	R»=0.40	a=11.99	b=0.001411	
Hyperbolic :	Y= a+b/X	R»=0.21	a=17.10	b=-3.980	
Logarithmic:	Y= a+b*LN(X)	R»=0.91	a=4.834	b=2.381	

Optimum :

H.-Bulkley :	ó= óo+a*D■	R»=0.94	a=4.182	n=0.2143	óo=4.230

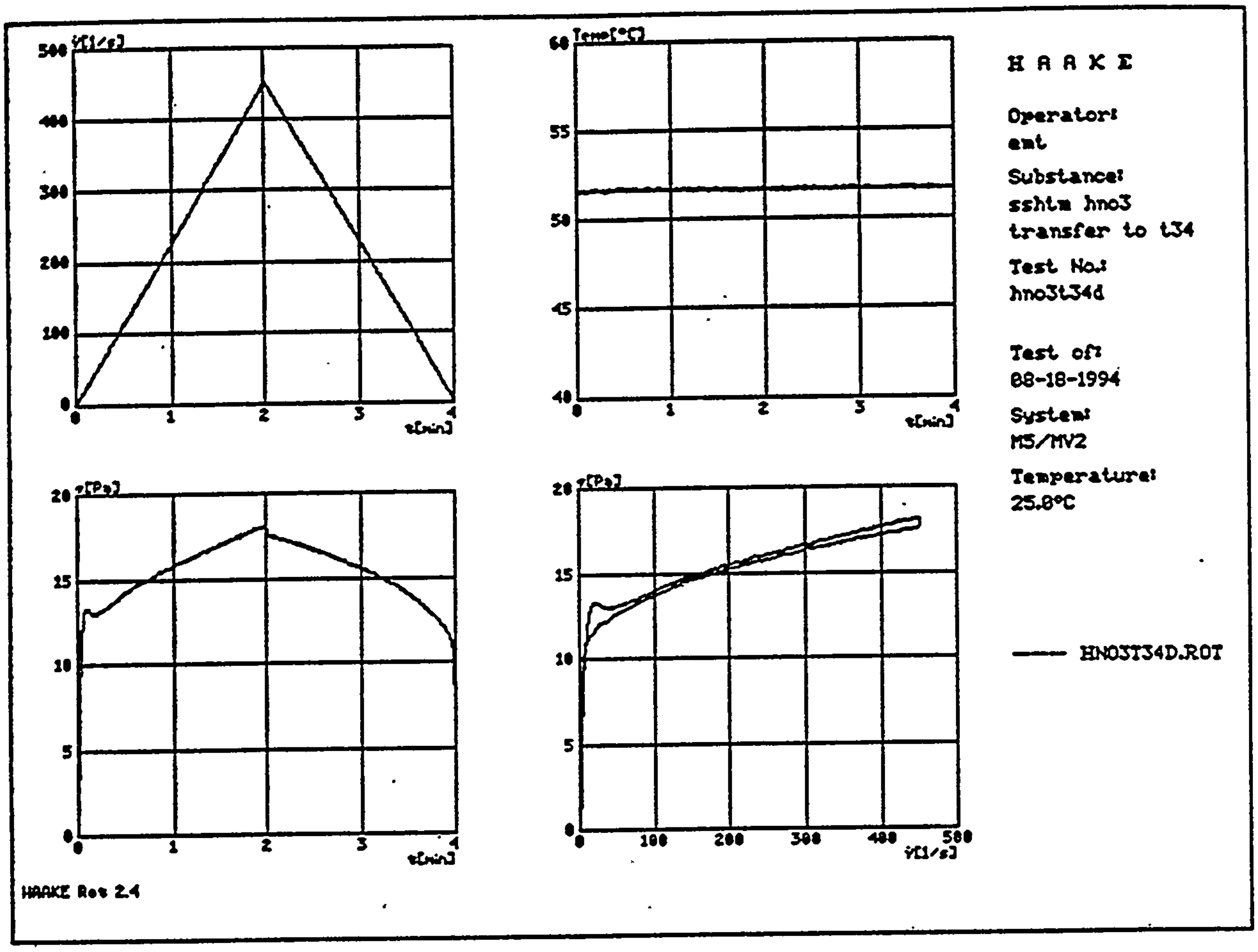
Rheogram and Data for Test Number hno3t34c




```
Bingham      : ó= óo+Eta*D      R»=0.81   óo=11.67   Eta=0.01621
Ostwald      : ó= a*D∎          R»=0.69   a=4.601   n=0.2314
H.-Bulkley   : ó= óo+a*D∎       R»=0.94   a=5.637   n=0.1761   óo=1.297
Casson       : «ó-«óo = «(Eta*D)  R»=0.74   óo=9.160   Eta=0.003797
Linear       : Y= a+b*X         R»=0.81   a=11.67   b=0.01621
Inv. linear: Y= 1/(a+b*X)       R»=0.02   a=1.454   b=-0.004644
Exponential: Y= a*EXP(b*X)      R»=0.16   a=9.859   b=0.001711
Hyperbolic : Y= a+b/X           R»=0.27   a=15.45   b=-4.255
Logarithmic: Y= a+b*LN(X)       R»=0.96   a=4.180   b=2.185

Optimum :
Logarithmic: Y= a+b*LN(X)       R»=0.96   a=4.180   b=2.185
```

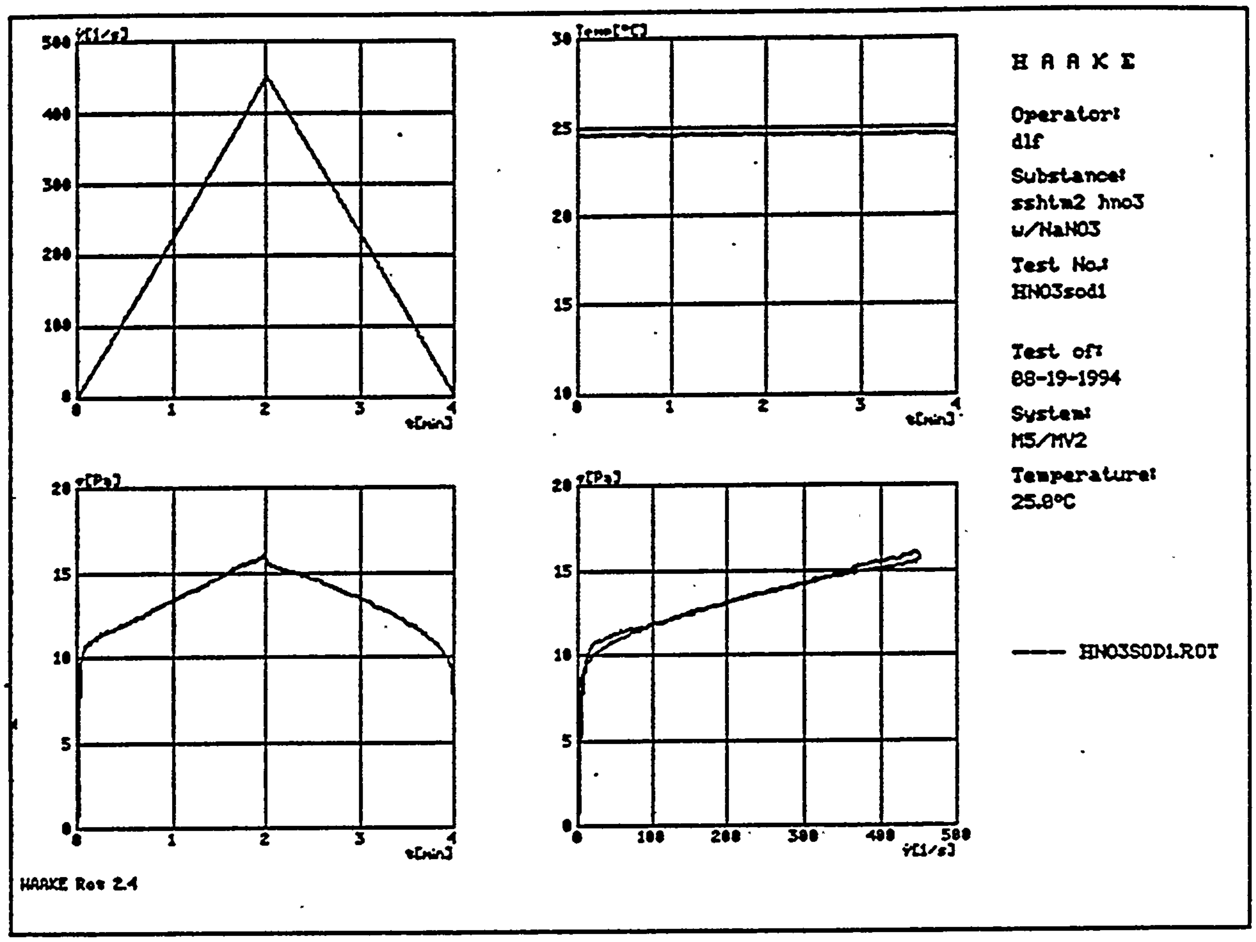
Rheogram and Data for Test Number hno3t34d



Bingham : ó= óo+Eta*D R»=0.77 óo=12.30 Eta=0.01422
Ostwald : ó= a*D■ R»=0.79 a=6.299 n=0.1738
H.-Bulkley : ó= óo+a*D■ R»=0.82 a=5.822 n=0.1639 óo=1.915
Casson : «ó-«óo = «(Eta*D) R»=0.73 óo=10.11 Eta=0.00279
Linear : Y= a+b*X R»=0.77 a=12.30 b=0.01422
Inv. linear: Y= 1/(a+b*X) R»=0.07 a=0.1105 b=-0.000166
Exponential: Y= a*EXP(b*X) R»=0.30 a=11.46 b=0.001219
Hyperbolic : Y= a+b/X R»=0.29 a=15.63 b=-4.367
Logarithmic: Y= a+b*LN(X) R»=0.92 a=5.703 b=1.923

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.92 a=5.703 b=1.923

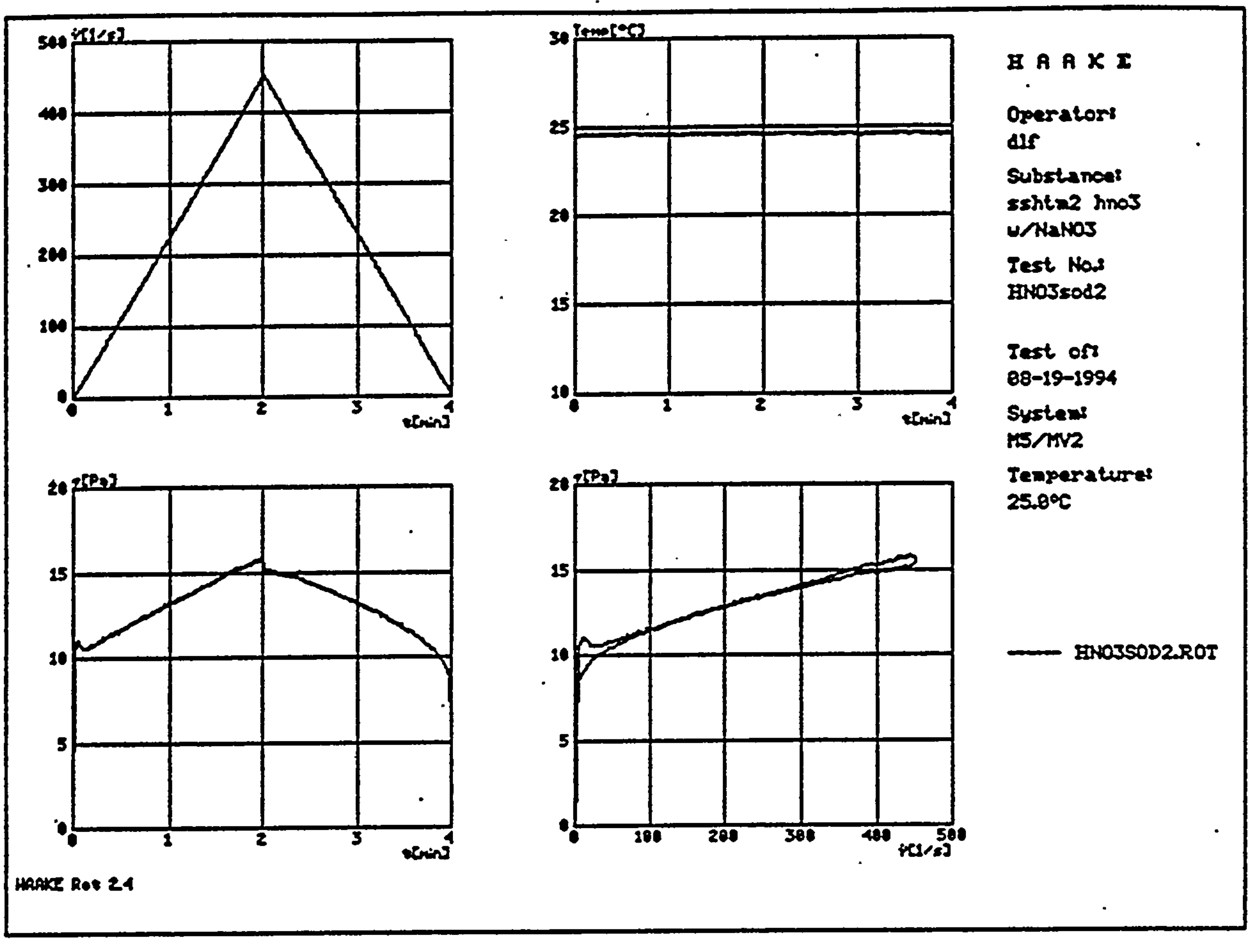
Rheogram and Data for Test Number HNO3sod1




```
Bingham      : ó= óo+Eta*D      R»=0.81  óo=10.14   Eta=0.01386
Ostwald      : ó= a*D■          R»=0.77  a=4.863   n=0.1932
H.-Bulkley   : ó= óo+a*D■       R»=0.49  a=2.309   n=0.3135   óo=1.378
Casson       : «ó-«óo = «(Eta*D)   R»=0.76  óo=8.133   Eta=0.003014
Linear       : Y= a+b*X         R»=0.81  a=10.14   b=0.01386
Inv. linear: Y= 1/(a+b*X)       R»=0.02  a=0.3013   b=-769.1E-06
Exponential: Y= a*EXP(b*X)      R»=0.23  a=9.104   b=0.00149
Hyperbolic : Y= a+b/X           R»=0.36  a=13.47   b=-9.763
Logarithmic: Y= a+b*LN(X)       R»=0.91  a=3.866   b=1.842

Optimum :
Logarithmic: Y= a+b*LN(X)       R»=0.91  a=3.866   b=1.842
```

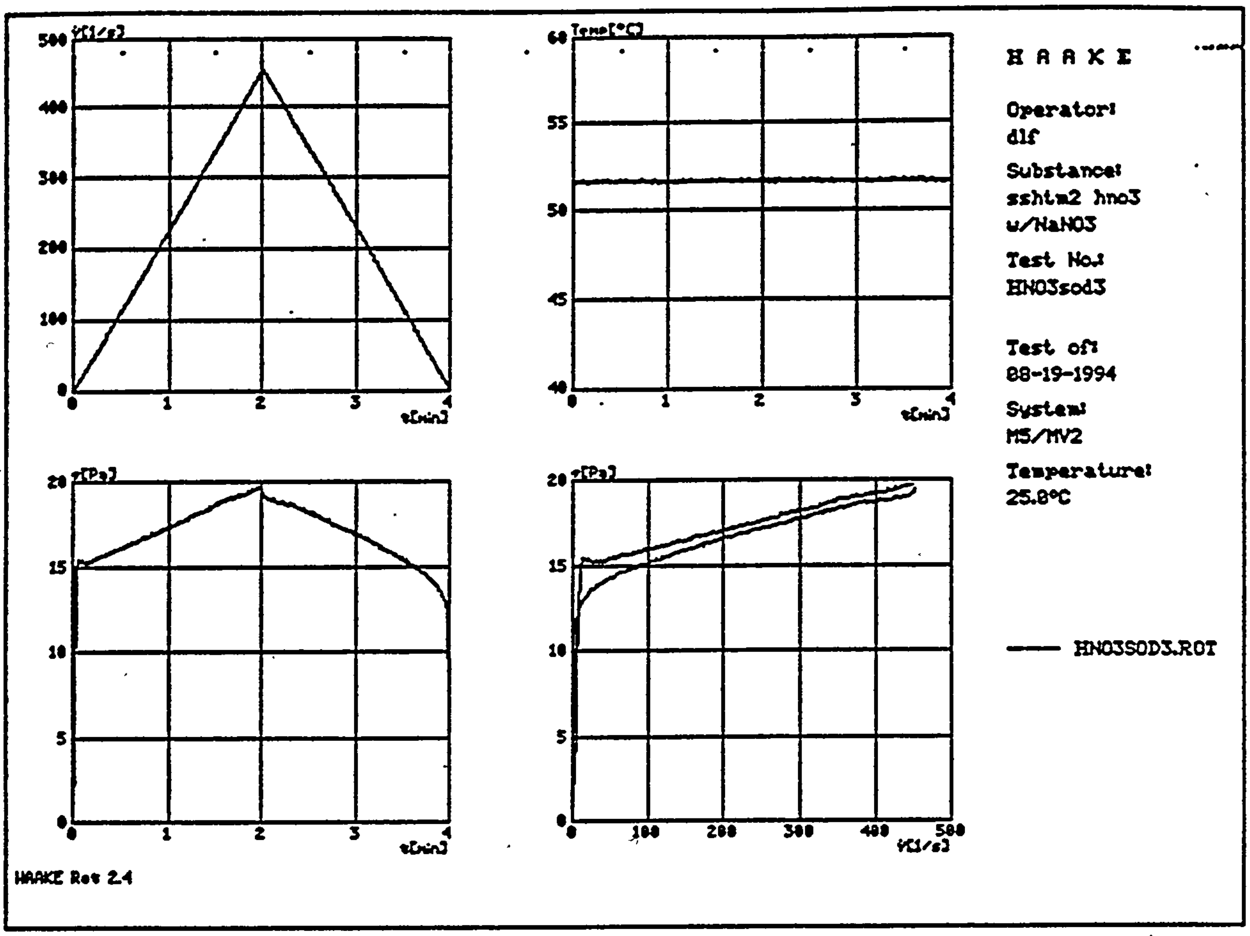
Rheogram and Data for Test Number HNO3sod2




```
Bingham     : ó= óo+Eta*D      R»=0.88   óo=10.14   Eta=0.0132
Ostwald     : ó= a*D■          R»=0.79   a=5.831   n=0.1564
H.-Bulkley  : ó= óo+a*D■       R»=0.86   a=3.612   n=0.1823   óo=3.812
Casson      : «ó-«óo = «(Eta*D)   R»=0.81   óo=8.444   Eta=0.002487
Linear      : Y= a+b*X         R»=0.88   a=10.14   b=0.0132
Inv. linear: Y= 1/(a+b*X)     R»=0.08   a=0.1223   b=-0.000169
Exponential: Y= a*EXP(b*X)    R»=0.38   a=9.662   b=0.001245
Hyperbolic : Y= a+b/X         R»=0.24   a=13.21   b=-3.129
Logarithmic: Y= a+b*LN(X)     R»=0.84   a=4.997   b=1.593

Optimum :
Bingham     : ó= óo+Eta*D      R»=0.88   óo=10.14   Eta=0.0132
```

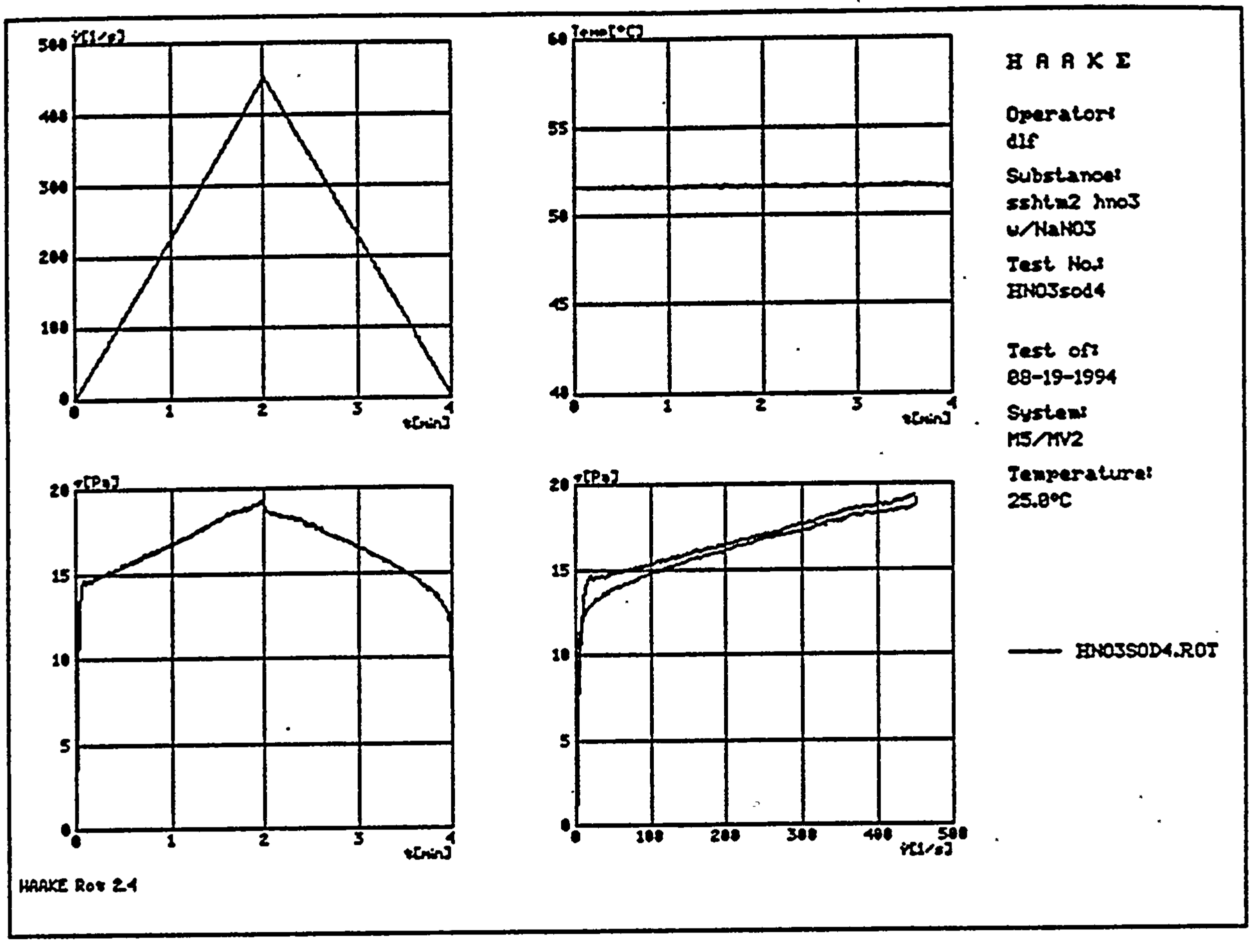
Rheogram and Data for Test Number HNO3sod3




```
Bingham      : ó= óo+Eta*D      R»=0.68  óo=14.31   Eta=0.01287
Ostwald      : ó= a*D■          R»=0.69  a=8.081   n=0.1459
H.-Bulkley   : ó= óo+a*D■       R»=0.62  a=6.183   n=0.1787  óo=1.567
Casson       : «ó-«óo = «(Eta*D)  R»=0.63  óo=12.20  Eta=0.002084
Linear       : Y= a+b*X         R»=0.68  a=14.31   b=0.01287
Inv. linear: Y= 1/(a+b*X)      R»=0.09  a=0.08563  b=-101.2E-06
Exponential: Y= a*EXP(b*X)     R»=0.29  a=13.47   b=991.3E-06
Hyperbolic  : Y= a+b/X          R»=0.45  a=17.45   b=-11.82
Logarithmic: Y= a+b*LN(X)      R»=0.86  a=7.842   b=1.836

Optimum :
Logarithmic: Y= a+b*LN(X)      R»=0.86  a=7.842   b=1.836
```

Rheogram and Data for Test Number HNO3sod4

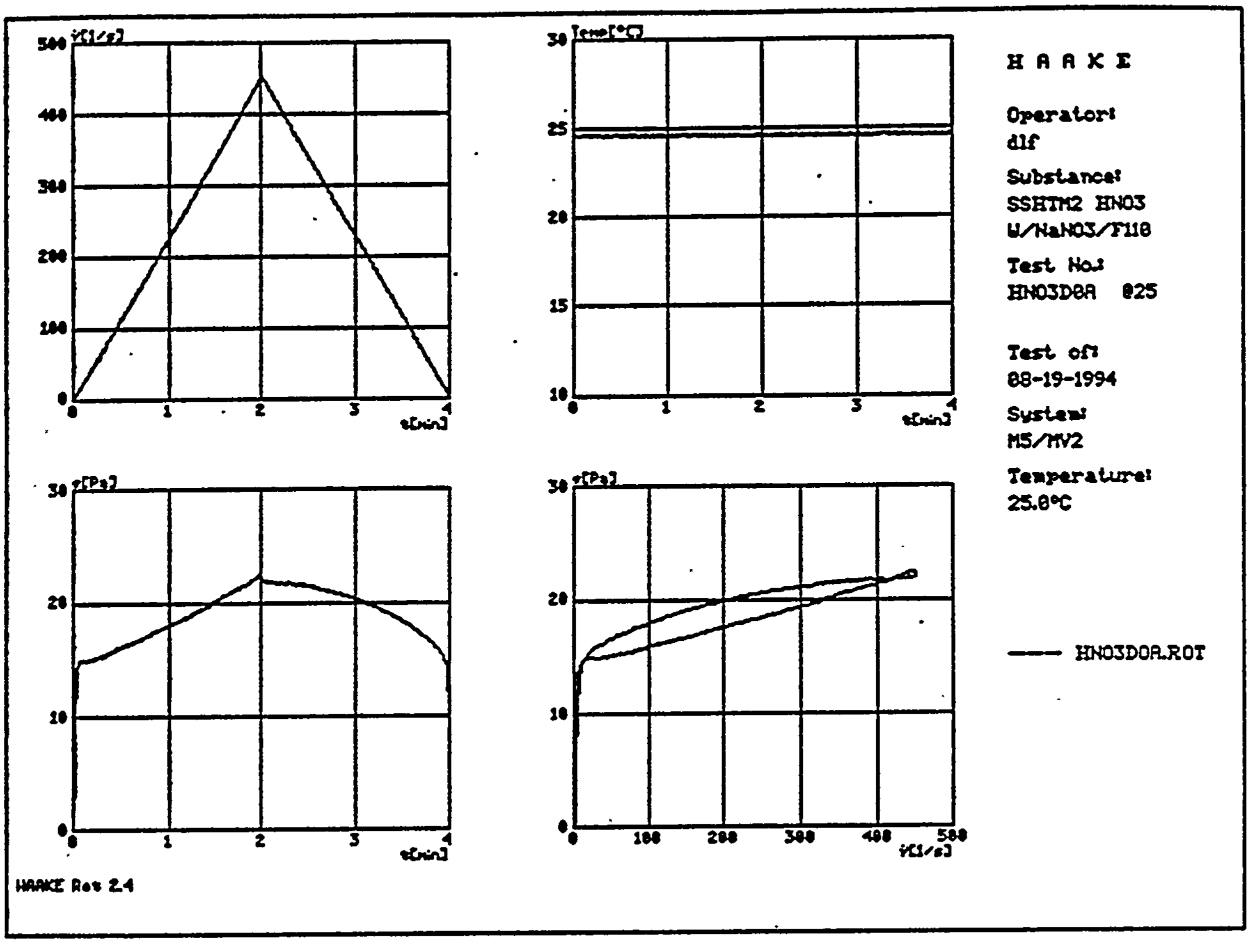


Bingham	: ó= óo+Eta*D	R»=0.71	óo=13.66	Eta=0.01334	
Ostwald	: ó= a*D■	R»=0.71	a=7.199	n=0.1620	
H.-Bulkley	: ó= óo+a*D■	R»=0.77	a=7.080	n=0.1406	óo=2.039
Casson	: «ó-«óo = «(Eta*D)	R»=0.64	óo=11.49	Eta=0.002325	
Linear	: Y= a+b*X	R»=0.71	a=13.66	b=0.01334	
Inv. linear	: Y= 1/(a+b*X)	R»=0.05	a=0.1043	b=-0.000158	
Exponential	: Y= a*EXP(b*X)	R»=0.25	a=12.64	b=0.001107	
Hyperbolic	: Y= a+b/X	R»=0.35	a=16.80	b=-4.558	
Logarithmic	: Y= a+b*LN(X)	R»=0.89	a=7.233	b=1.850	

Optimum :

Logarithmic	: Y= a+b*LN(X)	R»=0.89	a=7.233	b=1.850

Rheogram and Data for Test Number HNO3D0A @25

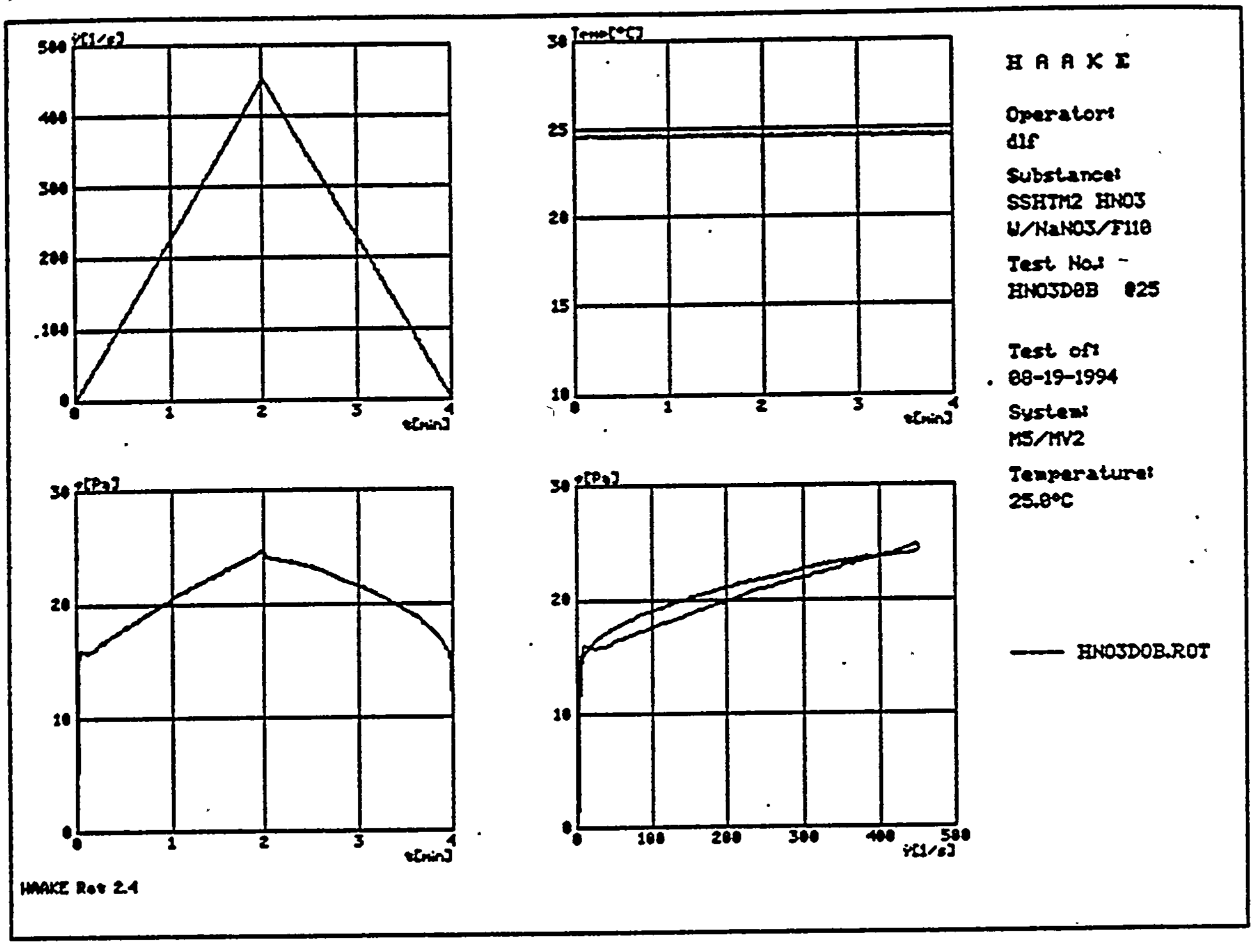


Bingham	: ó= óo+Eta*D	R»=0.83	óo=13.58	Eta=0.01962	
Ostwald	: ó= a*D■	R»=0.66	a=5.755	n=0.2192	
H.-Bulkley	: ó= óo+a*D■	R»=0.82	a=7.045	n=0.1654	óo=1.412
Casson	: «ó-«óo = «(Eta*D)	R»=0.71	óo=10.84	Eta=0.004343	
Linear	: Y= a+b*X	R»=0.83	a=13.58	b=0.01962	
Inv. linear	: Y= 1/(a+b*X)	R»=0.02	a=0.8100	b=-0.002527	
Exponential	: Y= a*EXP(b*X)	R»=0.17	a=11.68	b=0.001685	
Hyperbolic	: Y= a+b/X	R»=0.26	a=18.15	b=-5.157	
Logarithmic	: Y= a+b*LN(X)	R»=0.86	a=5.446	b=2.463	

