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13. ABSTRACT (Maximum 200 words) A STUDY ON THE DISPOSAL OF THE ROCKY MOUNTAIN ARSENAL BASIN F CONTENTS, CONDUCTED IN 1978 BY CHEMICAL SYSTEMS LABORATORY, CONCLUDED THAT NONE OF THE DEVELOPED DISPOSAL SCHEMES WERE COST EFFECTIVE. IT WAS RECOMMENDED THAT OTHER ALTERNATIVES FOR BASIN F CONTROL BE INVESTIGATED. THESE LABORATORY INVESTIGATIONS, OUTLINED IN TEST PLAN RMA NO. 1, DATED SEPTEMBER 1979 (SEE APPENDIX 1) WERE TO BE USED TO ESTABLISH A DATA BASE FOR EVAPORATION OF BASIN F WITHOUT OUTSIDE INFLUENCES (LEAKAGE, INPUT TO BASIN ETC.) (ENGINEERING DATA (VISCOSITY, DENSITY, SURFACE TENSION, ETC.) REQUIRED FOR THE FEASIBILITY EVALUATION OF VARIOUS CANDIDATE PROCESSES WERE TO BE GATHERED FOR INPUT INTO THE PROCESS ENGINEERING EFFORT. THE TWO TASK OBJECTIVES ARE: 1) DETERMINE PHYSICAL PROPERTIES OF BASIN F FLUID AT VARIOUS STAGES OF ARTIFICIAL EVAPORATION (DISTILLATION) 2) DETERMINE NATURAL EVAPORATION RATE DATA OF BASIN F FLUID AT AMBIENT CONDITIONS.				
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TEST SUMMARY

Basin F

LABORATORY EVAPORATION STUDIES OF
ROCKY MOUNTAIN ARSENAL BASIN F FLUID

RMA BASIN TEST NO. 1

DECEMBER 1979

Rocky Mountain Arsenal
Information Center
Commerce City, Colorado

FILE COPY

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BASIN F PRECIPITATION DATA (IN.)

	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC
1964										0.05	0.26	0.16
1965	0.55	1.27	1.20	0.98	1.47	4.02	5.62	0.79	2.51	0.39	0.20	0.45
1966	0.13	1.28	0.32	1.10	0.39	1.72	1.93	0.31	1.90	0.96	0.32	0.17
1967	0.72	0.39	0.79	4.03	3.41	2.54	2.79	0.74	0.61	1.06	1.01	1.06
1968	0.51	0.74	0.85	2.39	1.09	1.11	1.68	2.41	0.41	0.43	0.71	0.51
1969	0.17	0.43	1.10	1.30	5.35	2.45	0.43	0.71	1.37	5.09	0.62	0.32
1970	0.10	0.01	1.31	0.95	0.68	3.79	1.72	0.24	2.50	0.88	1.19	0.09
1971	0.35	0.78	0.53	2.33	1.18	0.21	0.84	0.19	2.78	0.44	0.16	5.25
1972	0.36	0.44	0.48	3.52	0.29	3.08	0.36	2.11	1.56	0.31	2.19	0.70
1973	1.31	0.16	1.70	2.94	5.06	0.20	2.39	1.20	2.85	0.47	0.83	2.84
1974	1.03	1.50	1.32	2.08	0.06	1.95	2.20	0.39	1.11	2.00	1.43	0.29
1975	0.22	0.37	0.83	1.50	2.73	2.11	2.00	2.82	0.27	0.30	1.88	0.18
1976	0.69	0.54	1.34	1.08	1.34	0.63	3.31	2.49	1.88	0.93	0.31	0.14
1977	0.00	0.43	1.24	2.13	0.34	1.02						
1981										0.22		

REPORT OF LABORATORY EVAPORATION STUDIES OF ROCKY MOUNTAIN ARSENAL BASIN F FLUID

I. TEST PURPOSE:

A study on the disposal of the Rocky Mountain Arsenal Basin F contents conducted in 1978 by the Producibility Engineering and Technology Branch, Munitions Division, Chemical Systems Laboratory, concluded that none of the developed disposal schemes were cost effective. It was recommended that other alternatives for Basin F control be investigated. These laboratory investigations, outlined in Test Plan RMA No. 1 dated September 1979 (See Appendix 1), were to be used to establish a data base for evaporation of Basin F without outside influences (leakage, input to basin, etc). Engineering data (viscosity, density, surface tension, etc) required for the feasibility evaluation of various candidate processes were to be gathered for input to the process engineering effort.

II. OBJECTIVES:

A. Task 1. Determine physical properties of Basin F fluid at various stages of artificial evaporation (distillation).

B. Task 2. Determine natural evaporation rate data of Basin F fluid at ambient conditions.

III. TEST ACCOMPLISHMENTS:

A. Task 1, Physical Properties. Specimens of Basin F fluid waste were distilled at various specified temperatures while samples of the still bottoms were taken and tested for density, viscosity, and surface tension. Approximately 500 ml of the liquid waste material were poured into a 500 ml - 3 necked flask set up as shown in Figure 1. Temperature levels of 60°C, 80°C, 100°C, and 120°C were to be achieved and each temperature level was to be maintained for the entire day or until a 20% weight loss was attained in the reactor (determined by the amount of distillate recovered). Reactor, distillate, and sample container weights were to be recorded so that an overall material balance could be calculated.

1. Run 1-1: During the period 20 thru 28 August 1979, two heating intervals were completed, one at 60°C and the other at 80°C. This run was discontinued because of procedural errors in weighing. It was noted however, that no distillation occurred at these temperatures and no material was recovered in either the distillate receiver or the organic trap in dry ice/acetone bath.

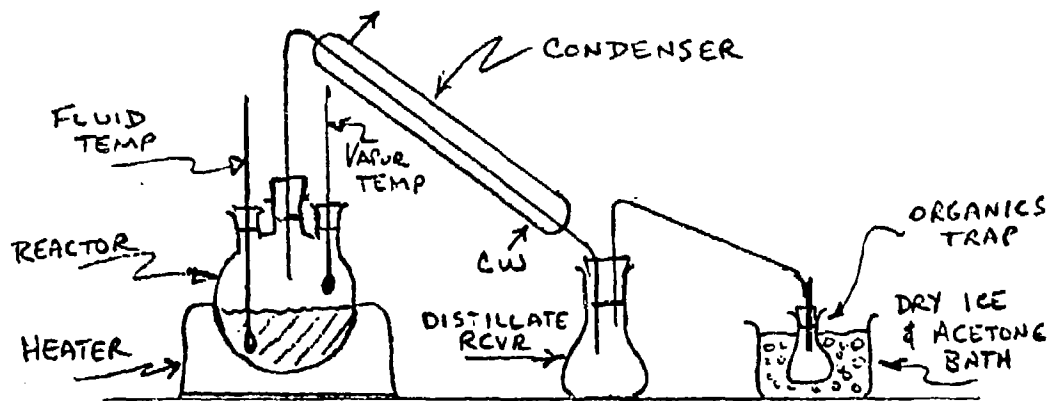


FIG 1. TASK 1 SETUP, COLLECTION OF BASIN F
FLUID DISTILLATION CUTS

2. Run 1-2: The same set up was used in this run as in Run 1-1; however, dry ice was only available on 7 September 1979 so the run was normally conducted either without a dry ice/acetone bath or with a regular ice bath substitute. Run 1-2 was conducted from 4-12 September 1979. Data collected from this run are shown in Table 1. In the middle of the 7 September run, the material in the reactor boiled over into the distillate receiver. The test was stopped and all material in the system was returned to the reactor. The run was restarted and distillate came off at a liquor temperature range of 107°C to 110°C. Material would not distill at vapor temperatures below 100°C and the temperature controller had to be continuously adjusted to allow the vapor temperature to rise and fall to prevent recurrence of the boil over. At reactor liquor temperatures of approximately 114°C, vigorous boiling occurred and solids precipitation were noted. When the liquor temperature reached 135°C a noticeable amount of vapors traveled through the condenser. Heating was discontinued and the run was terminated. The vapors appeared like smoke and swirled in the reactor. A white solid residue formed on the condensing tube wall. No material was noted in the organics trap. Samples of the reactor liquor were withdrawn after the heating intervals of 60°C, 80°C, 100°C, 110°C, 112°C, and 115°C for density, viscosity and surface tension measurements by Research Division. The report of their findings are shown in Appendix 2 and are also summarized in Table 1.

