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SYNTHETIC FUELS FROM U. S. OIL SHALES, A TECHNICAL AND ECONOMIC VERIFICATION OF THE HYTORT PROCESS

Final Report for January 1—March 31, 1981

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Work Performed Under Contract No. AC20-79ET14102

Institute of Gas Technology
Chicago, Illinois

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SYNTHETIC FUELS FROM U.S. OIL SHALES A TECHNICAL AND ECONOMIC VERIFICATION OF THE HYTORT PROCESS

Project 61040 Final Quarterly Report
and Final Report
For the Period January 1 Through March 31, 1981

Prepared by
Institute of Gas Technology
IIT Center, 3424 S. State Street
Chicago, Illinois 60616

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UNITED STATES DEPARTMENT OF ENERGY

Under Contract No. DE-AC20-79ET14102

EXECUTIVE SUMMARY

The following is a summary by task of the HYTORT process development work conducted during the final quarter of the project (January 1, 1981, through March 31, 1981. As directed by DOE, this final quarterly report is submitted in lieu of a final project report. In addition, some of the work originally planned was not done per instructions from DOE.

Task 1. Laboratory Program

Seventeen thermobalance tests were conducted during this quarter, nine with six different types of oil shale and eight with samples of Kentucky New Albany shale having equivalent particle diameters ranging from 0.08 to 0.65 inch. The total organic carbon conversion decreased by only about 5% with particle diameter increases in this range. However, the organic carbon conversion to oil decreased substantially (from about 70% to 50%) for this particle diameter increase.

During an examination of a Michigan Antrim shale core from Dow/DOE Well #110, the fossil alga Foerstia was discovered, which is important to stratigraphers because it marks a "time line" in the Late Devonian. Discovery of this fossil, which has never before been seen from Michigan, will permit more precise correlation between the Michigan, Appalachian, and Illinois Basins.

During this quarter, shakedown and preliminary testing of the laboratory-scale reactor unit was completed.

Tests were conducted on eight different oil shale types during this quarter in the differential scanning calorimeter. Tests were conducted in hydrogen and in nitrogen at 1 atm, in 500 psig nitrogen and (with selected shales) in hydrogen/nitrogen (50/50) atmospheres at pressures up to 800 psig.

Task 2. Bench-Scale Program

Two successful runs were made during this quarter in the 4-inch bench-scale unit (BSU) with samples of the Dowelltown member and of the Gassaway member of the Chattanooga shale. Tests have now been conducted with shales from the seven major Eastern shale rock units selected for testing.

Modification of the 6-inch-diameter bench-scale unit for fluidized-bed operation and for feeding of raw shale fines to the bottom of the reactor was completed in this quarter. One test was conducted to study steam-oxygen gasification of raw shale fines.

Two hydrotreating runs were made this quarter with Tennessee shale oil produced in the 4-inch bench-scale unit. The hydrotreated oil was of very high quality, with an API gravity of 43.7° and with 95% of the oil boiling below 605°F.

Experimental work was completed during this quarter in the test system designed to evaluate a technique for enhancing mist formation to reduce oil refluxing. Tests continued to show that oil vapor mist formation was significantly enhanced by injecting a side stream of nitrogen or carbon dioxide (that had first been passed through a high-voltage arc) into a gas/oil vapor mixture.

Tests were conducted on three types of Eastern shales in the liquid-sealed lockhopper test unit during this quarter.

Task 3. Process Development Unit (PDU) Tests

During this quarter, the PDU was re-piped to convert the unit back to two-stage operation. One test was then conducted using a sample of the Gassaway member of the Chattanooga shale from Tennessee. Results of all tests conducted have now been analyzed and reported.

Task 4. Process Environmental Assessment

The Pace Company Phase I report was revised and is appended to this report. Trace-element analyses for feed and residue shales from the PDU test program have now been completed and are presented in this report.

Task 5. Process Design and Economics

A process design and cost estimate for conversion of Kentucky Sunbury oil shale to syncrude was completed. Shale oil costs are only about 15% higher than those obtained in the earlier study for Colorado oil shale feedstock. Thus, the HYTORT Process makes both Eastern and Western oil shales economically attractive sources of oil.

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PROGRAM OBJECTIVE

The objective of this program is to fully demonstrate the technical and economic feasibility of the HYTORT process concept as it applies to both Eocene and Devonian shales. Achievement of this objective will significantly widen the Nation's synthetic fuels resource base by adding a new resource — Devonian shale — to the list of possible feedstocks for a synthetic fuels industry. It will also establish a high-efficiency alternative to conventional retorting of Western oil shales.

INTRODUCTION

Until recently, many U.S. oil shales (for example, the Devonian shales of the Eastern United States) were considered to be unsatisfactory feedstocks for synthetic oil production because of their relatively low oil yields in the Fischer Assay test and in conventional thermal retorting. Because oil shales that are chemically more similar to Devonian shale than to Eocene shale actually constitute the bulk of the Nation's oil shale resources, and because many of these shales have organic carbon contents comparable with much of the Eocene shale deposits, there is a need for processes capable of economically processing both of these types of shale.

Research at the Institute of Gas Technology (IGT) has led to the development of the HYTORT process concept, which can be applied to the resources of Devonian shales of the Eastern United States as well as to the Western shales, to produce oil or combustible gas at costs competitive with or less than those for retorting of Colorado shales. The HYTORT process concept has been successfully scaled up through small-scale laboratory equipment, a bench-scale unit capable of processing 100 pounds of shale per hour, and a Process Development Unit (PDU) with a shale capacity of 1 ton/h. In addition, preliminary process design and economic analysis work shows that the HYTORT process exhibits attractive efficiency and economics. The main areas that required further research and that were studied in this program are as follows:

- Hydroretorting kinetics of those Devonian and Eocene shales not previously tested in the HYTORT process and the nature of primary products of hydroretorting
- Analysis of process heat effects (both chemical and physical) in the shale hydroretorting step
- H_2 manufacture by steam-oxygen gasification of raw shale fines or spent shale
- Upgrading of raw HYTORT shale oil
- Control of oil mist size in the hydroretorting reactor
- Performance of liquid-sealed lockhoppers on Eastern shale feed
- Assessment of the environmental aspects of the HYTORT process

- Process design and economics for production of syncrude from Eastern Devonian (Kentucky Sunbury) and Western Eocene (Colorado) shales.

The above process development work was covered by the following tasks and subtasks:

Task 1 - Laboratory Program

Subtask 1.1. Thermobalance Tests
Subtask 1.2. Laboratory-Scale Reactor Tests
Subtask 1.3. Calorimetric Studies

Task 2 - Bench-Scale Program

Subtask 2.1. Bench-Scale Reactor Tests
Subtask 2.2. Hydrogen Production Studies
Subtask 2.3. Shale Oil Upgrading
Subtask 2.4. Mist-Size Control Studies
Subtask 2.5. Liquid-Sealed Lockhopper Tests

Task 3 - Process Development Unit (PDU) Tests

Task 4 - Process Environmental Assessment

Task 5 - Process Design and Economics

To minimize total program costs, the objectives of some of these tasks have been modified from those defined originally, and this final quarterly report has been submitted in lieu of a final project report. Work conducted prior to this current quarter has been previously reported in five quarterly reports, Nos. FE-14120-1 and -2 and DOE/ET/14102-1, -2 and -3.

TECHNICAL PROGRESS REPORT

Task 1. Laboratory Program

Subtask 1.1. Thermobalance Tests

In this part of the program, tests were conducted to evaluate the chemical reactivity and determine the kinetics of reactions of selected Eastern and Western oil shales under a variety of operating conditions. Seventeen thermobalance runs were made during this quarter. Nine runs were made with different shales, and eight runs were made with "large" pieces of Kentucky New Albany shale. Feed and residue analyses are given in Tables 1 and 2. Carbon conversions are given in Tables 3 and 4.

All runs made this quarter utilized a nominal heating rate of 20°F/min from room temperature to 1400°F with a 10-minute hold at 1400°F. Gas pressure was 35 atm for all runs. CO and CO_2 were measured with infrared analyzers, and a flame hydrocarbon analyzer was used to measure the light hydrocarbon gases (LHC). The carbon conversion to liquid is found by the difference —

$$\underset{C \rightarrow \text{liq}}{\%\ \text{Conv}} = \underset{C\ \text{Conv}}{\%\ \text{Total}} - \underset{C \rightarrow \text{light hydrocarbons}}{\%\ \text{Conv}} - \underset{C \rightarrow CO + CO_2}{\%\ \text{Conv}} \qquad (1)$$

A series of tests was made to study the effects of particle size. The operating conditions were held constant: pure hydrogen at 35 atm, heat-up rate of 20°F/min, maximum temperature of 1400°F, 10-minute hold at 1400°F. Dimensional limitations of the thermobalance limited particle thickness to 0.5 inch. Particle size variation was achieved by cutting the shale rock into approximately rectangular parallelepipeds with lengths up to 2 inches. With respect to heat or mass transfer within the particles, these particles can be treated as spheres of a size for which the time to reach a prescribed temperature is comparable. A sample of dimensions $L_1 \cdot L_2 \cdot L_3$ behaves approximately like a sphere with an equivalent diameter —

$$D_{eq} = 2[\sum_i 1/L_i^2]^{-1/2} \qquad (2)$$

Table 1. FEED AND RESIDUE ANALYSES FOR NEW ALBANY SHALE — LARGE PARTICLES

	Proximate Analysis		Ultimate Analysis (dry basis)						
Material	Moisture	High Temperature H_2O	Ash	Total C	Organic C	Mineral C	Hydrogen	Sulfur	Nitrogen
	%								
S-81-3 Feed	0.6	3.2	77.07	14.96	14.86	0.10	1.72	4.93	0.51
Residue	0.2	1.1	96.55	3.48	3.36	0.12	0.18	1.97	0.12
S-81-4 Feed	0.7	3.6	77.08	14.85	14.66	0.20	1.71	5.48	0.32
Residue	0.2	0.7	95.95	3.85	3.80	0.05	0.19	2.29	0.12
S-81-5 Feed	0.7	3.6	77.37	14.75	14.50	0.25	1.69	5.50	0.52
Residue	0.2	0.6	95.77	3.79	3.62	0.17	0.19	2.12	0
S-81-6 Feed	0.7	2.9	77.70	13.58	13.37	0.21	1.54	6.89	0.41
Residue	0.4	1.4	96.36	2.91	2.75	0.15	0.23	1.78	0
S-81-7 Feed	0.6	3.4	76.99	15.48	15.21	0.27	1.81	4.32	0.42
Residue	0.1	0.8	96.13	3.84	3.66	0.18	0.18	1.78	0
S-81-8 Feed	0.6	3.1	80.56	11.97	11.77	0.20	1.42	5.11	0.34
Residue	0.3	0.7	97.24	2.69	2.58	0.12	0.21	2.09	0.15
S-81-9 Feed	0.6	3.2	79.59	12.80	12.51	0.29	1.53	5.00	0.26
Residue	0.3	0.9	96.66	3.14	3.05	0.09	0.22	2.05	0
S-81-10 Feed	0.7	3.3	78.74	13.37	13.13	0.23	1.59	5.10	0.43
Residue	0.4	0.8	97.17	3.15	3.00	0.15	0.22	1.97	0