Optimum :

Logarithmic	: Y= a+b*LN(X)	R»=0.86	a=5.446	b=2.463

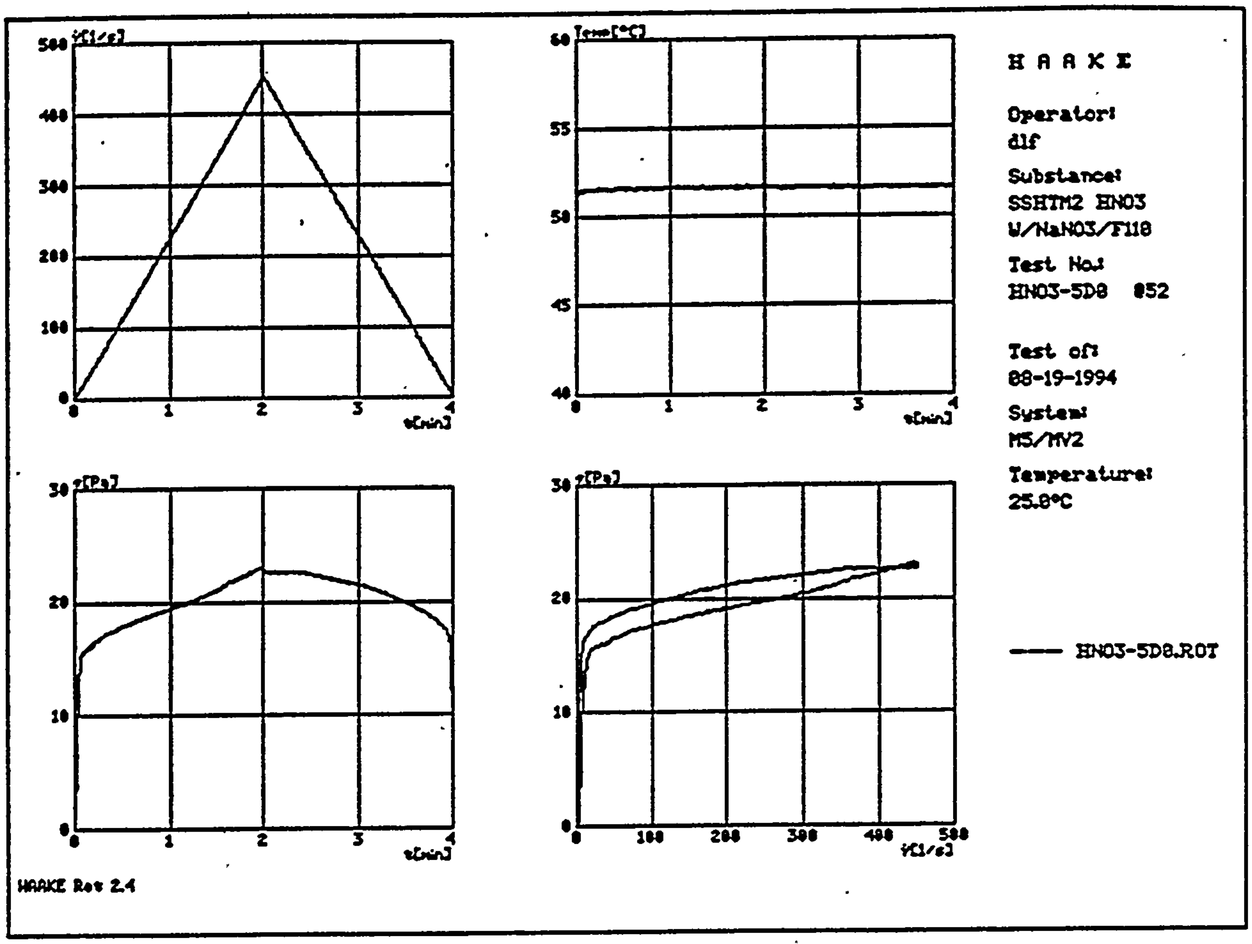
Rheogram and Data for Test Number HNO3D0B @25




```
Bingham      : ó= óo+Eta*D      R»=0.88   óo=15.13   Eta=0.02243
Ostwald      : ó= a*D■          R»=0.79   a=7.651    n=0.1868
H.-Bulkley   : ó= óo+a*D■       R»=0.87   a=5.966    n=0.1912   óo=4.078
Casson       : «ó-«óo = «(Eta*D)    R»=0.81   óo=12.17   Eta=0.004862
Linear       : Y= a+b*X         R»=0.88   a=15.13    b=0.02243
Inv. linear  : Y= 1/(a+b*X)     R»=0.06   a=0.09441  b=-160.5E-06
Exponential  : Y= a*EXP(b*X)    R»=0.35   a=14.19    b=0.001423
Hyperbolic   : Y= a+b/X         R»=0.23   a=20.35    b=-6.060
Logarithmic  : Y= a+b*LN(X)     R»=0.87   a=6.063    b=2.768

Optimum :
Bingham      : ó= óo+Eta*D      R»=0.88   óo=15.13   Eta=0.02243
```

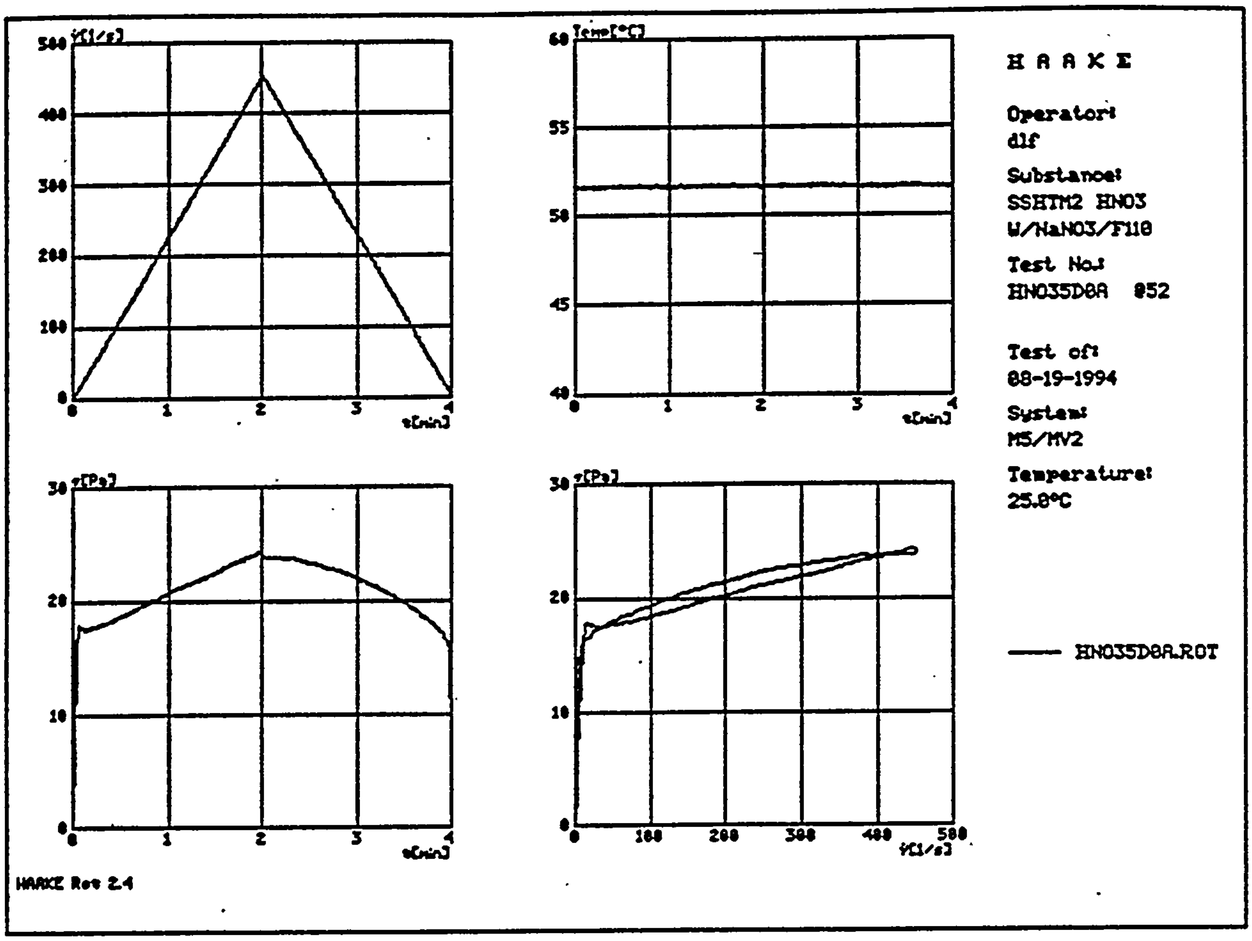
Rheogram and Data for Test Number HNO3-5D0 @52



Bingham	: ő= őo+Eta*D	R»=0.75	őo=15.05	Eta=0.01857	
Ostwald	: ő= a*D■	R»=0.65	a=6.032	n=0.2230	
H.-Bulkley	: ő= őo+a*D■	R»=0.88	a=8.197	n=0.1565	őo=0.8297
Casson	: «ő-«őo = «(Eta*D)	R»=0.67	őo=12.03	Eta=0.004051	
Linear	: Y= a+b*X	R»=0.75	a=15.05	b=0.01857	
Inv. linear	: Y= 1/(a+b*X)	R»=0.02	a=1.619	b=-0.005233	
Exponential	: Y= a*EXP(b*X)	R»=0.13	a=12.55	b=0.001643	
Hyperbolic	: Y= a+b/X	R»=0.31	a=19.41	b=-6.583	
Logarithmic	: Y= a+b*LN(X)	R»=0.93	a=6.097	b=2.576	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.93 a=6.097 b=2.576

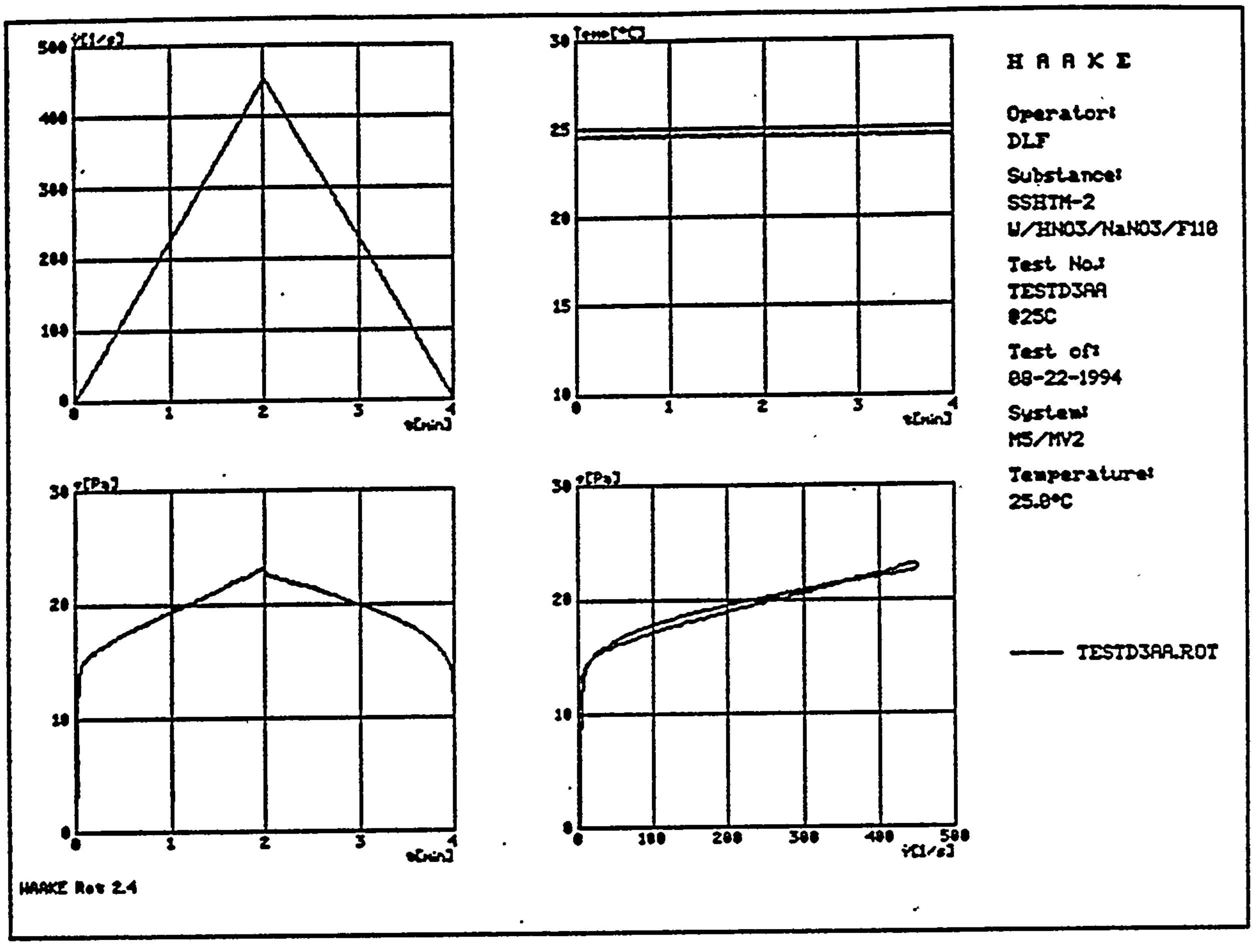
Rheogram and Data for Test Number HNO35D0A @52




```
Bingham     : ó= óo+Eta*D      R»=0.76  óo=16.15   Eta=0.01918
Ostwald     : ó= a*D▪          R»=0.75  a=7.999  n=0.1811
H.-Bulkley  : ó= óo+a*D▪       R»=0.67  a=6.168  n=0.2178  óo=1.326
Casson      : «ó-«óo = «(Eta*D)  R»=0.69  óo=13.22  Eta=0.003813
Linear      : Y= a+b*X         R»=0.76  a=16.15  b=0.01918
Inv. linear: Y= 1/(a+b*X)      R»=0.07  a=0.08432  b=-127.8E-06
Exponential: Y= a*EXP(b*X)     R»=0.31  a=15.02  b=0.001246
Hyperbolic : Y= a+b/X          R»=0.30  a=20.66  b=-6.599
Logarithmic: Y= a+b*LN(X)      R»=0.88  a=7.363  b=2.571

Optimum :
Logarithmic: Y= a+b*LN(X)      R»=0.88  a=7.363  b=2.571
```

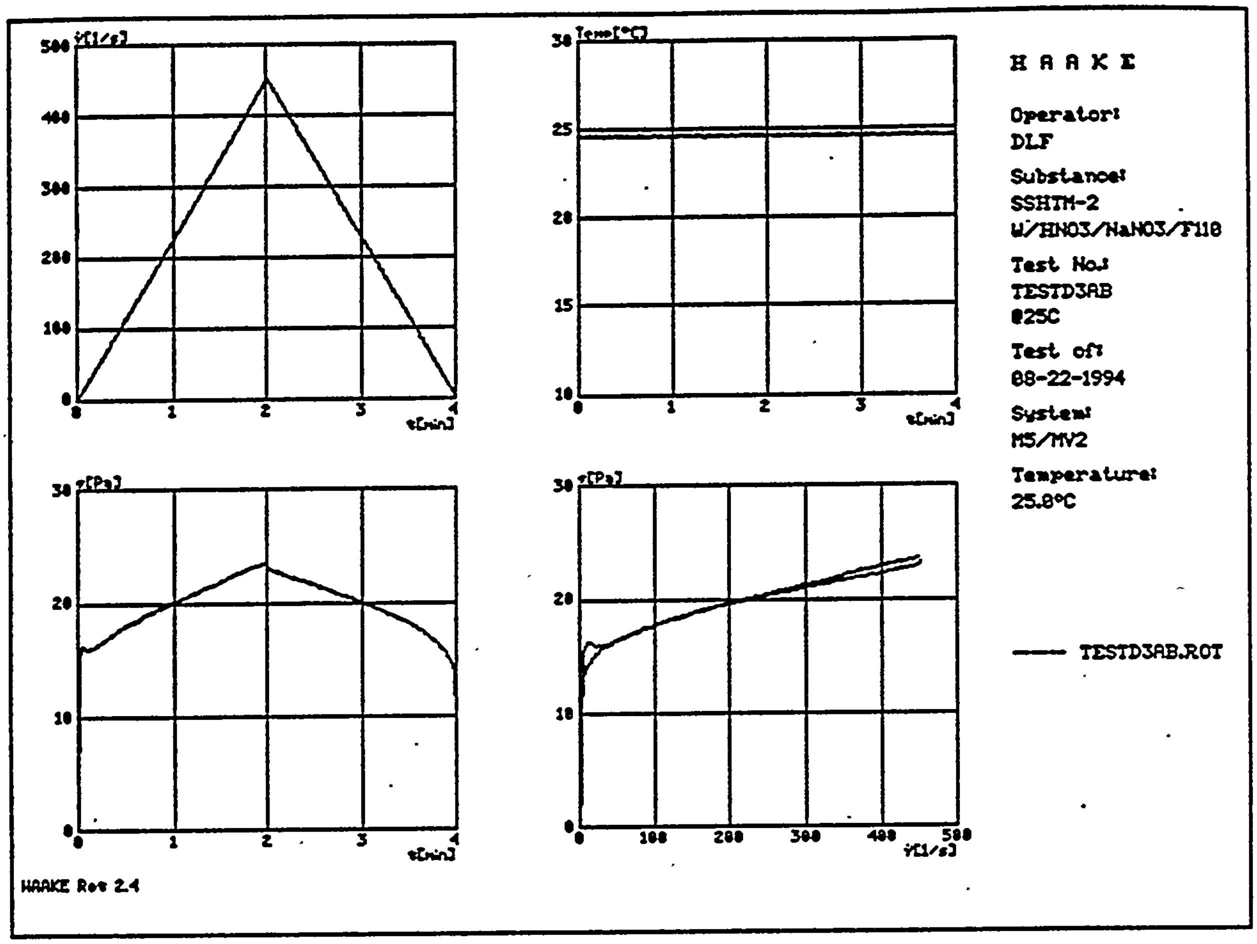
Rheogram and Data for Test Number TESTD3AA @25C




```
Bingham      : б= бo+Eta*D       R»=0.81   бo=14.60   Eta=0.01999
Ostwald      : б= a*D■           R»=0.62   a=5.495   n=0.2395
H.-Bulkley   : б= бo+a*D■        R»=0.88   a=7.198   n=0.1708   бo=1.675
Casson       : «б-«бo = «(Eta*D)    R»=0.70   бo=11.53   Eta=0.004573
Linear       : Y= a+b*X          R»=0.81   a=14.60   b=0.01999
Inv. linear: Y= 1/(a+b*X)        R»=0.02   a=0.8623   b=-0.002711
Exponential: Y= a*EXP(b*X)       R»=0.16   a=12.32   b=0.0017
Hyperbolic : Y= a+b/X            R»=0.29   a=19.27   b=-5.934
Logarithmic: Y= a+b*LN(X)        R»=0.93   a=5.622   b=2.644

Optimum :
Logarithmic: Y= a+b*LN(X)        R»=0.93   a=5.622   b=2.644
```

Rheogram and Data for Test Number TESTD3AB @25C

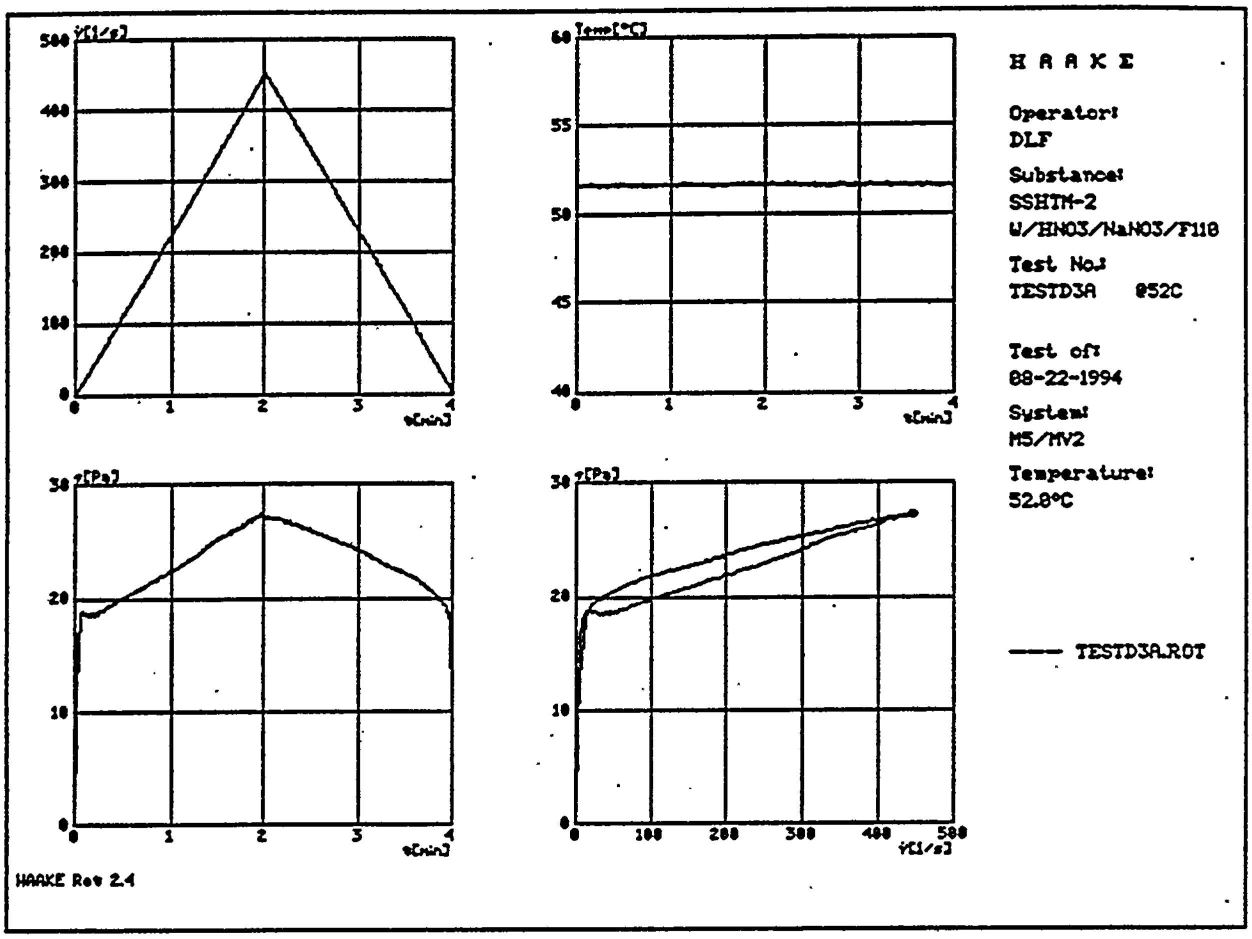


Bingham	: ó= óo+Eta*D	R»=0.86	óo=15.54	Eta=0.0190	
Ostwald	: ó= a*D■	R»=0.79	a=8.830	n=0.1561	
H.-Bulkley	: ó= óo+a*D■	R»=0.90	a=5.527	n=0.1799	óo=5.780
Casson	: «ó-«óo = «(Eta*D)	R»=0.81	óo=12.95	Eta=0.003534	
Linear	: Y= a+b*X	R»=0.86	a=15.54	b=0.0190	
Inv. linear	: Y= 1/(a+b*X)	R»=0.07	a=0.08242	b=-116.6E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.35	a=14.71	b=0.001212	
Hyperbolic	: Y= a+b/X	R»=0.26	a=19.97	b=-5.070	
Logarithmic	: Y= a+b*LN(X)	R»=0.88	a=7.726	b=2.372	

Optimum :

H.-Bulkley	: ó= óo+a*D■	R»=0.90	a=5.527	n=0.1799	óo=5.780

Rheogram and Data for Test Number TESTD3A @52C

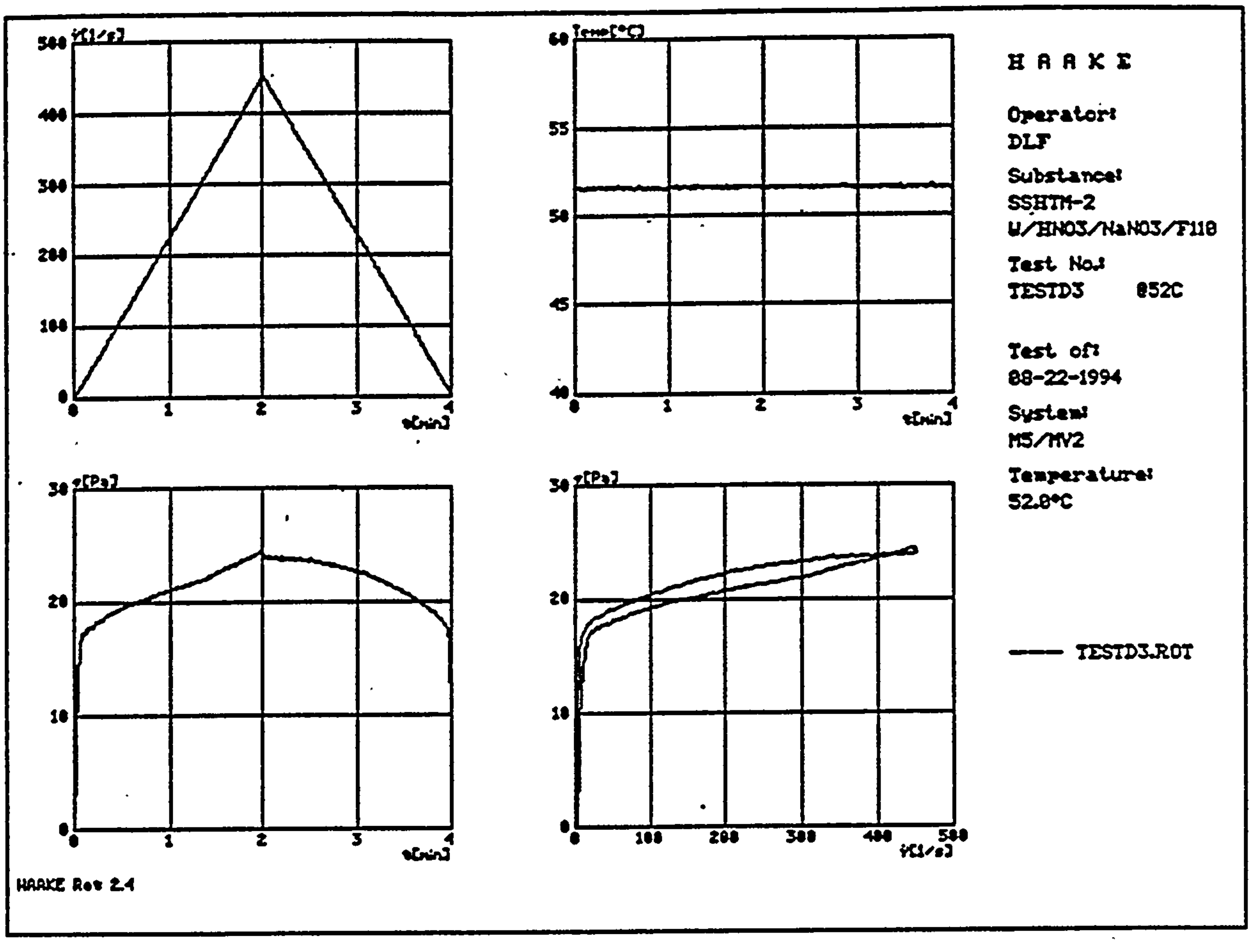


Bingham	: ó= óo+Eta*D	R»=0.89	óo=17.09	Eta=0.02383	
Ostwald	: ó= a*D■	R»=0.86	a=9.788	n=0.1600	
H.-Bulkley	: ó= óo+a*D■	R»=0.80	a=5.698	n=0.2211	óo=4.408
Casson	: «ó-«óo = «(Eta*D)	R»=0.87	óo=14.09	Eta=0.004706	
Linear	: Y= a+b*X	R»=0.89	a=17.09	b=0.02383	
Inv. linear	: Y= 1/(a+b*X)	R»=0.19	a=0.0638	b=-73.66E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.54	a=16.63	b=0.001238	
Hyperbolic	: Y= a+b/X	R»=0.39	a=23.02	b=-37.13	
Logarithmic	: Y= a+b*LN(X)	R»=0.87	a=6.864	b=3.052	

Optimum :

Bingham	: ó= óo+Eta*D	R»=0.89	óo=17.09	Eta=0.02383

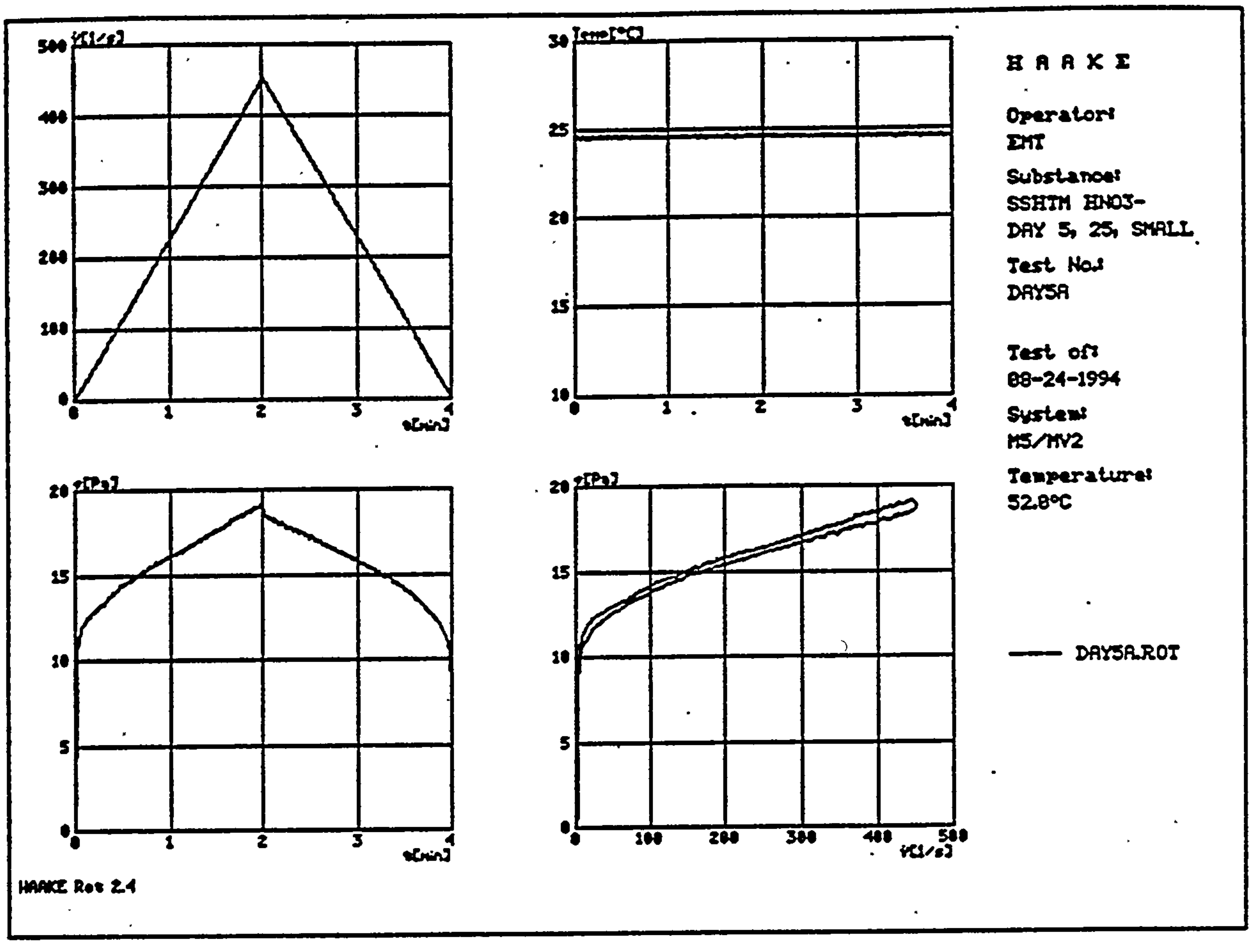
Rheogram and Data for Test Number TESTD3 @52C




```
Bingham     : ó= óo+Eta*D      R»=0.69  óo=16.53   Eta=0.01848
Ostwald     : ó= a*D■          R»=0.50  a=4.675   n=0.2855
H.-Bulkley  : ó= óo+a*D■       R»=0.84  a=9.467   n=0.1479  óo=0.3832
Casson      : «ó-«óo = «(Eta*D)  R»=0.57  óo=13.14   Eta=0.004092
Linear      : Y= a+b*X         R»=0.69  a=16.53   b=0.01848
Inv. linear: Y= 1/(a+b*X)      R»=0.02  a=0.3376  b=-973.2E-06
Exponential: Y= a*EXP(b*X)     R»=0.15  a=15.07   b=0.001263
Hyperbolic  : Y= a+b/X         R»=0.32  a=20.86   b=-4.946
Logarithmic: Y= a+b*LN(X)      R»=0.94  a=7.142   b=2.658

Optimum :
Logarithmic: Y= a+b*LN(X)      R»=0.94  a=7.142   b=2.658
```