3. Run 1-3: This run, conducted on 17 and 18 September 1979, was a repeat of Run 2 with the following changes:

a. A 1000 ml flask replaced the 500 ml flask to prevent liquid from "boiling over" into the distillate receiver through the condenser tube as experienced in Run 1-2.

b. Distillation at temperature levels of 60°C, 80°C, and 100°C was deleted. Based on the experience gained during the prior runs it was decided to heat over the intervals of 105-107°C, 108-110°C, 110-112°C, 112-114°C, and 114°C end.

c. Samples of reactor liquor were withdrawn when distillation ceased at each heating interval.

d. Dry ice was still not available at Chemical Systems Laboratory; therefore, only a regular ice bath was used at the organic trap.

e. Distillation began at a slightly higher reactor temperature (107°C) than previously noted (105°C), probably due to the larger flask size, thus eliminating the first planned temperature interval. The liquid distilled rapidly during the final heating interval and the run was terminated at a reactor liquor temperature of 122°C. No distillate was recovered in the organics trap. The data for Run 3 are summarized in Table 1.

TABLE #1
RMA BASIN F LIQUID WASTE DISTILLATION STUDIES

DATE	TASK RUN #	HEATING TIME (HR - MIN)	TEMP INTERVAL (°C)	INITIAL WEIGHT (g)	FINAL WEIGHT (g)	VAPOR LOSS (g)	VAPOR LOSS %	TOTAL LOSS %	DISTILLATE RECOVERED (g)	SAMPLE WEIGHT (g)	SAMPLE WEIGHT LOSS (g)	SAMPLE #	DENSITY	VISCOSITY (CENTI-STOKES)	SURFACE TENSION (DYNES/CM)
SEPT															
4	1	6 - 58	60 ± 5	574.0 ¹	572.3	1.7	0.3	0.3	0.0	2.3	0.0	60 - 2	1.124	1.56	53.1
5		7 - 01	80 ± 5	546.0	545.3	0.7	0.1	0.4	0.0	27.6	+0.1	80 - 2	1.125	1.55	-
6		6 - 00	100 ± 5	517.8	516.3	1.5	0.3	0.7	0.0	28.8	0.2	100 - 2	1.151	1.50	-
7		1 - 17	105 - 107	487.3	474.0	13.3	2.3	3.0	11.4	-	-	-	-	-	-
7	1-2	4 - 13	107 - 110	473.6	355.0	119.1	20.7	23.7	115.0	-	-	-	-	-	-
7 ³		5 - 30	105 - 110	487.3	355.0	132.4	23.1	-	126.4	31.0	0.0	² 110 - 2a	1.174	2.05	49.8
10		6 - 47	110 - 112	324.0	286.0	38.0	6.6	30.4	34.0	29.7	+0.5	² 110 - 2b	1.246	2.40	50.9
11		2 - 00	112 - 114	256.8	173.8	83.0	14.5	44.8	80.2	27.2	1.6	² 115 - 2a	1.235	2.79	-
12		1 - 01	114 - 135	145.0	87.3	57.7	10.1	54.9	55.0	POT RESIDUE	-	-	-	-	-
17		1 - 28	107 - 110	579.5	503.0	76.5	13.2	13.2	74.8	26.0	0.0	107 - 3	1.173	1.74	50.9
17	1-3	1 - 12	110 - 112	477.0	405.0	72.0	12.4	25.6	71.0	27.0	0.0	110 - 3	1.218	3.25	-
17		1 - 45	112 - 114	378.0	259.0	119.0	20.5	46.2	119.6	31.3	0.2	112 - 3	1.234	2.62	47.6
18		0 - 22	114 - 122	227.5	125.8	101.7	17.5	63.7	100.8	POT RESIDUE	-	-	-	-	-

¹ All weights estimated to nearest quarter of a gram.

² Samples contained sand, gravel, etc.

³ This line of data represents the summation of the day's testing.

4. Run 1-4: The equipment set up for this run, conducted on 25 September 1979, was the same as for Run 1-3 except that dry ice was again available at Chemical Systems Laboratory so the organic trap was placed in a dry ice/acetone bath. Reactor liquor sample withdrawals were eliminated so that actual percent solids data at each desired temperature interval could be determined for comparison with those assumed in prior runs. The surplus of reactor liquor (caused by the elimination of samples) present at the final temperature interval (114°C - end) necessitated the addition of a 114-117°C temperature interval. The final interval for the run then was 117°C - end. Distillation occurred at 107°C and the run was terminated at a reactor liquor temperature of 127°C. Experimental data for Run 1-4 are located in Table 2. A negligible amount of distillate was noted in the organics trap.

5. Run 1-5: This run was a repeat of Run 1-4. The liquid distilled at 105°C and the distillation was terminated at a reactor liquor temperature of 132°C. Again a negligible amount of material was found in the organics trap. The data for this run are also shown in Table 2.

6. A summary of the mass balances for Runs 1-2 thru 1-5 are given in Table 3 and shows accountability of test material in each run. Table 4 shows data for the final drying of residue remaining in the reactor at the conclusion of the run.

B. Task 2, Evaporation. Specimens of Basin F fluid waste were evaporated under ambient conditions. Samples were withdrawn during the evaporation process to characterize the physical changes in the liquid. A known weight of the fluid waste was poured into a circular dish (18.3 cm diameter; exposed surface area of 263 cm²). The dish was then placed in a laboratory hood and the fluid waste was allowed to evaporate naturally. Temperature and humidity within the hood was monitored continuously. Material weights, temperatures, and humidities were recorded daily while samples were withdrawn at two or three day intervals for density, viscosity and surface tension determinations.

1. Run 2-1: This run was started on 30 August 1979 and was concluded on 14 September. Six hundred and ten grams of Basin F fluid was poured into the evaporation dish at the start of the test. The dish rested upon a Mettler balance during the evaporation period and direct weight readings were made. On 6 September, it was noted that the final weight recorded was inconsistent with expectations. The balance had to be zeroed-in and this accounted for a slight error in the weight. Furthermore, the edge of Hurricane David passed through this region and resulted in a high, essentially constant, relative humidity (approximately 70%) within the test hood. The high humidity together with the weighing error contributed to the incongruent weight readings for that data. The practice of leaving the pan on the scale throughout the run was discontinued at this point to facilitate zeroing of the balance. By 12 September, a considerable amount of solids had accumulated on the bottom of the dish and was crystalline and lumpy in form. The data for this run are reported in Table 5.