Table 2. FEED AND RESIDUE COMPOSITIONS FOR THERMOBALANCE TESTS

	Proximate Analysis		Ultimate Analysis (dry basis), wt %						
Material	Moisture	High Temperature H_2O	Ash	Total C	Organic C	Mineral C	Hydrogen	Sulfur	Nitrogen
Utah Feed	2.0	2.4	57.91	24.43	19.97	4.45	2.75	0.40	0.83
S-80-71 Residue	0.1	1.8	89.89	2.92	2.06	0.86	0.57	2.32	0
S-80-72 Residue	0.5	1.0	77.18	10.67	6.50	4.17	0.24	0.47	0.34
Wy. Green River Feed	0.5	3.3	68.50	16.06	12.15	3.91	1.89	0.58	0.45
S-80-73 Residue	0.2	2.7	96.75	0.84	0.66	0.18	0.42	0.72	0
Gassaway Feed	0.6	3.0	82.60	9.97	9.91	0.06	1.09	6.43	0.34
S-80-74 Residue	0.1	0.9	95.87	3.64	3.58	0.06	0.20	2.88	0.09
Wy Washakie Feed	4.8	2.9	77.43	13.05	11.70	1.35	1.70	0.55	0.46
S-80-75 Residue	0.2	1.2	98.80	0.78	0.65	0.13	0.23	1.44	0.10
Gassaway Feed	0.8	3.3	78.48	13.18	12.82	0.36	1.42	5.54	0.37
S-81-1 Residue	0.2	1.1	95.68	3.44	3.33	0.11	0.19	3.00	0
S-81-2 Residue	0.3	0.4	88.68	8.40	8.14	0.27	0.16	3.47	0.19
Dowelltown Feed	2.8	3.7	91.27	3.81	3.53	0.28	0.73	0.23	0.11
S-81-11 Residue	0.4	1.1	98.85	1.03	0.95	0.08	0.18	0.09	0
S-81-12 Residue	0.3	0.9	96.86	2.64	2.57	0.06	0.14	0.24	0.12

Table 3. CARBON CONVERSIONS FOR NEW ALBANY SHALE — LARGE PARTICLES

Run	D_{eq} (in)	Total Carbon Conversion	Organic Carbon Conversion	C → LHC	C → CO	C → CO_2	C → liq
		%					
S-81-3	0.55	81.4	81.9	26.1	0.4	0.5	54.4
S-81-4	0.65	79.4	79.4	28.9	0.6	0.5	49.4
S-81-5	0.49	79.6	80.2	28.6	0.2	0.4	50.4
S-81-6	0.37	82.8	83.4	24.8	0.2	0.5	57.3
S-81-7	0.62	80.2	80.8	26.1	0.5	0.6	53.0
S-81-8	0.34	81.5	82.0	24.4	0.8	0.7	55.6
S-81-9	0.50	80.0	80.1	28.2	0.7	0.7	50.4
S-81-10	0.36	81.0	81.5	22.6	0.5	0.8	57.1

Table 4. CARBON CONVERSIONS FOR THERMOBALANCE TESTS

Thermobalance Run	Shale	Gas	Total Carbon Conversion	Organic Carbon Conversion	C → LHC*	C → CO	C → CO_2	C → liq
			%					
S-80-71	Utah Loc 168	H_2	92.6	93.6	18.4	9.1	2.2	62.9
S-80-72		N_2	66.9	75.3	7.0	6.5	4.3	49.1
S-80-73	Wy. Green River Loc 176	H_2	96.3	96.2	14.4	2.1	0.5	79.3
S-80-74	Gassaway Loc 180	H_2	68.7	69.0	28.6	0.4	0	39.7
S-80-75	Wy. Washakie Loc 177	H_2	95.5	95.8	14.3	5.4	3.4	72.4
S-81-1	Gassaway Loc 148/4	H_2	78.8	78.9	28.1	0.7	1.1	48.9
S-81-2		N_2	43.7	43.9	4.9	2.2	2.4	34.2
S-81-11	Dowelltown Loc 148/5	H_2	75.9	76.0	30.0	0.6	1.1	44.2
S-81-12		N_2	36.5	33.2	3.8	3.3	4.3	25.1

*Light gaseous hydrocarbons.

Because all large pieces could not be considered to be of the same composition, the raw shale composition of each run sample was estimated from other samples taken on either side of the run sample in the original rock. The variation in organic carbon was from 11.8 to 15.2 weight percent in the samples analyzed.

For an increase in D_{eq} from 0.08 to 0.65 inch, the total conversion decreases only about 5%. The distribution between gas and liquid is much more sensitive to particle size (Figure 1). While the yield of liquid decreases, the yield of gas increases approximately linearly with $(D_{eq})^{1/2}$; and by extrapolating on that basis, we estimate the yields from very small (-6 + 10 mesh) particles to be 82% of the carbon as liquid and 4% as gas.

Overall sulfur conversions for the large-piece (0.34-0.65 inch equivalent diameter) samples were 4.5% to 7.5% greater than the sulfur conversions (about 62%) observed with the small particles. The large-piece samples also showed higher conversions of high- temperature water than the corresponding small particle samples.

Both the liquid and low-temperature-CH_4-producing reactions have seemed to be faster for the large-piece particles. Observed temperatures for the maximum rates of reaction were as much as 60°F lower than those of the earlier tests with small particles. A comparison of the main weight loss peaks ($dw/d\theta$ vs. T) of a large and small particle test for the same New Albany (HK-2) shale tested under the same conditions is given in Figure 2.

Solids analyses and conversions for the tests made with six different shales this quarter are given in Tables 2 and 4. Table 5 presents the same information for the nine Eastern shales studied during this project. The Utah and Wyoming Green River shales had organic carbon conversions well above 90% in H_2 but a wide range of organic carbon conversions to liquid (63% to 79%). H_2 did improve the organic carbon conversion and the organic carbon conversion was lower than with the Utah shale using H_2, but the improvement over the conversion in N_2 was greater. H_2 also improved the conversion of organic carbon to liquid more with the Eastern shales than with the Utah shale. The Dowelltown and the two Gassaway shales all showed low conversions of organic carbon to liquid (40% to 49%) even in a 500 psig H_2 atmosphere. Table 5 shows that in 500 psig H_2 the organic carbon recovery as liquid is high for the

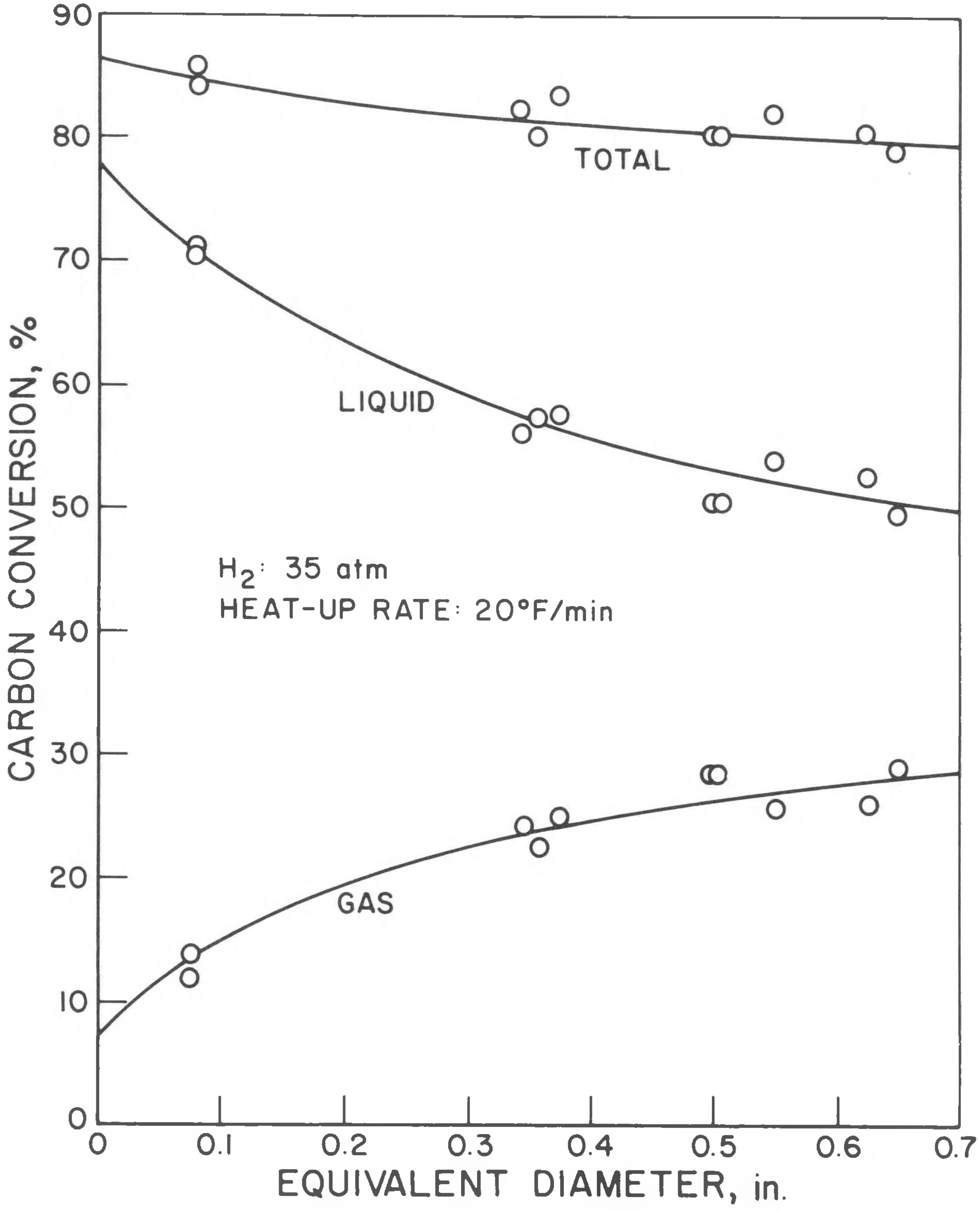

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Figure 1. VARIATION IN YIELD WITH PARTICLE SIZE FOR KENTUCKY NEW ALBANY SHALE (Location 64)