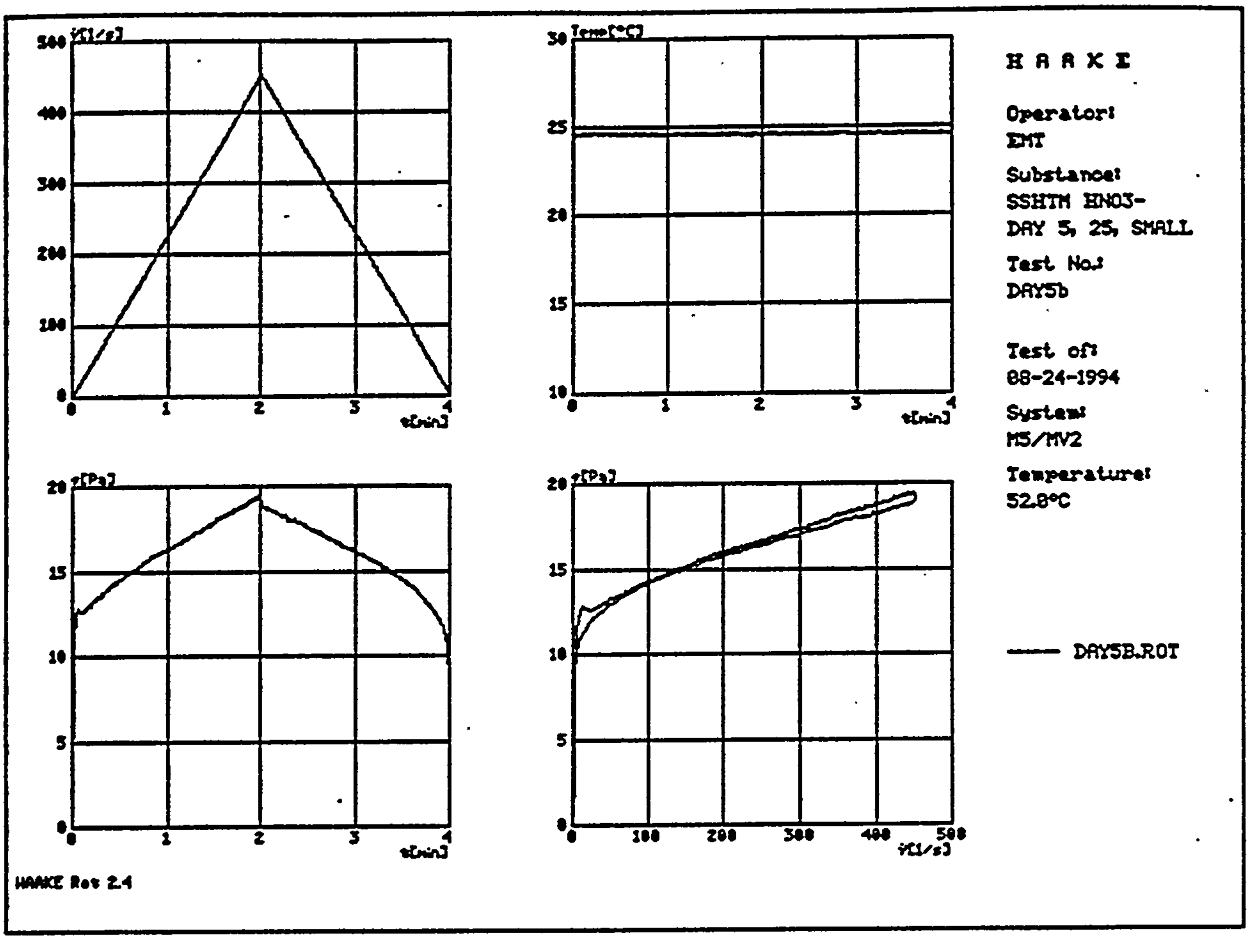
Rheogram and Data for Test Number DAY5A




```
Bingham      : б= бo+Eta*D        R»=0.85   бo=12.09   Eta=0.01674
Ostwald      : б= a*D■            R»=0.76   a=5.670    n=0.1982
H.-Bulkley   : б= бo+a*D■         R»=0.95   a=4.770    n=0.1883   бo=3.207
Casson       : «б-«бo = «(Eta*D)  R»=0.78   бo=9.691   Eta=0.003656
Linear       : Y= a+b*X           R»=0.85   a=12.09    b=0.01674
Inv. linear  : Y= 1/(a+b*X)       R»=0.02   a=3.291    b=-0.01077
Exponential  : Y= a*EXP(b*X)      R»=0.14   a=10.23    b=0.001701
Hyperbolic   : Y= a+b/X           R»=0.25   a=15.98    b=-3.824
Logarithmic  : Y= a+b*LN(X)       R»=0.93   a=4.890    b=2.152

Optimum :
H.-Bulkley   : б= бo+a*D■         R»=0.95   a=4.770    n=0.1883   бo=3.207
```

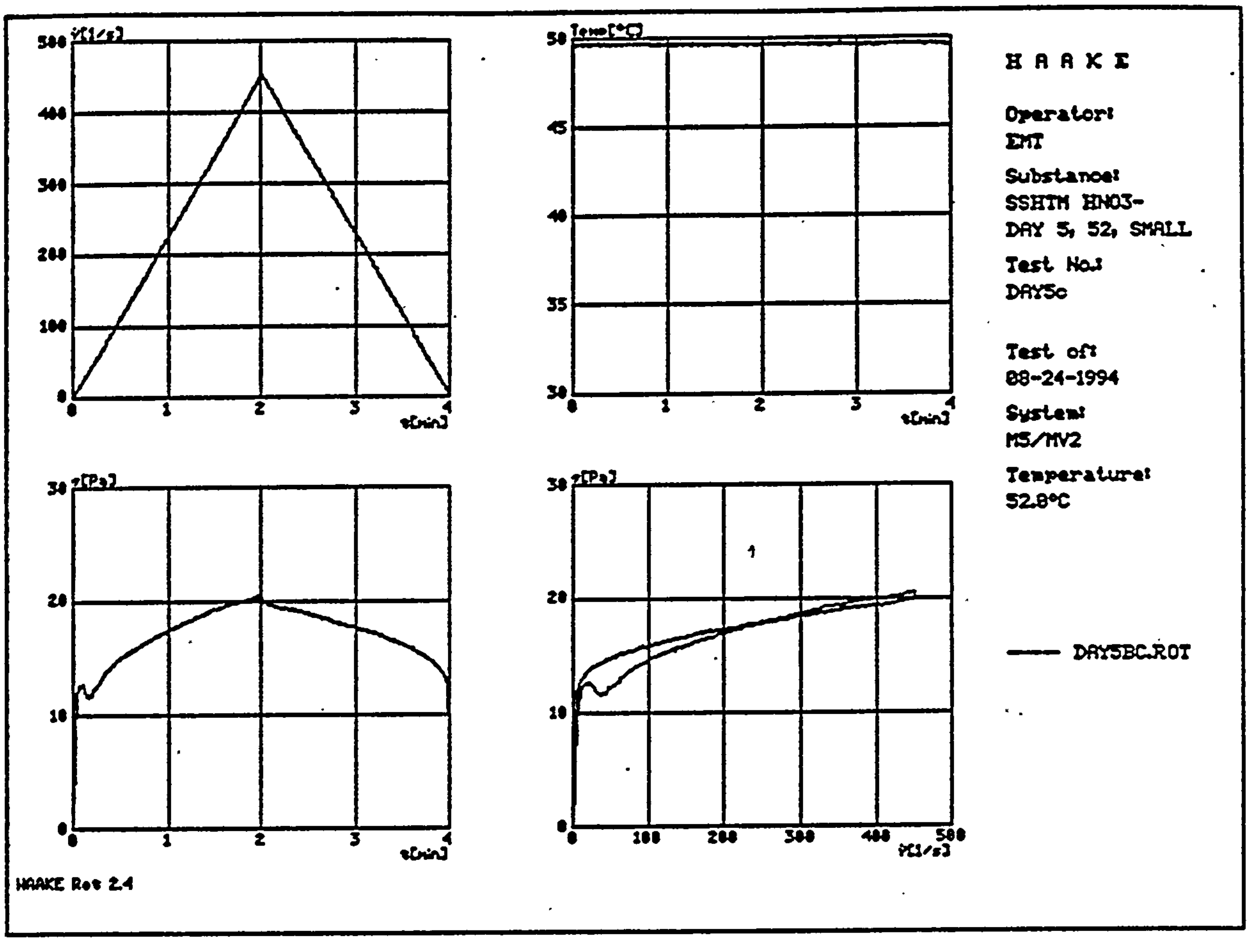
Rheogram and Data for Test Number DAY5b



Bingham	: ő= őo+Eta*D	R»=0.88	őo=12.37	Eta=0.01663	
Ostwald	: ő= a*D■	R»=0.83	a=6.786	n=0.1668	
H.-Bulkley	: ő= őo+a*D■	R»=0.92	a=4.424	n=0.1917	őo=4.150
Casson	: «ő-«őo = «(Eta*D)	R»=0.84	őo=10.14	Eta=0.003339	
Linear	: Y= a+b*X	R»=0.88	a=12.37	b=0.01663	
Inv. linear	: Y= 1/(a+b*X)	R»=0.08	a=0.1012	b=-143.6E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.39	a=11.75	b=0.001285	
Hyperbolic	: Y= a+b/X	R»=0.25	a=16.25	b=-4.953	
Logarithmic	: Y= a+b*LN(X)	R»=0.90	a=5.477	b=2.086	

Optimum :
H.-Bulkley : ő= őo+a*D■ R»=0.92 a=4.424 n=0.1917 őo=4.150

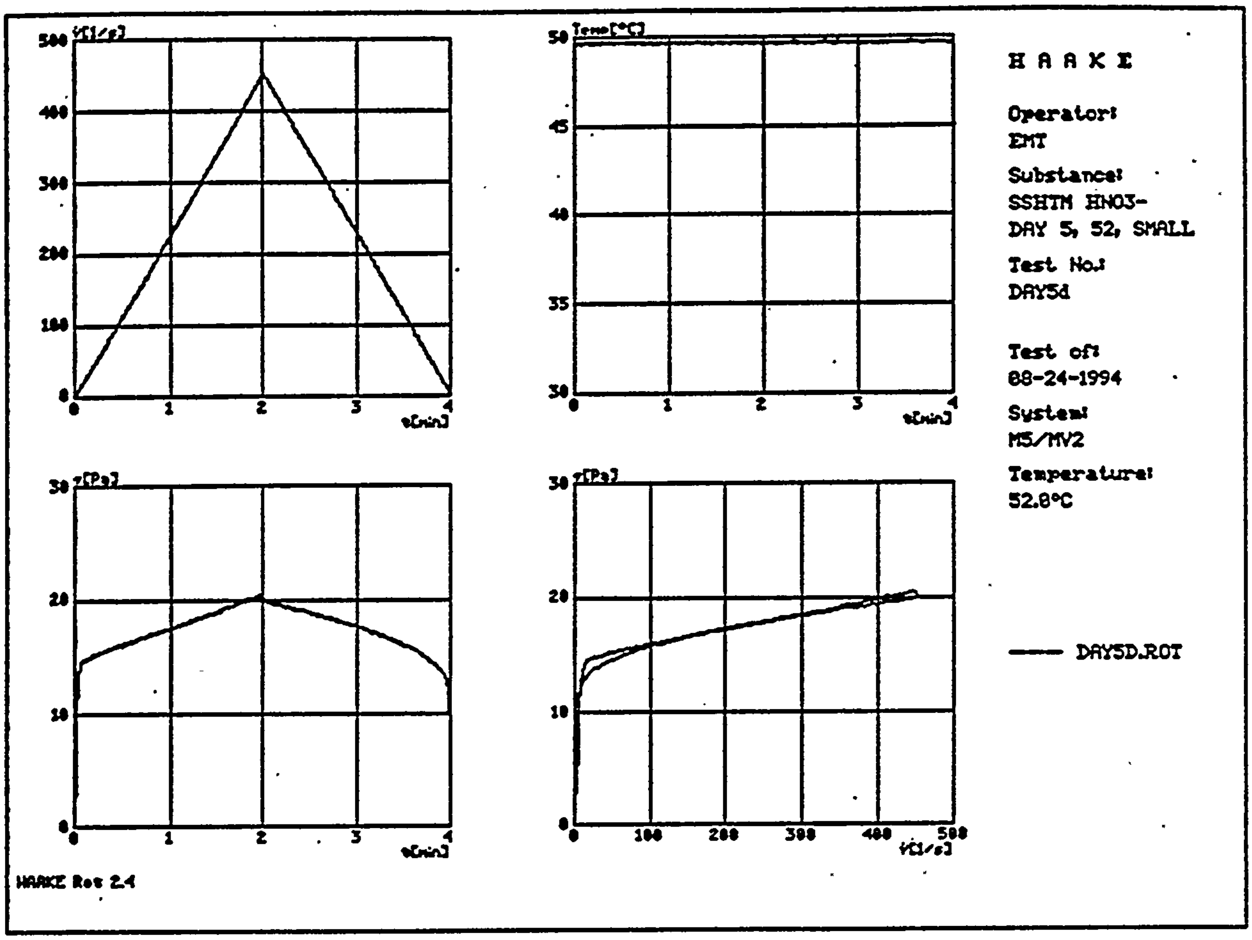
Rheogram and Data for Test Number DAY5c




```
Bingham     : ó= óo+Eta*D      R»=0.88   óo=12.00   Eta=0.02128
Ostwald     : ó= a*D■          R»=0.92   a=5.661    n=0.2091
H.-Bulkley  : ó= óo+a*D■       R»=0.92   a=3.991    n=0.2410   óo=2.779
Casson      : «ó-«óo = «(Eta*D)   R»=0.89   óo=9.158   Eta=0.005542
Linear      : Y= a+b*X         R»=0.88   a=12.00    b=0.02128
Inv. linear: Y= 1/(a+b*X)      R»=0.12   a=0.1004   b=-151.3E-06
Exponential: Y= a*EXP(b*X)     R»=0.50   a=11.45    b=0.00156
Hyperbolic : Y= a+b/X          R»=0.19   a=16.94    b=-5.884
Logarithmic: Y= a+b*LN(X)      R»=0.92   a=2.943    b=2.716

Optimum :
Logarithmic: Y= a+b*LN(X)      R»=0.92   a=2.943    b=2.716
```

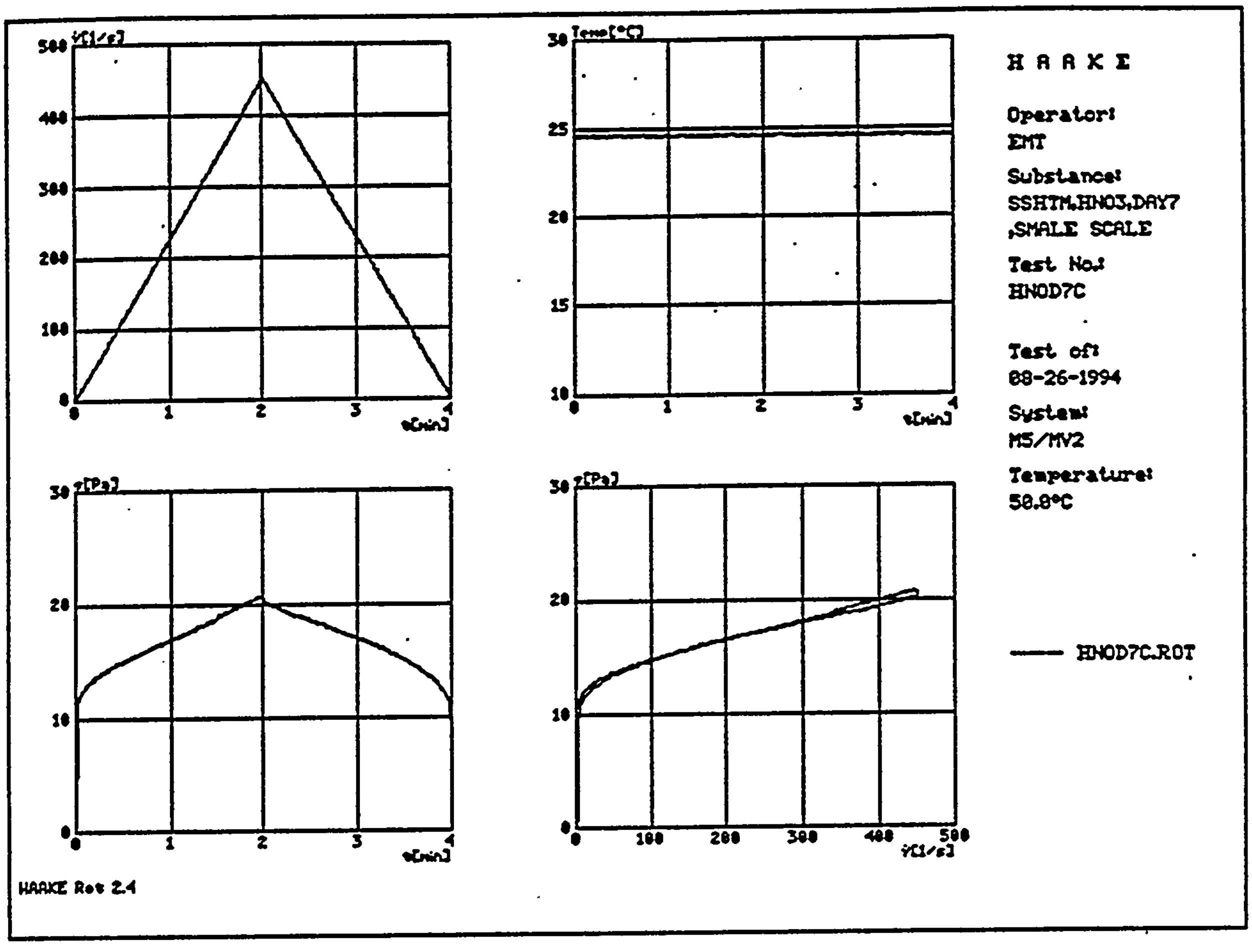
Rheogram and Data for Test Number DAY5d




```
Bingham     : ó= óo+Eta*D      R»=0.80  óo=14.06   Eta=0.01482
Ostwald     : ó= a*D■          R»=0.80  a=8.172   n=0.1460
H.-Bulkley  : ó= óo+a*D■       R»=0.42  a=2.849   n=0.3037   óo=3.468
Casson      : «ó-«óo = «(Eta*D)  R»=0.77  óo=11.89   Eta=0.002547
Linear      : Y= a+b*X         R»=0.80  a=14.06   b=0.01482
Inv. linear: Y= 1/(a+b*X)     R»=0.13  a=0.08091  b=-88.55E-06
Exponential: Y= a*EXP(b*X)    R»=0.40  a=13.48   b=0.001049
Hyperbolic : Y= a+b/X         R»=0.38  a=17.63   b=-11.75
Logarithmic: Y= a+b*LN(X)     R»=0.90  a=7.156   b=2.008

Optimum :
Logarithmic: Y= a+b*LN(X)     R»=0.90  a=7.156   b=2.008
```

Rheogram and Data for Test Number HNOD7C

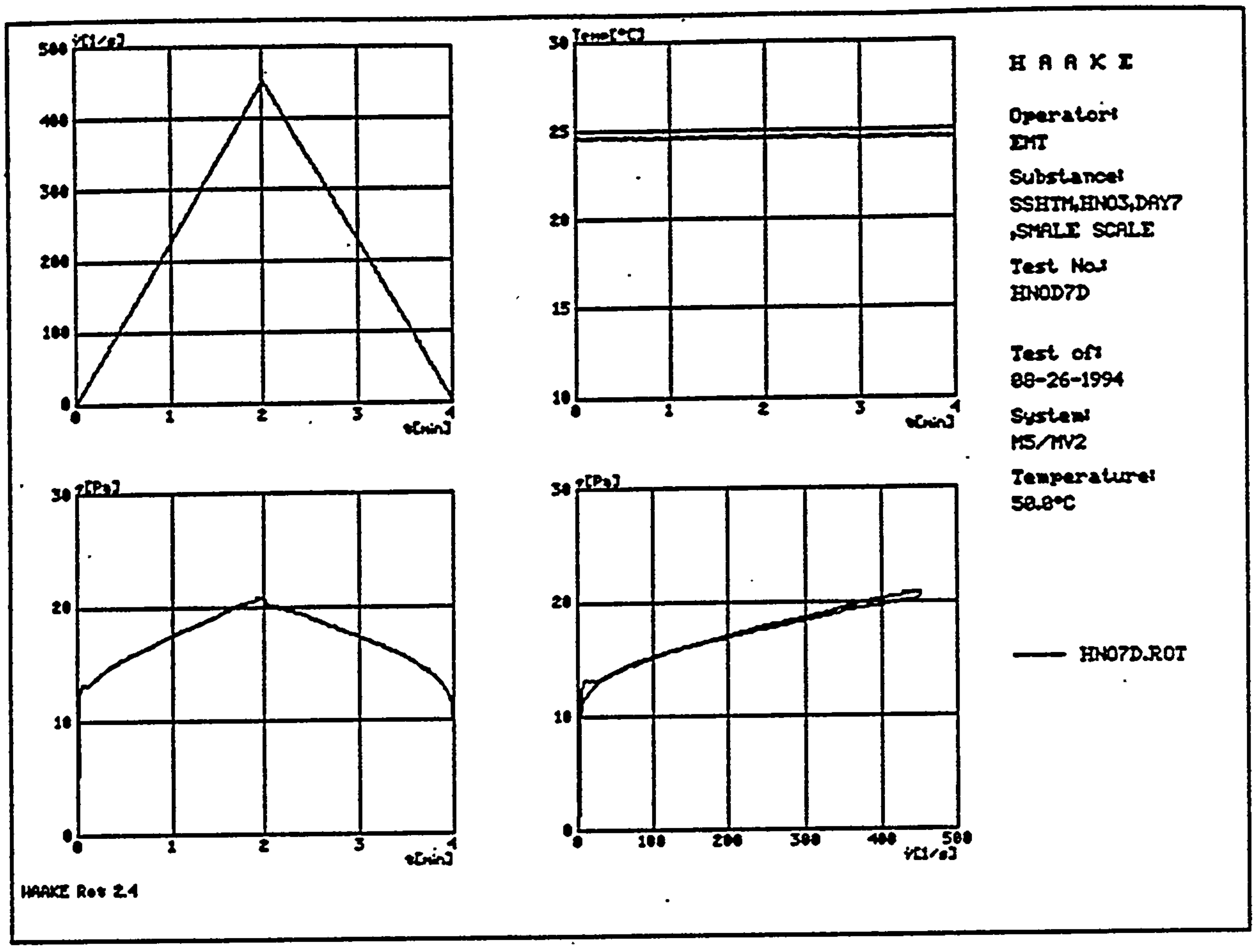


Bingham	: б= бo+Eta*D	R»=0.89	бo=12.47	Eta=0.01902	
Ostwald	: б= a*D■	R»=0.76	a=5.910	n=0.2006	
H.-Bulkley	: б= бo+a*D■	R»=0.94	a=4.624	n=0.2000	бo=3.688
Casson	: «б-«бo = «(Eta*D)	R»=0.81	бo=9.931	Eta=0.004283	
Linear	: Y= a+b*X	R»=0.89	a=12.47	b=0.01902	
Inv. linear	: Y= 1/(a+b*X)	R»=0.02	a=1.422	b=-0.004554	
Exponential	: Y= a*EXP(b*X)	R»=0.17	a=10.80	b=0.001716	
Hyperbolic	: Y= a+b/X	R»=0.24	a=16.90	b=-5.318	
Logarithmic	: Y= a+b*LN(X)	R»=0.90	a=4.626	b=2.378	

Optimum :

H.-Bulkley	: б= бo+a*D■	R»=0.94	a=4.624	n=0.2000	бo=3.688

Rheogram and Data for Test Number HNOD7D

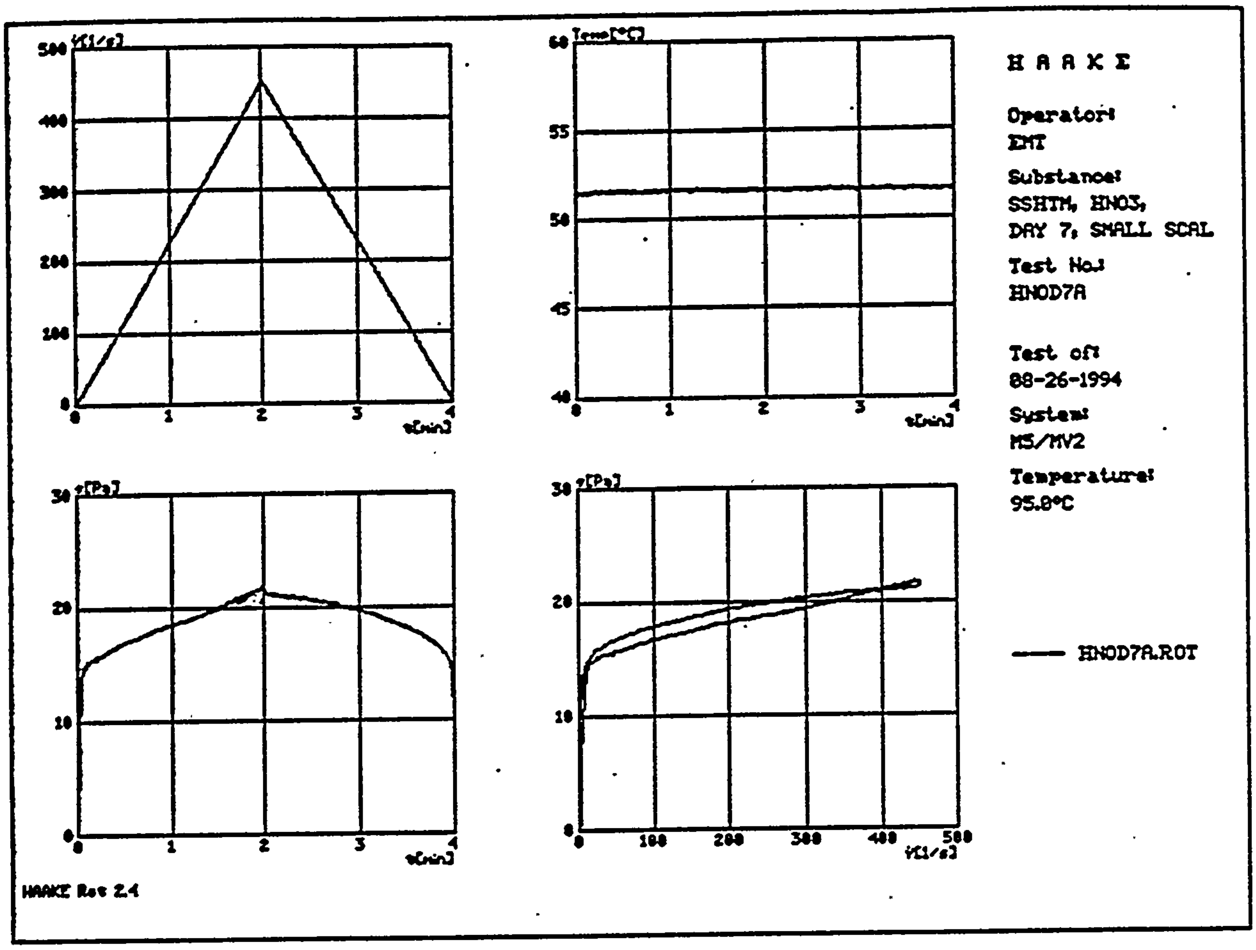


Bingham	: ó= óo+Eta*D	R»=0.88	óo=13.00	Eta=0.01874	
Ostwald	: ó= a*D▪	R»=0.82	a=6.707	n=0.1817	
H.-Bulkley	: ó= óo+a*D▪	R»=0.93	a=4.665	n=0.1988	óo=4.123
Casson	: «ó-«óo = «(Eta*D)	R»=0.83	óo=10.49	Eta=0.00402	
Linear	: Y= a+b*X	R»=0.88	a=13.00	b=0.01874	
Inv. linear	: Y= 1/(a+b*X)	R»=0.05	a=0.1113	b=-190.3E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.34	a=12.18	b=0.001398	
Hyperbolic	: Y= a+b/X	R»=0.24	a=17.37	b=-5.656	
Logarithmic	: Y= a+b*LN(X)	R»=0.90	a=5.215	b=2.353	

Optimum :