TABLE #2
RMA BASIN F LIQUID WASTE DISTILLATION STUDIES

DATE	TASK RUN #	HEATING TIME (HR - MIN)	TEMP INTERVAL (°C)	INITIAL WEIGHT (g)	FINAL WEIGHT (g)	VAPOR LOSS (g)	VAPOR LOSS %	TOTAL LOSS %	DISTILLATE RECOVERED (g)	% LOSS FOR EACH PHASE
SEPT 25	1-4	0 - 44	107 - 110	581.0	469.5	111.5	19.2	19.2	109.5	19.2
		0 - 14	110 - 112	469.5	393.0	76.5	13.2	32.4	75.5	16.3
		0 - 20	112 - 114	393.0	292.0	101.0	17.4	49.7	100.2	25.7
		0 - 20	114 - 117	292.0	195.8	96.2	16.6	66.3	94.0	32.9
		0 - 09	117 - 127	195.8	153.0	42.8	7.4	73.7	41.7	21.9
SEPT 27	1-5	0 - 56	107 - 110	585.0	410.8	174.2	29.8	29.8	171.0	29.8
		0 - 31	110 - 112	410.8	343.0	67.8	11.6	41.4	67.0	16.5
		0 - 31	112 - 114	343.0	237.2	105.8	18.1	59.5	104.2	30.8
		0 - 14	114 - 118	237.2	189.0	48.2	8.3	67.7	48.5	20.3
		0 - 17	118 - 128	189.0	147.5	41.5	7.1	74.8	37.2	22.0

TABLE #3

MASS BALANCES FOR DISTILLATION STUDIES

TASK-RUN	POT RESIDUE	VAPOR LOSS	DISTILLATE RECOVERED	SAMPLE WEIGHT	SAMPLING LOSS	MATERIAL LOSS	TOTAL
RUN # 1-2 Weight in grams	87.3	315.0	295.6	170.6	1.2	19.3	574.1
	15.2	54.9	51.5	29.7	0.2	3.4	100.1
RUN # 1-3	125.8	369.2	366.2	84.3	0.2	3.0	579.5
	21.7	63.7	63.2	14.5	< 0.1	0.5	99.9
RUN # 1-4	153.0	428.0	420.9	NO	-	6.8	581.0
	26.3	73.7	72.4	SAMPLES	-	1.2	100.0
RUN # 1-5	147.5	437.5	427.9	NO	-	9.1	585.0
	25.2	74.8	73.1	SAMPLES	-	1.6	100.0

TABLE #4

SOLID RESIDUE DRYING DATA

TASK RUN	WEIGHT BEFORE DRYING (g)	WEIGHT AFTER DRYING (g)	MOISTURE LOSS (g)	MOISTURE LOSS %
1-2	88.5	82.7	5.8	6.6
1-3	118.8	108.3	10.5	8.8
1-4	151.8	143.6	8.2	5.4
1-5	145.6	139.8	5.8	4.0

TABLE #5
 RHA BASIN F LIQUID WASTE EVAPORATION STUDY RUN #2-1

DATE	TIME DATA RECORDED	TEMP. OF	RELATIVE HUM. %	WEIGHT INITIAL g	WEIGHT FINAL g	LOSS g	LOSS %	TOTAL LOSS %	EVAPORATION RATE g/cm ² - DAY	WEIGHT g	SAMPLING LOSS g	SAMPLE #	DATA DENSITY g/cm ³	VISCOSITY CENTISTOKES	SURFACE TENSION DYNES/CM	
																MATERIAL PUT IN HOOD
AUG 30																
31	10:10	75	63	610.0	585.2	24.8	4.1	4.1	0.09	16.5	1.7	E-1	1.162	1.62	54.3	
SEPT 4	10:10	79	66	567.0	482.0	85.0	13.9	18.0	0.08	24.5	0.5	E-2	1.149	1.85	55.8	
5	10:10	77	62	457.0	443.5	13.5	2.2	20.2	0.05	-	-	-	-	-	-	
6	10:11	76	66	443.5	444.0	+0.5	-	-	-	-	-	-	-	-	-	
7	10:10	74	60	444.0	420.0	24.0	3.9	24.1	0.09	30.7	+0.2	E-3	1.217	2.12	50.6	
10	10:10	70	65	389.5	310.0	79.5	13.0	37.2	0.10	30.0	1.5	E-4	1.246	2.74	49.3	
11	10:11	70	66	278.5	263.5	15.0	2.5	39.6	0.06	-	-	-	-	-	-	
12	10:11	65	62	263.5	250.5	13.0	2.1	41.8	0.05	26.4	1.1	E-5	1.179	3.09	47.1	
13	10:10	69	68	223.0	216.0	7.0	1.1	42.9	0.03							
14	10:12	-	-	216.0	216.0	0.0		CONCLUDED EXPERIMENT								

2. Run 2-2: Run 2-2 was started on 13 September 1979 and was a repeat of the first run but the initial weight of the Basin F fluid was doubled to 1256.5 gm to minimize the effect of sampling. By 26 September, less than half of the initial starting material remained in the dish. Because a large part of this loss was due to sampling, the collection of samples was discontinued. The remainder of this run was spent monitoring the loss due to evaporation. Another change to the experimental set up inadvertently occurred to the control of the temperature/humidity system of the laboratory hood. Beginning 1 October 1979, the ventilation of the hood was no longer maintained 24 hours per day as in the past. For reasons of maintenance work or energy conservation, routine shutdowns of the ventilation system were made by building mechanics every evening and on weekends and holidays. Irregular conditions of temperature and humidity were experienced until the end of the run on 15 October. The data for this run are found in Table 6.

3. Run 2-3: Because of the disproportionate loss of starting material due to sampling in the first two trials, no samples were collected during Run 2-3. The run started on 27 September 1979 and was terminated on 29 October. The loss of control of the ventilation system noted in Run 2-2 pertained to this run as well and it was impossible to maintain the test environment within the laboratory hood. Table 7 summarizes the data collected in this run.

4. The mass balances for Runs 2-1, 2-2, and 2-3 are summarized in Table 8. The data for the final drying of the residue remaining in the dish at the conclusion of the run are shown in Table 9.

IV. TEST EVALUATION:

Between 20 August and 29 October 1979, tests on the Basin F fluid were conducted at the Chemical Systems Laboratory pilot plant, building E5625. The first task, to determine physical properties of the fluid at various stages of distillation, was completed on 27 September. The second task, to determine natural evaporation rate data of the fluid at ambient conditions, was completed on 29 October 1979.

A. Task 1, Physical Properties: Distillation of the Basin F liquid waste occurred when the material was heated to a pot temperature of 105° - 107°C and a vapor temperature of 100°C. If the material was not sufficiently heated to maintain the distillation, the vapor temperature dropped rapidly followed by a slowly decreasing pot temperature. With sufficient heat, the liquid distilled slowly at one degree temperature intervals. Between 110° and 115°C the material boiled vigorously and appeared as though exploding upward from beneath the liquid's surface. At approximately 114°C solids precipitated out of the liquid being splashed on the interior reactor walls and a very dense liquid layer formed on the bottom of the pot. At temperatures approaching 120°C white smokelike vapors formed inside the flask. The vapors

TABLE # 6
RMA BASIN F LIQUID WASTE EVAPORATION STUDY RUN #2-2

DATE	TIME DATA RECORDED	TEMP. OF	RELATIVE HUM. %	WEIGHTS		EVAPORATION DATA			SAMPLE DATA							
				INITIAL g	FINAL g	LOSS g	LOSS %	TOTAL LOSS %	RATE g/CM ² - DAY	WEIGHT g	SAMPLING LOSS g	SAMPLE #	DENSITY g/CM ³	VISCOSITY CENTISTOKES	SURFACE TENSION DYNES/CM	
SEPT. 13	15:50															
14	15:49	82	66	1256.5	1221.5	35.0	2.8	2.8	0.13	27.3	0.2	1	1.167	1.52	50.9	
17	15:52	77	47	1194.0	1020.2	173.8	13.8	16.6	0.22	28.2	1.0	2	1.192	3.56		
18	15:51	77	57	991.0	951.2	39.8	3.2	19.8	0.15	29.2	0.5	3	1.202	1.93		
19	15:30	72	47	921.0	870.2	51.3	4.1	23.7	0.20	29.3	0.4	4	1.210	1.97		
20	15:30	67	43	840.5	791.2	49.3	3.9	27.8	0.19	29.5	0.5	5	1.230	2.27		
24	15:50	66	58	761.2	703.5	57.7	4.6	32.4	0.05	28.4	1.1	6	1.248	2.49		
25	15:45	68	65	674.0	651.0	23.0	1.8	34.2	0.09	29.5	0.3	7	1.250	2.54	49.9	
26	15:50	73	60	621.2	618.0	3.2	0.3	34.5	0.01	28.8	0.2	8	1.252	2.62	47.0	
28	15:48	71	62	589.0	570.5	18.5	1.5	35.9	0.04	--	--	--	--	--	--	--
OCT 1	15:51	72	66	570.5	559.0	11.5	0.9	36.9	0.01	--	--	--	--	--	--	--
2	15:47	75	64	559.0	553.5	5.5	0.4	37.3	0.02	--	--	--	--	--	--	--
4	15:55	75	65	553.5	508.0	45.5	3.6	40.9	0.09	--	--	--	--	--	--	--
9	10:30	68	58	508.0	408.0	100.0	8.0	48.9	0.08	--	--	--	--	--	--	--
10	15:50	63	48	408.0	382.0	26.0	2.1	50.9	0.10	--	--	--	--	--	--	--
11	15:55	74	41	382.0	345.0	36.5	2.9	53.8	0.14	--	--	--	--	--	--	--
12	15:52	74	48	345.5	313.0	32.5	2.6	56.4	0.12	--	--	--	--	--	--	--
15	15:48	81	30	313.0	267.0	46.0	3.7	60.1	0.06	CONCLUDED EXPERIMENT		--	--	--	--	--