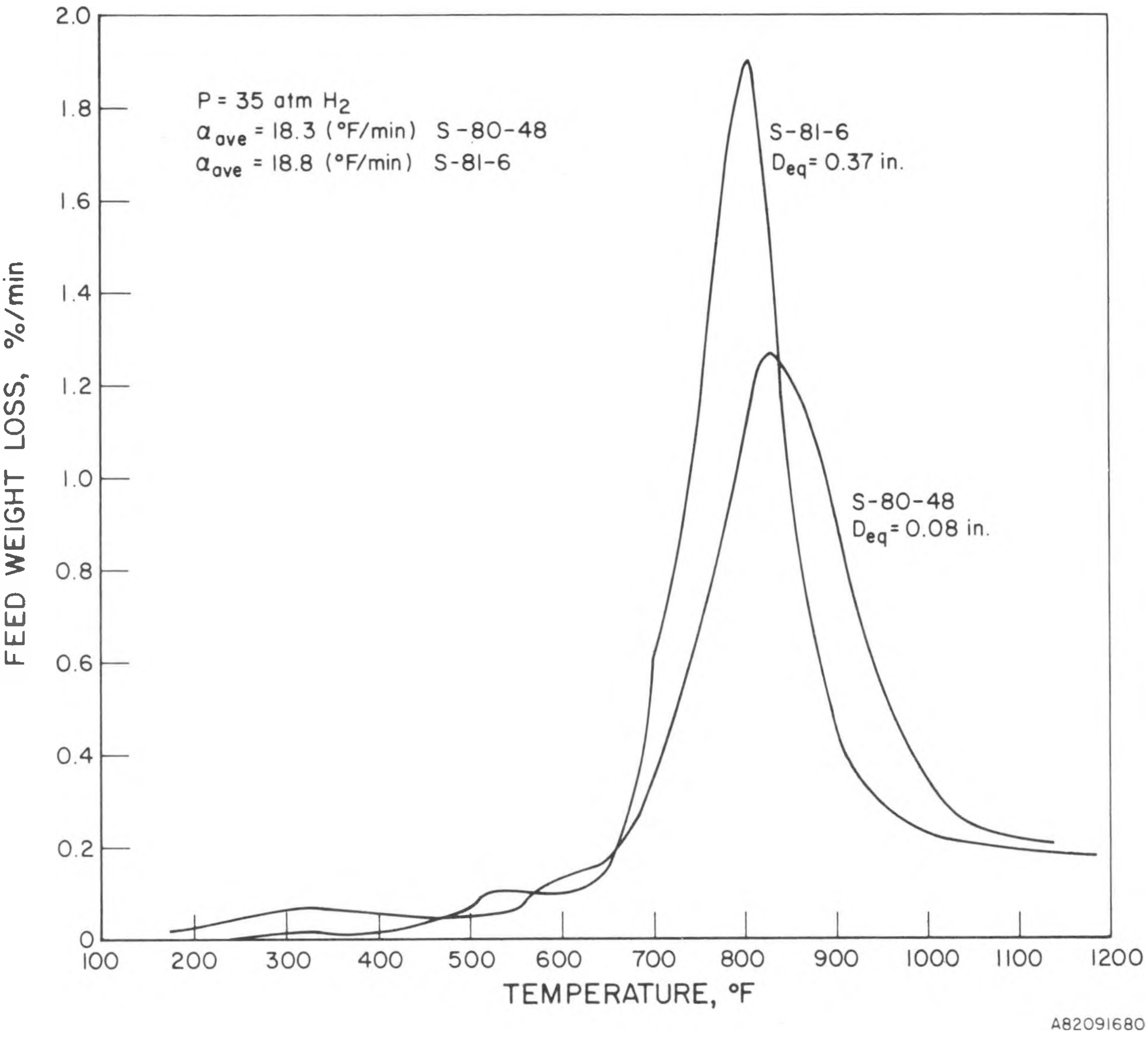

Figure 2. RATE OF WEIGHT LOSS OF KENTUCKY NEW ALBANY (HK-2) LARGE AND SMALL PARTICLES

Table 5. HYDRORETORTING ASSAYS OF EASTERN SHALES

Shale	Sunbury	New Albany (HK-1)		New Albany (HK-2)		Cleveland		Dowelltown		Gassaway	Gassaway		Antrim		Lower Huron	
State	Ky	Ky		Ky		Ohio		Tenn		Tenn	Tenn		Mich		Ohio	
Location No.	142B	64		64		139		148/5		180	148/4		147		154	
Run	S-80-45	S-80-24	S-80-20	S-80-48	S-80-49	S-80-9	S-80-10	S-80-11	S-80-12	S-80-74	S-81-1	S-81-2	S-80-15	S-80-16	S-80-46	S-80-47
Maximum Temp, °F*	1400	1400	1400	1200	1200	1400	1400	1400	1400	1400	1400	1400	1400	1400	1200	1400
Atmosphere	H_2	H_2	N_2	H_2	N_2	H_2	N_2	H_2	N_2	H_2	H_2	N_2	H_2	N_2	H_2	N_2
P, psig	500	500	500	500	500	500	500	500	500	500	500	500	500	500	500	500
Organic C Recovery, %																
Oil	64.2	67.6	29.1	71.8	34.9	41.6	19.8	47.7	27.1	39.8	50.3	35.2	34.1	42.8	62.8	29.3
LHC†	14.5	20.1	10.8	13.1	7.6	27.1	6.9	28.3	6.1	29.3	28.6	8.7	34.5	3.9	14.9	5.4
Total	78.7	87.7	39.9	84.9	42.5	68.7	26.7	76.0	33.2	69.0	78.9	43.9	68.6	46.7	77.7	34.7
Raw Shale Analysis																
Lab No.	122908	122559	122559	123765	123765	119492	119492	123833	123833	124492	124618	124618	120032	120032	123540	123540
Moisture, wt %	3.1	0.9	0.9	1.4	1.4	5.8	5.8	2.8	2.8	0.6	0.8	0.8	2.7	2.7	3.5	3.5
Ultiate Analysis, wt %																
(Dry basis) Org. C	15.53	14.42	14.42	14.12	14.12	9.85	9.85	3.53	3.53	9.91	12.82	12.82	7.49	7.49	11.78	11.78
CO_2	0.09	0.82	0.82	0.71	0.71	0.61	0.61	1.03	1.03	0.22	1.33	1.33	1.38	1.38	3.05	3.05
H	1.79	1.64	1.64	1.59	1.59	1.06	1.06	0.73	0.73	1.09	1.42	1.42	0.94	0.94	1.48	1.48
S	2.33	5.51	5.51	5.38	5.38	0.98	0.98	0.23	0.23	6.43	5.54	5.54	2.61	2.61	0.82	0.82
N	0.53	0.36	0.36	0.48	0.48	0.48	0.48	0.11	0.11	0.34	0.37	0.37	0.35	0.35	0.58	0.58
O (HTW)**	3.67	3.03	3.03	2.33	2.33	2.14	2.14	3.40	3.40	2.69	2.92	2.92	1.96	1.96	2.64	2.64
Ash	76.42	76.26	77.26	77.34	77.34	83.22	93.22	91.27	91.27	82.60	78.48	78.48	85.86	85.86	78.62	78.62
Total	100.36	102.04	102.04	101.95	101.95	98.34	98.34	100.30	100.30	103.28	102.88	102.88	100.59	100.59	98.97	98.97
Spent Shale Analysis																
Lab No.	123852	122541	122000	123854	123855	119609	119648	124905	124906	124563	124619	124620	120045	120114	123851	123850
Moisture, wt %	1.2	0.6	0.1	0.8	0.8	0.0	0.0	0.4	0.3	0.1	0.2	0.3	0.1	0.0	1.2	1.0
Ultimate Analysis, wt %																
(Dry basis) Org C	4.24	2.23	9.81	2.65	9.28	3.56	7.85	0.95	2.57	3.58	3.33	8.14	2.70	4.36	3.23	8.69
CO_2	0.17	0.33	0.25	0.70	0.61	0.20	0.11	0.29	0.23	0.23	0.41	0.97	0.15	0.17	0.69	2.47
H	0.31	0.17	0.23	0.25	0.30	0.22	0.21	0.18	0.14	0.20	0.19	0.16	0.25	0.18	0.37	0.34
S	0.95	2.69	3.85	3.06	3.96	0.22	0.77	0.09	0.24	2.88	3.04	3.47	1.28	1.90	1.02	0.80
N	0.06	0.05	0.32	0.11	0.42	--	--	0.00	0.12	0.09	0.00	0.19	0.06	0.17	0.10	0.42
O (HTW)**	0.87	0.51	1.04	0.92	0.71	--	--	0.96	0.81	0.80	0.97	0.33	1.17	0.95	1.22	0.80
Ash	95.04	97.35	87.27	95.11	87.16	95.85	90.91	98.85	96.86	95.87	95.68	88.68	96.25	93.56	94.38	86.99
Total	101.64	103.33	102.77	102.80	102.44	--	--	101.32	100.97	103.65	103.62	101.94	101.86	101.29	101.01	100.51

* All heating rates were 20°F/min.

† Light, gaseous hydrocarbons.

** By difference from 100%.

Lower Huron, New Albany, and Sunbury shales studied but much lower for the Cleveland, Gassaway, Dowelltown, and Antrim shales studied.

Summaries of all thermobalance runs made during the program are given in Appendix A.

The IGT shale sampling program included only one location in northern Michigan. In an effort to increase the amount of sampling, we requested and were given an Antrim core by the Dow Chemical Company. That core was from the Dow/DOE well #110 (Sec 8, T9N-R15E, Sanilac County, Michigan), which was assigned IGT's location number 181. The core was brought to Chicago and cut in half vertically.

The acquisition of a Dow core was deemed important because other Dow cores from the same site had been tested by many investigators pursuant to the Dow/Antrim Project. Thermobalance hydroretorting tests on that core could result in cross-correlations with other data such as Fischer Assay results[1] and pyrochromatographic studies,[2] although these have not been attempted to date.

Half the core was jaw crushed to about -80 mesh and sample intervals about 6 feet in length were choosen on the basis of lithologic character. Each sample was riffled to save 1 gallon of crushed shale. Two 8-ounce splits and one 4-ounce split were riffled out of the gallon sample. The 4-ounce sample was ground and used for analysis of carbon, hydrogen, and nitrogen. One 8-ounce sample of crushed material will remain with the half core and the other 8-ounce sample will remain with the gallon in IGT lab storage. The results of the analysis are shown in Table 6 and also as a histogram plotted alongside the Core-Gamma Correlation Log of the core made by Core Labs, Inc., in July 1979 (Figure 3). The general correspondence of gamma ray intensity with increased organic carbon in apparent. Mineral carbon was run on only five samples to verify the relatively inconsequential levels of carbonate carbon.

During the reexamination of the core for sample selection, the fossil alga Foerstia (Protosalvinia) (Figure 4) was discovered in a 5-foot zone at a depth of 1321 to 1326 feet. This fossil is important to stratigraphers because it marks a "time line" in the Late Devonian. It has never before been seen from Michigan and this discovery will now permit more precise correlation

Table 6. CARBON, HYDROGEN, AND NITROGEN IN CORE FROM SANILAC COUNTY, MICHIGAN (Dow Chemical Co./DOE Well #110, Sec 8, T9N-R15E), IGT LOCATION 181. CORE BEGINS IN BEDFORD SHALE AND ENDS LOW IN ANTRIM

Sample Number	Depth, feet	Thickness, feet	H, wt %	N, wt %	Carbon, wt %		
					Total	Organic	Mineral
1	1178-1186.3	8.3	0.76	0.09	1.03		
2	1186.3-1188.9	2.6	0.52	0.07	1.90		
3	1188.9-1189.3	0.4	0.43	0.05	0.56		
	Top of Antrim 1189.3						
4	1189.3-1195	5.7	1.60	0.40	11.61	11.50	0.11
5	1195-1200	5	1.45	0.35	9.96	9.83	0.13
6	1200-1206	6	1.31	0.30	8.41		
7	1206-1212.2	6.2	1.31	0.31	8.67		
8	1212.2-1219.5	7.3	1.18	0.27	8.12		
9	1219.5-1226.5	7	1.27	0.29	8.72		
10	1226.5-1233	6.5	1.29	0.27	8.68		
	*						
11	1234-1240	6	1.07	0.22	6.30		
12	1240-1248	8	0.96	0.22	5.44		
13	1248-1250	2	0.84	0.17	4.23	4.0	0.23
14	1250-1257	7	0.86	0.19	4.62		
15	1257-1264	7	0.83	0.17	4.18		
16	1264-1272	8	0.77	0.17	4.50		
17	1272-1281	9	0.82	0.18	4.81		
18	1281-1287	6	0.89	0.19	5.58		
19	1287-1293	6	0.92	0.20	5.71		
20	1293-1299	6	0.96	0.20	6.35		
21	1299-1305	6	1.02	0.20	6.68		
22	1305-1311	6	0.89	0.21	6.90	6.53	0.38
23	1311-1317	6	0.89	0.21	6.15		
24	1317-1323	6	0.85	0.20	5.88		
25	1323-1329	6	0.91	0.21	6.31		
26	1329-1334.4	5.4	0.98	0.22	7.12	6.77	0.35
	*						
27	1337.9-1340.8 *1341.2-1341.8	3.5	0.96	0.22	7.25		

* Core missing.