H.-Bulkley : ó= óo+a*D▪ R»=0.93 a=4.665 n=0.1988 óo=4.123

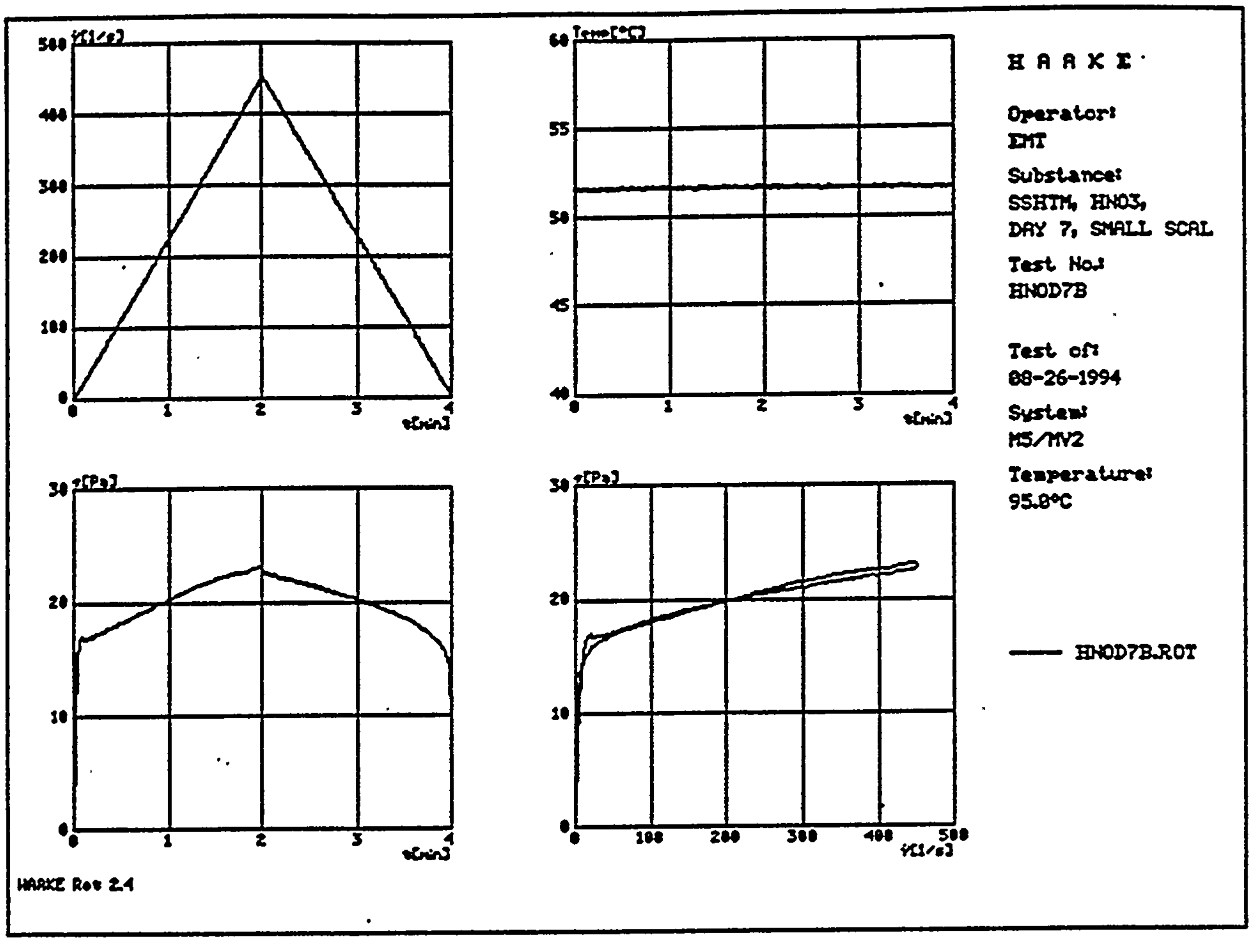
Rheogram and Data for Test Number HNOD7A



Bingham	: ó= óo+Eta*D	R»=0.70	óo=14.17	Eta=0.01774	
Ostwald	: ó= a*D■	R»=0.74	a=5.316	n=0.2372	
H.-Bulkley	: ó= óo+a*D■	R»=0.63	a=5.017	n=0.2302	óo=1.611
Casson	: «ó-«óo = «(Eta*D)	R»=0.62	óo=11.08	Eta=0.004193	
Linear	: Y= a+b*X	R»=0.70	a=14.17	b=0.01774	
Inv. linear	: Y= 1/(a+b*X)	R»=0.04	a=0.1133	b=-205.4E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.26	a=13.28	b=0.001268	
Hyperbolic	: Y= a+b/X	R»=0.27	a=18.27	b=-1.191	
Logarithmic	: Y= a+b*LN(X)	R»=0.94	a=6.571	b=2.287	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.94 a=6.571 b=2.287

Rheogram and Data for Test Number HNOD7B

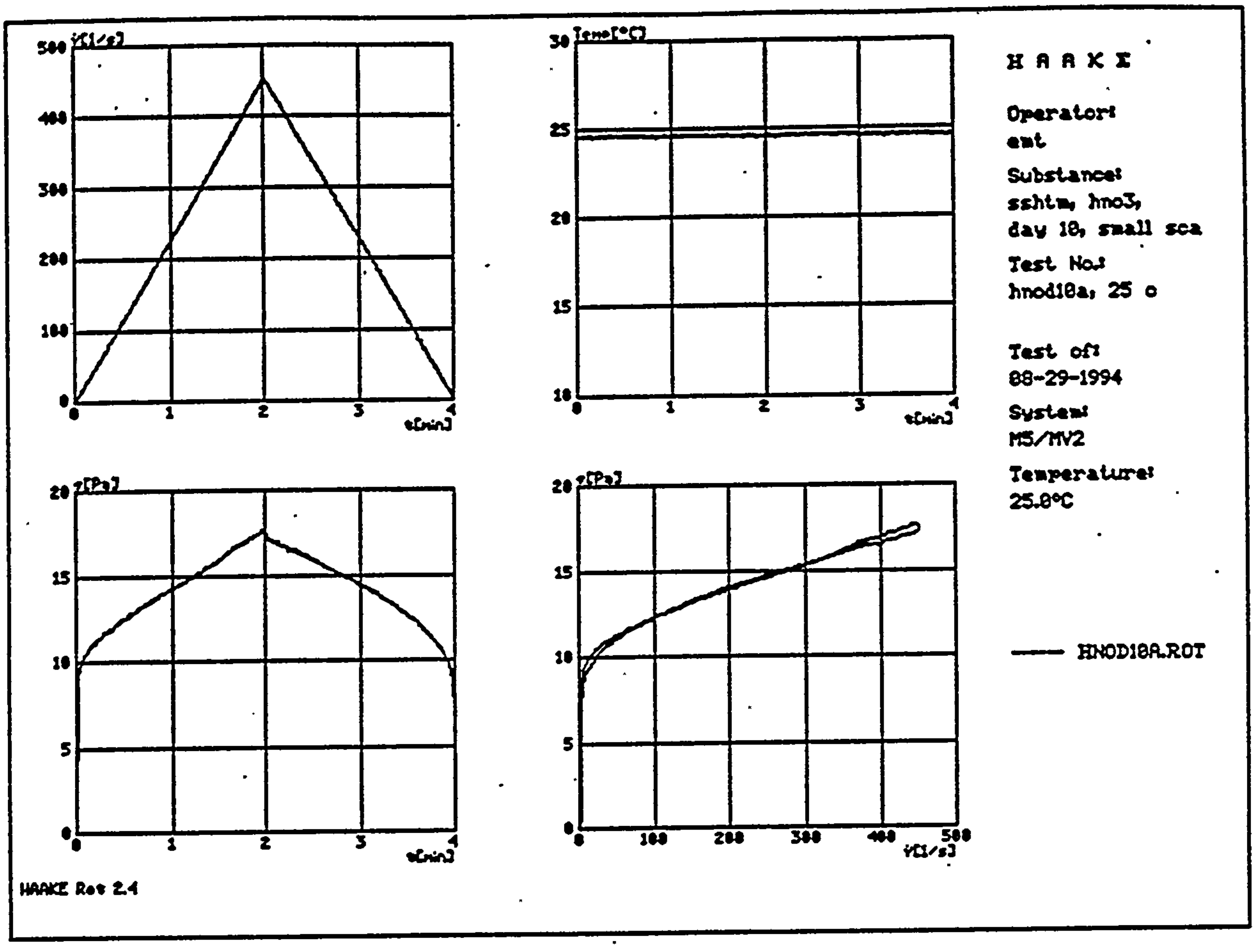


Bingham	: ó= óo+Eta*D	R»=0.81	óo=15.94	Eta=0.01781	
Ostwald	: ó= a*D■	R»=0.83	a=9.188	n=0.1496	
H.-Bulkley	: ó= óo+a*D■	R»=0.76	a=6.124	n=0.1943	óo=3.125
Casson	: «ó-«óo = «(Eta*D)	R»=0.80	óo=13.34	Eta=0.003216	
Linear	: Y= a+b*X	R»=0.81	a=15.94	b=0.01781	
Inv. linear	: Y= 1/(a+b*X)	R»=0.15	a=0.07069	b=-78.01E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.43	a=15.30	b=0.00109	
Hyperbolic	: Y= a+b/X	R»=0.51	a=20.47	b=-33.58	
Logarithmic	: Y= a+b*LN(X)	R»=0.91	a=7.421	b=2.453	

Optimum :

Logarithmic	: Y= a+b*LN(X)	R»=0.91	a=7.421	b=2.453

Rheogram and Data for Test Number hnod10a, 25 c

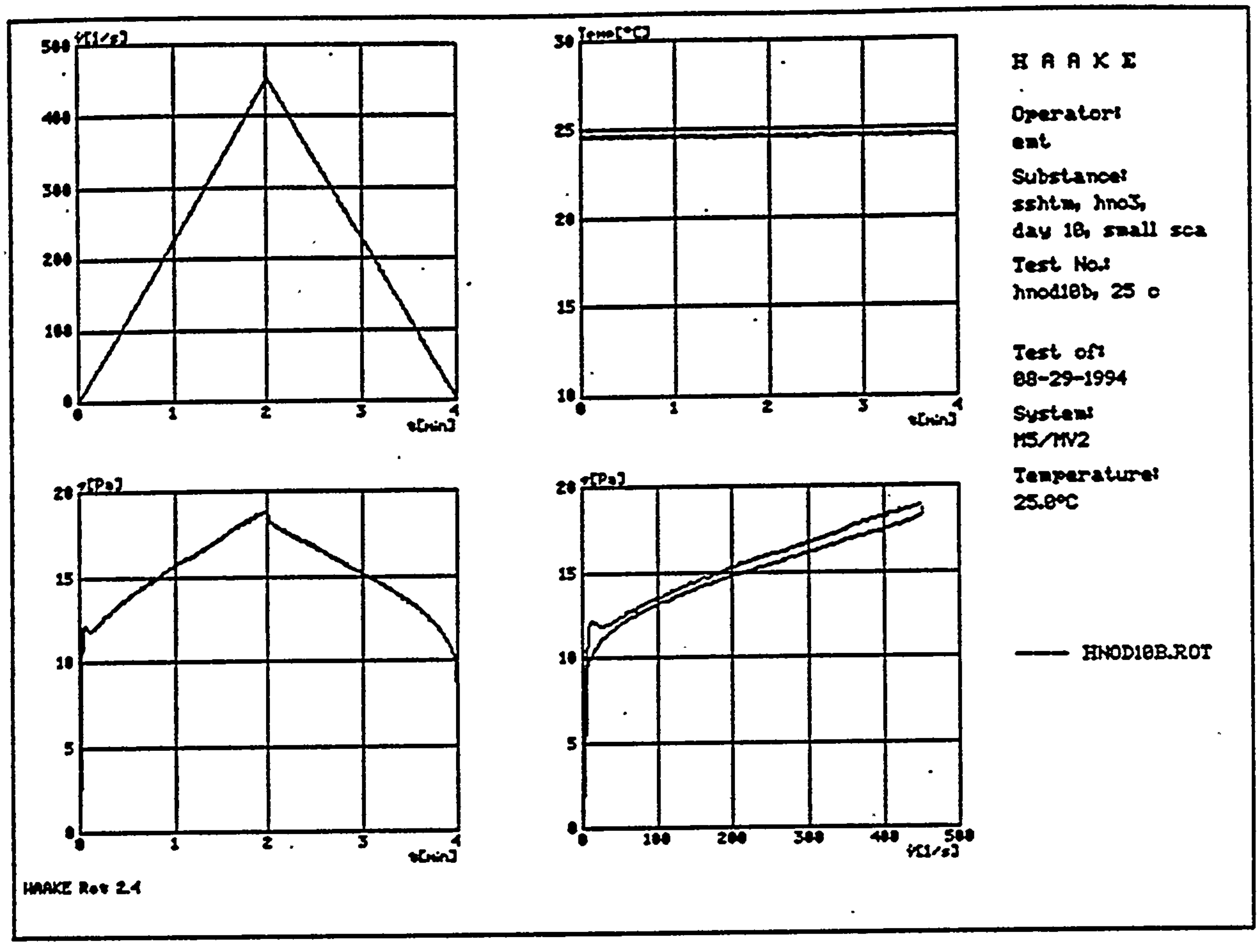


Bingham	: ő= őo+Eta*D	R»=0.91	őo=10.28	Eta=0.01713	
Ostwald	: ő= a*D■	R»=0.79	a=4.806	n=0.2076	
H.-Bulkley	: ő= őo+a*D■	R»=0.94	a=3.423	n=0.2192	őo=3.432
Casson	: «ő-«őo = «(Eta*D)	R»=0.84	őo=8.078	Eta=0.004067	
Linear	: Y= a+b*X	R»=0.91	a=10.28	b=0.01713	
Inv. linear	: Y= 1/(a+b*X)	R»=0.02	a=0.5927	b=-0.001757	
Exponential	: Y= a*EXP(b*X)	R»=0.22	a=9.156	b=0.001717	
Hyperbolic	: Y= a+b/X	R»=0.21	a=14.25	b=-3.885	
Logarithmic	: Y= a+b*LN(X)	R»=0.89	a=3.445	b=2.097	

Optimum :

H.-Bulkley	: ő= őo+a*D■	R»=0.94	a=3.423	n=0.2192	őo=3.432

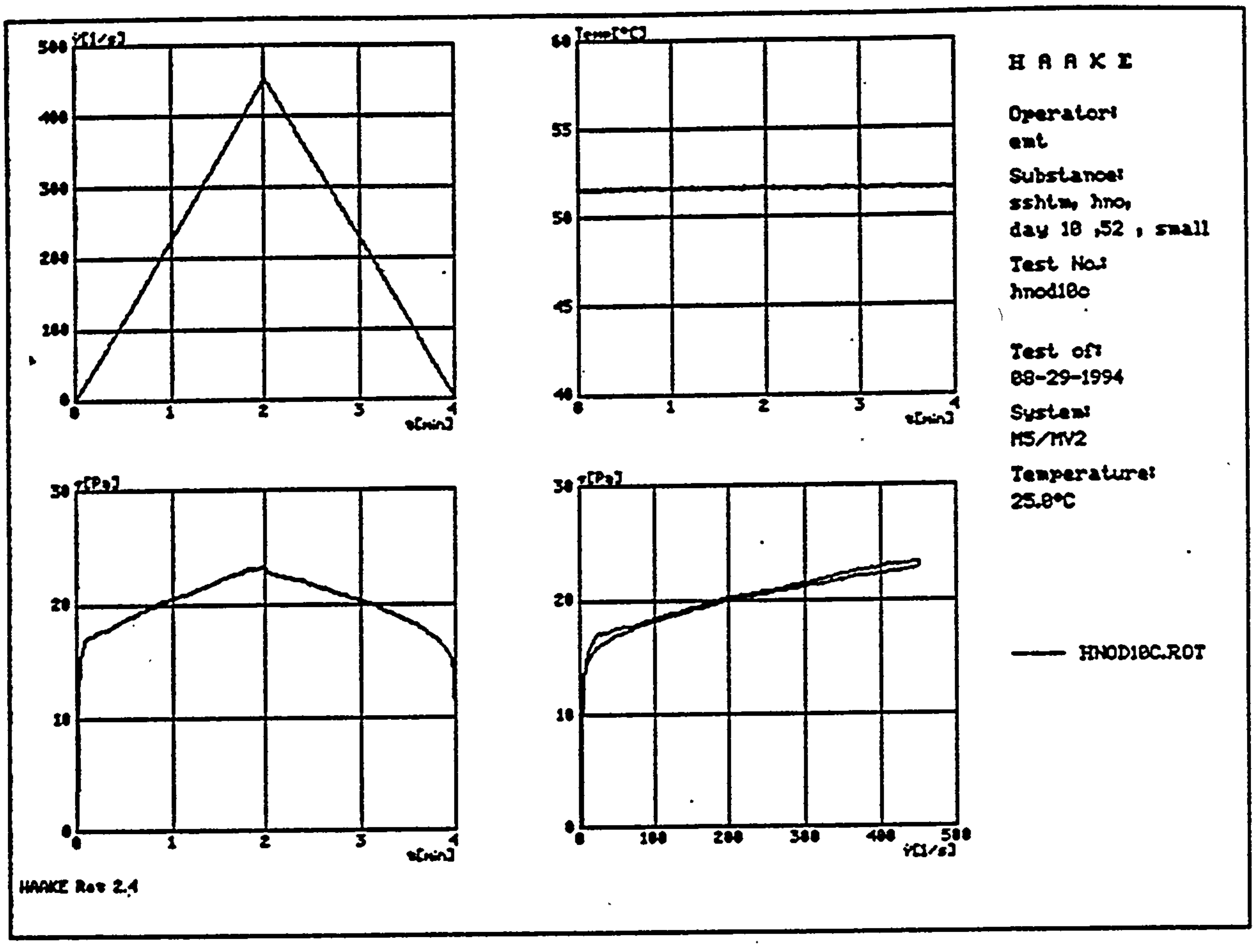
Rheogram and Data for Test Number hnod10b, 25 c




```
Bingham     : ó= óo+Eta*D      R»=0.85  óo=11.35  Eta=0.01797
Ostwald     : ó= a*D■          R»=0.86  a=5.615   n=0.1947
H.-Bulkley  : ó= óo+a*D■       R»=0.64  a=3.065   n=0.2685  óo=2.995
Casson      : «ó-«óo = «(Eta*D)  R»=0.82  óo=8.934  Eta=0.004235
Linear      : Y= a+b*X         R»=0.85  a=11.35   b=0.01797
Inv. linear: Y= 1/(a+b*X)      R»=0.17  a=0.09619  b=-119.4E-06
Exponential: Y= a*EXP(b*X)     R»=0.53  a=11.21   b=0.001321
Hyperbolic : Y= a+b/X          R»=0.23  a=15.51   b=-3.099
Logarithmic: Y= a+b*LN(X)      R»=0.90  a=4.487   b=2.148

Optimum :
Logarithmic: Y= a+b*LN(X)      R»=0.90  a=4.487   b=2.148
```

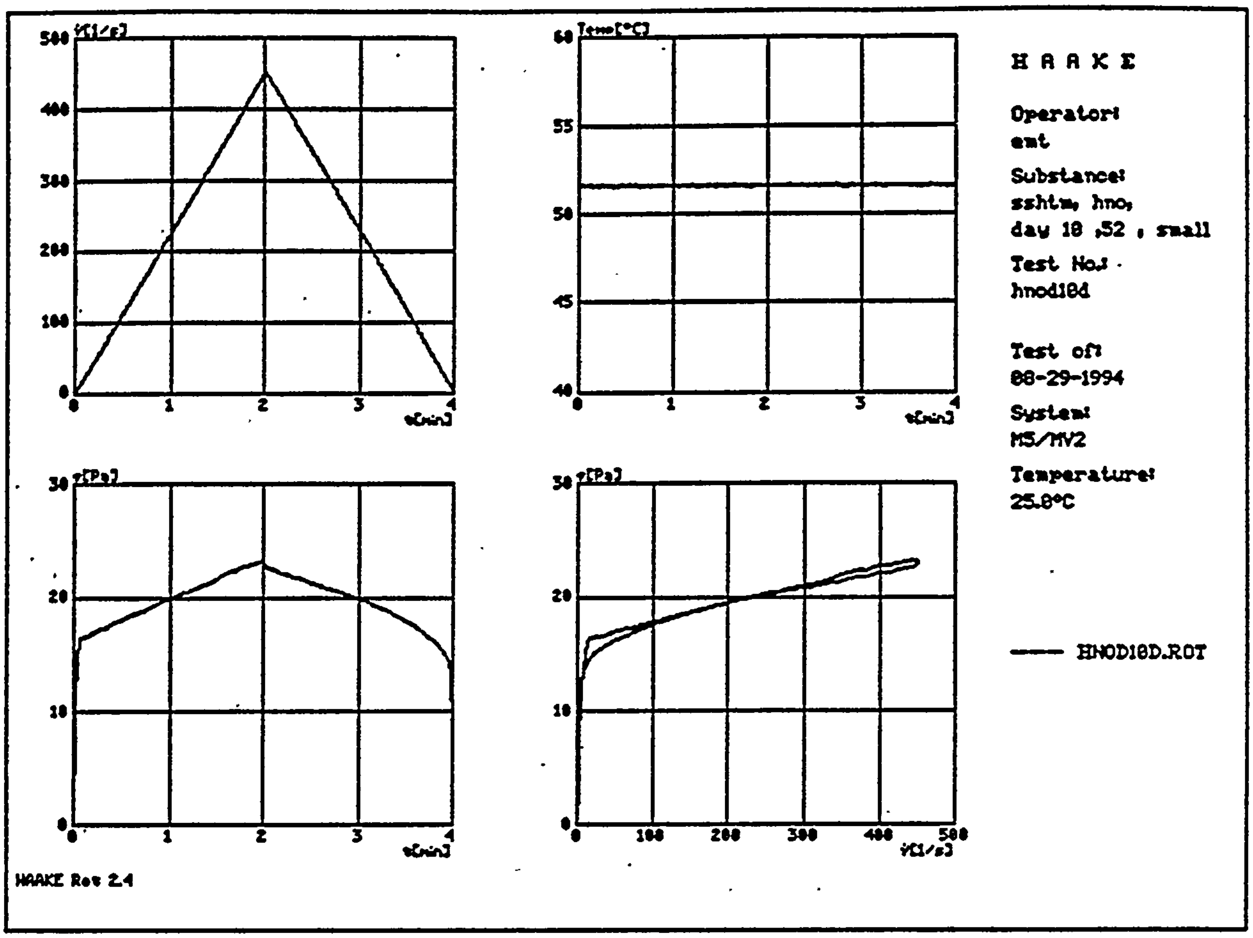
Rheogram and Data for Test Number hnod10c




```
Bingham     : ő= őo+Eta*D      R»=0.73  őo=16.22  Eta=0.01747
Ostwald     : ő= a*D■          R»=0.52  a=6.093   n=0.2299
H.-Bulkley  : ő= őo+a*D■       R»=0.85  a=8.696   n=0.1435  őo=1.930
Casson      : «ő-«őo = «(Eta*D) R»=0.59  őo=13.21  Eta=0.003518
Linear      : Y= a+b*X         R»=0.73  a=16.22   b=0.01747
Inv. linear: Y= 1/(a+b*X)     R»=0.02  a=0.1388  b=-306.3E-06
Exponential: Y= a*EXP(b*X)    R»=0.19  a=15.26   b=0.001128
Hyperbolic : Y= a+b/X         R»=0.38  a=20.35   b=-6.743
Logarithmic: Y= a+b*LN(X)     R»=0.92  a=7.733   b=2.437

Optimum :
Logarithmic: Y= a+b*LN(X)     R»=0.92  a=7.733   b=2.437
```

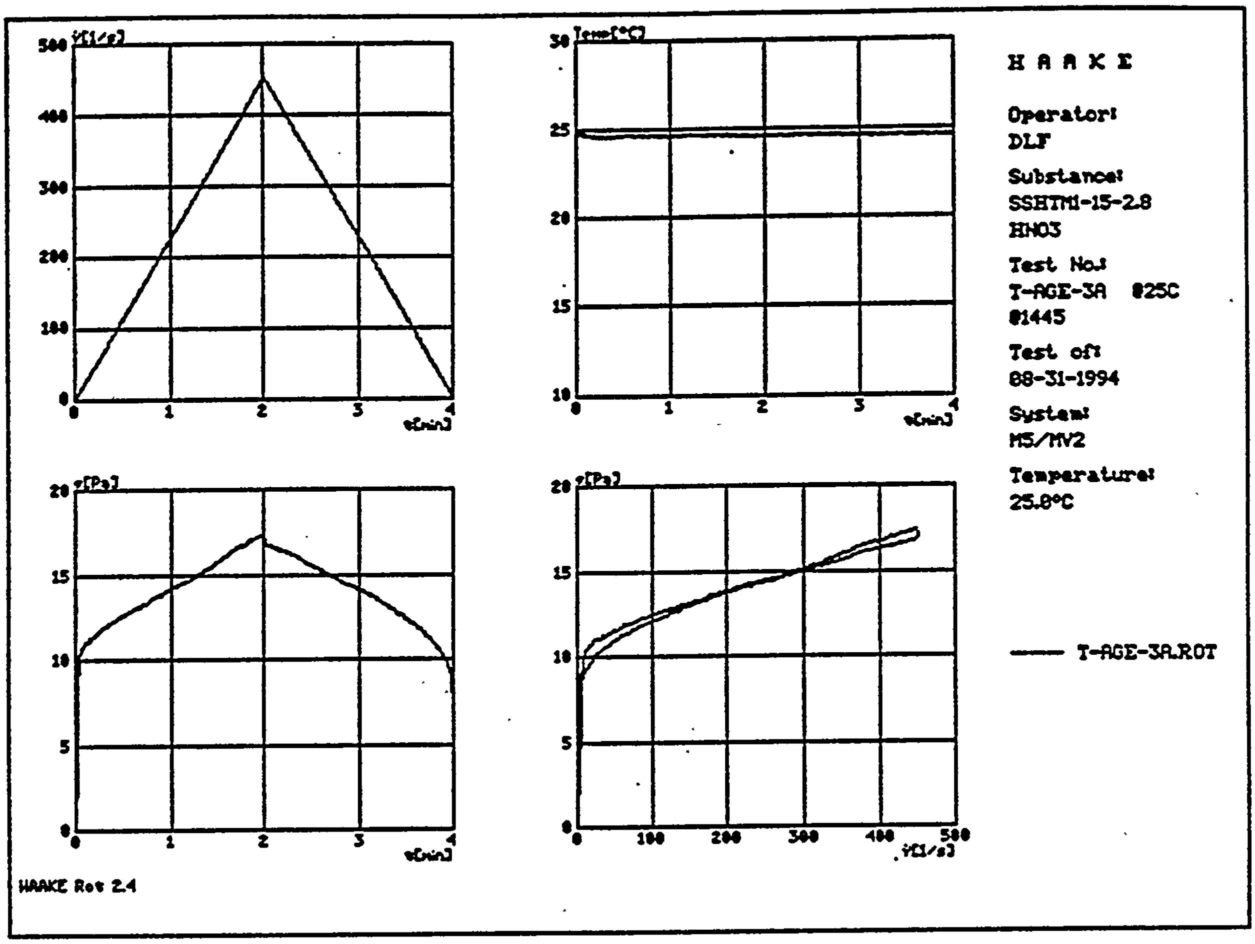
Rheogram and Data for Test Number hnod10d




```
Bingham     : б= бo+Eta*D     R»=0.80  бo=15.57  Eta=0.01838
Ostwald     : б= a*D■         R»=0.79  a=8.165   n=0.1700
H.-Bulkley  : б= бo+a*D■      R»=0.84  a=7.090   n=0.1656  бo=3.005
Casson      : «б-«бo = «(Eta*D)  R»=0.74  бo=12.85  Eta=0.00354
Linear      : Y= a+b*X        R»=0.80  a=15.57   b=0.01838
Inv. linear: Y= 1/(a+b*X)     R»=0.07  a=0.08464  b=-122.9E-06
Exponential: Y= a*EXP(b*X)    R»=0.32  a=14.59   b=0.001214
Hyperbolic : Y= a+b/X         R»=0.29  a=19.86   b=-5.176
Logarithmic: Y= a+b*LN(X)     R»=0.91  a=7.355   b=2.422

Optimum :
Logarithmic: Y= a+b*LN(X)     R»=0.91  a=7.355   b=2.422
```

Rheogram and Data for Test Number T-AGE-3A @25C @1445

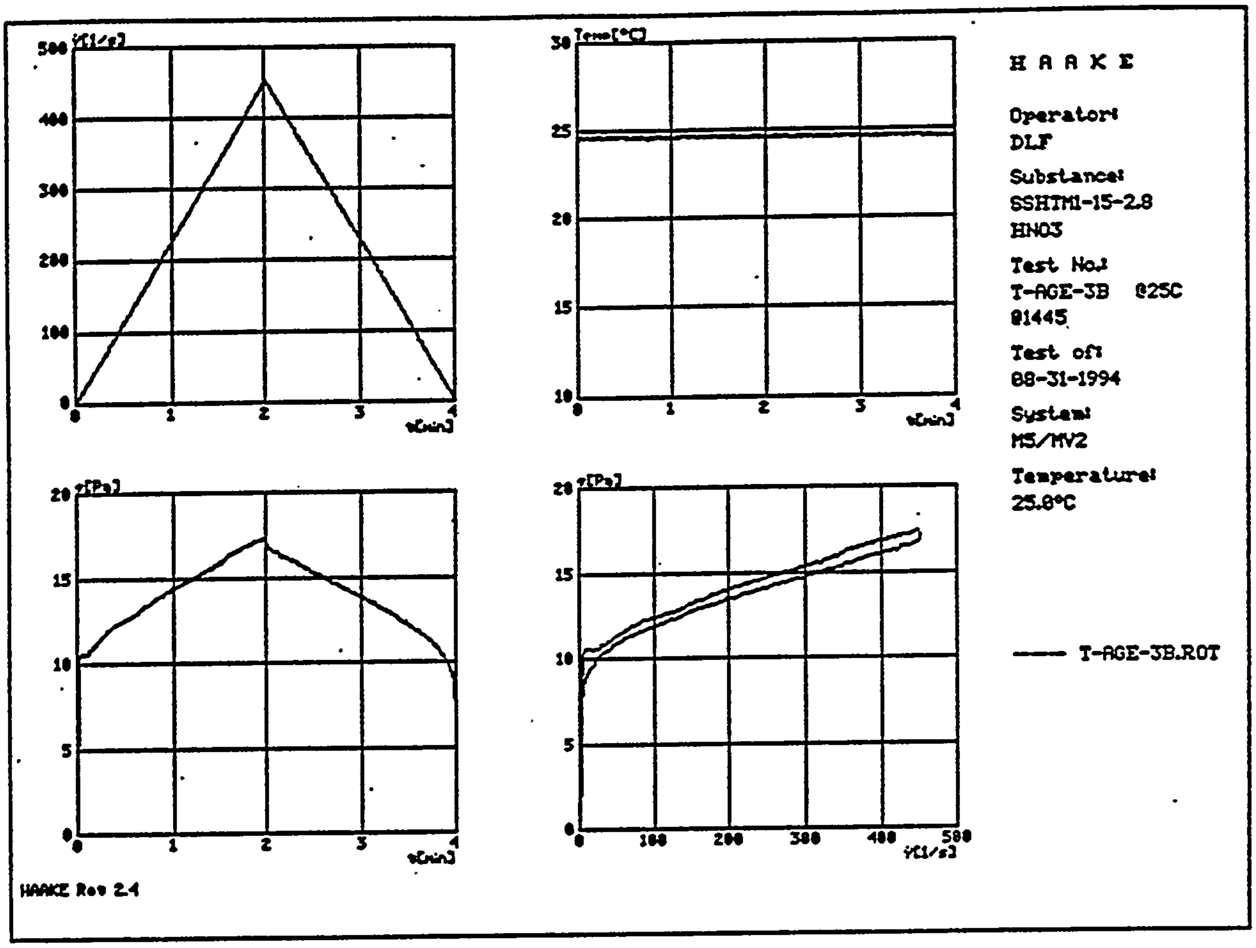


Bingham	: ő= őo+Eta*D	R»=0.91	őo=10.54	Eta=0.01583	
Ostwald	: ő= a*D■	R»=0.88	a=5.756	n=0.1727	
H.-Bulkley	: ő= őo+a*D■	R»=0.92	a=3.548	n=0.2116	őo=3.442
Casson	: «ő-«őo = «(Eta*D)	R»=0.88	őo=8.539	Eta=0.003366	
Linear	: Y= a+b*X	R»=0.91	a=10.54	b=0.01583	
Inv. linear	: Y= 1/(a+b*X)	R»=0.11	a=0.1107	b=-148.4E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.48	a=10.15	b=0.001346	
Hyperbolic	: Y= a+b/X	R»=0.26	a=14.28	b=-9.162	
Logarithmic	: Y= a+b*LN(X)	R»=0.89	a=3.927	b=1.994	

Optimum :