TABL 7

RMA BASIN F LIQUID WASTE EVAPORATION STUDY RUN #3

DATE	TIME DATA RECORDED	TEMP OF F	RELATIVE HUM %	WEIGHTS		LOSS g	EVAPORATION DATA		RATE g/cm DAY
				INITIAL g	FINAL g		LOSS %	TOTAL LOSS %	
Sep 27	09:35			1181.5	Material Put in Hood				
Sep 28	10:00	67	63	1181.5	1145.5	36.0	3.0	3.0	0.14
Oct 1	09:30	66	63	1145.5	1065.0	80.5	6.8	9.9	0.10
Oct 2	09:35	71	65	1065.0	1052.0	13.0	1.1	11.0	0.05
Oct 3	09:33	68	59	1052.0	1028.5	23.5	2.0	12.9	0.09
Oct 4	09:32	69	56	1028.5	994.5	34.0	2.9	15.8	0.13
Oct 5	09:50	--	--	994.5	962.5	32.0	2.7	18.5	0.12
Oct 9	10:30	68	58	962.5	844.5	118.0	10.0	28.5	0.11
Oct 10	10:00	51	46	844.5	817.5	27.0	2.3	30.8	0.10
Oct 11	09:30	68	43	817.5	793.0	24.5	2.1	32.9	0.09
Oct 12	09:32	74	43	793.0	750.0	43.0	3.6	36.5	0.16
Oct 15	09:35	71	35	750.0	655.0	95.0	8.0	44.6	0.12
Oct 16	09:35	73	41	655.0	594.4	60.5	5.1	49.7	0.23
Oct 17	09:35	65	52	594.5	547.0	47.5	4.0	53.7	0.18
Oct 18	09:35	68	52	547.0	513.5	33.5	2.8	56.5	0.13
Oct 19	09:35	63	50	513.5	485.5	28.0	2.4	58.9	0.11
Oct 22	09:35	76	54	485.5	424.5	61.0	5.2	64.1	0.08
Oct 23	09:35	80	53	424.5	404.0	20.5	1.7	65.8	0.08
Oct 24	09:35	61	48	404.0	380.7	23.3	2.0	67.8	0.09
Oct 26	09:18	53	47	380.7	341.0	39.7	3.4	71.2	0.08
Oct 29	09:35	64	47	341.0	326.3	14.7	1.2	72.4	0.02

TABLE #8

MASS BALANCES FOR EVAPORATION STUDIES

	SOLID RESIDUE	VAPOR EVAPORATED	SAMPLE WEIGHT	SAMPLING LOSS	TOTAL MATERIAL
RUN #1					
WEIGHT (GM)	216.0	267.3	128.1	4.6	610.0
PERCENT (%)	35.4	42.8	21.0	0.8	100.0
RUN #2					
WEIGHT (GM)	267.0	755.1	230.2	4.2	1256.5
PERCENT (%)	21.2	60.1	18.3	0.3	99.9
RUN #3					
WEIGHT (GM)	326.3	855.2	NO	NO	1181.5
PERCENT (%)	27.6	72.4	SAMPLES	SAMPLES	100.0

TABLE #9
SOLID RESIDUE DRYING DATA

TASK RUN	WEIGHT BEFORE DRYING gm	WEIGHT AFTER DRYING, gm	SOLIDS FRACTION	ACTUAL FINAL WEIGHT gm	CALC TOTAL SOLIDS WEIGHT gm
2-1	33.0	27.2	0.824	216.0	178.0
2-2	109.2	95.2	0.872	267.0	232.8
2-3	122.3	110.6	0.904	326.3	295.1

completely filled the flask and were carried over into the distillate receiver prior to termination of some runs. The typical characteristic of this distillation is shown by the relationship of temperature level to percent solids in the fluid (Table 10 and Figure 2). The effect of sample losses are shown by the lower percent solids for Runs 1-2 and 1-3 compared to the Runs 1-4 and 1-5 during which no samples were drawn. Odors were not noticed exiting the dry ice/acetone trap, but all the distillate phases and remaining solid residue were quite unpleasant to smell. The distillate product was very clear initially but gradually turned yellow. The amount of material collected in the organics trap was negligible in all cases. During two of the distillation runs, samples of the reactor liquor were collected for physical property measurements of density, viscosity, and surface tension. The relationship of these properties to total percent solids in the remaining reactor liquor were determined and are shown in Table 11. Good correlations were noted for density (Figure 3) and viscosity (Figure 4) but a relatively poor one was found for surface tension (Figure 5).

B. Task 2, Evaporation: An attempt was made during this task to determine the natural evaporation rate of Basin F fluid at ambient conditions. Unfortunately, the sampling in Runs 2-1 and 2-2 represented a significant loss to the experimental system. Therefore the data is somewhat suspect. Furthermore the loss of control of the ventilation system during Runs 2-2 and 2-3 seriously disrupted the ambient conditions planned for this task. A correlation has been attempted, however, between the evaporation rates and the residual fluid at increasing average percent solids levels. Very poor results were obtained. An analysis was also made between the rates and relative humidity. Again a very poor correlation was obtained. The valves used in these analyses are provided in Table 12.

TABLE 10
TEMPERATURE LEVEL VERSUS PERCENT SOLIDS

TEMPERATURE °F	% SOLIDS			
	<u>RUN 1-2</u>	<u>RUN 1-3</u>	<u>RUN 1-4</u>	<u>RUN 1-5</u>
60	14.5			
80	15.2			
100	16.0			
110	23.3	21.5	30.6	34.0
112	28.9	26.7	36.5	40.8
114	47.6	41.8	49.2	58.9
117			73.3	
118				74.0
127			93.9	
128				94.8