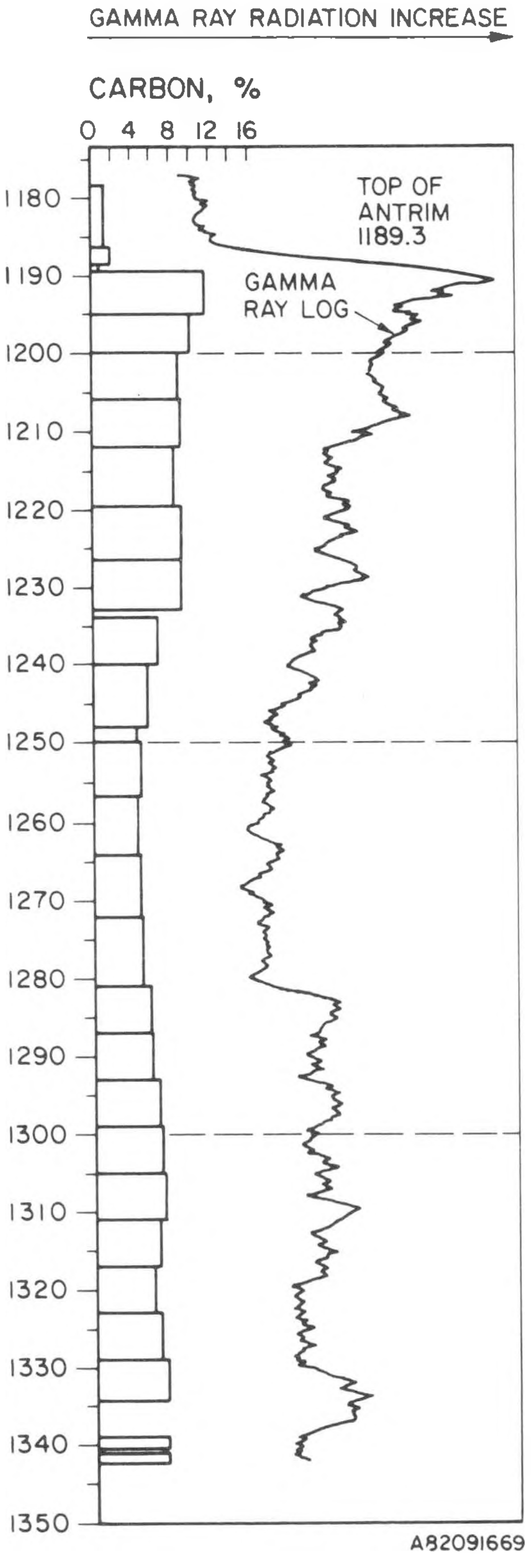

Figure 3. TOTAL CARBON COMPARED TO NATURAL GAMMA OF CORE FROM DOW/DOE WELL #110, SANILAC COUNTY, MICHIGAN

Figure 4. FOSSIL ALGA <u>FOERSTIA</u> FROM ANTRIM SHALE CORE, SANILAC COUNTY, MICHIGAN

between the Michigan Basin, the Appalachian Basin, and the Illinois Basin. Foerstia occurs in Unit 2 as defined by Ells[3] in his stratigraphic gamma ray cross sections of the Michigan Basin. Because of this identification we now know Unit 2 in Sanilac County, Michigan, correlates with the Middle Huron of Ohio — a positive time relationship not understood before.

Subtask 1.2. Laboratory-Scale Reactor Tests

In this part of the program, a small integral reactor was designed and constructed to determine oil shale hydroretorting product distributions and properties under integral reactor conditions. During this quarter, construction of the laboratory-scale reactor test unit was completed and pressure tests were carried out. Preliminary tests were conducted on the major equipment and the safety and control devices. The gas flow meters in the main and purge streams, the bed level controller, and the feed screw were calibrated. Because of early termination of this project no experimental results are available from the operation of this unit.

A flow diagram of the laboratory-scale reactor unit is shown in Figure 5. The unit consists of an integral hydroretorting reactor with equipment for feeding and measuring the flow rates of residue shale and feed gas (hydrogen) and for collecting and/or measuring the flow rates of residue shale, liquid product, and product gas. Simple controls are used to maintain reactor temperatures at the desired values, to maintain constant reactor pressure, and to collect representative samples of the feed and product gases.

Shale is fed by a screw feeder from a pressurized hopper that, before testing, is filled with a sufficient amount of shale for the entire run (about 20 lb). The cold shale enters the top of the reactor and is preheated by counter-current contact with the hot product gas. The residue shale is discharged from the bottom of the reactor into a pressurized residue receiver by a second screw feeder.

The feed gas enters the bottom of the reactor after passing through a preheater, and the product gas and vapor leave the top of the reactor tube. A small purge flow of the feed gas is continuously passed into the feed hopper during the test to make up for the volume of shale lost from the feed hopper as shale is fed into the reactor and to keep exit gas containing liquid product from entering the feed hopper.

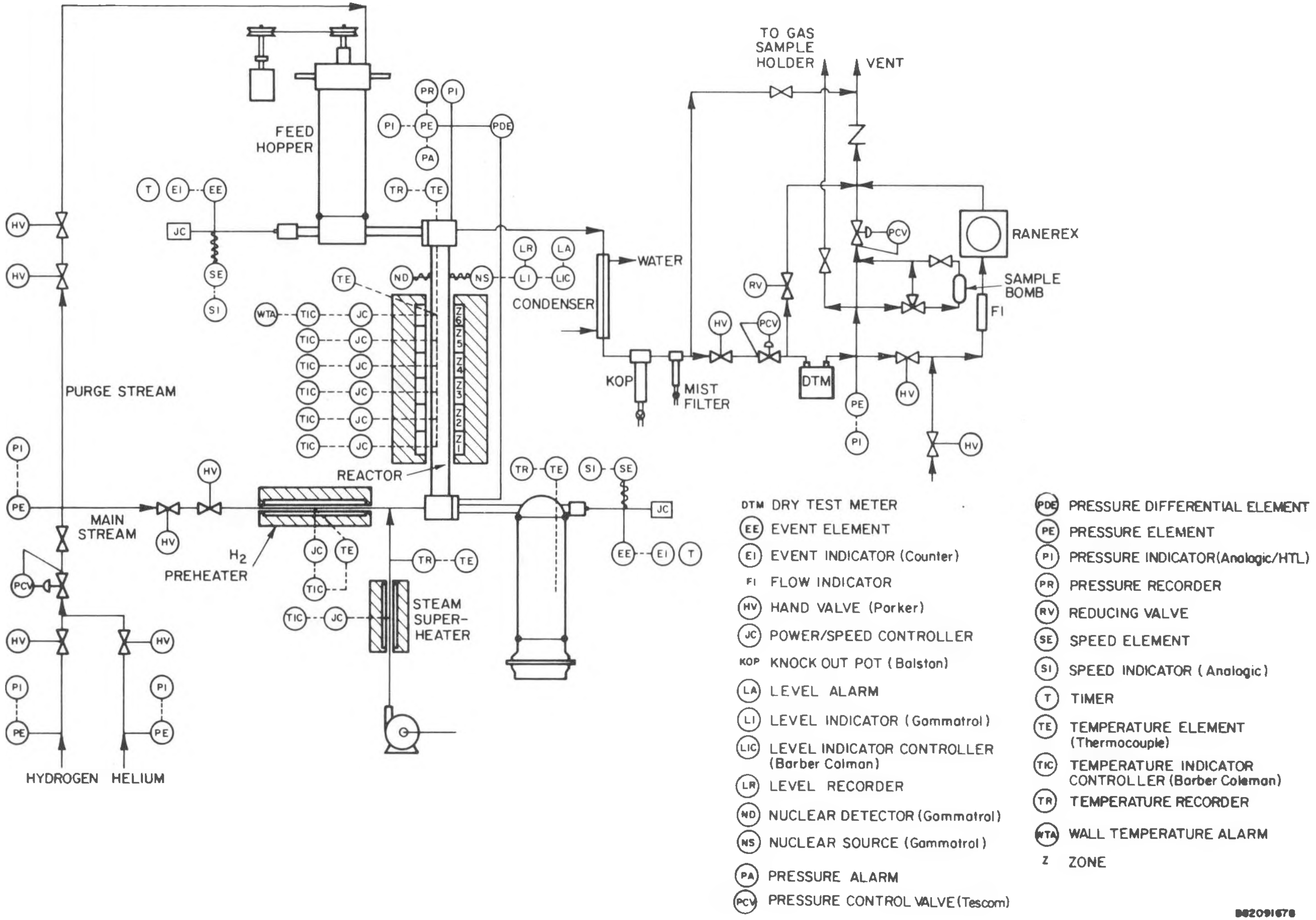

Figure 5. FLOW AND INSTRUMENTATION DIAGRAM FOR LABORATORY-SCALE REACTOR UNIT

The reactor (Figure 6) consists of a pressure shell containing the reactor tube, which is heated by a six-zoned electric heater. Each zone is 3 inches long and the heater extends from about 10 to 28 inches from the bottom of the reactor tube. An additional electric heater is used to heat a portion of the reactor above the main heater. The temperature of each zone is controlled individually by a temperature controller. The reactor is about 39-1/8 inches long and contains a tapered tube with an ID of 1.10 inches at the top and 1.77 inches at the bottom. The top 9 inches of this tube are not tapered and it has a thickness of 0.075 inches. Six separate thermocouples are used to measure the temperatures of the shale bed. Thermocouples are enclosed in a 5/8-inch OD thermowell. Reactor temperatures are recorded continously along with the temperatures of other major equipment.

Both screw feeders are calibrated. The shale is fed at the desired rate by setting the shale feed screw speed of rotation at the desired value. The rate of shale discharge then is set manually as required to give a constant shale-bed level. The shale-bed level is indicated by a nuclear-type level detector and is continuously recorded. This level can be controlled to within about 1 inch. The total weight of shale fed during the run is measured by weighing the shale initially charged to the feed hopper and that present at the end of the run. The residue shale is weighed after the run. Feed and residue shale rates are calculated by dividing these weights by the shale feeding time.

The exit gas volume is measured by a direct-reading dry-test meter. An "aliquot" sample of the exit gas is taken by feeding a portion of it into a water-sealed holder during the steady-state test portion of each run. "Spot" samples are also taken throughout each steady-state test period. The exit gas is cooled in a single-stage condenser. The condensed liquids are then collected in a knockout pot. The pot is drained and the liquid is weighed at approximately 20-minute intervals during the steady-state portion of each run to obtain product oil rates. We define steady-state as a condition wherein the temperature profile in the shale bed is uniform; feed gas, purge gas, and the exit gas flow rates are constant; reactor pressure is stable; and the shale feed rate and bed height are essentially constant.