H.-Bulkley	: ő= őo+a*D■	R»=0.92	a=3.548	n=0.2116	őo=3.442

Rheogram and Data for Test Number T-AGE-3B @25C @1445

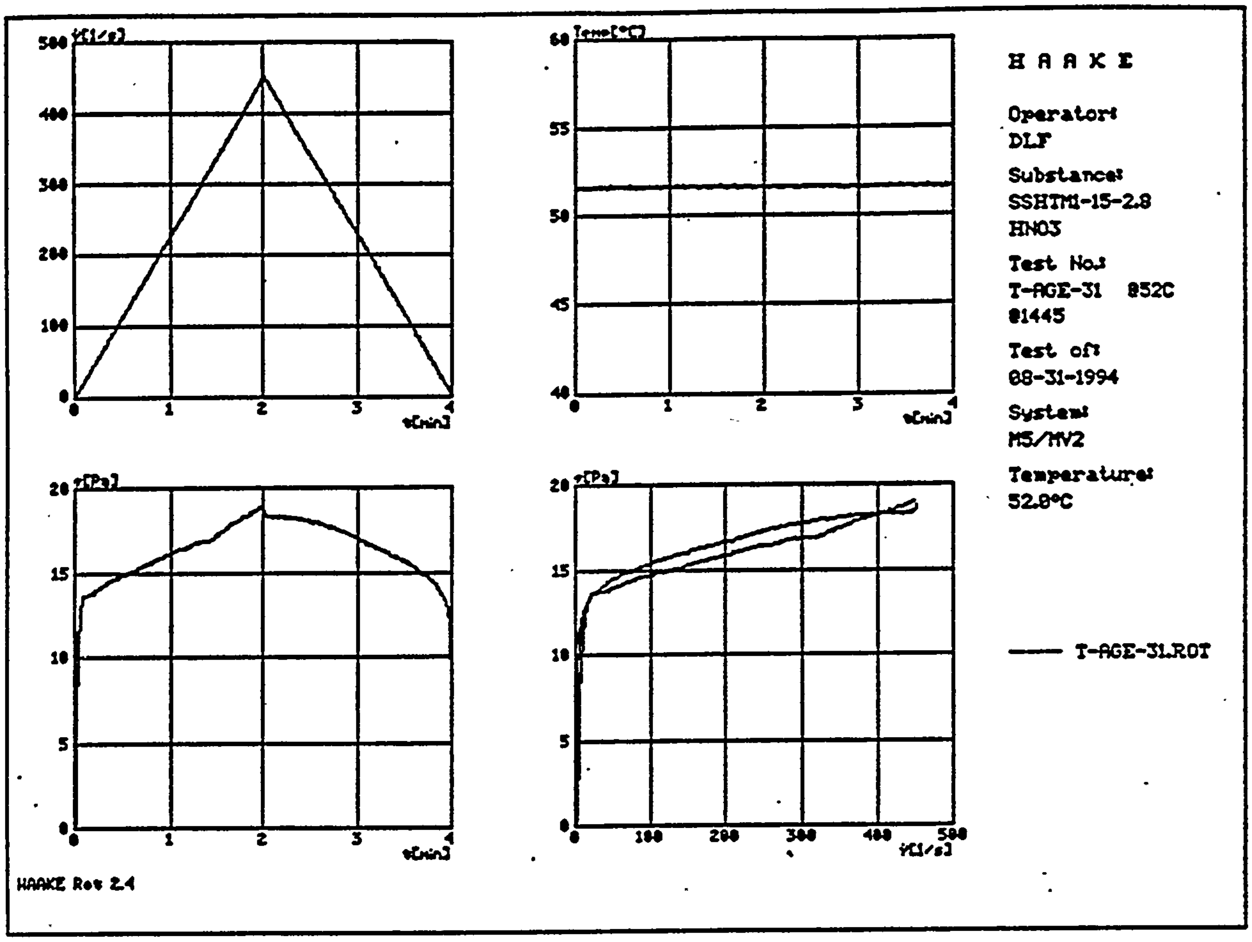


Bingham	: ó= óo+Eta*D	R»=0.92	óo=10.54	Eta=0.01619	
Ostwald	: ó= a*D■	R»=0.89	a=5.875	n=0.1700	
H.-Bulkley	: ó= óo+a*D■	R»=0.93	a=2.864	n=0.2327	óo=4.556
Casson	: «ó-«óo = «(Eta*D)	R»=0.90	óo=8.525	Eta=0.003476	
Linear	: Y= a+b*X	R»=0.92	a=10.54	b=0.01619	
Inv. linear	: Y= 1/(a+b*X)	R»=0.13	a=0.1088	b=-143.7E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.50	a=10.19	b=0.001358	
Hyperbolic	: Y= a+b/X	R»=0.20	a=14.30	b=-4.294	
Logarithmic	: Y= a+b*LN(X)	R»=0.88	a=4.155	b=1.967	

Optimum :

H.-Bulkley	: ó= óo+a*D■	R»=0.93	a=2.864	n=0.2327	óo=4.556

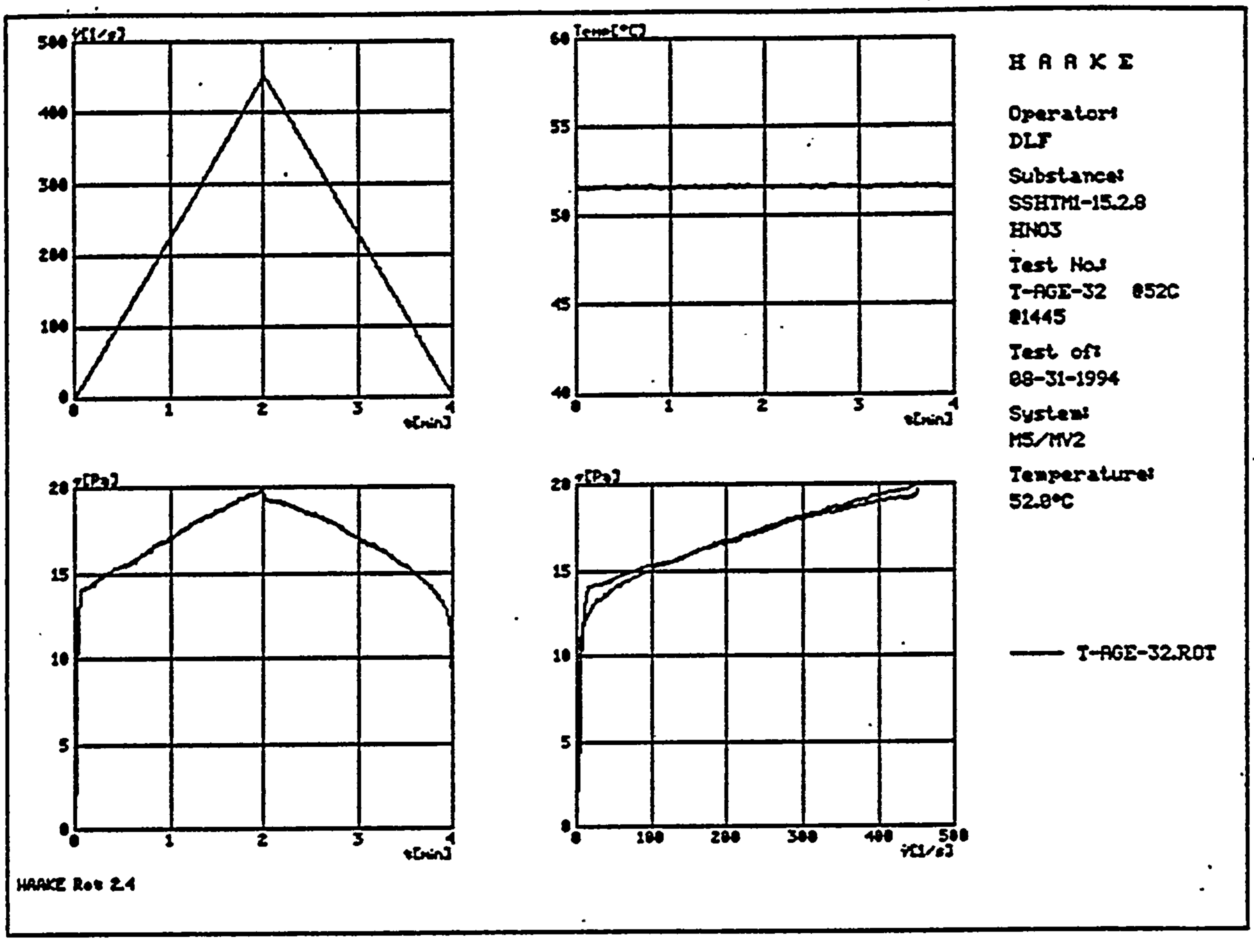
Rheogram and Data for Test Number T-AGE-31 @52C @1445



Bingham	: ó= óo+Eta*D	R»=0.71	óo=12.74	Eta=0.01424	
Ostwald	: ó= a*D■	R»=0.59	a=5.234	n=0.2143	
H.-Bulkley	: ó= óo+a*D■	R»=0.83	a=7.329	n=0.1444	óo=0.5038
Casson	: «ó-«óo = «(Eta*D)	R»=0.62	óo=10.33	Eta=0.002933	
Linear	: Y= a+b*X	R»=0.71	a=12.74	b=0.01424	
Inv. linear	: Y= 1/(a+b*X)	R»=0.03	a=0.1191	b=-0.0002	
Exponential	: Y= a*EXP(b*X)	R»=0.25	a=12.15	b=0.001113	
Hyperbolic	: Y= a+b/X	R»=0.38	a=16.13	b=-7.907	
Logarithmic	: Y= a+b*LN(X)	R»=0.91	a=5.615	b=2.026	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.91 a=5.615 b=2.026

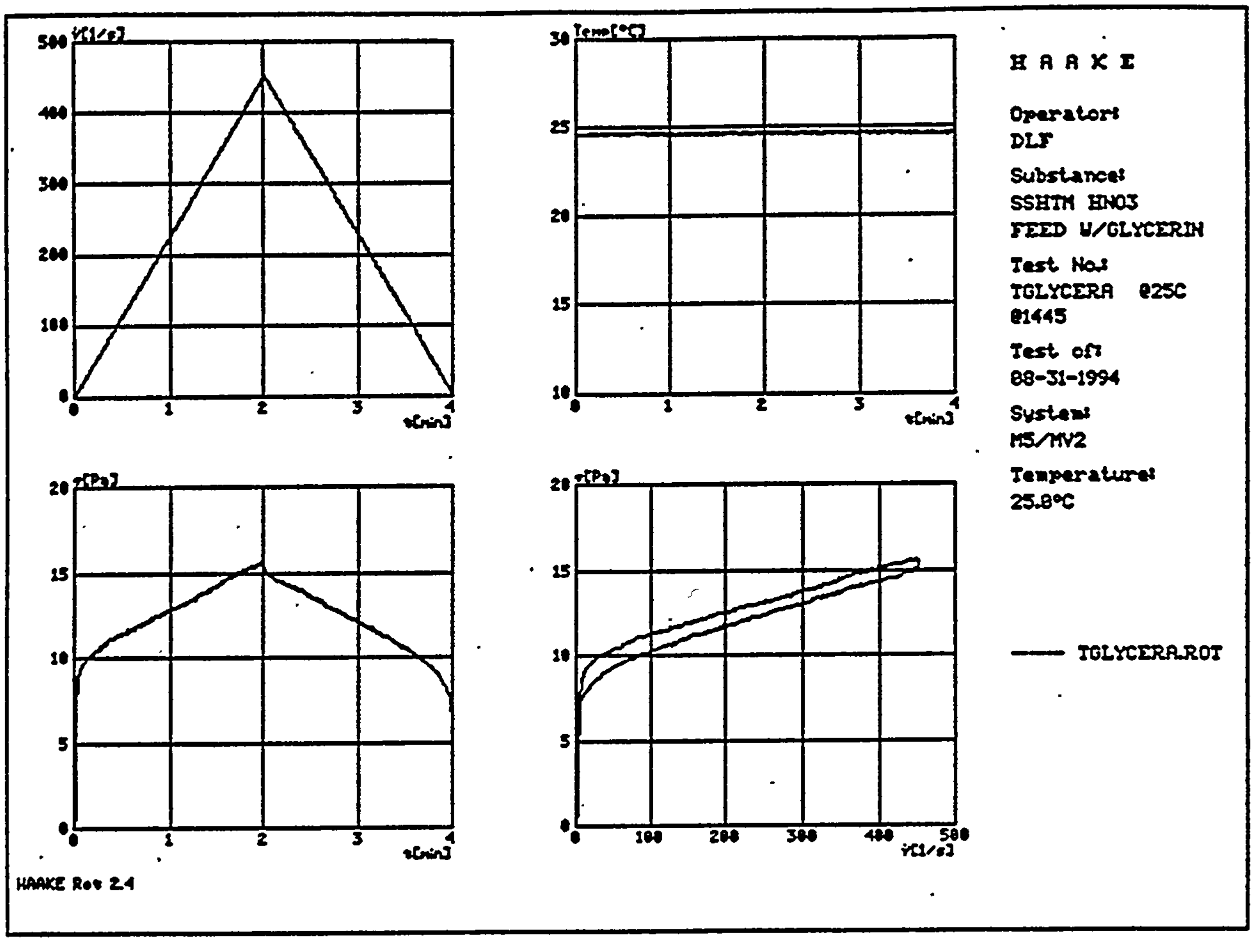
Rheogram and Data for Test Number T-AGE-32 @52C @1445



Bingham	: б= бo+Eta*D	R»=0.80	бo=13.40	Eta=0.01561	
Ostwald	: б= a*D■	R»=0.80	a=7.234	n=0.1638	
H.-Bulkley	: б= бo+a*D■	R»=0.60	a=4.087	n=0.2431	бo=2.407
Casson	: «б-«бo = «(Eta*D)	R»=0.76	бo=11.09	Eta=0.002967	
Linear	: Y= a+b*X	R»=0.80	a=13.40	b=0.01561	
Inv. linear	: Y= 1/(a+b*X)	R»=0.10	a=0.08975	b=-113.6E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.37	a=12.71	b=0.001164	
Hyperbolic	: Y= a+b/X	R»=0.36	a=17.14	b=-11.39	
Logarithmic	: Y= a+b*LN(X)	R»=0.91	a=6.116	b=2.116	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.91 a=6.116 b=2.116

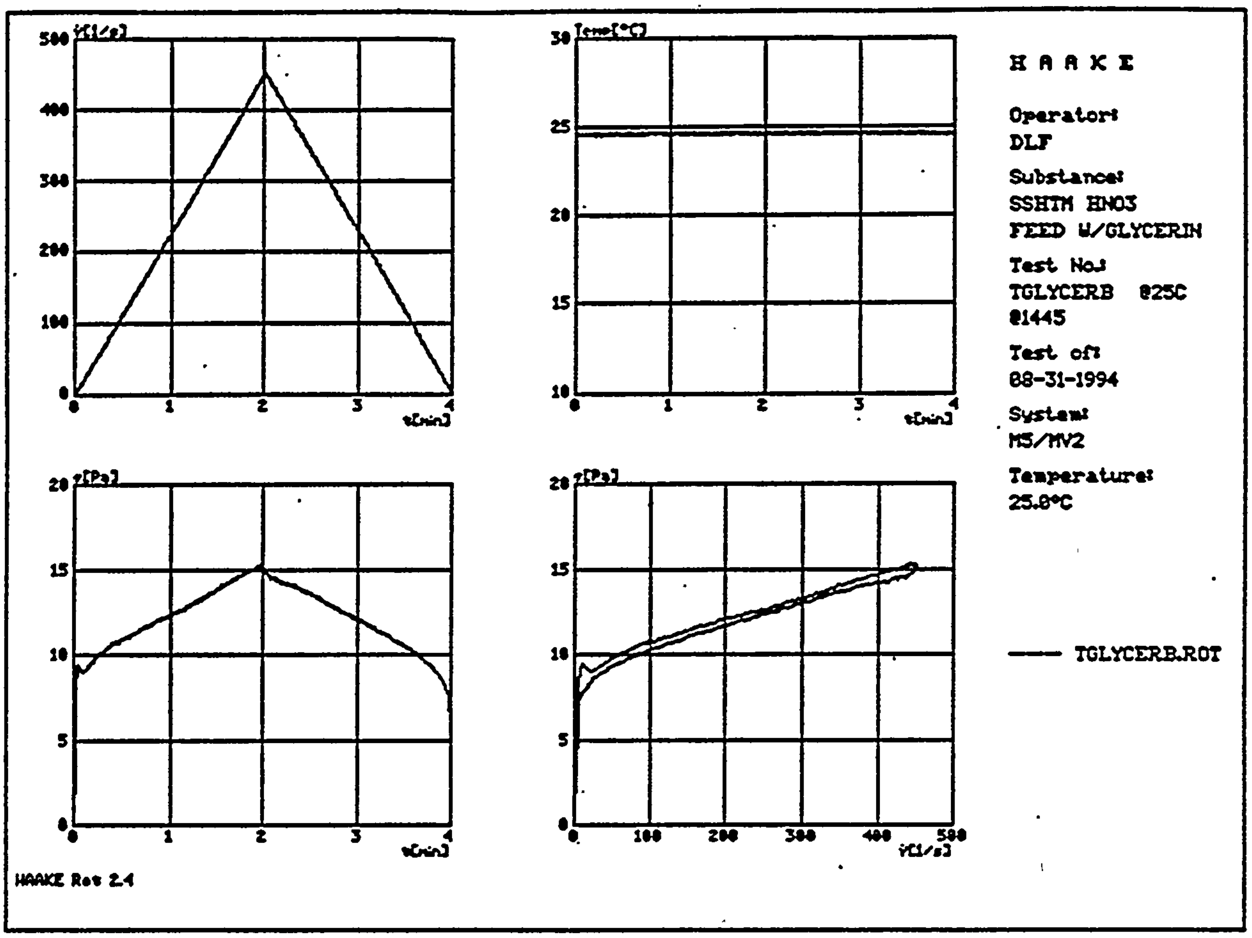
Rheogram and Data for Test Number TGLYCERA @25C @1445




```
Bingham     : ó= óo+Eta*D       R»=0.85  óo=9.434   Eta=0.01458
Ostwald     : ó= a*D■           R»=0.80  a=4.334   n=0.2071
H.-Bulkley  : ó= óo+a*D■        R»=0.86  a=3.870   n=0.2034   óo=1.587
Casson      : «ó-«óo = «(Eta*D)   R»=0.80  óo=7.414   Eta=0.003431
Linear      : Y= a+b*X          R»=0.85  a=9.434   b=0.01458
Inv. linear: Y= 1/(a+b*X)      R»=0.02  a=1.638   b=-0.005215
Exponential: Y= a*EXP(b*X)     R»=0.18  a=8.171   b=0.001731
Hyperbolic  : Y= a+b/X          R»=0.32  a=12.91   b=-9.010
Logarithmic: Y= a+b*LN(X)      R»=0.92  a=3.041   b=1.897

Optimum :
Logarithmic: Y= a+b*LN(X)      R»=0.92  a=3.041   b=1.897
```

Rheogram and Data for Test Number TGLYCERB @25C @1445

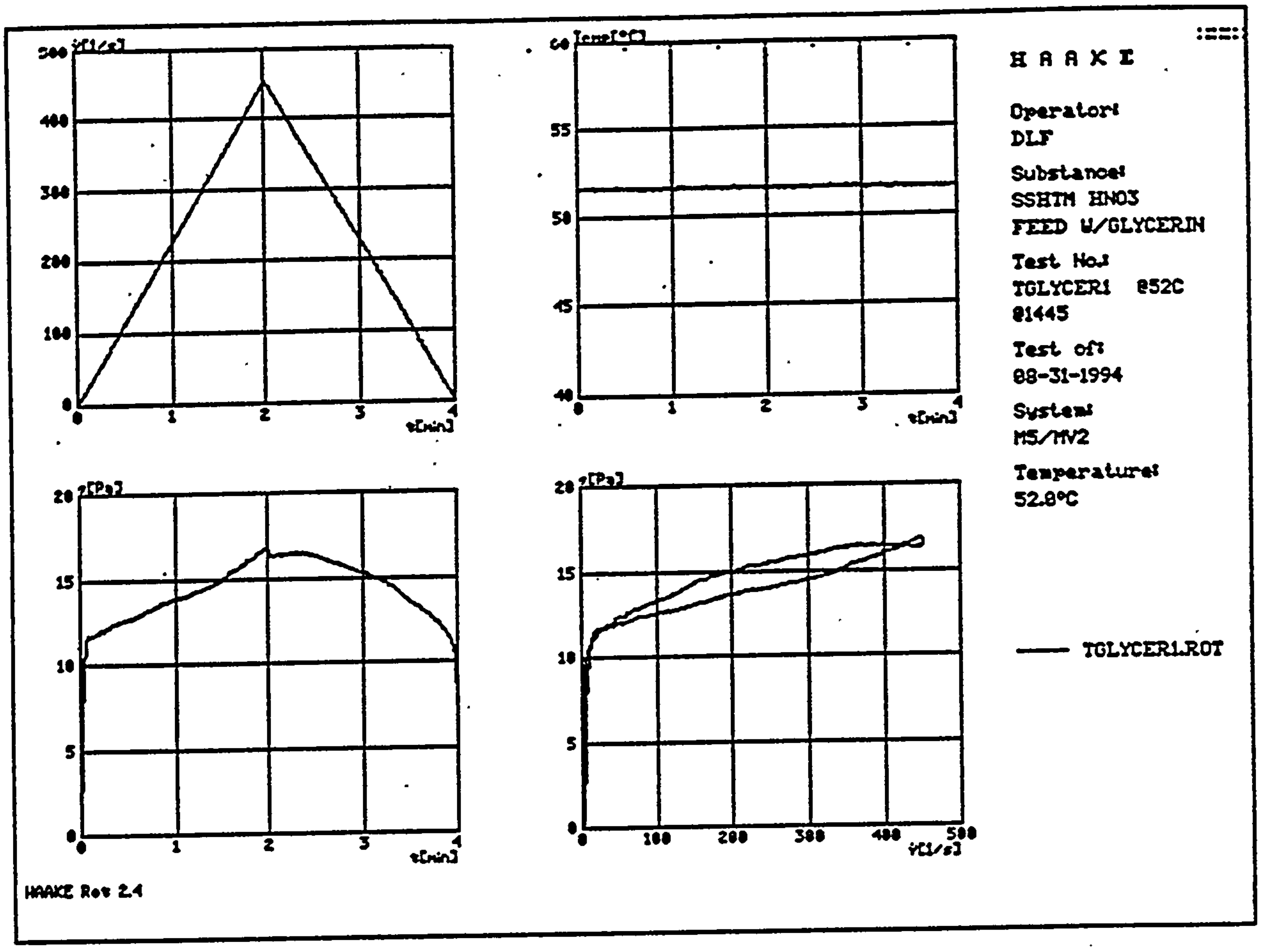


Bingham	: ő= őo+Eta*D	R»=0.93	őo=9.046	Eta=0.01435	
Ostwald	: ő= a*D■	R»=0.88	a=4.991	n=0.1734	
H.-Bulkley	: ő= őo+a*D■	R»=0.92	a=2.724	n=0.2249	őo=3.485
Casson	: «ő-«őo = «(Eta*D)	R»=0.90	őo=7.299	Eta=0.003119	
Linear	: Y= a+b*X	R»=0.93	a=9.046	b=0.01435	
Inv. linear	: Y= 1/(a+b*X)	R»=0.14	a=0.1249	b=-163.7E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.52	a=8.779	b=0.001377	
Hyperbolic	: Y= a+b/X	R»=0.23	a=12.43	b=-7.790	
Logarithmic	: Y= a+b*LN(X)	R»=0.87	a=3.252	b=1.768	

Optimum :

Bingham	: ő= őo+Eta*D	R»=0.93	őo=9.046	Eta=0.01435

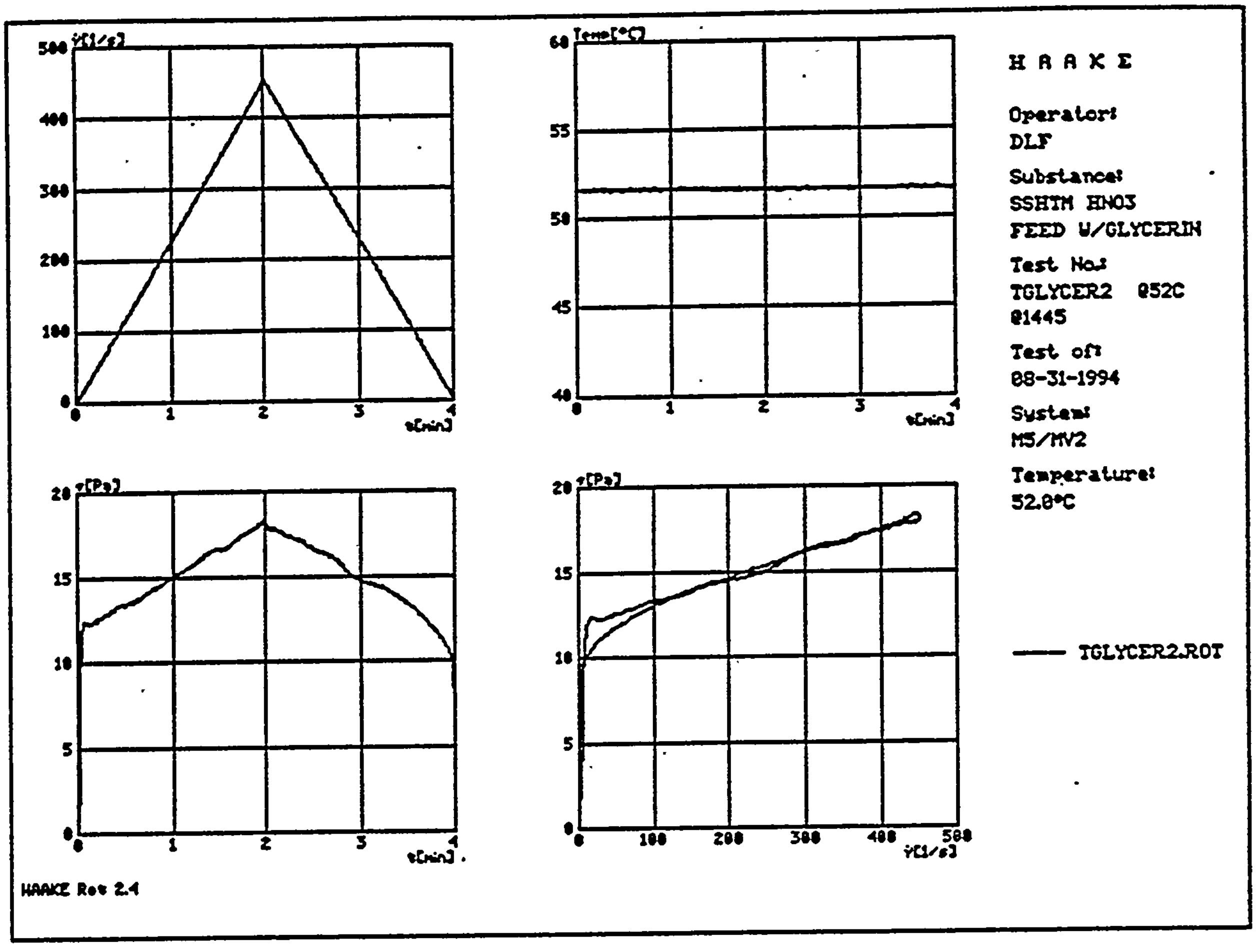
Rheogram and Data for Test Number TGLYCER1 @52C @1445




```
Bingham      : б= бo+Eta*D       R»=0.76   бo=10.84   Eta=0.01305
Ostwald      : б= a*D■           R»=0.63   a=4.678    n=0.2079
H.-Bulkley   : б= бo+a*D■        R»=0.84   a=6.161    n=0.1453   бo=0.7337
Casson       : «б-«бo = «(Eta*D)   R»=0.65   бo=8.821   Eta=0.002654
Linear       : Y= a+b*X          R»=0.76   a=10.84    b=0.01305
Inv. linear  : Y= 1/(a+b*X)      R»=0.04   a=0.1289   b=-201.8E-06
Exponential  : Y= a*EXP(b*X)     R»=0.29   a=10.45    b=0.001137
Hyperbolic   : Y= a+b/X          R»=0.31   a=13.90    b=-4.380
Logarithmic  : Y= a+b*LN(X)      R»=0.88   a=4.850    b=1.751

Optimum :
Logarithmic  : Y= a+b*LN(X)      R»=0.88   a=4.850    b=1.751
```

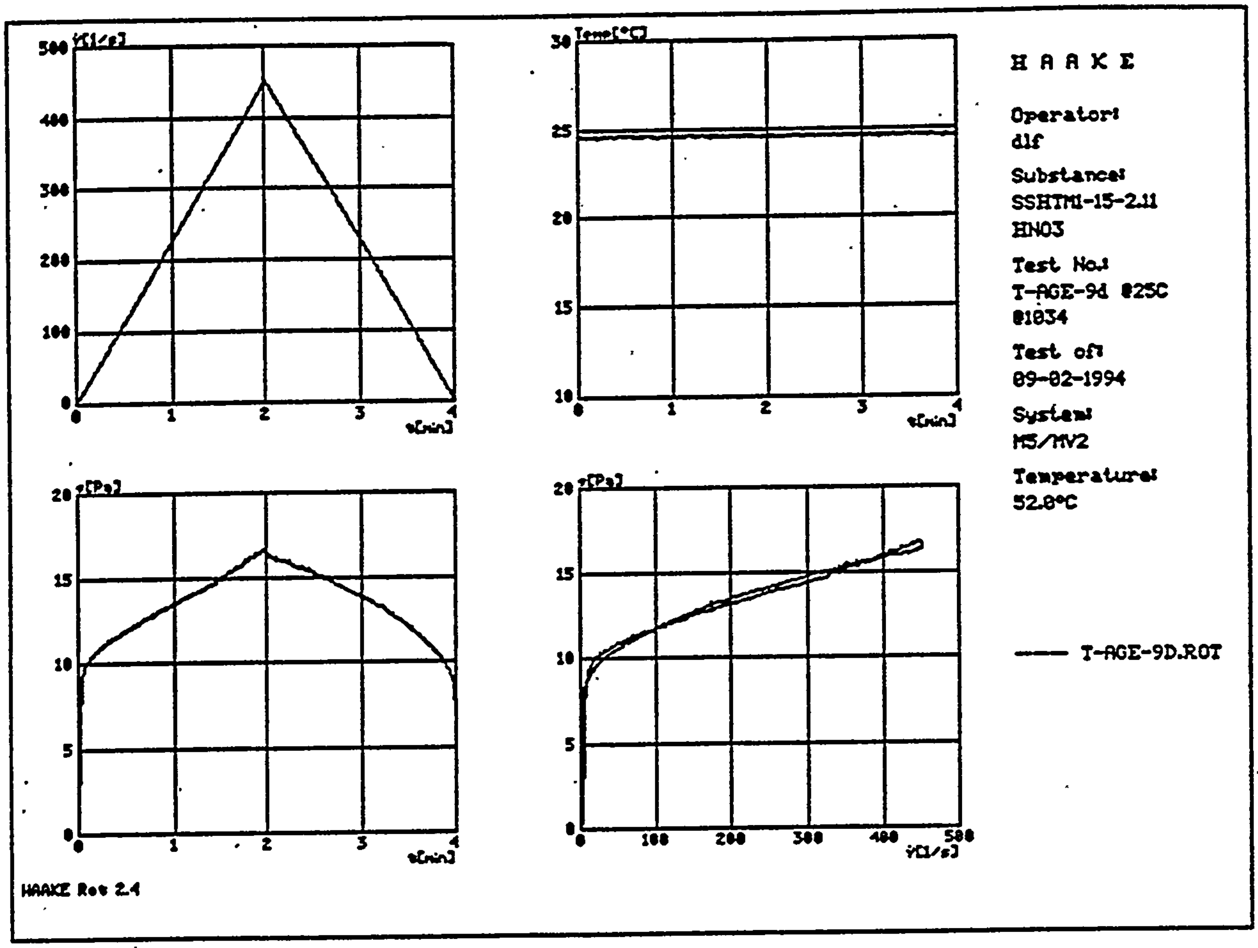
Rheogram and Data for Test Number TGLYCER2 @52C @1445



Bingham	: ó= óo+Eta*D	R»=0.86	óo=11.47	Eta=0.01548	
Ostwald	: ó= a*D■	R»=0.80	a=6.237	n=0.1686	
H.-Bulkley	: ó= óo+a*D■	R»=0.85	a=5.042	n=0.1743	óo=2.510
Casson	: «ó-«óo = «(Eta*D)	R»=0.81	óo=9.428	Eta=0.003081	
Linear	: Y= a+b*X	R»=0.86	a=11.47	b=0.01548	
Inv. linear	: Y= 1/(a+b*X)	R»=0.10	a=0.1042	b=-138.4E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.42	a=10.96	b=0.001269	
Hyperbolic	: Y= a+b/X	R»=0.30	a=15.15	b=-9.454	
Logarithmic	: Y= a+b*LN(X)	R»=0.87	a=4.905	b=1.970	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.87 a=4.905 b=1.970