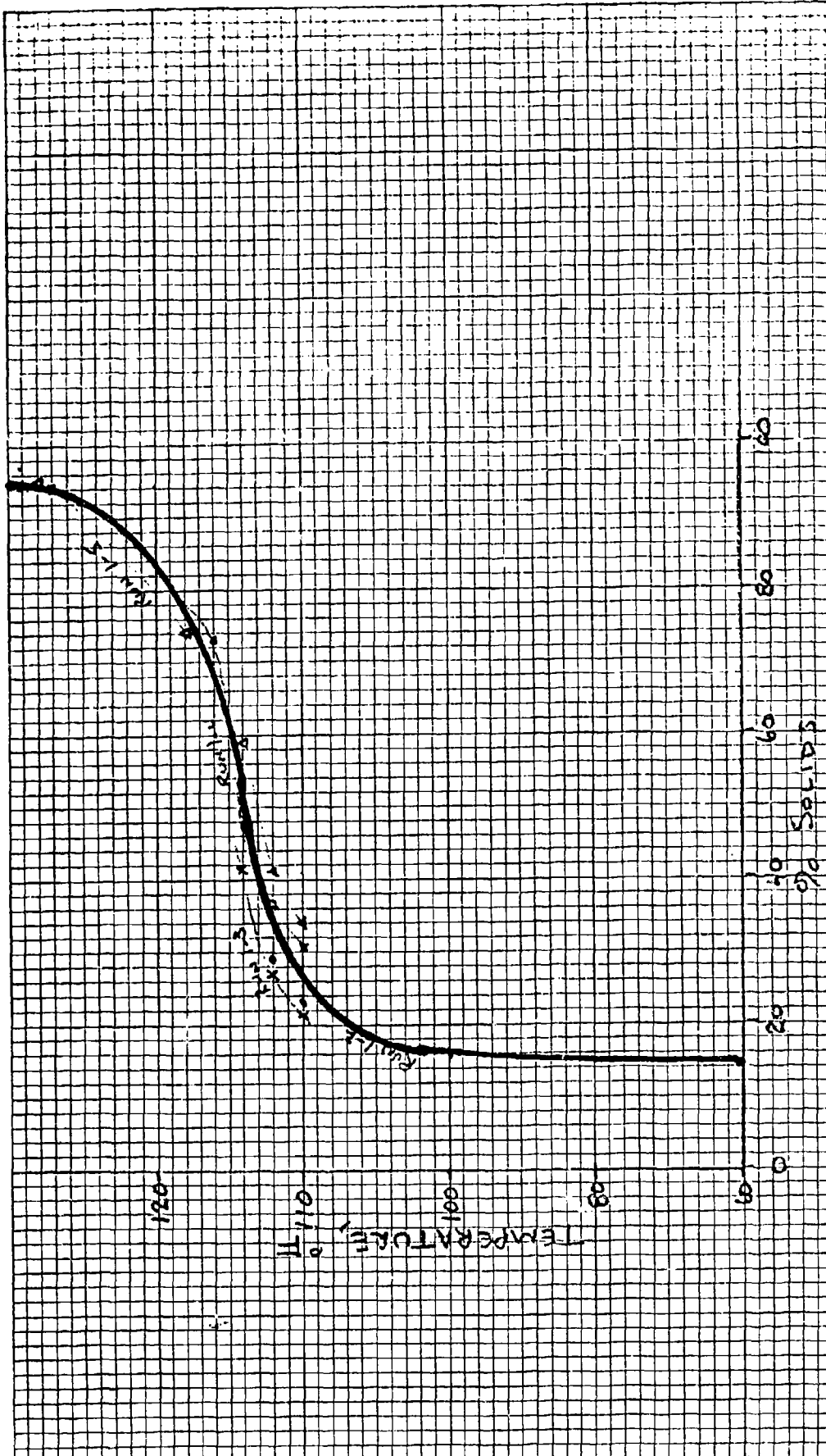


FIGURE 2. Distillation characteristic of Basin F Fluid, Runs 12 through 15

TABLE 11

PHYSICAL PROPERTIES OF BASIN F FLUID AT DIFFERENT SOLIDS CONCENTRATIONS

TASK RUN	SAMPLE NO.	REACTOR LIQUOR WT	% SOLIDS	DENSITY	VISCOSITY	SURFACE TENSION
				g/ml	cs	dynes/cm
1-2	Pure	574.0	14.4	1.158	1.57	50.6
	60-2	572.3	14.5	1.124	1.56	53.1
	80-2	545.3	15.2	1.126	1.55	---
	100-2	516.3	16.0	1.151	1.50	---
	110-2a	355.0	23.3	1.174	2.05	49.8
	110-2b	286.0	28.9	1.246	2.40	50.9
	115-2a	173.8	47.6	1.235	2.79	---
1-3	107.3	503.0	21.5	1.173	1.74	50.9
	110.3	405.0	26.7	1.218	3.25	---
	112-3	259.0	41.8	1.234	2.62	47.6
1	Pure	610.0	14.4	1.158	1.57	50.6
	E-1	585.2	30.4	1.162	1.62	54.3
	E-2	482.0	36.9	1.149	1.85	55.3
	E-3	420.0	42.4	1.217	2.12	50.6
	E-4	310.0	57.4	1.246	2.74	49.3
	E-5	250.5	71.1	1.179	3.09	47.1
2-2	1	1221.5	19.1	1.167	1.52	---
	2	1020.2	22.8	1.192	3.56	50.9
	3	951.2	24.5	1.202	1.93	---
	4	870.2	26.8	1.210	1.97	---
	5	791.2	29.4	1.230	2.27	---
	6	703.5	33.1	1.248	2.49	---
	7	651.0	35.8	1.250	2.54	49.9
	8	618.0	37.7	1.252	2.62	47.0

$$Y = A + B/X$$

$$A = 1.29299033774$$

$$B = 2.30578110563$$

$$R\text{-SQUARE} = 0.850731194758$$

$$RES\ ERROR = 3.554631352E-4$$

$$MAX(ABS(RESIDUAL)) = 0.0327944755996$$

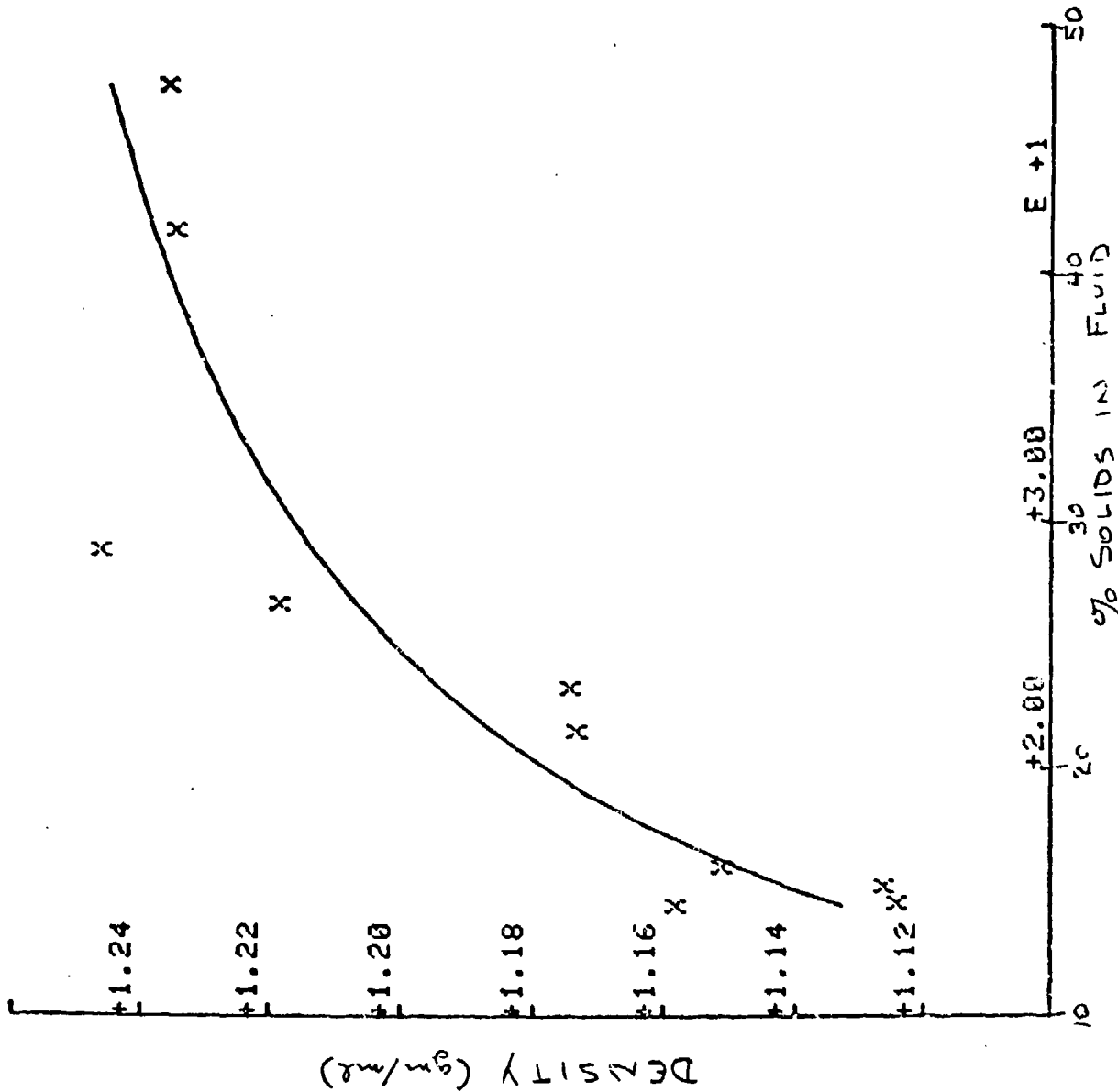


FIGURE 3. Density vs. Percent Solids in Basin F Fluid, Composite data
Runs 1-2 and 1-3.