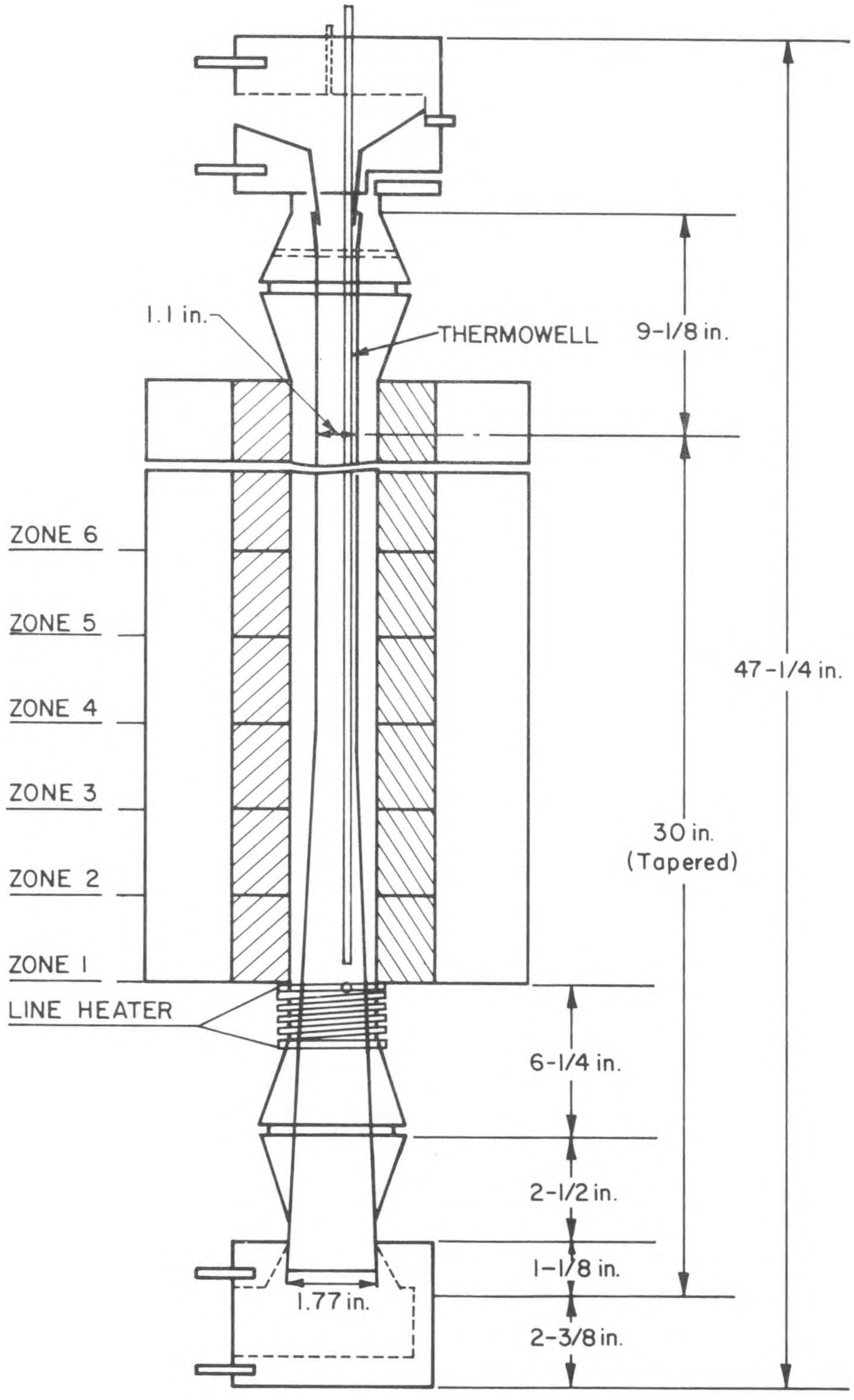

Figure 6. HIGH-TEMPERATURE REACTOR DETAILS

The reactor is filled with fine silica sand prior to each run to establish a bed and the desired temperature profile prior to initiating shale feeding. This also avoids rapid heating of the initial shale feed and a varying bed height at the start of a run. It also subjects all feed shale to a similar time-temperature history.

The feed and residue shale as well as the fine silica sand used in these test runs are weighed on a balance that can be read to 0.1 gram. Liquid products from the runs are also weighed on this balance.

Subtask 1.3. Calorimetric Studies

In this subtask tests were conducted to determine the heat capacities and heats of reactions of selected Eastern and Western oil shales under hydroretorting conditions. Eighteen tests were conducted with various Eastern and Western oil shales in the Du Pont Model 990 Differential Scanning Calorimeter (DSC) this quarter. Because the amount of shale required for each test is on the order of 20 mg or less, all samples were ground to a very fine particle size to assure the most representative, homogeneous sample possible for the DSC measurements. The screen analysis given in Table 7 is an example of the sample particle size range.

Table 7. TYPICAL SCREEN ANALYSIS

Particles Size, U.S.S. Sieve Size	wt %
+70	53.1
-70+100	6.7
-100+140	6.4
-140+200	5.7
-200	28.1
Total	100.1

Tests were conducted with eight different oil shales. The samples tested consisted of one Western oil shale (from Colorado) and seven Eastern oil shales. The Eastern oil shales examined were two Kentucky New Albany, two Gassaway, one Cleveland, one Lower Huron, and one Sunbury shale. A summary of conditions used for tests is given in Table 8. In all tests the sample was heated at 20°C/min, and the gas purge rate was set at approximately 0.4 SCF/h. Residue shale tests were made by taking the sample through a second heating cycle without exposing the sample chamber to air.

Table 8. SUMMARY OF DSC TESTS

Shale	Test	Atm	P, psig	Remarks
Kentucky New Albany (HK-2)	80-149	H_2	0	
	80-150	H_2	0	Residue of 80-149
	80-177	H_2	500	
	80-178	H_2	500	Residue of 80-177
	81-15	H_2/N_2 (50/50)	0	
	81-21	H_2/N_2 (50/50)	200	
	81-28	H_2/N_2 (50/50)	400	
	81-31	H_2/N_2 (50/50)	600	
	81-56	H_2/N_2 (50/50)	800	
	81-4	N_2	0	
Colorado (CS-1)	80-144	H_2	0	
	80-134	H_2	0	
	80-135	H_2	0	Residue of 80-134
	80-138	H_2	500	
	80-139	H_2	500	Residue of 80-138
	80-145	N_2	0	
Sunbury (SS-1)	80-168	H_2	0	
	81-2	H_2	0	
	81-3	H_2	0	Residue of 81-2
Cleveland (CL-2)	80-196	H_2	0	
	80-194	H_2	0	
	80-195	H_2	0	Residue of 80-194
Kentucky New Albany (NA-1A)	80-174	H_2	0	
	80-172	H_2	0	
	80-173	H_2	0	Residue of 80-172
Gassaway (GA-2)	80-182	H_2	0	
	80-162	H_2	0	
	80-163	H_2	0	Residue of 80-162
Gassaway (GA-3)	80-155	H_2	0	
	80-156	H_2	0	Residue of 80-155
	80-198	H_2	0	
	80-199	H_2	0	Residue of 80-198
Lower Huron (HU-2)	80-166	H_2	0	
	80-167	H_2	0	Residue of 80-166

All feed shales and residues were tested in 0 psig H_2. New Albany (HK-2) and Colorado (CS-1) feed and residue were tested in 500 psig H_2 and 0 psig N_2. New Albany (HK-2) shale was tested under a range of pressures in an H_2/N_2 (50/50) atmosphere.

The apparent specific heats of the raw and spent shales studies were determined and are shown in Figures 7 through 18. There is still a question of the accuracy of the results. Under even low-pressure hydroretorting conditions, the variation in the sapphire calibration alone is of the order of 10%. The variation is even greater under high hydrogen pressure.

The main wide peak between 425° and 500°C of the atmospheric pressure tests (Figures 7 through 13, 15, 16, and 18) probably corresponds to the major oil generation period. The very sharp peak observed near 540°C with all Eastern shales at low pressure seems, on the basis of other shale analyses, to be related to the presence of pyrites. A phase change within the shale may be occurring at this temperature rather than the decomposition of pyrite. The sharp peak for most DSC residues at about 150°C may also indicate a phase change in the residue.

For the high-pressure tests with Kentucky New Albany (HK-2) shale (Figures 13 through 15), the sharp peak at 540°C did not appear. The main endotherm occurred over a larger temperature range — possibly because of the imposition of some exothermic processes. This effect is most clearly observed in Figure 15 where the apparent specific heat is shown for samples exposed to a range of pressures from 0 to 800 psig in H_2/N_2 (50/50) mixtures.

The Colorado shale (Figures 16 and 17) showed two large endothermic peaks near 350° and 475°C. At 500 psig H_2, the endothermic peaks were broadened but not to the extent observed with the Kentucky New Albany shale. The sharp endotherm observed near 540°C in tests with Eastern shales was absent in the 0 psig H_2 test with Colorado (CS-1) shale. The size of the 540°C endotherm in 0 psig H_2 (Figures 7 through 13 and 16) correlates well with the amount of sulfur in the feed shale (Table 9). The sulfur and the pyrite contents of Colorado (CS-1) shale was 2.5 to 7.5 times lower than in the Eastern shales studied.