Rheogram and Data for Test Number T-AGE-9d @25C @1034

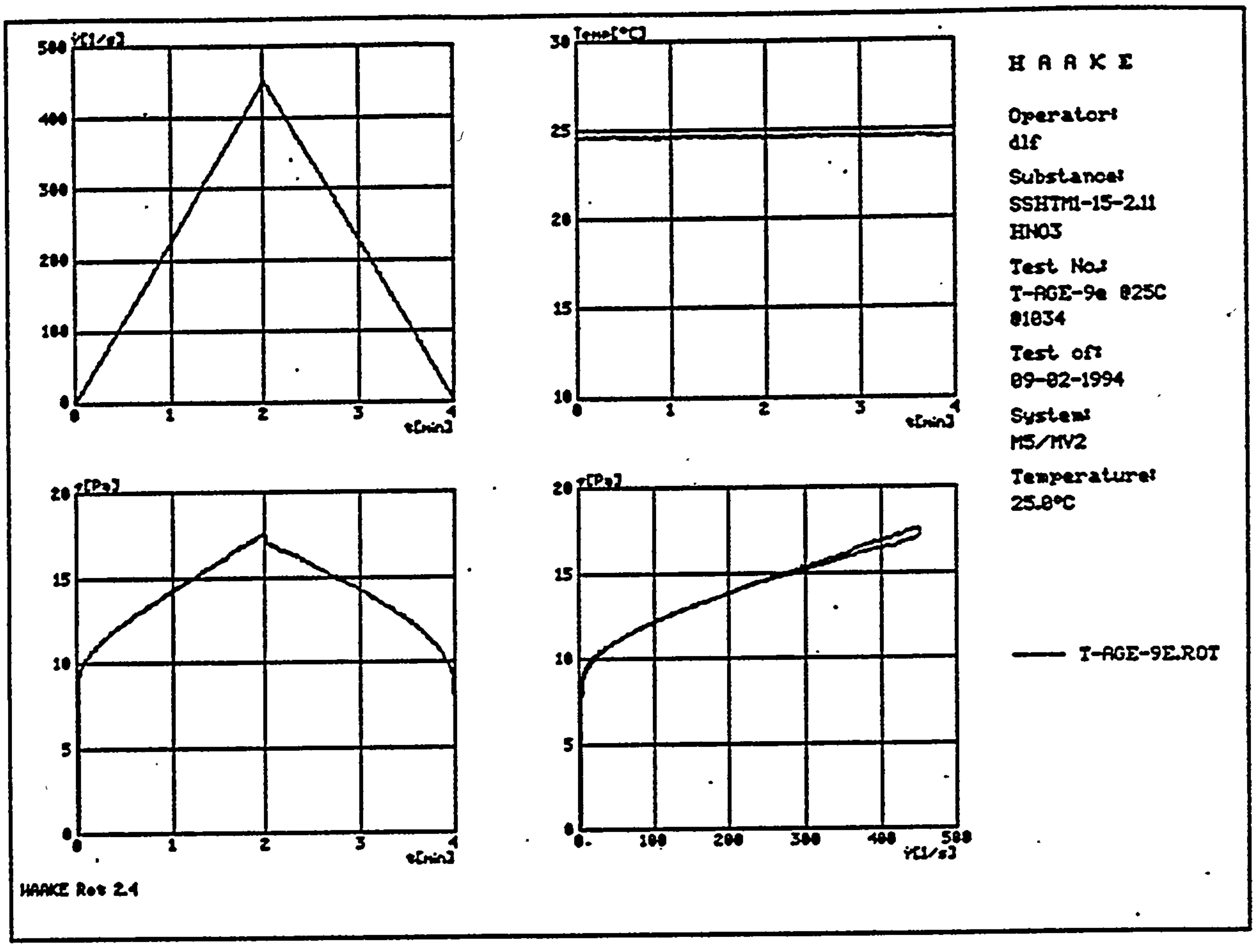


Bingham	: ó= óo+Eta*D	R»=0.87	óo=9.799	Eta=0.01579	
Ostwald	: ó= a*D■	R»=0.75	a=4.045	n=0.2297	
H.-Bulkley	: ó= óo+a*D■	R»=0.93	a=4.453	n=0.1905	óo=1.395
Casson	: «ó-«óo = «(Eta*D)	R»=0.79	óo=7.625	Eta=0.00384	
Linear	: Y= a+b*X	R»=0.87	a=9.799	b=0.01579	
Inv. linear	: Y= 1/(a+b*X)	R»=0.02	a=1.161	b=-0.003637	
Exponential	: Y= a*EXP(b*X)	R»=0.19	a=8.478	b=0.001778	
Hyperbolic	: Y= a+b/X	R»=0.22	a=13.46	b=-3.438	
Logarithmic	: Y= a+b*LN(X)	R»=0.92	a=3.112	b=2.008	

Optimum :

H.-Bulkley	: ó= óo+a*D■	R»=0.93	a=4.453	n=0.1905	óo=1.395

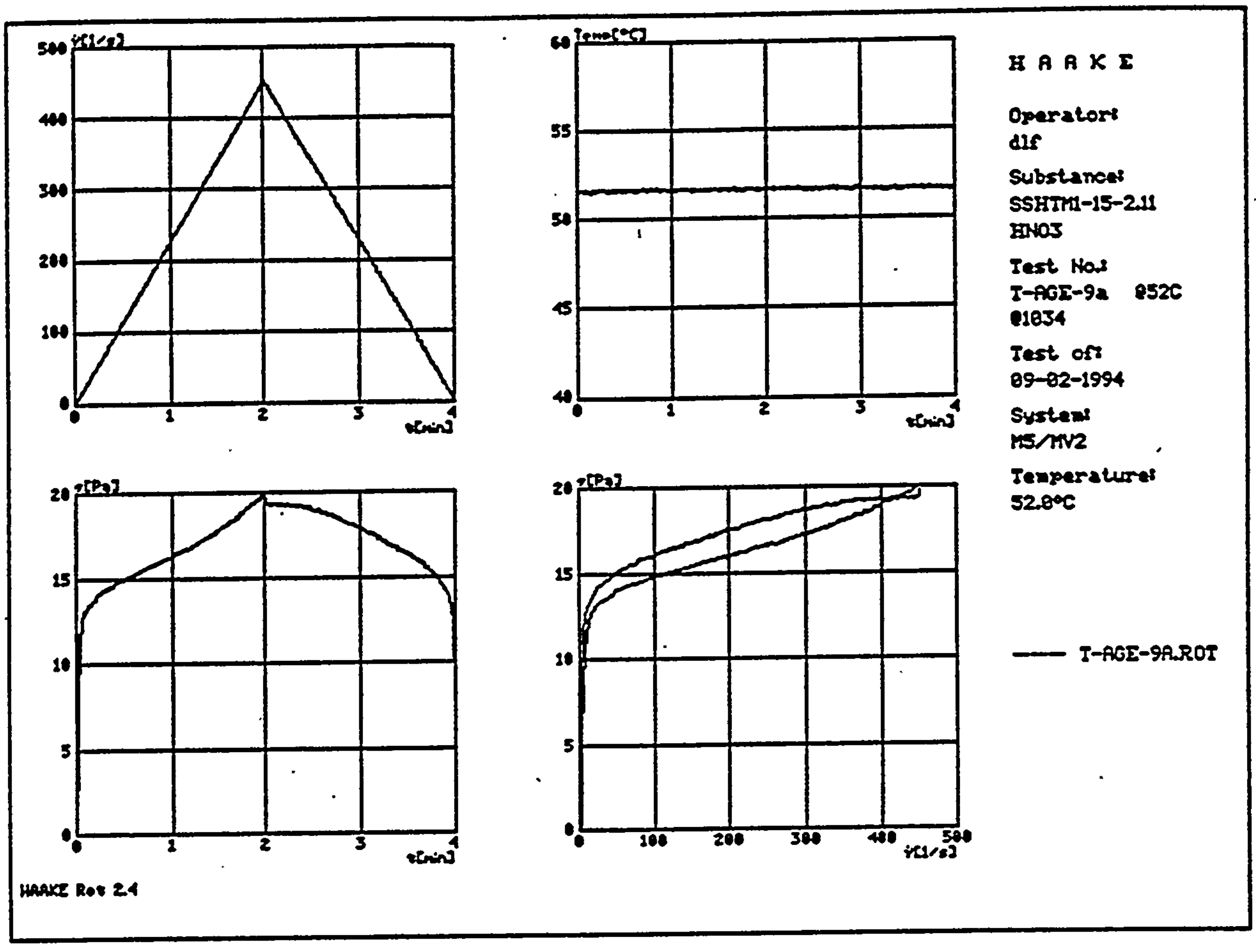
Rheogram and Data for Test Number T-AGE-9e @25C @1034




```
Bingham      : ó= óo+Eta*D       R»=0.90   óo=10.09   Eta=0.01757
Ostwald      : ó= a*D■           R»=0.69   a=4.091    n=0.2369
H.-Bulkley   : ó= óo+a*D■        R»=0.94   a=3.817    n=0.2128   óo=2.511
Casson       : «ó-«óo = «(Eta*D)  R»=0.81   óo=7.768   Eta=0.004449
Linear       : Y= a+b*X          R»=0.90   a=10.09    b=0.01757
Inv. linear: Y= 1/(a+b*X)        R»=0.04   a=0.1376   b=-0.000239
Exponential: Y= a*EXP(b*X)       R»=0.41   a=9.869    b=0.001455
Hyperbolic : Y= a+b/X            R»=0.26   a=14.22    b=-7.670
Logarithmic: Y= a+b*LN(X)        R»=0.90   a=2.781    b=2.210

Optimum :
H.-Bulkley : ó= óo+a*D■          R»=0.94   a=3.817    n=0.2128   óo=2.511
```

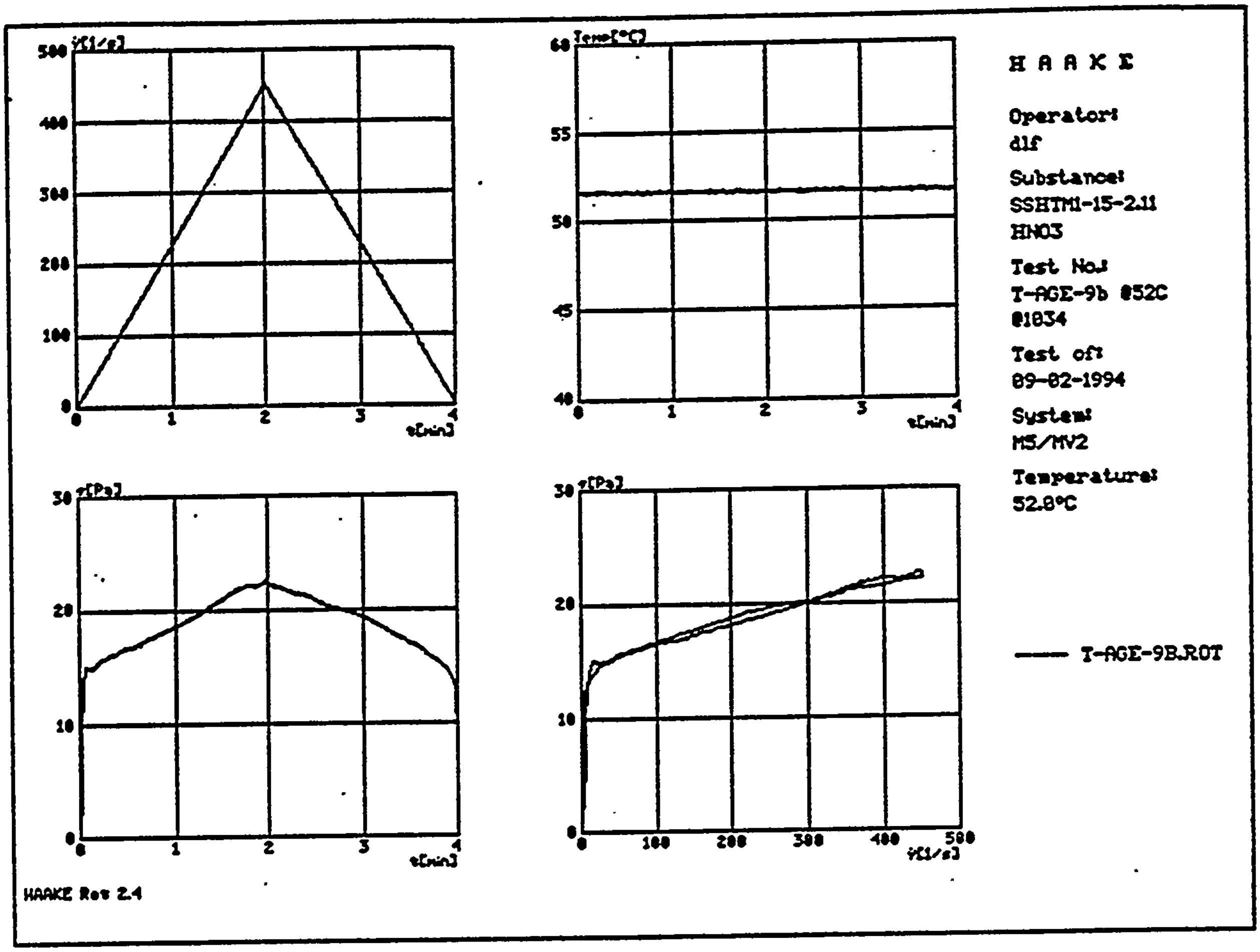
Rheogram and Data for Test Number T-AGE-9a @52C @1034



Bingham	: ó= óo+Eta*D	R»=0.78	óo=12.59	Eta=0.0162	
Ostwald	: ó= a*D■	R»=0.60	a=4.667	n=0.2397	
H.-Bulkley	: ó= óo+a*D■	R»=0.87	a=6.467	n=0.1624	óo=1.267
Casson	: «ó-«óo = «(Eta*D)	R»=0.66	óo=10.03	Eta=0.003575	
Linear	: Y= a+b*X	R»=0.78	a=12.59	b=0.0162	
Inv. linear	: Y= 1/(a+b*X)	R»=0.02	a=0.6906	b=-0.00211	
Exponential	: Y= a*EXP(b*X)	R»=0.16	a=10.67	b=0.001626	
Hyperbolic	: Y= a+b/X	R»=0.29	a=16.37	b=-4.008	
Logarithmic	: Y= a+b*LN(X)	R»=0.92	a=5.227	b=2.161	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.92 a=5.227 b=2.161

Rheogram and Data for Test Number T-AGE-9b @52C @1034

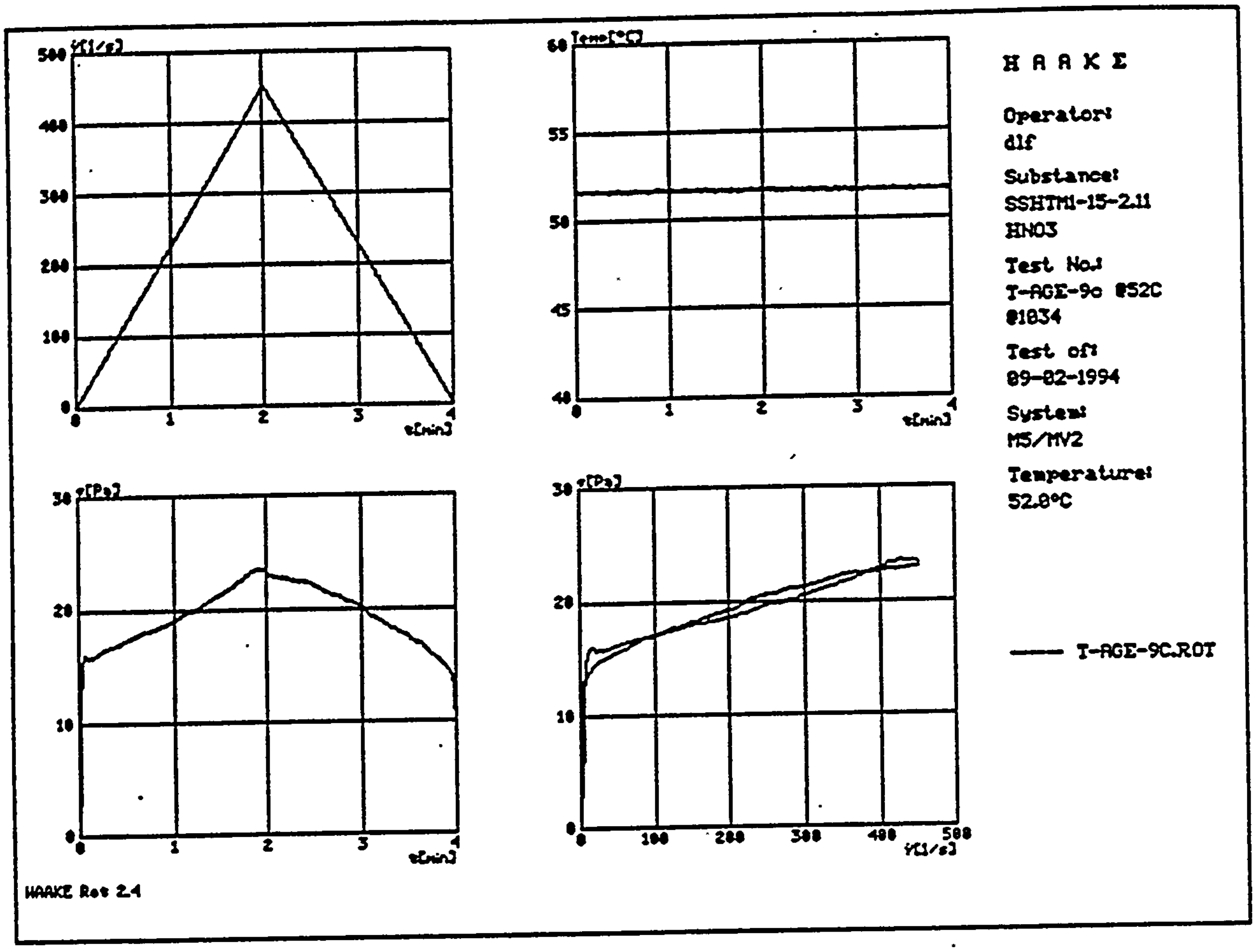


Bingham	: $\tau = \tau_0 + Eta \cdot D$	R»=0.86	τ_0=14.03	Eta=0.02016	
Ostwald	: $\tau = a \cdot D^n$	R»=0.82	a=7.291	n=0.1801	
H.-Bulkley	: $\tau = \tau_0 + a \cdot D^n$	R»=0.63	a=4.124	n=0.2610	τ_0=2.477
Casson	: $\sqrt{\tau} - \sqrt{\tau_0} = \sqrt{Eta \cdot D}$	R»=0.82	τ_0=11.36	Eta=0.004272	
Linear	: Y= a+b*X	R»=0.86	a=14.03	b=0.02016	
Inv. linear	: Y= 1/(a+b*X)	R»=0.11	a=0.0850	b=-115.6E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.43	a=13.41	b=0.001329	
Hyperbolic	: Y= a+b/X	R»=0.29	a=18.82	b=-12.10	
Logarithmic	: Y= a+b*LN(X)	R»=0.88	a=5.376	b=2.586	

Optimum :

Logarithmic	: Y= a+b*LN(X)	R»=0.88	a=5.376	b=2.586

Rheogram and Data for Test Number T-AGE-9c @52C @1034



Bingham	:	б= бo+Eta*D	R»=0.87	бo=14.74	Eta=0.01994	
Ostwald	:	б= a*D■	R»=0.80	a=8.357	n=0.1607	
H.-Bulkley	:	б= бo+a*D■	R»=0.84	a=6.278	n=0.1739	бo=3.763
Casson	:	«б-«бo = «(Eta*D)	R»=0.81	бo=12.23	Eta=0.00383	
Linear	:	Y= a+b*X	R»=0.87	a=14.74	b=0.01994	
Inv. linear	:	Y= 1/(a+b*X)	R»=0.14	a=0.07716	b=-94.64E-06	
Exponential	:	Y= a*EXP(b*X)	R»=0.46	a=14.23	b=0.001236	
Hyperbolic	:	Y= a+b/X	R»=0.28	a=19.47	b=-11.74	
Logarithmic	:	Y= a+b*LN(X)	R»=0.84	a=6.597	b=2.477	

Optimum :

Bingham	:	б= бo+Eta*D	R»=0.87	бo=14.74	Eta=0.01994

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

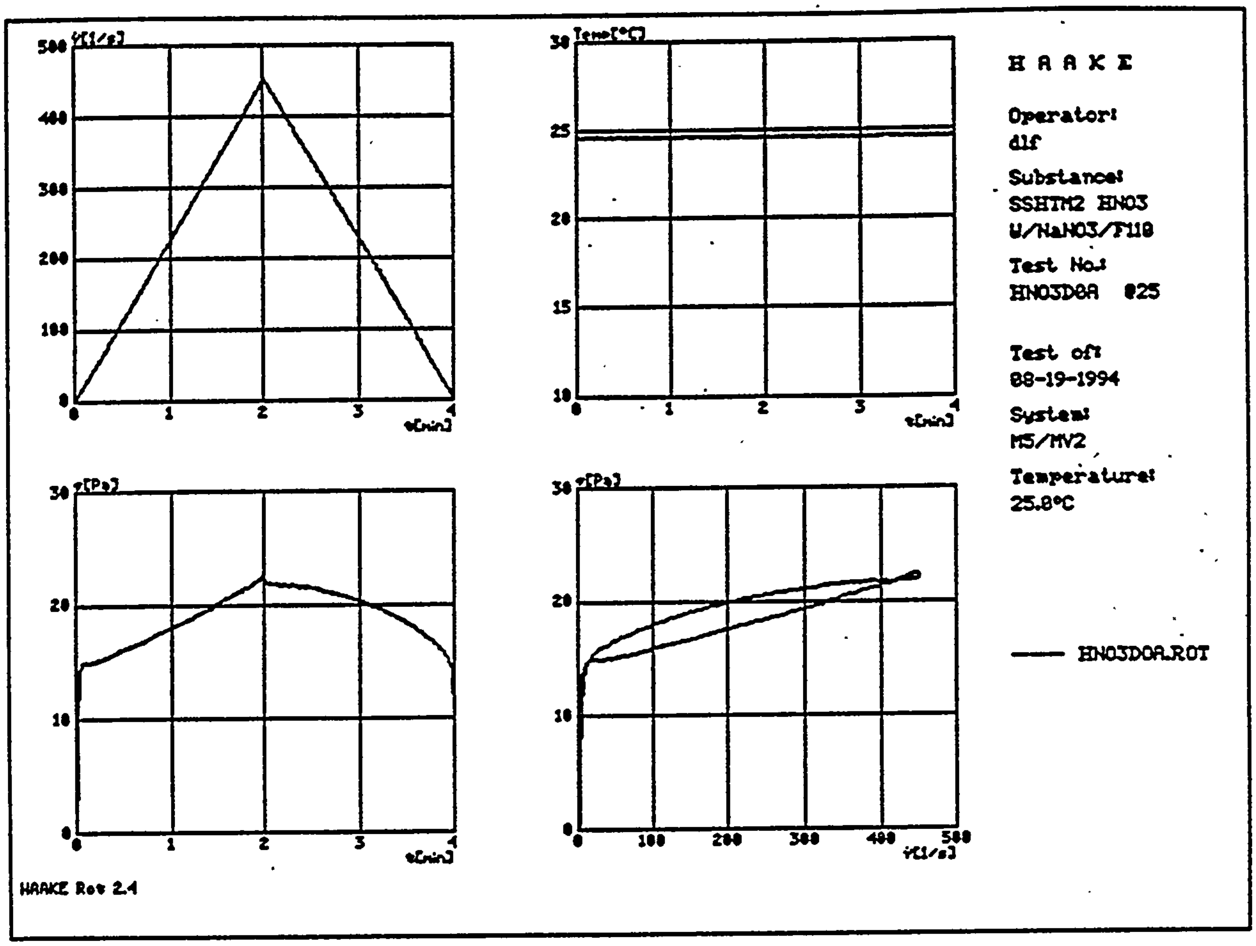
For the HTM feed prepared in the small-scale system but aged in the laboratory, yield stress and viscosity changed with sample age. From day 0 to day 20, yield stress varied between 13.6 Pa and 26.4 Pa (25°C) and the plastic viscosity varied between 18.2 mPa•s to 24.5 mPa•s (25°C). The Bingham model fit the rheological data with correlation coefficients in the range of 0.64 to 0.93. The solids loading of the sample aged at 25°C was 37 ± 1 weight percent throughout the testing period. The pH remained steady at 7.5 ± 0.2 (25°C) for the aging system at 25°C. Summary Table A4.4.2 containing all data from the requisite rheograms and each individual rheogram follows.

Table A4.4.2 Physical Property and Rheology Data for Alternate HTM Flowsheet 1, Laboratory Aged Small-Scale Melter Feed Preparation Samples.

Alternate HTM Flowsheet 1[d]		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]
Aged Small-Scale Samples[e]:								
After silica addition, Day 0:								
File ID/Date:	HNO3D0A/8-19-94	354	----	36.6	1.29	13.6	19.6	0.83
	HNO3D0B/8-19-94	----	----		----	15.1	22.4	0.88
Day 3 after silica addition:								
File ID/Date:	HNO3-D1/8-22-94	----	7.5	37.0	----	26.4	24.5	0.69
	HNO3-D1A/8-22-94	----			----	26.2	23.6	0.68
Day 5 after silica addition:								
File ID/Date:	D5-25A/8-24-94	----	7.4	36.1	----	22.6	19.6	0.73
	HNO-D5A/8-24-94	----			----	23.4	17.3	0.61
Day 7 after silica addition:								
File ID/Date:	HNOD7E/8-26-94	----	7.7	37.0	----	22.5	21.6	0.64
	HNOD7F/8-26-94	----			----	21.8	18.1	0.69
Day 10 after silica addition:								
File ID/Date:	hnod15e/8-29-94	----	7.6	37.8	----	20.8	20.9	0.93
	hnod10f/8-29-94	----			----	24.2	23.8	0.74
Day 13 after silica addition:								
File ID/Date:	hnod14A/9-1-94	----	7.4	37.7	----	17.1	18.2	0.72
	hnod14B/9-1-94	----			----	18.0	19.0	0.77
Day 20 after silica addition:								
File ID/Date:	hnod28A/9-8-94	----	7.5	36.5	----	20.1	22.4	0.69
	hnod28b/9-8-94	----	7.5	36.5	----	20.2	22.3	0.81
	hnod28c/9-8-94	----			----	21.0	20.3	0.76

(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 1 is the no reductant, nitric acid process flowsheet.
(e) Rheology measurements were taken at 25°C for the aged small-scale feed samples.

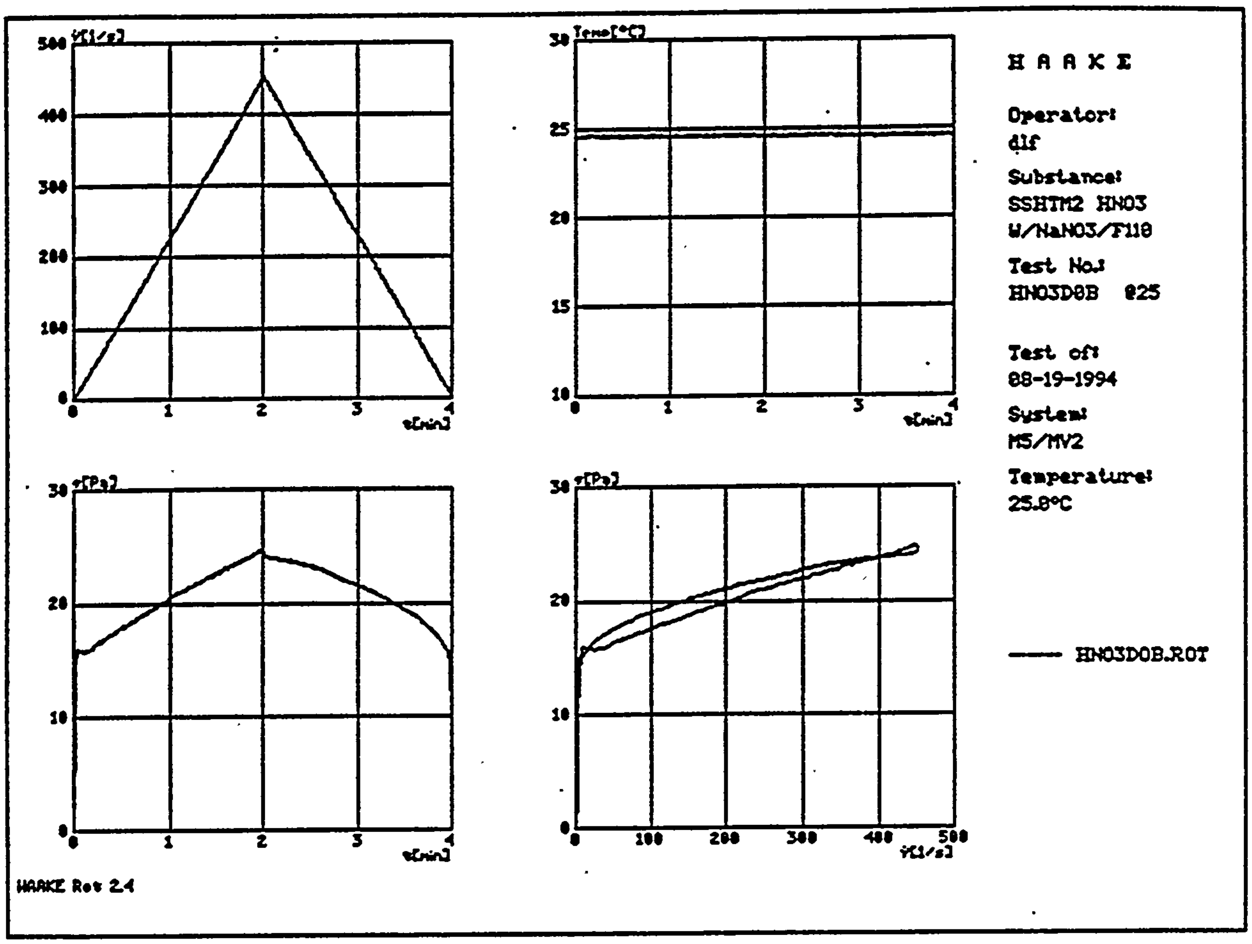
Rheogram and Data for Test Number HNO3D0A @25




```
Bingham      : ő= őo+Eta*D      R»=0.83  őo=13.58   Eta=0.01962
Ostwald      : ő= a*D■          R»=0.66  a=5.755    n=0.2192
H.-Bulkley   : ő= őo+a*D■       R»=0.82  a=7.045    n=0.1654   őo=1.412
Casson       : «ő-«őo = «(Eta*D)    R»=0.71   őo=10.84   Eta=0.004343
Linear       : Y= a+b*X         R»=0.83  a=13.58    b=0.01962
Inv. linear: Y= 1/(a+b*X)       R»=0.02  a=0.8100   b=-0.002527
Exponential: Y= a*EXP(b*X)      R»=0.17  a=11.68    b=0.001685
Hyperbolic  : Y= a+b/X          R»=0.26  a=18.15    b=-5.157
Logarithmic: Y= a+b*LN(X)       R»=0.86  a=5.446    b=2.463

Optimum :
Logarithmic: Y= a+b*LN(X)       R»=0.86  a=5.446    b=2.463
```