$$Y = A + B/X$$

A = 3.46338693386

B = -28.753733404

R-SQUARE = 0.714787002725

RES ERROR 0.125707985065

MAX(ABS(RESIDUAL)) 0.86353192022

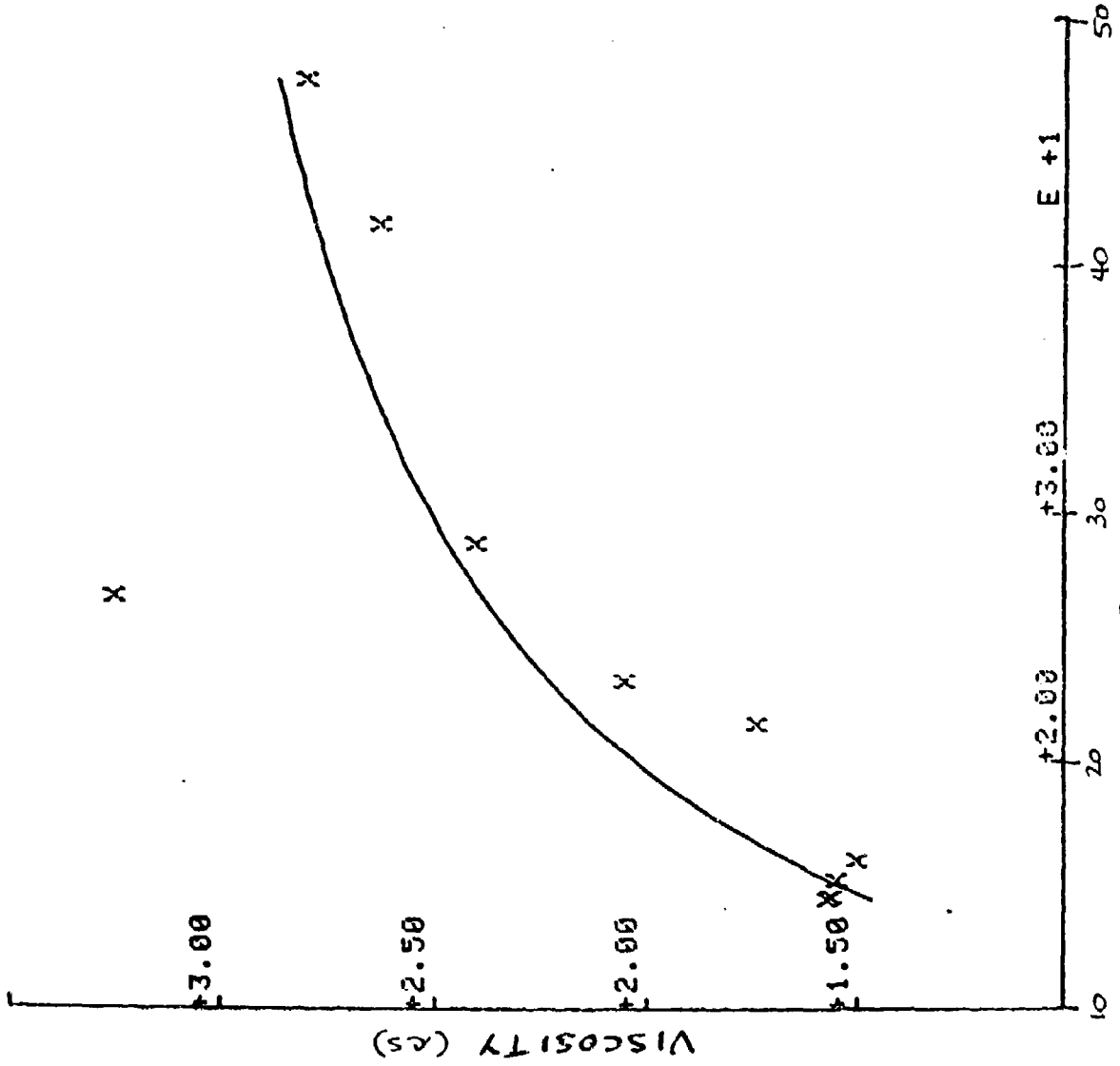


FIGURE 4. Viscosity vs. percent solids in Basin F Fluid, Composite data Runs 1-2 and 1-3.

$$Y = A + B \cdot X$$

A = 53.9265535373

B = -0.143070091577

R-SQUARE = 0.678497940812

RES ERROR 1.28507052242

MAX(ABS(RESIDUAL)) 1.26634421857

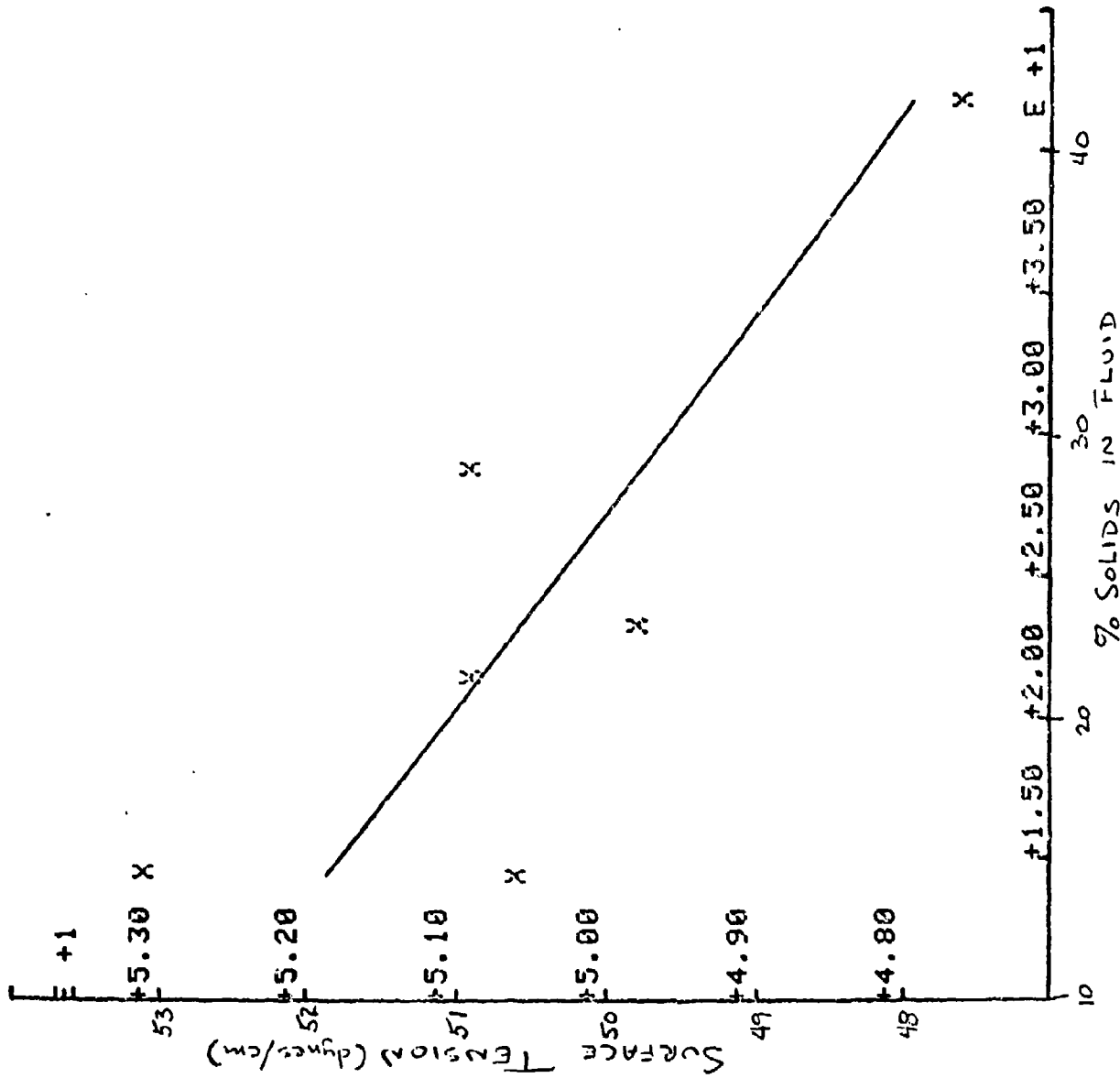


FIGURE 5. Surface Tension vs. percent solids in Basin F Fluid, Composite data Runs 1-2 and 1-3.