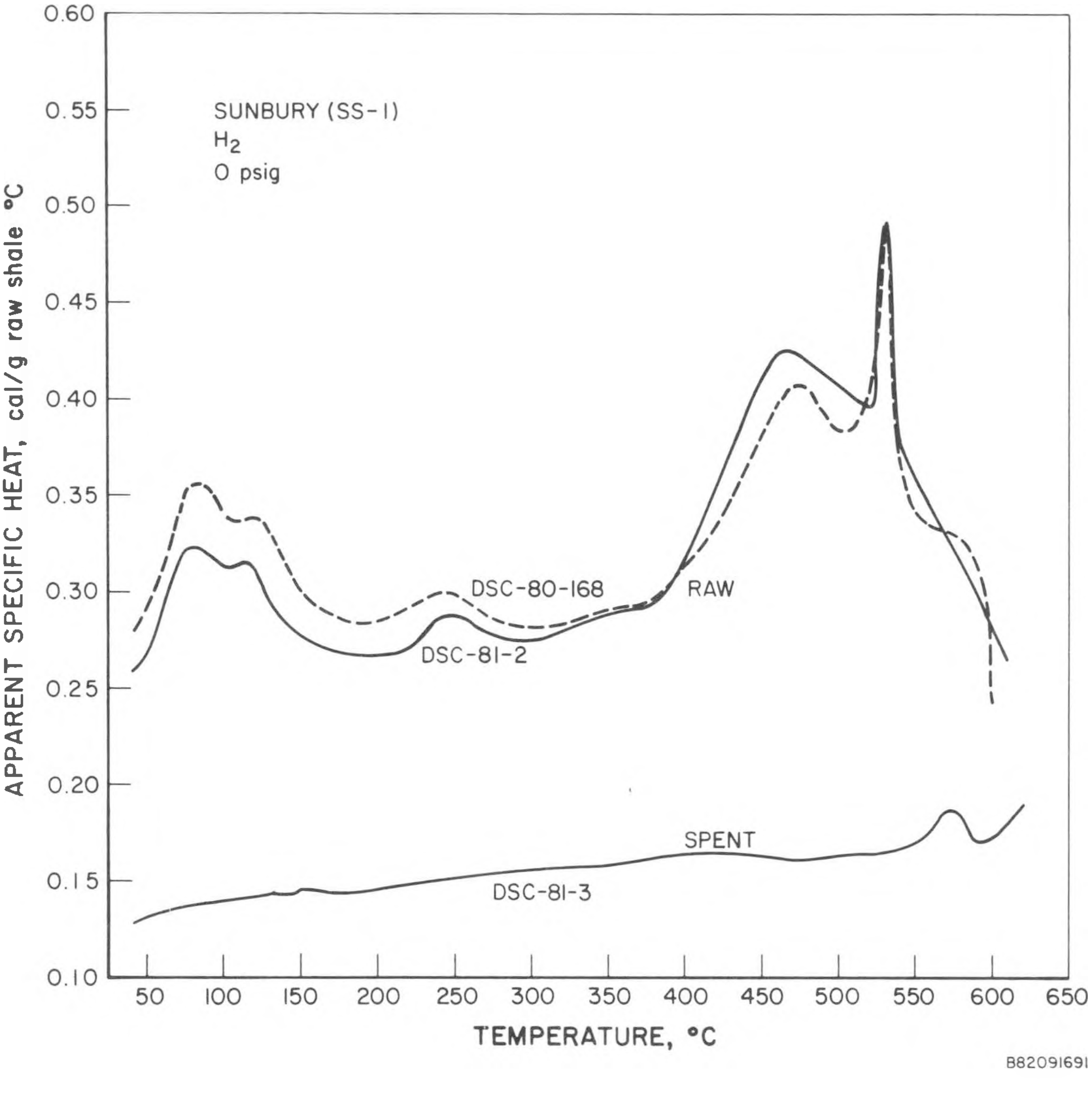

Figure 7. APPARENT SPECIFIC HEATS OF SUNBURY SHALE

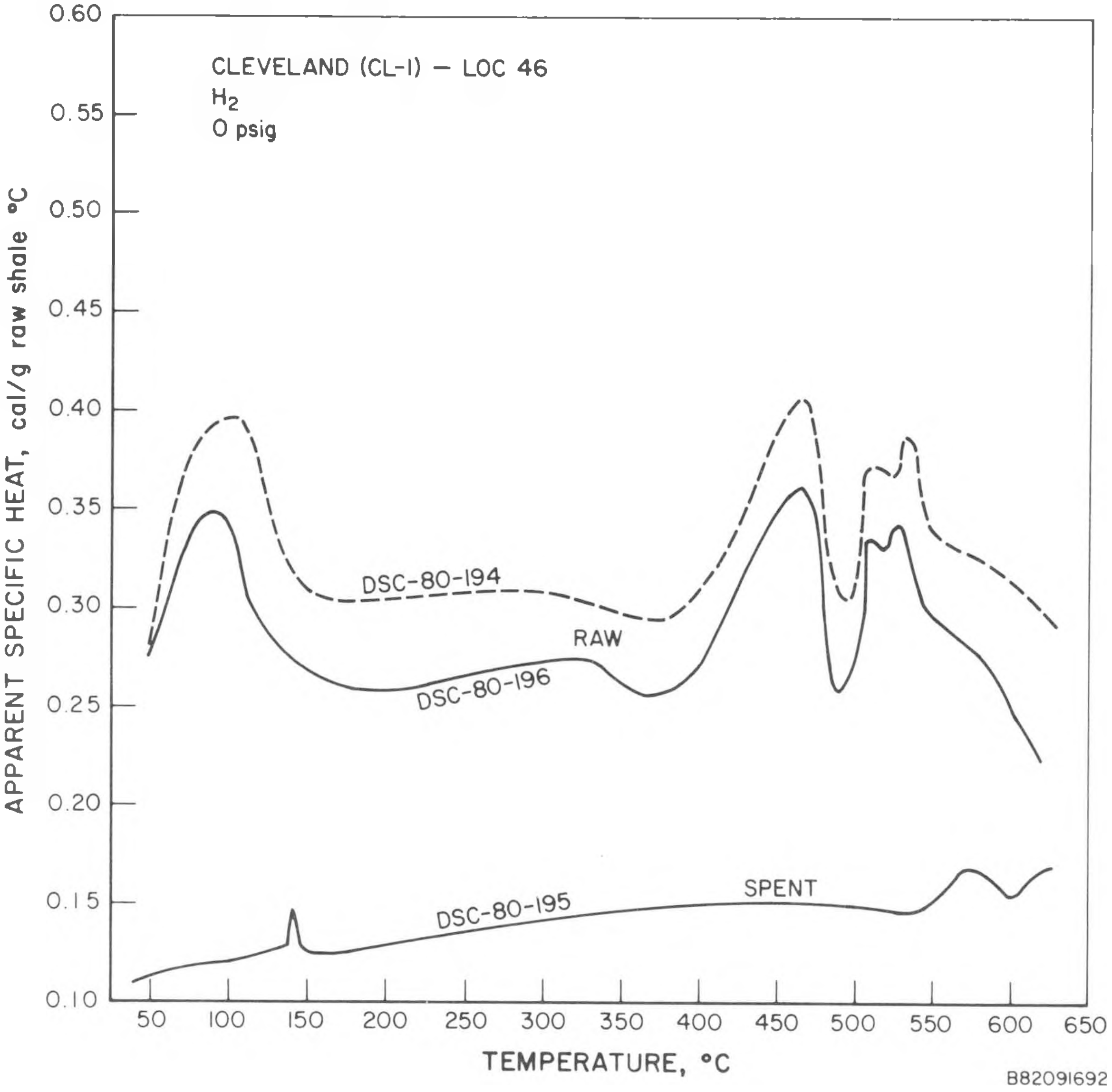

Figure 8. APPARENT SPECIFIC HEATS OF CLEVELAND SHALE

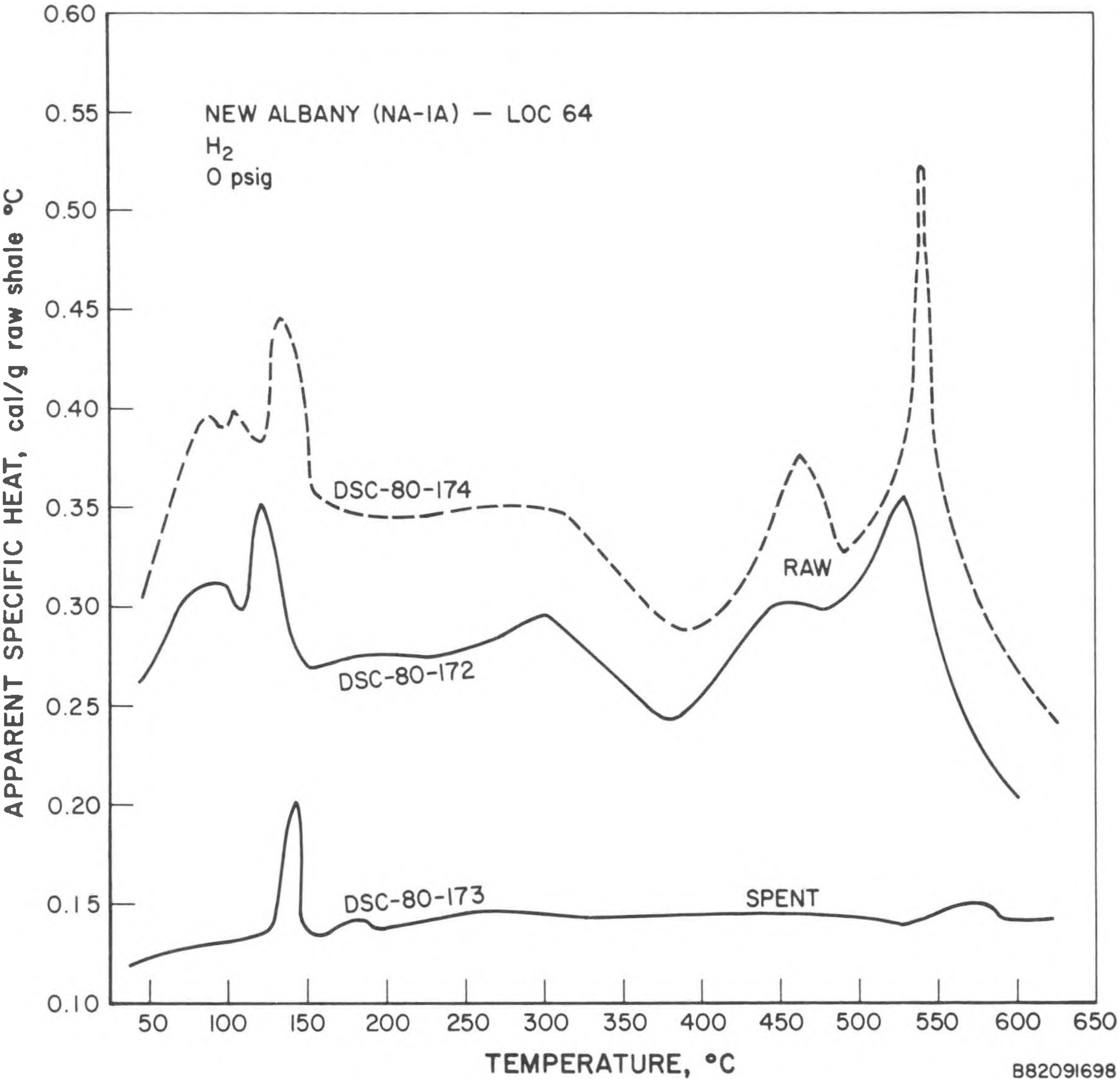

Figure 9. APPARENT SPECIFIC HEATS OF NEW ALBANY SHALE

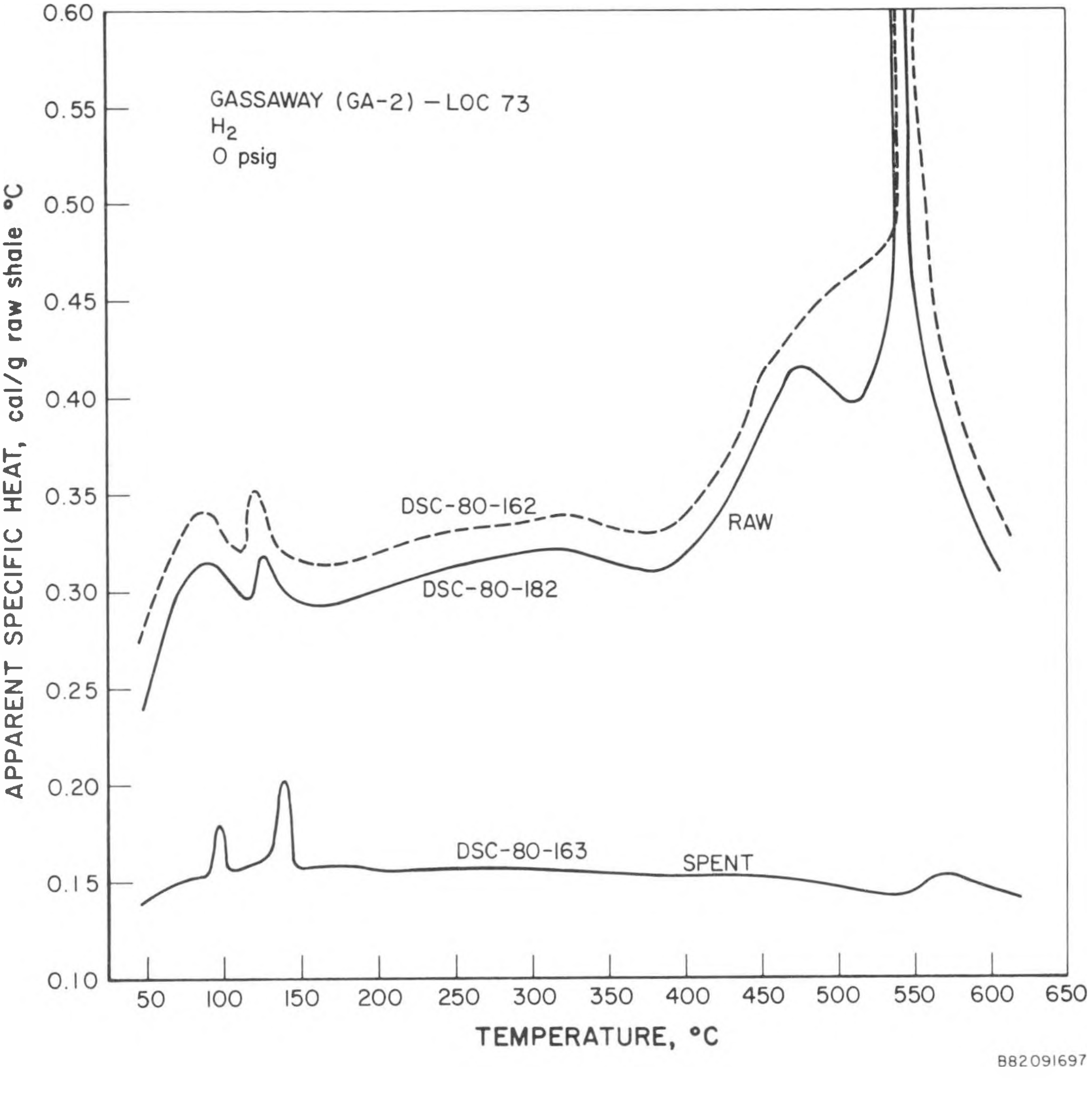

Figure 10. APPARENT SPECIFIC HEATS OF GASSAWAY SHALE (GA-2)