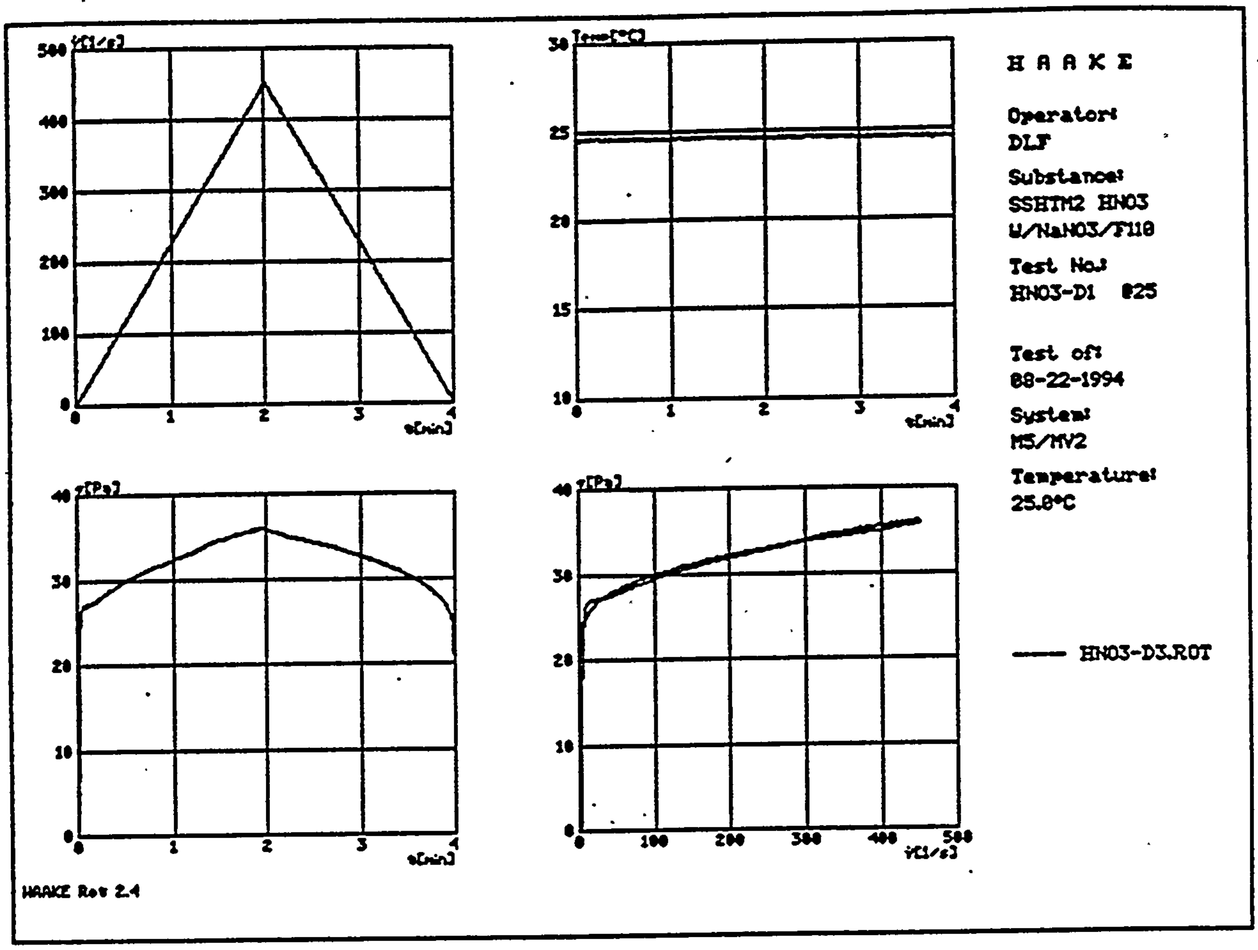
Rheogram and Data for Test Number HNO3D0B @25




```
Bingham      : ő= őo+Eta*D     R»=0.88   őo=15.13   Eta=0.02243
Ostwald      : ő= a*D■         R»=0.79   a=7.651   n=0.1868
H.-Bulkley   : ő= őo+a*D■      R»=0.87   a=5.966   n=0.1912   őo=4.078
Casson       : «ő-«őo = «(Eta*D)   R»=0.81   őo=12.17   Eta=0.004862
Linear       : Y= a+b*X        R»=0.88   a=15.13   b=0.02243
Inv. linear: Y= 1/(a+b*X)     R»=0.06   a=0.09441   b=-160.5E-06
Exponential: Y= a*EXP(b*X)    R»=0.35   a=14.19   b=0.001423
Hyperbolic : Y= a+b/X         R»=0.23   a=20.35   b=-6.060
Logarithmic: Y= a+b*LN(X)     R»=0.87   a=6.063   b=2.768

Optimum :
Bingham      : ő= őo+Eta*D     R»=0.88   őo=15.13   Eta=0.02243
```

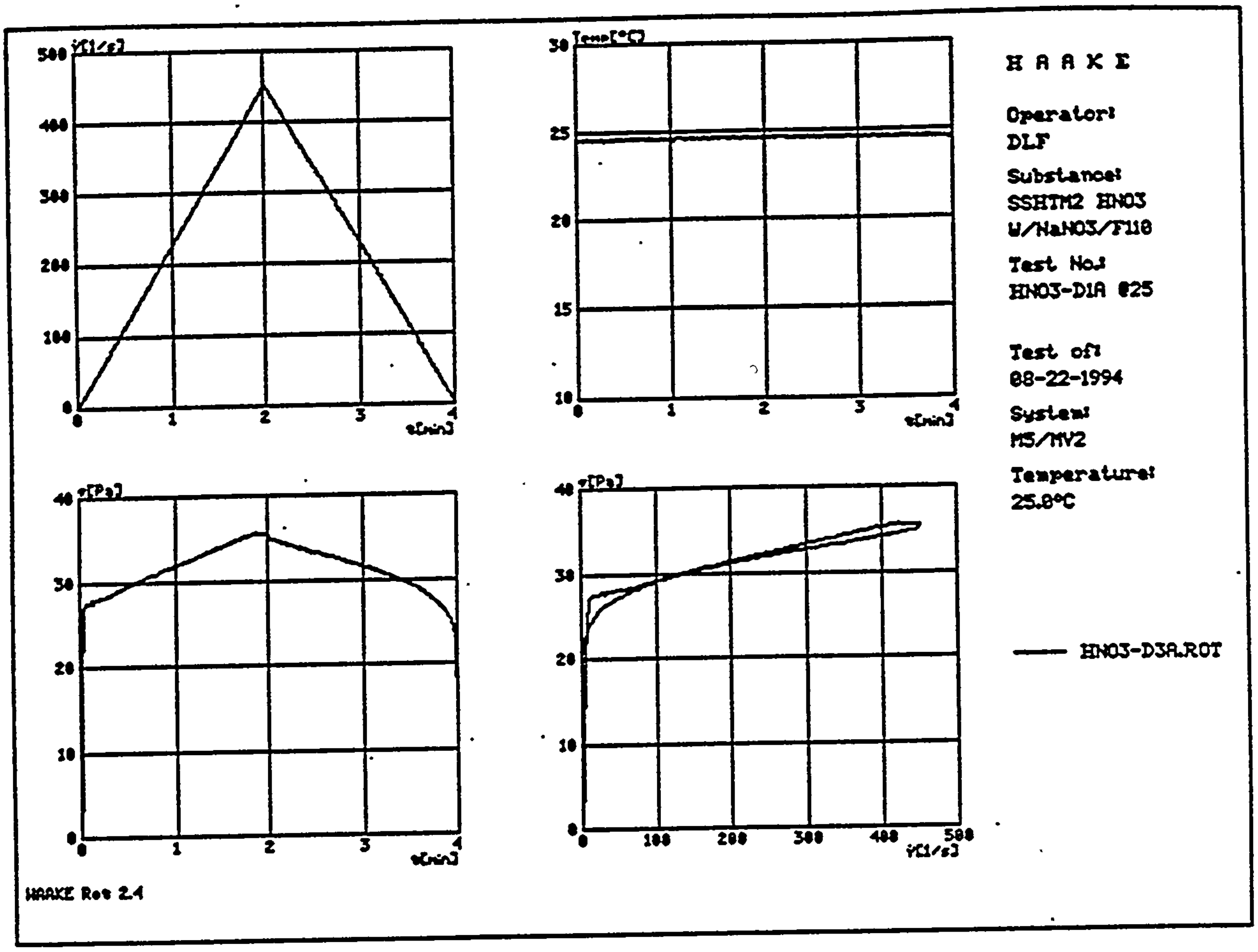
Rheogram and Data for Test Number HNO3-D1 @25



Bingham   : б= бo+Eta*D   R»=0.69 бo=26.36 Eta=0.02447
Ostwald   : б= a*D■   R»=0.54 a=11.86 n=0.1903
H.-Bulkley : б= бo+a*D■   R»=0.83 a=14.42 n=0.1279 бo=4.004
Casson   : «б-«бo = «(Eta*D) R»=0.57 бo=22.04 Eta=0.004357
Linear   : Y= a+b*X   R»=0.69 a=26.36 b=0.02447
Inv. linear: Y= 1/(a+b*X)   R»=0.03 a=0.05711 b=-0.000091
Exponential: Y= a*EXP(b*X)   R»=0.21 a=25.20 b=962.4E-06
Hyperbolic : Y= a+b/X   R»=0.41 a=32.15 b=-9.439
Logarithmic: Y= a+b*LN(X)   R»=0.90 a=14.20 b=3.466

Optimum :
Logarithmic: Y= a+b*LN(X)   R»=0.90 a=14.20 b=3.466

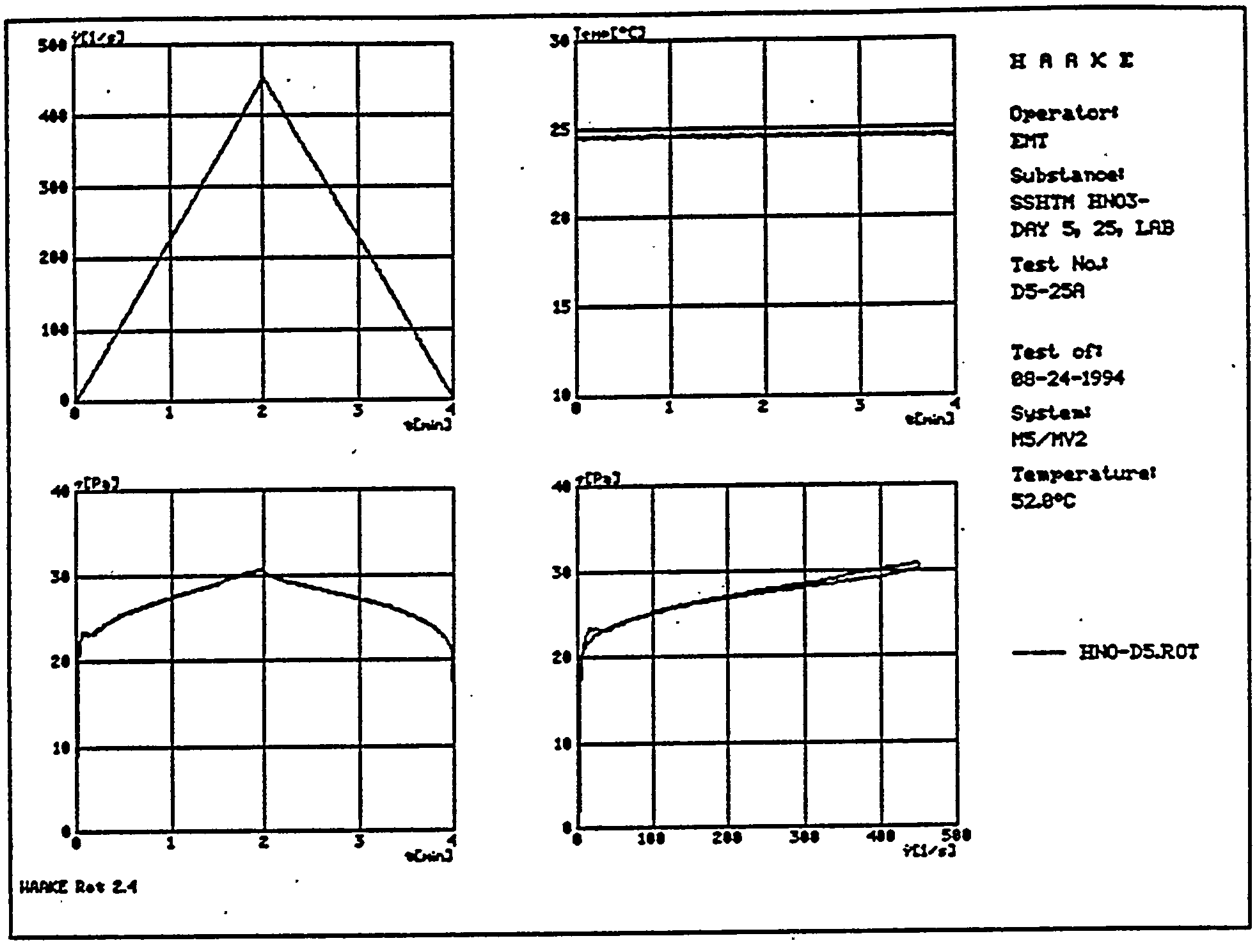
Rheogram and Data for Test Number HNO3-D1A @25



Bingham : ó= óo+Eta*D R»=0.68 óo=26.24 Eta=0.02358
Ostwald : ó= a*D■ R»=0.67 a=14.44 n=0.1509
H.-Bulkley : ó= óo+a*D■ R»=0.48 a=8.133 n=0.2345 óo=3.935
Casson : «ó-«óo = «(Eta*D) R»=0.62 óo=22.29 Eta=0.003895
Linear : Y= a+b*X R»=0.68 a=26.24 b=0.02358
Inv. linear: Y= 1/(a+b*X) R»=0.07 a=0.04941 b=-64.20E-06
Exponential: Y= a*EXP(b*X) R»=0.26 a=24.49 b=0.001019
Hyperbolic : Y= a+b/X R»=0.50 a=32.00 b=-20.85
Logarithmic: Y= a+b*LN(X) R»=0.88 a=14.37 b=3.369

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.88 a=14.37 b=3.369

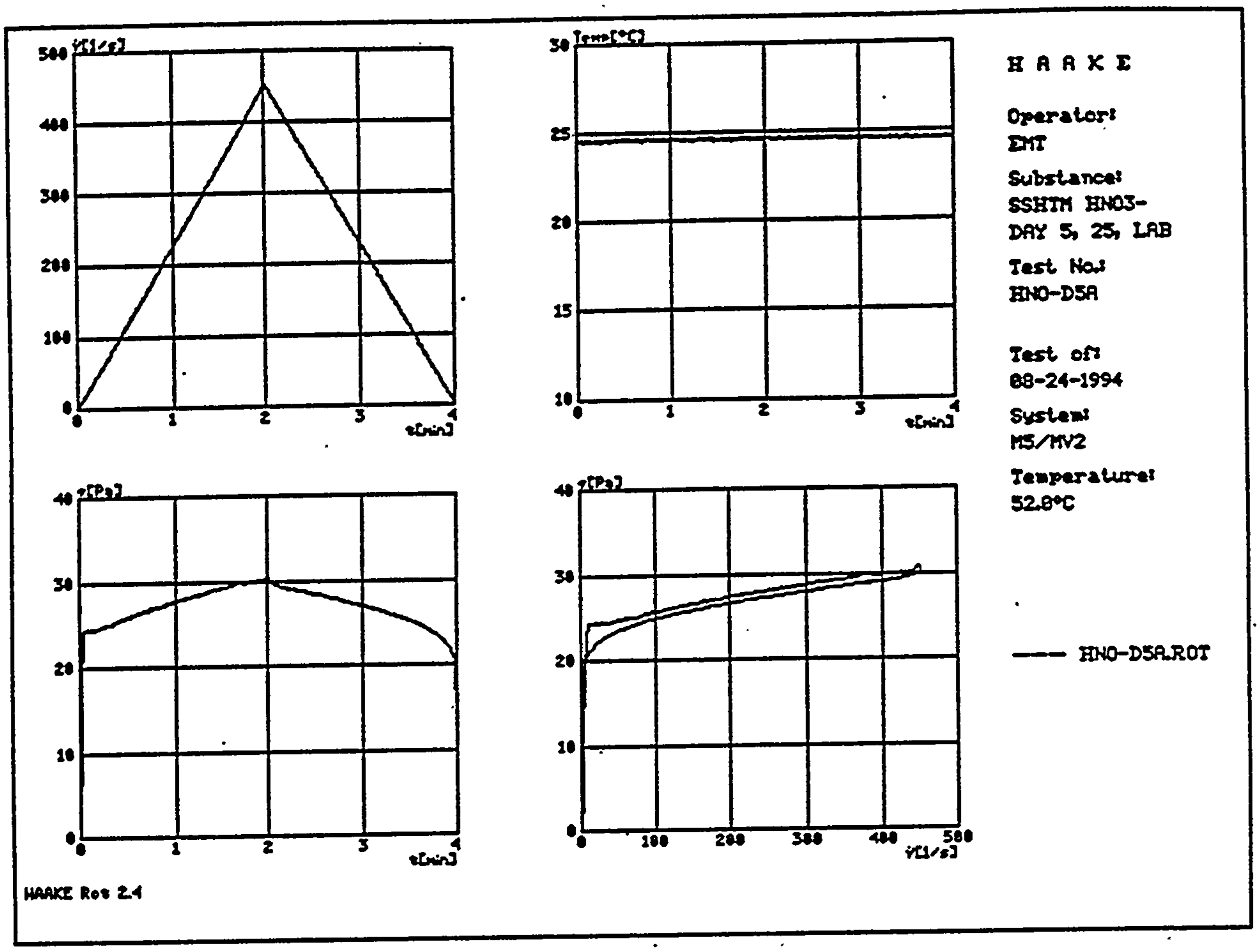
Rheogram and Data for Test Number D5-25A




```
Bingham     : б= бo+Eta*D      R»=0.73   бo=22.64   Eta=0.01959
Ostwald     : б= a*D■          R»=0.68   a=13.07   n=0.1406
H.-Bulkley  : б= бo+a*D■       R»=0.92   a=10.14   n=0.1346   бo=6.772
Casson      : «б-«бo = «(Eta*D)  R»=0.66   бo=19.46   Eta=0.003031
Linear      : Y= a+b*X         R»=0.73   a=22.64   b=0.01959
Inv. linear: Y= 1/(a+b*X)      R»=0.04   a=0.06968   b=-114.9E-06
Exponential: Y= a*EXP(b*X)     R»=0.20   a=20.88   b=0.001032
Hyperbolic  : Y= a+b/X         R»=0.42   a=27.29   b=-8.527
Logarithmic: Y= a+b*LN(X)      R»=0.91   a=13.20   b=2.718

Optimum :
H.-Bulkley  : б= бo+a*D■       R»=0.92   a=10.14   n=0.1346   бo=6.772
```

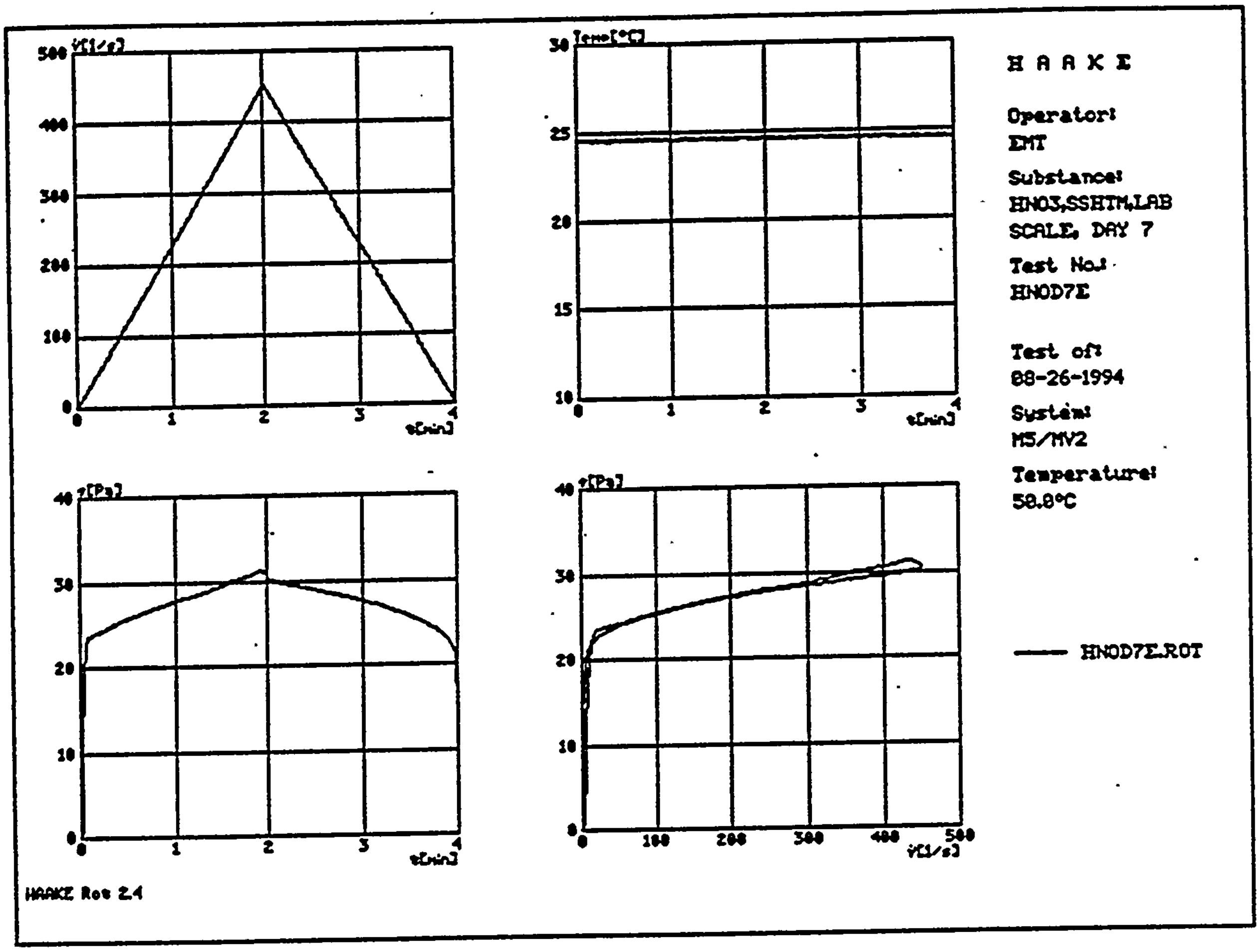
Rheogram and Data for Test Number HNO-D5A




```
Bingham      : б= бo+Eta*D       R»=0.61   бo=23.41   Eta=0.01734
Ostwald      : б= a*D■           R»=0.63   a=13.15   n=0.1413
H.-Bulkley   : б= бo+a*D■        R»=0.71   a=12.57   n=0.1172   бo=4.362
Casson       : «б-«бo = «(Eta*D)   R»=0.54   бo=20.27   Eta=0.002547
Linear       : Y= a+b*X          R»=0.61   a=23.41   b=0.01734
Inv. linear: Y= 1/(a+b*X)        R»=0.05   a=0.06008   b=-83.47E-06
Exponential: Y= a*EXP(b*X)       R»=0.19   a=21.64   b=927.2E-06
Hyperbolic : Y= a+b/X            R»=0.45   a=27.53   b=-6.862
Logarithmic: Y= a+b*LN(X)        R»=0.87   a=14.29   b=2.555

Optimum :
Logarithmic: Y= a+b*LN(X)        R»=0.87   a=14.29   b=2.555
```

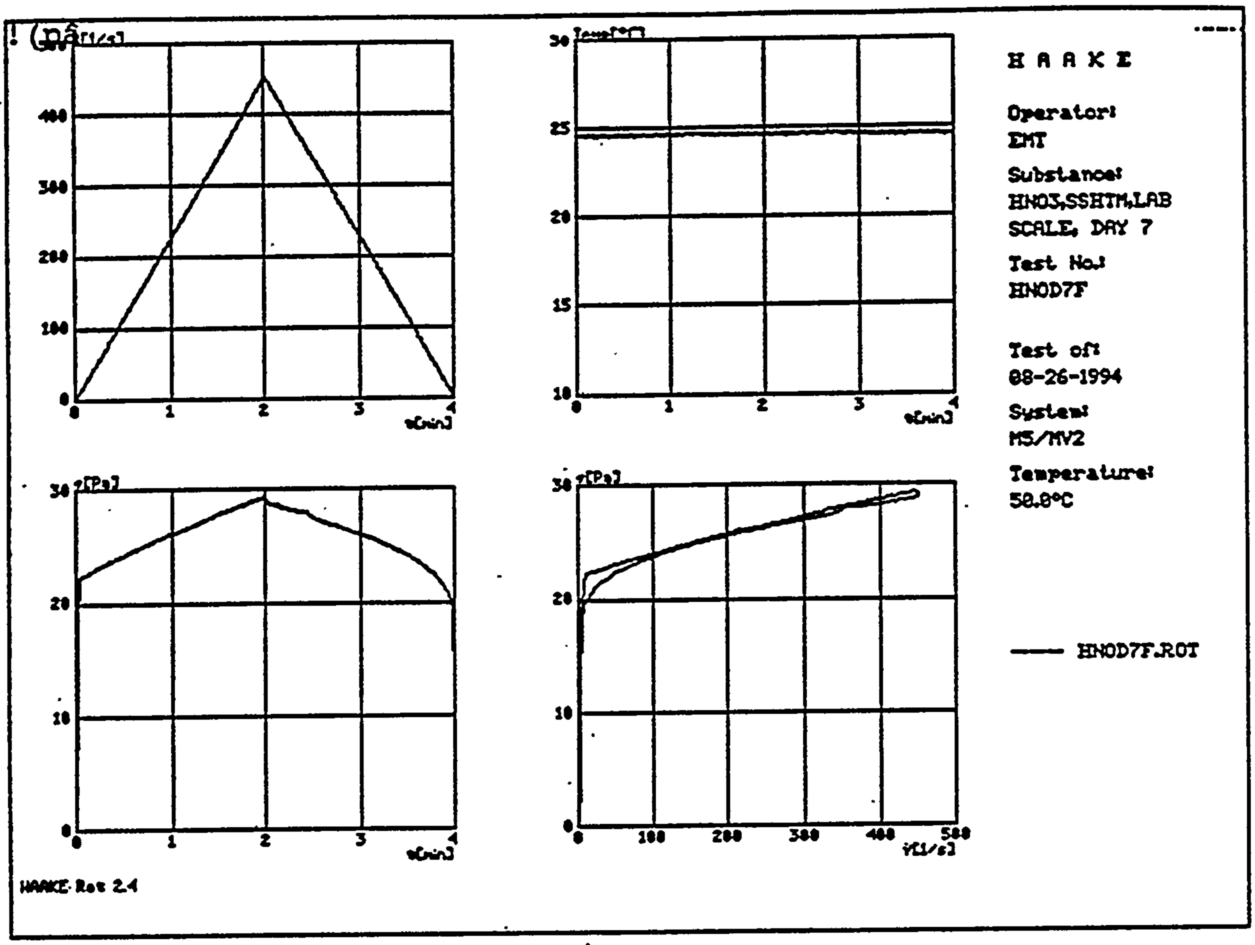
Rheogram and Data for Test Number HNOD7E



Bingham	: ó= óo+Eta*D	R»=0.64	óo=22.48	Eta=0.02155	
Ostwald	: ó= a*D■	R»=0.59	a=10.13	n=0.1909	
H.-Bulkley	: ó= óo+a*D■	R»=0.55	a=9.260	n=0.2047	óo=0.4773
Casson	: «ó-«óo = «(Eta*D)	R»=0.57	óo=18.52	Eta=0.004117	
Linear	: Y= a+b*X	R»=0.64	a=22.48	b=0.02155	
Inv. linear	: Y= 1/(a+b*X)	R»=0.02	a=0.3975	b=-0.001209	
Exponential	: Y= a*EXP(b*X)	R»=0.13	a=19.16	b=0.001369	
Hyperbolic	: Y= a+b/X	R»=0.50	a=27.76	b=-21.56	
Logarithmic	: Y= a+b*LN(X)	R»=0.90	a=10.77	b=3.246	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.90 a=10.77 b=3.246

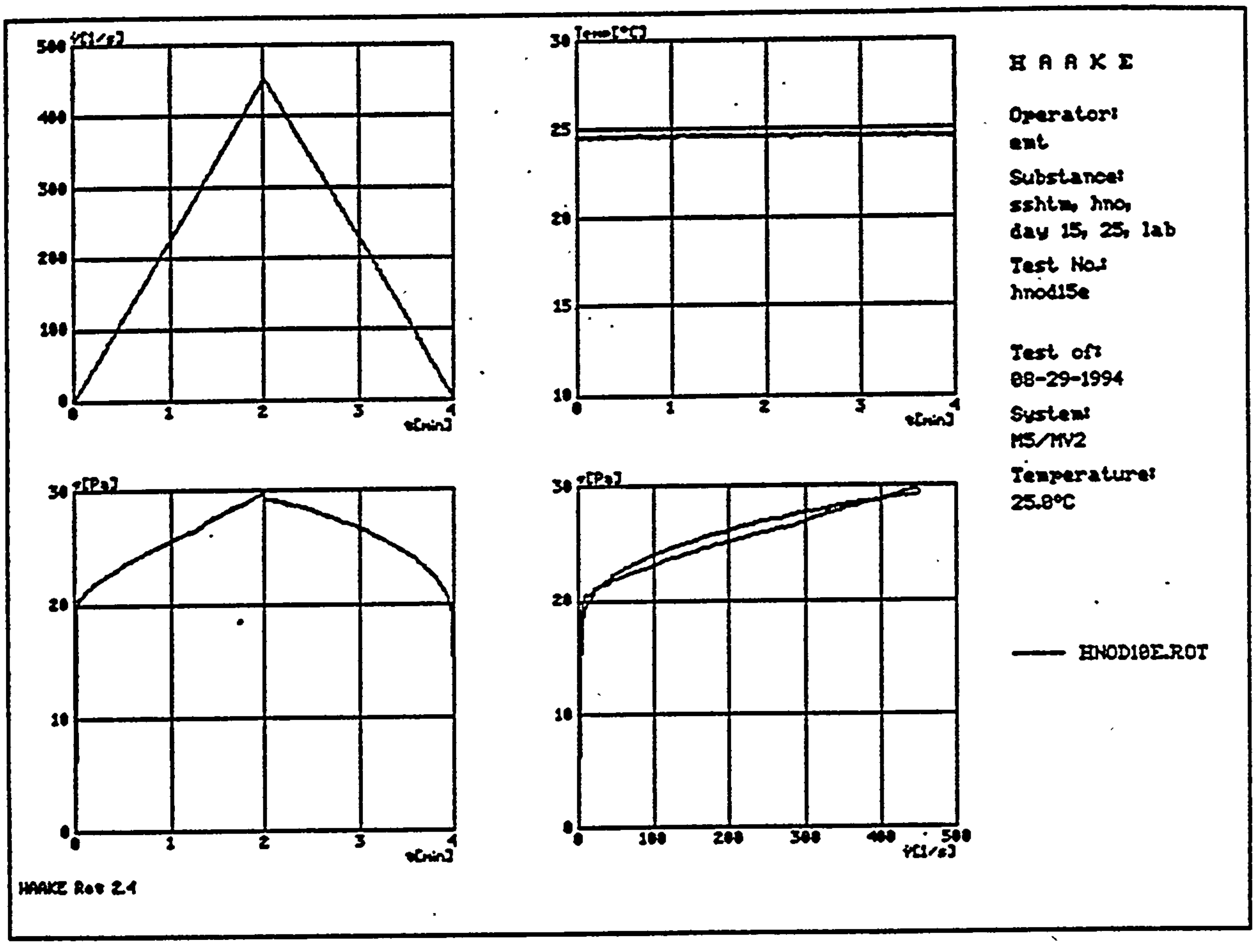
Rheogram and Data for Test Number HNOD7F



Bingham	: ó= óo+Eta*D	R»=0.69	óo=21.78	Eta=0.01814	
Ostwald	: ó= a*D■	R»=0.66	a=12.50	n=0.1405	
H.-Bulkley	: ó= óo+a*D■	R»=0.81	a=10.73	n=0.1266	óo=5.274
Casson	: «ó-«óo = «(Eta*D)	R»=0.62	óo=18.78	Eta=0.002746	
Linear	: Y= a+b*X	R»=0.69	a=21.78	b=0.01814	
Inv. linear	: Y= 1/(a+b*X)	R»=0.05	a=0.06416	b=-90.94E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.22	a=20.23	b=983.5E-06	
Hyperbolic	: Y= a+b/X	R»=0.43	a=26.08	b=-7.812	
Logarithmic	: Y= a+b*LN(X)	R»=0.88	a=12.92	b=2.538	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.88 a=12.92 b=2.538