TABLE 12

EVAPORATION RATE RELATIVE TO AVERAGE PERCENT SOLIDS AND RELATIVE HUMIDITY

<u>TASK-RUN</u>	<u>AVG WT, g</u>	<u>% SOLIDS (AVG)</u>	<u>EVAP RATE</u> <u>Z/cm² day</u>	<u>RELATIVE</u> <u>HUMIDITY, %</u>
2-1	597.6	29.8	0.09	63
	524.5	33.9	0.08	66
	450.3	39.5	0.05	68
	432.0	41.2	0.09	60
	349.8	50.9	0.10	65
	271.0	65.7	0.06	66
	257.0	69.3	0.05	62
	219.5	81.1	0.03	58
	2-2	1239.0	18.8	0.13
1107.1		21.0	0.22	47
971.1		24.0	0.15	57
895.6		26.0	0.20	47
815.9		28.5	0.19	43
732.4		31.8	0.05	58
662.5		35.1	0.09	65
619.6		37.6	0.01	60
579.8		40.2	0.04	62
564.8		41.2	0.01	66
556.3		41.9	0.02	64
530.8		43.9	0.09	65
458.0		50.8	0.08	58
395.0		58.9	0.10	48
363.5		64.0	0.14	41
329.3		70.7	0.12	48
290.0		80.3	0.06	30
2-3	1163.5	25.4	0.14	63
	1105.3	26.7	0.10	63
	1058.5	27.9	0.05	65
	1040.4	28.4	0.09	59
	1011.5	29.2	0.13	56
	978.5	30.2	0.12	--
	903.5	32.7	0.11	58
	831.0	35.5	0.10	46
	805.3	36.6	0.09	43
	771.5	38.3	0.16	43
	702.5	42.0	0.12	35
	624.8	47.2	0.23	41
	570.8	51.7	0.18	52
	530.3	55.6	0.13	52
	499.5	59.1	0.11	58
	455.0	64.9	0.08	54
	414.3	71.2	0.08	53
	392.4	75.2	0.09	48
	360.9	81.8	0.08	47
333.7	88.4	0.02	47	

APPENDIX 1

TEST PLAN

TEST PLAN

LABORATORY EVAPORATION STUDIES OF
ROCKY MOUNTAIN ARSENAL BASIN F FLUID

ROCKY MOUNTAIN ARSENAL BASIN TEST NO. 1

SEPTEMBER 1979

PREPARED BY:

CONCUR:



LEONARD M. LOJEK
DEMIL/DISPOSAL BRANCH
ENVIRONMENTAL TECHNOLOGY DIVISION



CARL E. GEPP
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MUNITIONS DIVISION



JOHN J. CALLAHAN
CHEMICAL BRANCH
RESEARCH DIVISION

APPROVED BY:



WILLIAM J. WEBER
CHIEF
ENVIRONMENTAL TECHNOLOGY DIVISION

LABORATORY EVAPORATION STUDIES OF
ROCKY MOUNTAIN ARSENAL BASIN F FLUID

I. INTRODUCTION:

A study on the disposal of the Rocky Mountain Arsenal Basin F contents conducted in 1978 by the Producibility Engineering and Technology Branch, Munitions Division, Chemical Systems Laboratory, concluded that none of the developed disposal schemes were cost effective. It was recommended that other alternatives for Basin F control be investigated. The laboratory investigations covered by this test plan will be used to establish a data base for evaporation of Basin F without outside influences (leakage, input to basin, etc.). Data relative to the feasibility of various candidate processes (viscosity, density, surface tension, etc) will be gathered for input to the process engineering effort.

II. OBJECTIVES:

A. Determine physical properties of Basin F fluid at various stages of artificial evaporation (distillation).

B. Determine natural evaporation rate data of Basin F fluid at ambient conditions.

III. TEST PROCEDURES: This investigation requires two tasks each with separate test assemblies.

A. TASK 1. Determination of the physical properties of the Basin F fluid (Distillation).

1. Insert a predetermined amount (by volume and weight) of the fluid into the reaction flask (Figure 1).

2. Heat the flask to accelerate the natural evaporation of the fluid. The heating element will be manually adjusted and controlled to maintain specific fluid temperatures (e.g., 60°C, 80°C, 100°C, 110°C, and 120°C). At each temperature level, the heat will be maintained until no further vapors (indicated by vapor temperature) can be driven off.

3. Cease heating at that point and collect samples of solutions from the reaction flask, the distillate receiver, and the organic trap.

4. Resume heating at the next higher temperature. The experimental trial will conclude when only precipitate or sludge remains in the reaction flask.

5. Finally place the sludge in an oven and completely dry it to determine final weight loss.

B. TASK 2. Determination of the natural evaporation rate of the Basin F fluid.

1. Place a predetermined amount (by volume and weight) of the fluid into an open face evaporation pan and allow it to evaporate naturally at ambient conditions (Figure 2).

2. Collect samples of the fluid at intervals of approximately three days.

3. Monitor and record barometric pressure, temperature and relative humidity throughout the trial.

IV. REQUIREMENTS:

A. FACILITIES. Testing will be conducted at Chemical Systems Laboratory, building E5625 (Pilot Plant), laboratory room. The test assemblies will be placed in laboratory hoods.

B. EQUIPMENT AND MATERIALS.

1. Reaction Flask.
2. Distillate Receiver Flask.
3. Volatile Organics Trap Flask.
4. Heating Mantle.
5. Cooling Water Condenser.
6. Evaporation Pan (Width to Depth Ratio Approximately Equal to 240:1).
7. Balance.
8. Graduate Cylinder.
9. Thermometers.

10. Viscometer.

11. Surface Tension Meter.

V. DATA REQUIRED:

A. TASK 1.

1. Weights of:
 - a. Reactor Solution Loss.
 - b. Distillate Recovery.
 - c. Liquid Samples Drawn.
 - d. Final Precipitate (Wet and Dry).
2. Room Temperature - continuous recorder.
3. Relative Humidity.
4. Barometric Pressure.
5. Physical Properties of Liquid Samples:
 - a. Density.
 - b. Surface Tension.
 - c. Viscosity.

VI. TENTATIVE TEST SCHEDULE: The testing will be initiated on or about 16 August 1979 and be completed by 28 September 1979. This test represents approximately six man weeks of effort. The two tasks are to be run simultaneously using the same test personnel.

VII. GENERAL RESPONSIBILITIES:

A. ENVIRONMENTAL TECHNOLOGY DIVISION, CHEMICAL SYSTEMS LABORATORY.

The Test Director will be assigned from the Environmental Technology Division. He will monitor the overall test program and coordinate the efforts of other Divisions involved. He will approve any changes required to the test plan. He will periodically review the progress of the test operations with the test personnel and verify the adequacy of the test data. He will be responsible for the preparation and distribution of the test report.

B. MUNITIONS DIVISION, CHEMICAL SYSTEMS LABORATORY. The experimental tasks will be conducted by personnel of the Process Technology Section, Producibility Engineering and Technology Branch. The Test Officer assigned by the Process Technology Section is responsible for running the experiments, collecting data and obtaining samples for analyses. He will assist the Test Director in preparing the test report.