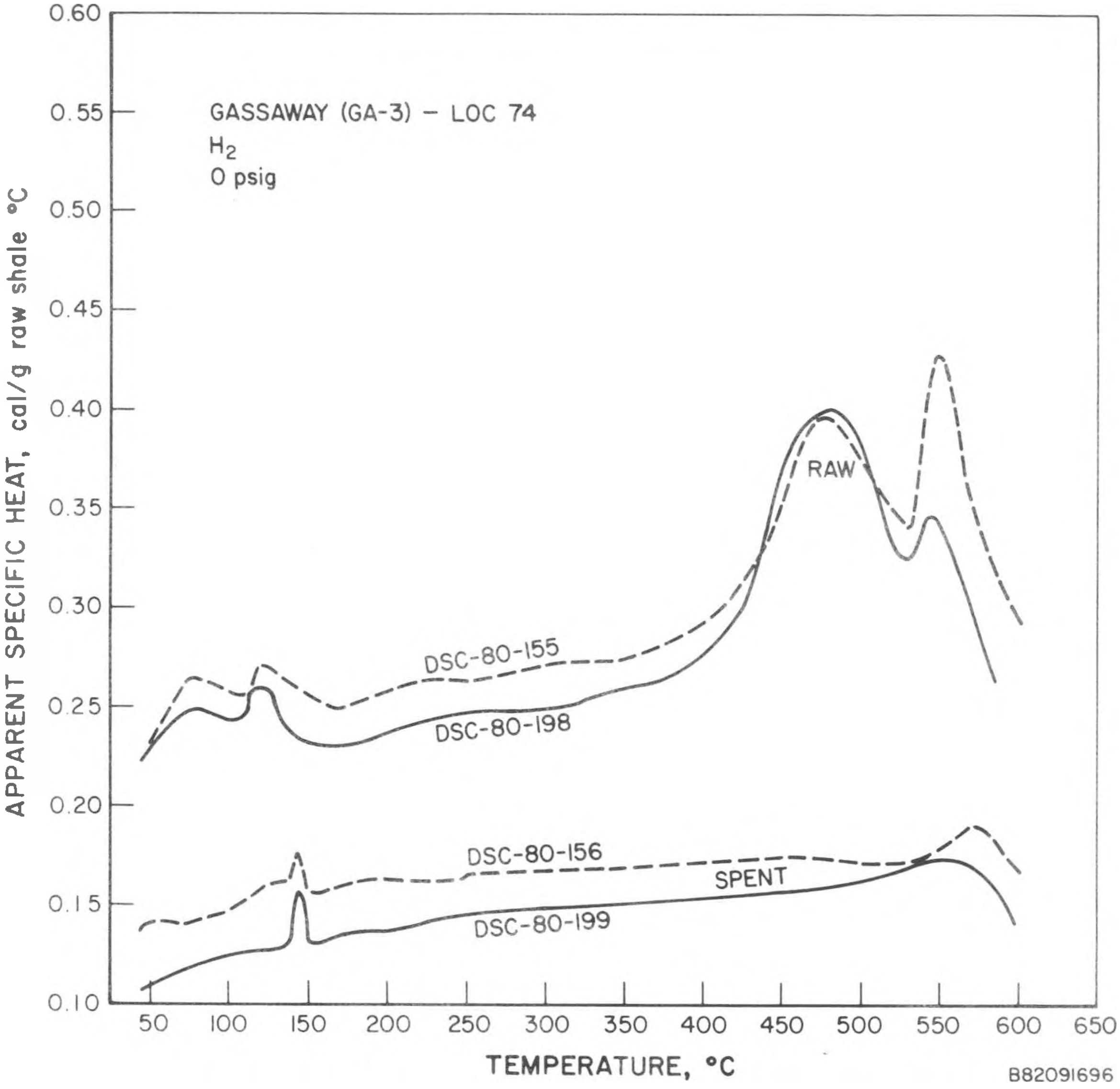

Figure 11. APPARENT SPECIFIC HEATS OF GASSAWAY SHALE (GA-3)