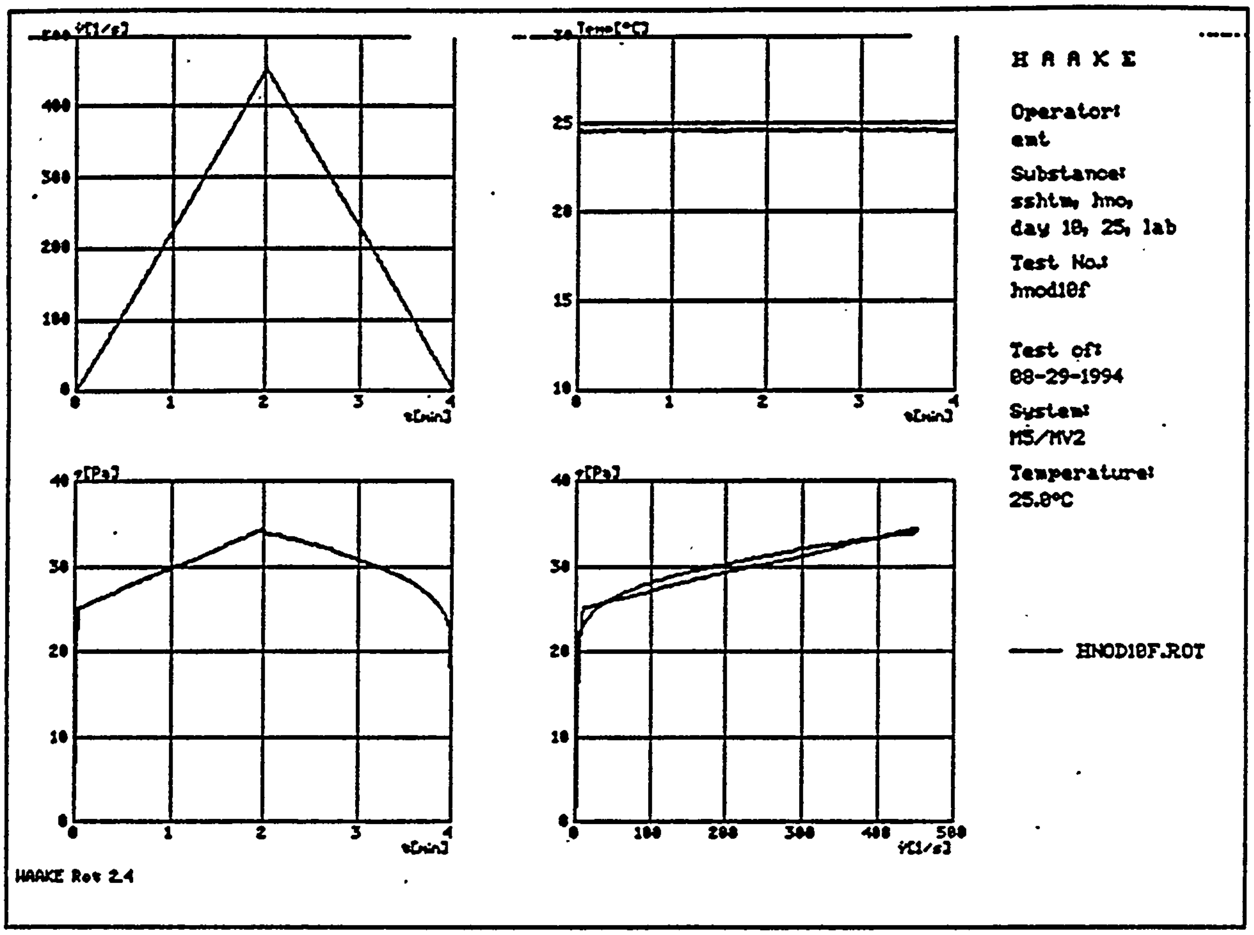
Rheogram and Data for Test Number hnod15e




```
Bingham      : б= бo+Eta*D        R»=0.93   бo=20.77   Eta=0.02089
Ostwald      : б= a*D■            R»=0.91   a=14.14   n=0.1139
H.-Bulkley   : б= бo+a*D■         R»=0.93   a=3.804   n=0.2344   бo=12.57
Casson       : «б-«бo = «(Eta*D)  R»=0.94   бo=18.07   Eta=0.003103
Linear       : Y= a+b*X           R»=0.93   a=20.77   b=0.02089
Inv. linear: Y= 1/(a+b*X)         R»=0.02   a=0.1247   b=-291.7E-06
Exponential: Y= a*EXP(b*X)        R»=0.18   a=19.17   b=0.001136
Hyperbolic : Y= a+b/X             R»=0.35   a=25.93   b=-29.83
Logarithmic: Y= a+b*LN(X)         R»=0.89   a=11.89   b=2.658

Optimum :
Casson       : «б-«бo = «(Eta*D)  R»=0.94   бo=18.07   Eta=0.003103
```

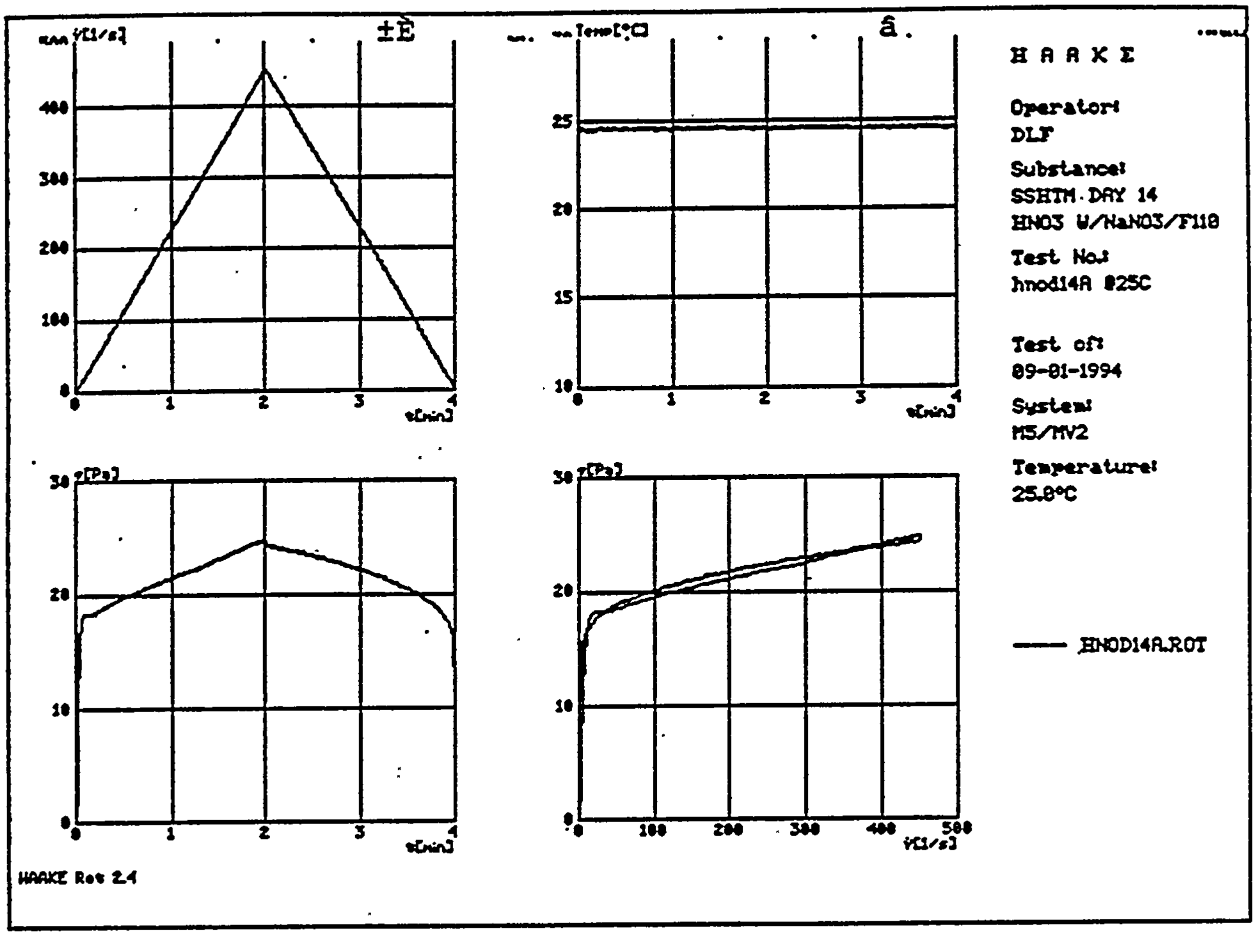
Rheogram and Data for Test Number hnod10f



Bingham :	ő= őo+Eta*D	R»=0.74	őo=24.23	Eta=0.02381	
Ostwald :	ő= a*D■	R»=0.66	a=12.85	n=0.1609	
H.-Bulkley :	ő= őo+a*D■	R»=0.80	a=12.09	n=0.1379	őo=4.967
Casson :	«ő-«őo = «(Eta*D)	R»=0.64	őo=20.48	Eta=0.004052	
Linear :	Y= a+b*X	R»=0.74	a=24.23	b=0.02381	
Inv. linear:	Y= 1/(a+b*X)	R»=0.04	a=0.06494	b=-109.6E-06	
Exponential:	Y= a*EXP(b*X)	R»=0.22	a=22.28	b=0.001132	
Hyperbolic :	Y= a+b/X	R»=0.37	a=29.84	b=-8.615	
Logarithmic:	Y= a+b*LN(X)	R»=0.88	a=13.23	b=3.209	

Optimum :
Logarithmic: Y= a+b*LN(X) R»=0.88 a=13.23 b=3.209

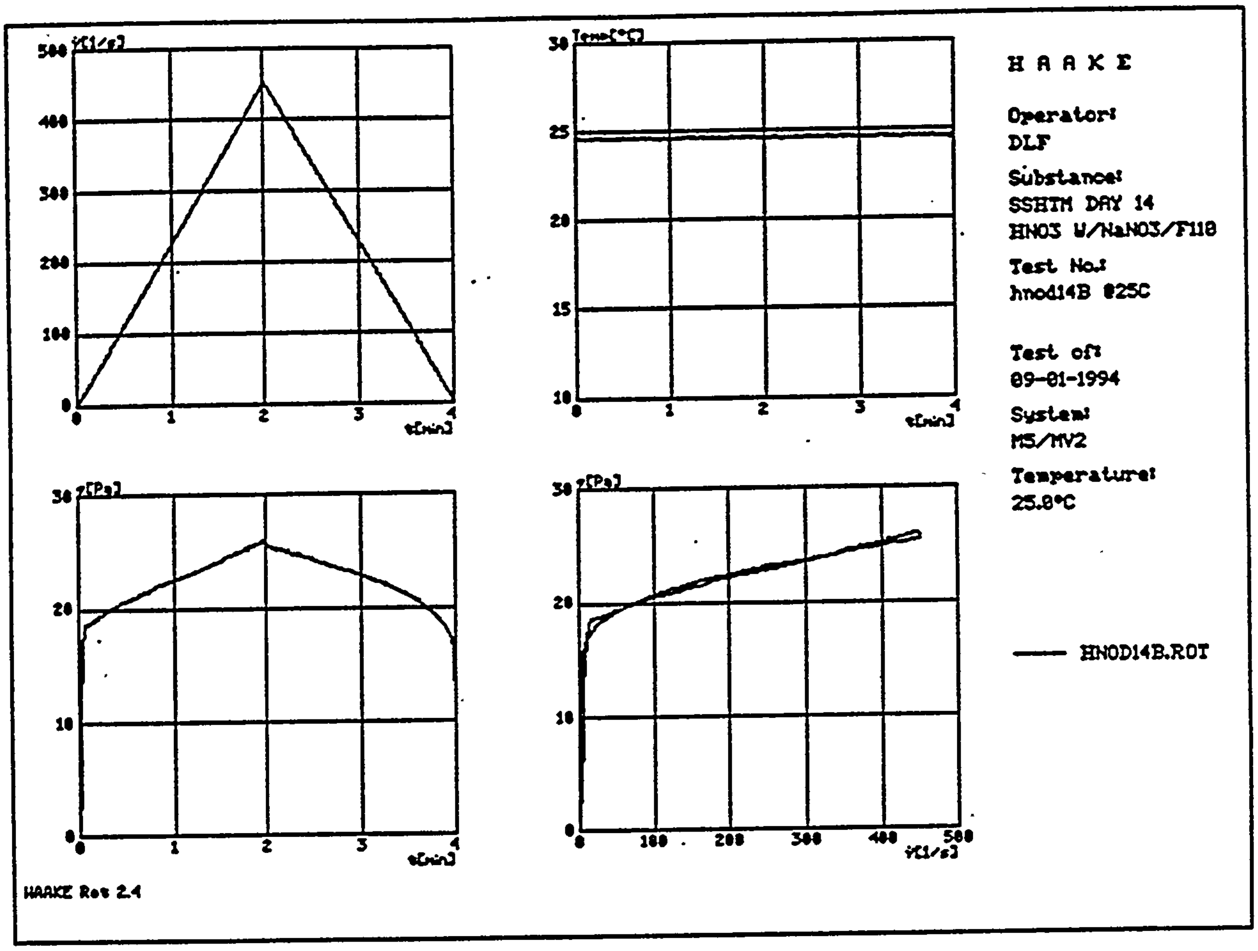
Rheogram and Data for Test Number hnod14a @25C




```
Bingham     : ó= óo+Eta*D      R»=0.72  óo=17.12  Eta=0.01817
Ostwald     : ó= a*D■          R»=0.71  a=8.614   n=0.1738
H.-Bulkley  : ó= óo+a*D■       R»=0.55  a=5.449   n=0.2428   óo=1.911
Casson      : «ó-«óo = «(Eta*D)  R»=0.66  óo=14.17  Eta=0.003422
Linear      : Y= a+b*X         R»=0.72  a=17.12   b=0.01817
Inv. linear: Y= 1/(a+b*X)      R»=0.06  a=0.08126  b=-122.4E-06
Exponential: Y= a*EXP(b*X)     R»=0.27  a=15.85   b=0.001169
Hyperbolic : Y= a+b/X          R»=0.39  a=21.45   b=-10.17
Logarithmic: Y= a+b*LN(X)      R»=0.90  a=8.257   b=2.541

Optimum :
Logarithmic: Y= a+b*LN(X)      R»=0.90  a=8.257   b=2.541
```

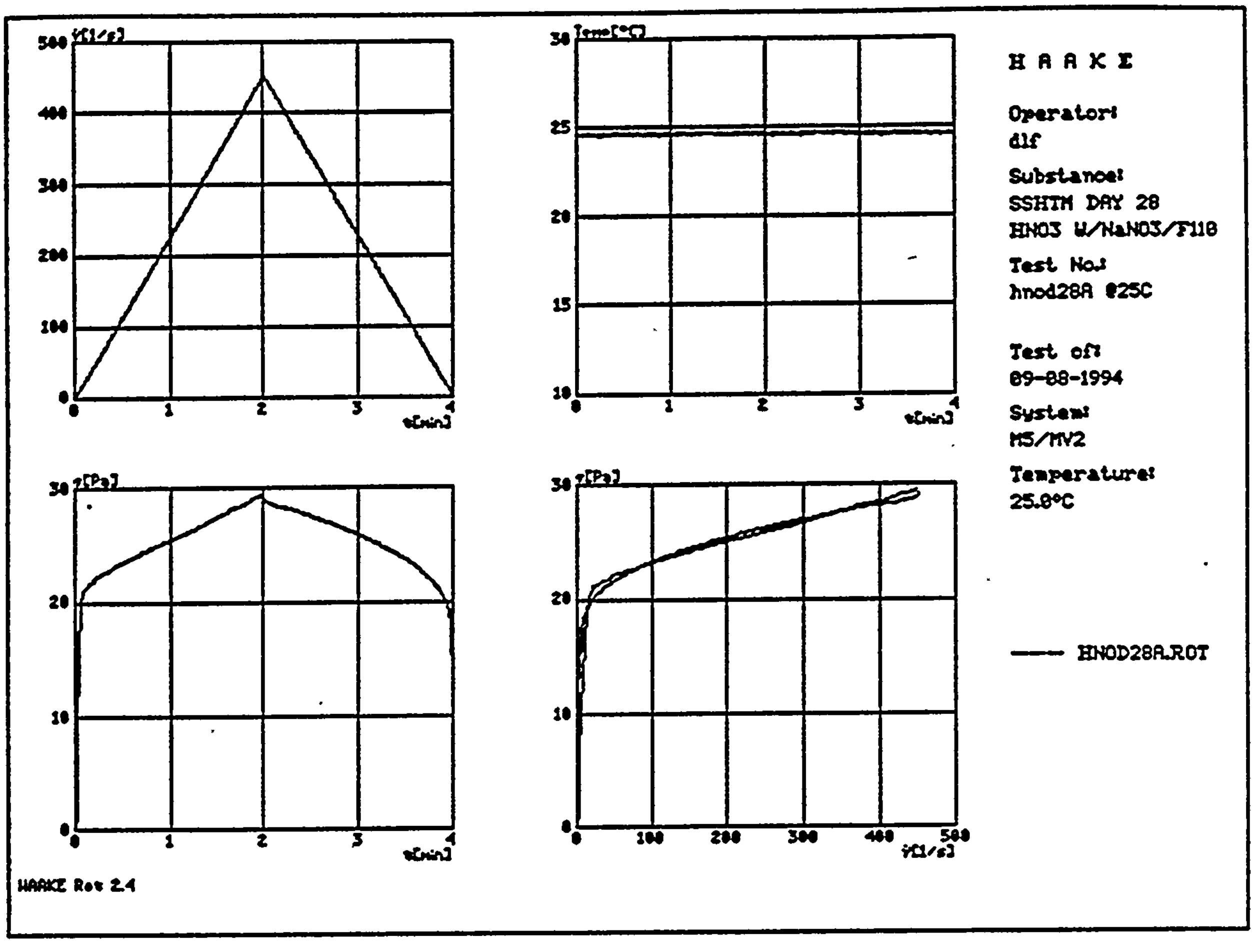
Rheogram and Data for Test Number hnod14B @25C




```
Bingham     : ó= óo+Eta*D        R»=0.77   óo=17.98   Eta=0.01897
Ostwald     : ó= a*D■            R»=0.77   a=9.858   n=0.1572
H.-Bulkley  : ó= óo+a*D■         R»=0.88   a=8.486   n=0.1536   óo=3.495
Casson      : «ó-«óo = «(Eta*D)  R»=0.73   óo=15.05   Eta=0.003408
Linear      : Y= a+b*X           R»=0.77   a=17.98   b=0.01897
Inv. linear: Y= 1/(a+b*X)        R»=0.09   a=0.0685   b=-86.76E-06
Exponential: Y= a*EXP(b*X)       R»=0.33   a=16.98   b=0.0011
Hyperbolic  : Y= a+b/X           R»=0.40   a=22.55   b=-14.50
Logarithmic: Y= a+b*LN(X)        R»=0.92   a=8.805   b=2.636

Optimum :
Logarithmic: Y= a+b*LN(X)        R»=0.92   a=8.805   b=2.636
```

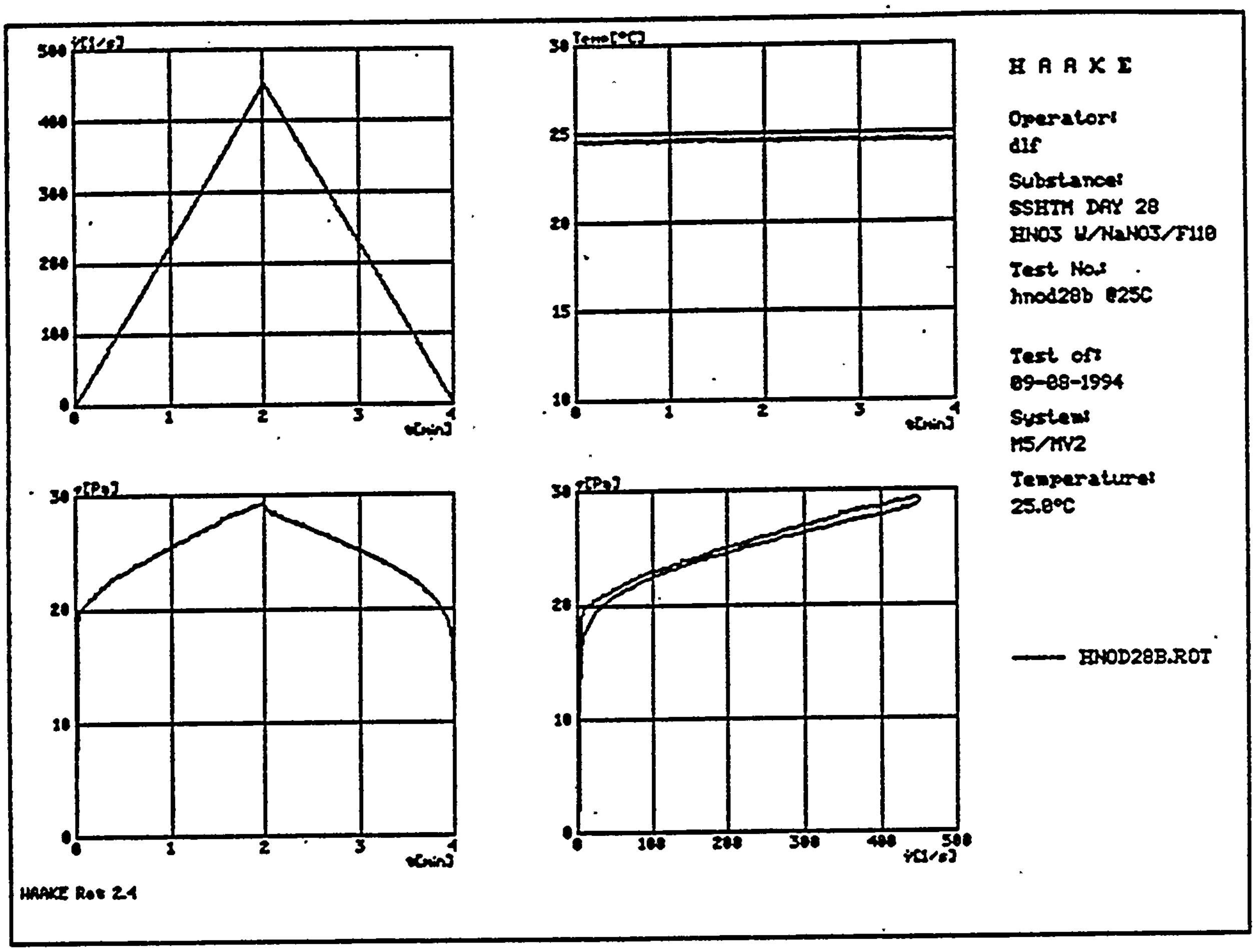
Rheogram and Data for Test Number hnod28A @25C




```
Bingham     : ó= óo+Eta*D       R»=0.69  óo=20.08  Eta=0.02238
Ostwald     : ó= a*D■           R»=0.70  a=8.807  n=0.2018
H.-Bulkley  : ó= óo+a*D■        R»=0.61  a=6.872  n=0.2404  óo=1.100
Casson      : «ó-«óo = «(Eta*D)  R»=0.64  óo=16.19  Eta=0.004705
Linear      : Y= a+b*X          R»=0.69  a=20.08  b=0.02238
Inv. linear: Y= 1/(a+b*X)       R»=0.02  a=0.7706  b=-0.002444
Exponential: Y= a*EXP(b*X)      R»=0.13  a=16.98  b=0.001513
Hyperbolic : Y= a+b/X           R»=0.38  a=25.42  b=-12.54
Logarithmic: Y= a+b*LN(X)       R»=0.93  a=8.542  b=3.252

Optimum :
Logarithmic: Y= a+b*LN(X)       R»=0.93  a=8.542  b=3.252
```

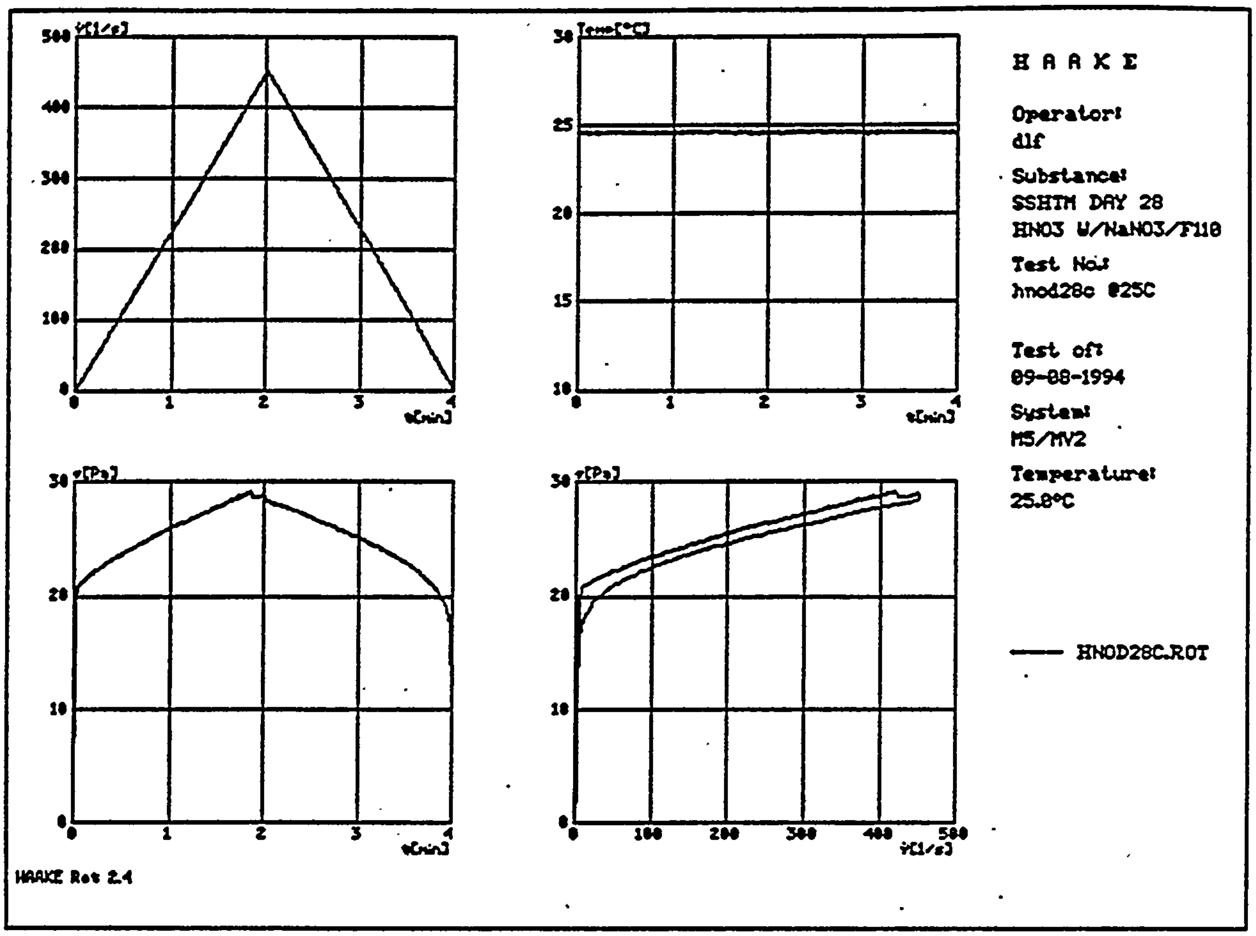
Rheogram and Data for Test Number hnod28b @25C



Bingham	: б= бo+Eta*D	R»=0.81	бo=20.20	Eta=0.02226	
Ostwald	: б= a*D■	R»=0.75	a=10.98	n=0.1605	
H.-Bulkley	: б= бo+a*D■	R»=0.92	a=8.077	n=0.1648	бo=6.281
Casson	: «б-«бo = «(Eta*D)	R»=0.75	бo=16.87	Eta=0.004052	
Linear	: Y= a+b*X	R»=0.81	a=20.20	b=0.02226	
Inv. linear	: Y= 1/(a+b*X)	R»=0.05	a=0.06937	b=-106.5E-06	
Exponential	: Y= a*EXP(b*X)	R»=0.28	a=18.84	b=0.001174	
Hyperbolic	: Y= a+b/X	R»=0.32	a=25.42	b=-7.124	
Logarithmic	: Y= a+b*LN(X)	R»=0.92	a=10.24	b=2.936	

Optimum :
H.-Bulkley : б= бo+a*D■ R»=0.92 a=8.077 n=0.1648 бo=6.281

Rheogram and Data for Test Number hnod28c @25C



Bingham	:	ó= óo+Eta*D	R»=0.76	óo=21.01	Eta=0.02026	
Ostwald	:	ó= a*D■	R»=0.70	a=11.53	n=0.1539	
H.-Bulkley	:	ó= óo+a*D■	R»=0.90	a=9.163	n=0.1467	óo=6.053
Casson	:	«ó-«óo = «(Eta*D)	R»=0.68	óo=17.79	Eta=0.003421	
Linear	:	Y= a+b*X	R»=0.76	a=21.01	b=0.02026	
Inv. linear	:	Y= 1/(a+b*X)	R»=0.05	a=0.07102	b=-113.1E-06	
Exponential	:	Y= a*EXP(b*X)	R»=0.23	a=19.44	b=0.001099	
Hyperbolic	:	Y= a+b/X	R»=0.36	a=25.77	b=-6.423	
Logarithmic	:	Y= a+b*LN(X)	R»=0.92	a=11.55	b=2.752	

Optimum :

Logarithmic	:	Y= a+b*LN(X)	R»=0.92	a=11.55	b=2.752

4.5 Standard Crucible and Melter Glass Redox Test Data

Introduction

This section addresses the measurement of a glass oxidation state indicator, the $Fe^{+2}/\Sigma Fe$ 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 1. The $Fe^{+2}/\Sigma Fe$ ratios of glass samples collected from the small-scale melter were also determined for this flowsheet. The measured $Fe^{+2}/\Sigma Fe$ ratios are given in Table A4.5.

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 glass[1]. 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 glasses[2].

Results

The relative quantities of oxidants are given in Table A4.5 (a reductant was not added during Alternate HTM Flowsheet 1 processing). A slightly lower, 10-20%, quantity of oxidants was present in the small-scale melter feed relative to the laboratory-scale melter feed. Sensitivity of the $Fe^{+2}/\Sigma Fe$ ratio to differences of this order of magnitude is unknown. The $Fe^{+2}/\Sigma Fe$ ratio of glasses prepared from the small-scale melter feed, using the standard crucible method, was 0.067 (mean value). The $Fe^{+2}/\Sigma Fe$ ratio of glasses collected from the bottom drain of the SSHTM and a canister pour ranged from 0.419 to 0.302. Comparison of the crucible and small-scale glass redox values suggest that glass prepared in the SSHTM with the nitric acid treated feed was significantly more reduced. 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 A4.5 Standard Crucible and Small-Scale Melter Glass Redox Test Data for Small-Scale Melter Feed Alternate HTM Flowsheet 1 Testing.

	Final		Moles Oxidant[a]		Moles Reductant[b]	Glass: $Fe^{+2}/\Sigma Fe$	
	pH (@ ≈22°C)	(gTO/L)	NO_3^-	NO_2^-		Crucible	Melter
Alternate HTM Flowsheet 1[c]							
small-scale melter feed[d]	7.9	354	1.05	0.35	N/A	N/A	N/A
dried melter feed/glass	N/A	N/A	----	----	N/A	0.0655	0.419[e]
	N/A	N/A	----	----	N/A	0.0717	0.302[f]
	N/A	N/A	----	----	N/A		0.302[g]

(a) Reported moles of NO_3^- are after addition of $NaNO_3$. The $NaNO_3$ contributed 0.47 moles of nitrate per 125 gWO.
(b) No reductant added for Alternate HTM Flowsheet 1.
(c) Alternate HTM Flowsheet 1 is the no reductant, nitric acid process flowsheet.
(d) Small-scale melter feed sample corresponds to rheology samples hno30a, hno3d0b, hno3-5d0, and hno35d0a taken from the small-scale feed preparation system on Aug. 19, 1994.
(e) Sample number SSHTM1-85; sampled on Aug. 30, 1994 at 0650 hours. Glass sample is a bottom drain sample taken during melter operation.
(f) Glass sample retrieved from SSHTM-1 Canister #3 on Nov. 4, 1994. This glass pour was the third of five total pours. Glass was not supposed to contain boron oxide, but was found to contain approximately 2.5 wt% B_2O_3. Glass sample poured on Aug. 30, 1994 at 0620 hours. Value is the mean of two duplicate analyses; one standard deviation is 0.001.
(g) Sample number SSHTM1-109; sampled on Sept. 1, 1994 at 0540 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.