C. RESEARCH DIVISION, CHEMICAL SYSTEMS LABORATORY. Personnel of the Physical Organics Section, Chemical Branch, will be responsible for the analyses of the physical properties (i.e., density, surface tension and viscosity) of all liquid samples collected during the experimentation. They will provide the physical property data for periodic review and for final incorporation into the test report.

VIII. SPECIAL SAFETY CRITERIA: No toxic agents are involved; however, routine laboratory precautions for handling organic chemicals will be taken.

1 Incl
Figures 1 and 2

DISTRIBUTION:

Chief, Environmental Technology Division, ATTN: DRDAR-CLT-D/Mr. Leonard M. Lojek
Chief, Research Division, ATTN: DRDAR-CLB-CP/Mr. John J. Callahan
Chief, Munitions Division, ATTN: DRDAR-CLN-TT/Mr. Carl E. Gepp
Chief, Munitions Division, ATTN: DRDAR-CLN-TT/Mr. James Hertzog
Commander, US Army Toxic and Hazardous Materials Agency, ATTN: DRXTH-IS/
Mr. Donald L. Campbell

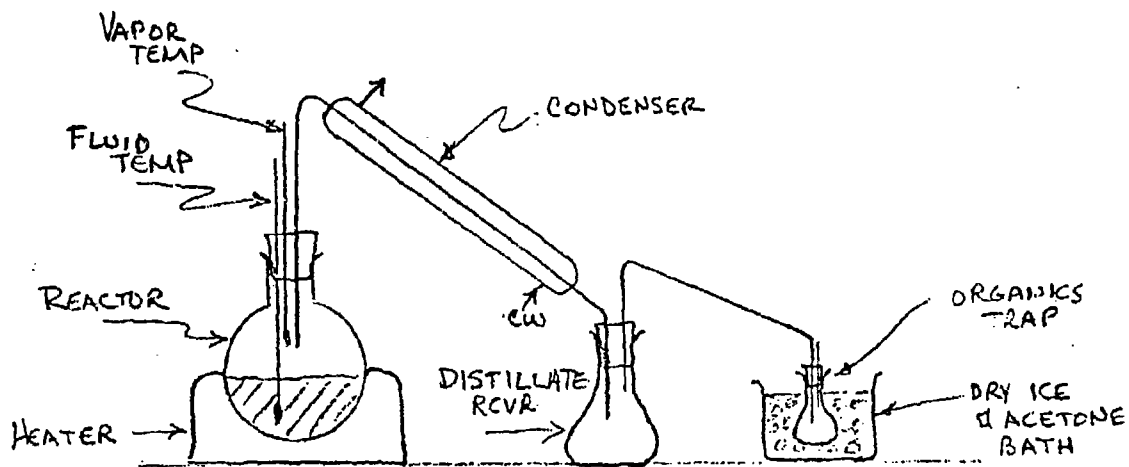


FIG. 1 Task 1 Set Up, Collection of Basin F Fluid Distillation Cuts.

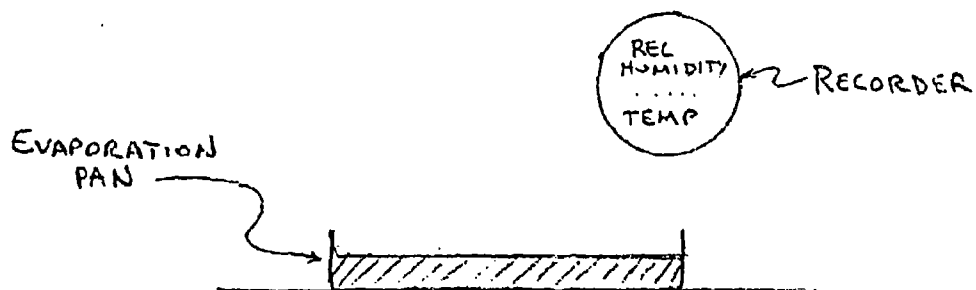


FIG. 2 Task 2 Simple Evaporation Test.

APPENDIX II

PHYSICAL PROPERTIES OF BASIN F FLUIDS

DISPOSITION FORM

For use of this form, see AR 340-15, the proponent agency is TAGCEN.

REFERENCE OR OFFICE SYMBOL

SUBJECT

DRDAR-CLB-CP

Physical Properties of Basin F Fluids

TO Chief, Envir Tech Div
ATTN: Mr. L. M. Lojek

FROM Chief, Rsch Div

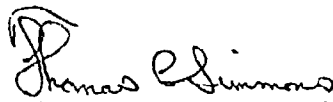
DATE 16 Nov 79

CMT 1

Mr. Callahan/cd/2243

Inclosed are the physical properties of Basin F liquids after various stages of artificial evaporation. The samples were provided by Munitions Division personnel in accordance with the Test Plan dated September 1979.

FOR THE CHIEF:

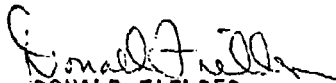

THOMAS C. SIMMONS, Ph.D.
Acting Chief, Chemical Branch

1 Incl
as

15 November 1979

SUBJECT: Physical Properties of Basin F Fluid at Various Stages of
Artificial Evaporation

1. All instruments were calibrated using freshly boiled, demineralized distilled water with the exception of the viscometers, which were calibrated with a standard oil obtained from Cannon Instrument Company of State College, PA.
2. A Mettler DMA55 Precision Digital Density Meter was used to determine the densities of all Basin F samples. The meter was calibrated at 25°C using dry air and water. The value obtained for air at 25°C was $0.00118 \pm 1 \times 10^{-5}$ g/ml; the value obtained for water was $0.99704 \pm 1 \times 10^{-5}$ g/ml.
3. The surface tensions of samples were measured by the capillary rise method. The capillary had a radius of 0.025 cm as determined by photomicrography and confirmed using freshly boiled, demineralized distilled water. The surface tension value for water obtained at 25°C was 72.0 ± 0.1 dynes/cm. Three samples were filtered through fritted glass to determine what influence the suspended solids had on the surface tension.
4. The experimental values are provided in the attached table.
5. NOTE: It was observed while filtering several of the Basin F samples that a considerable amount of foaming resulted from that procedure. This may be important when planning large scale procedures.


DONALD FIELDER
Research Chemist



JOHN J. CALLAHAN
Chief, Physical Organic Section

Table of Basin F Liquid Properties @ 25°C

<u>Sample Date</u>	<u>Sample Number</u>	<u>Density</u> g/ml	<u>Viscosity</u> cs	<u>Surface Tension</u> dynes/cm
-	Control	0.9970	0.893	72.0
8-28-79	60-1	1.1059	1.58	60.2
8-30-79	80-1	1.1014	1.61	62.5
8-30-79	E-1	1.1624	1.62	54.3
8-31-79	Pure	1.1578	1.57	50.6
9-4-79	E-2	1.1492	1.85	55.8
9-5-79	60-2	1.1238	1.56	53.1
9-6-79	80-2	1.1257	1.55	ND
9-7-79	100-2	1.1511	1.50	ND
9-7-79	E-3	1.2170	2.12	50.6
9-10-79	110-2a	1.1738	2.05	49.8
9-10-79	E-4	1.2464	2.74	49.3
9-11-79	110-2b	1.2461	2.40	50.9
9-12-79	E-5	1.1792	3.09	47.1
9-12-79	115-2a	1.2351	2.79	ND
9-14-79	#1	1.1666	1.52	ND
9-17-79	#2	1.1922	3.56	50.9
9-17-79	107-3	1.1728	1.74	50.9
9-17-79	112-3	1.2336	2.62	47.6 & 49.3*
9-17-79	110-3	1.2177	3.25	ND
9-18-79	#3	1.2021	1.93	ND
9-18-79	#4	1.2097	1.97	ND
9-20-79	#5	1.2302	2.27	ND
9-25-79	#6	1.2478	2.49	ND
9-25-79	#7	1.2498	2.54	49.9 & 50.6*
9-25-79	#8	1.2517	2.62	47.0 & 47.7*

ND = not determined

*Filtered