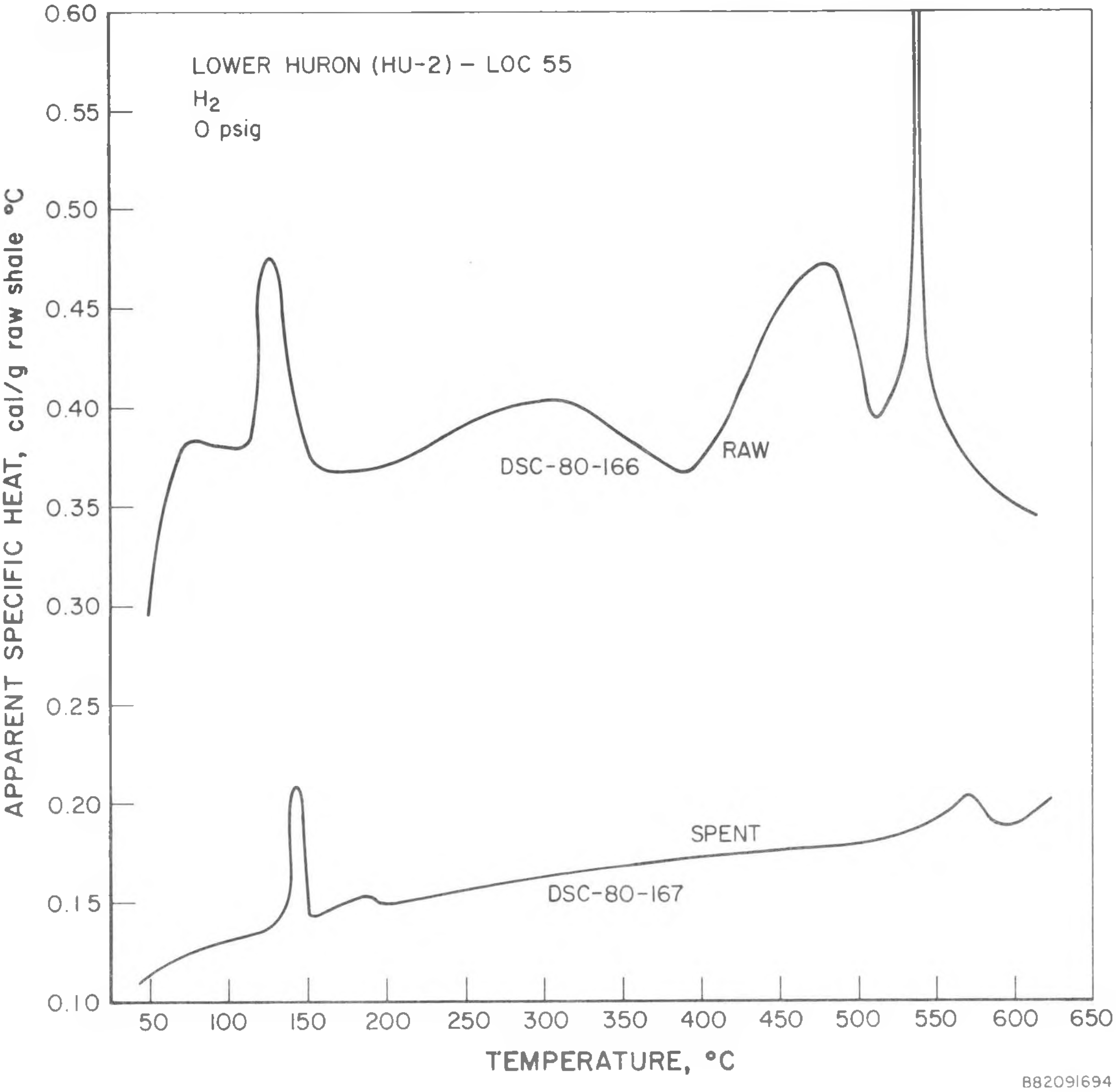

Figure 12. APPARENT SPECIFIC HEATS OF LOWER HURON SHALE

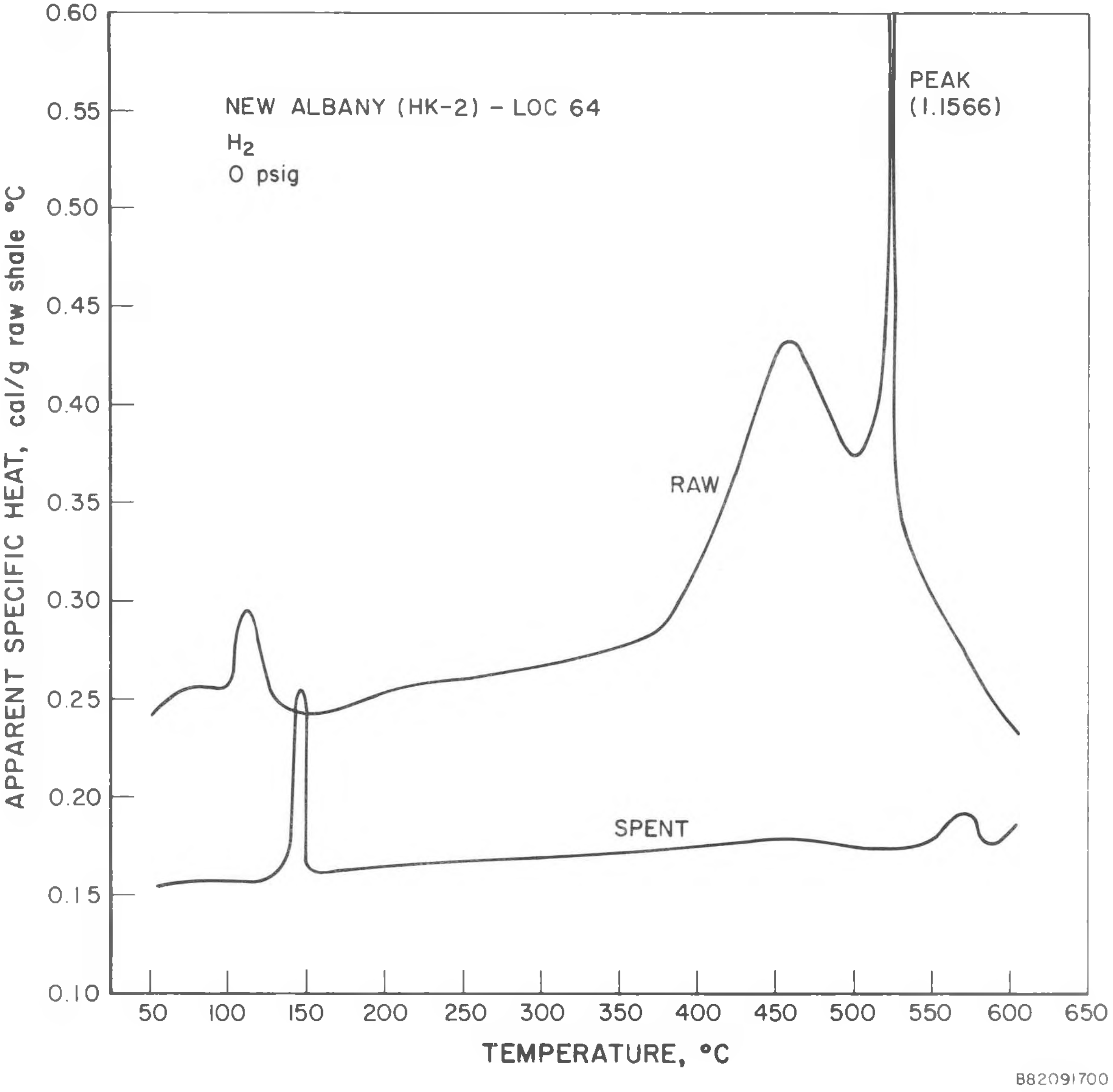

Figure 13. APPARENT SPECIFIC HEATS OF NEW ALBANY SHALE IN 0 psig H_2

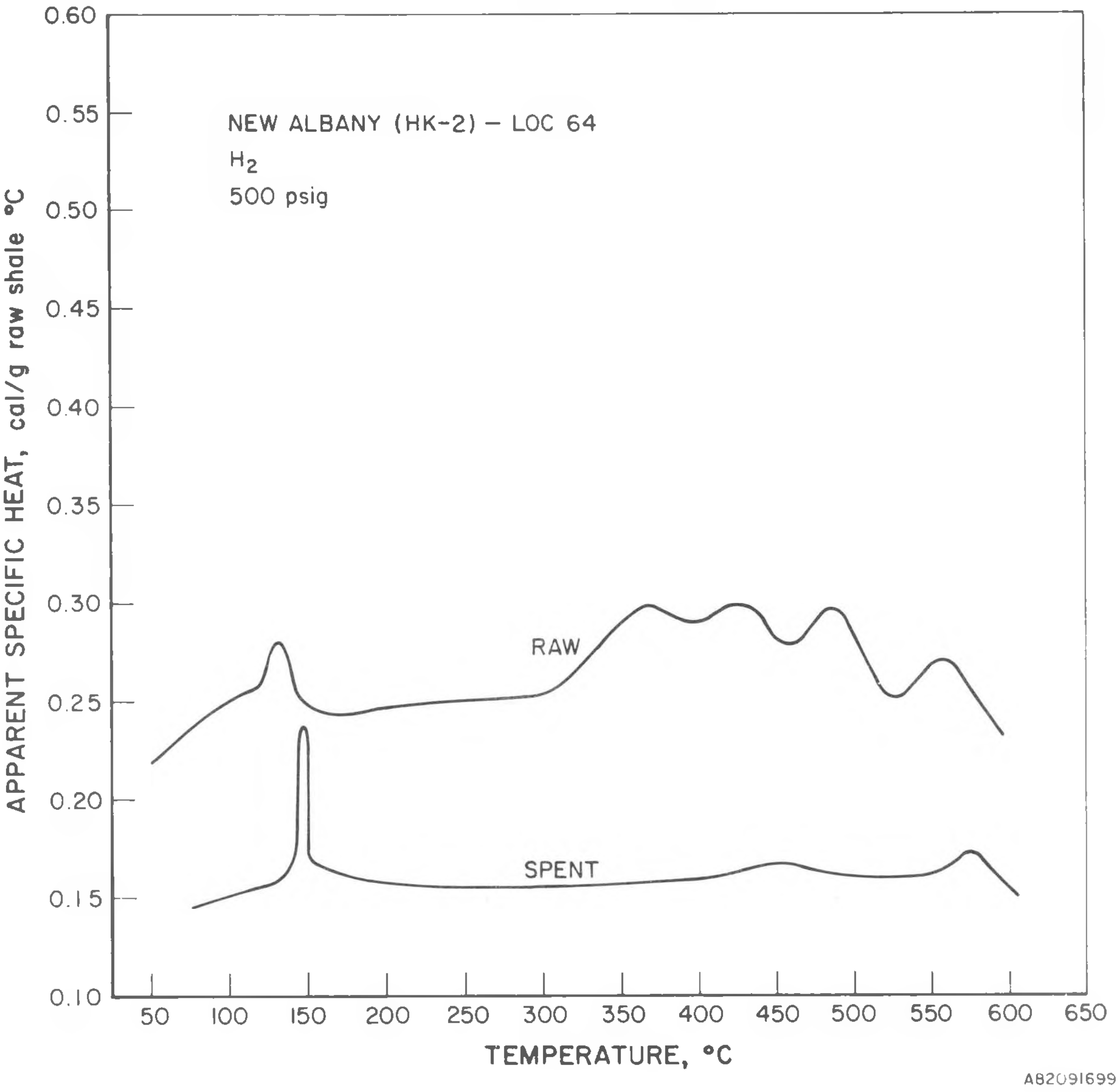

Figure 14. APPARENT SPECIFIC HEATS OF NEW ALBANY SHALE IN 500 psig H_2

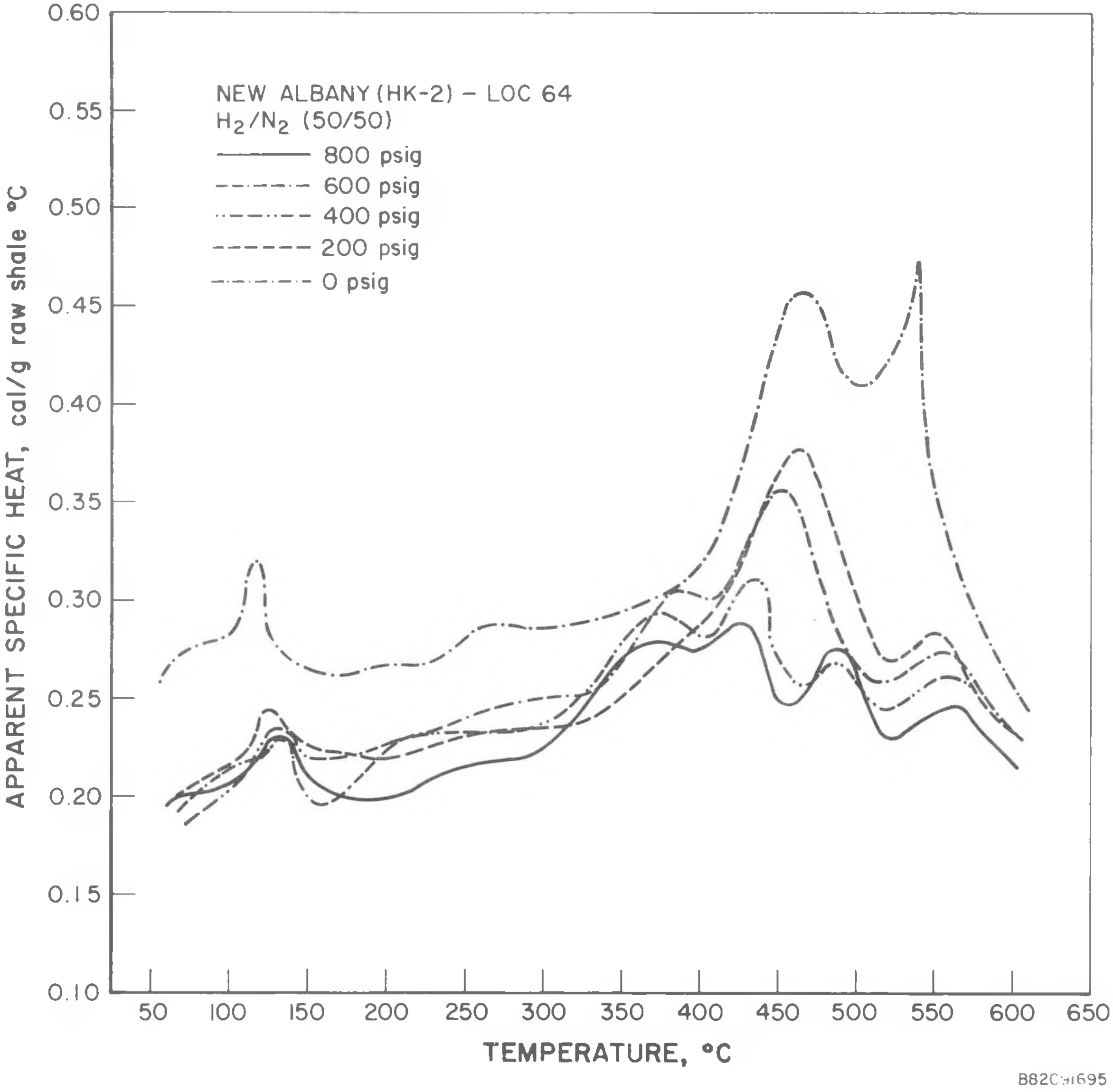

Figure 15. APPARENT SPECIFIC HEATS OF NEW ALBANY SHALE IN AN H_2/N_2 MIXTURE

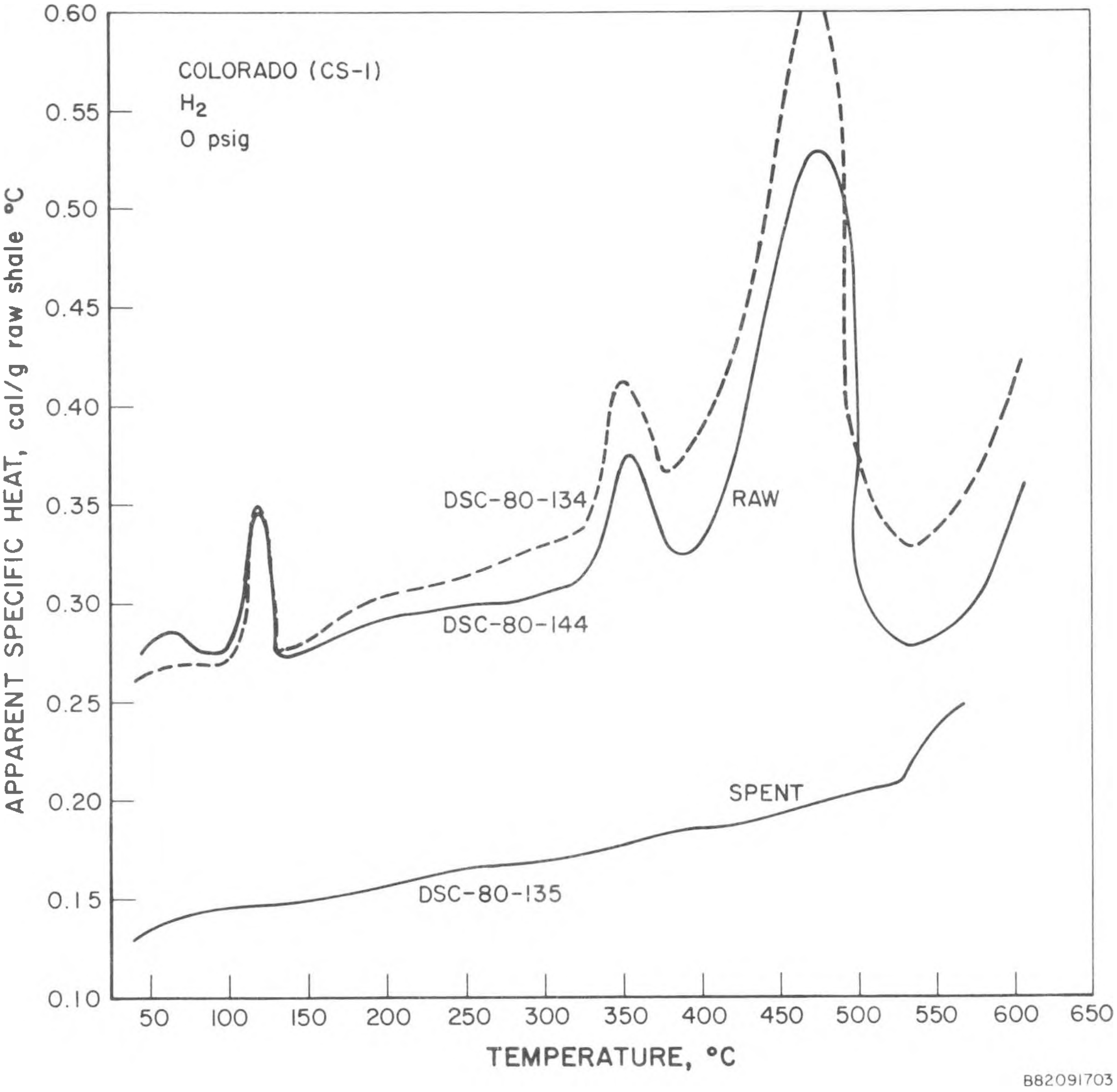

Figure 16. APPARENT SPECIFIC HEATS OF COLORADO SHALE

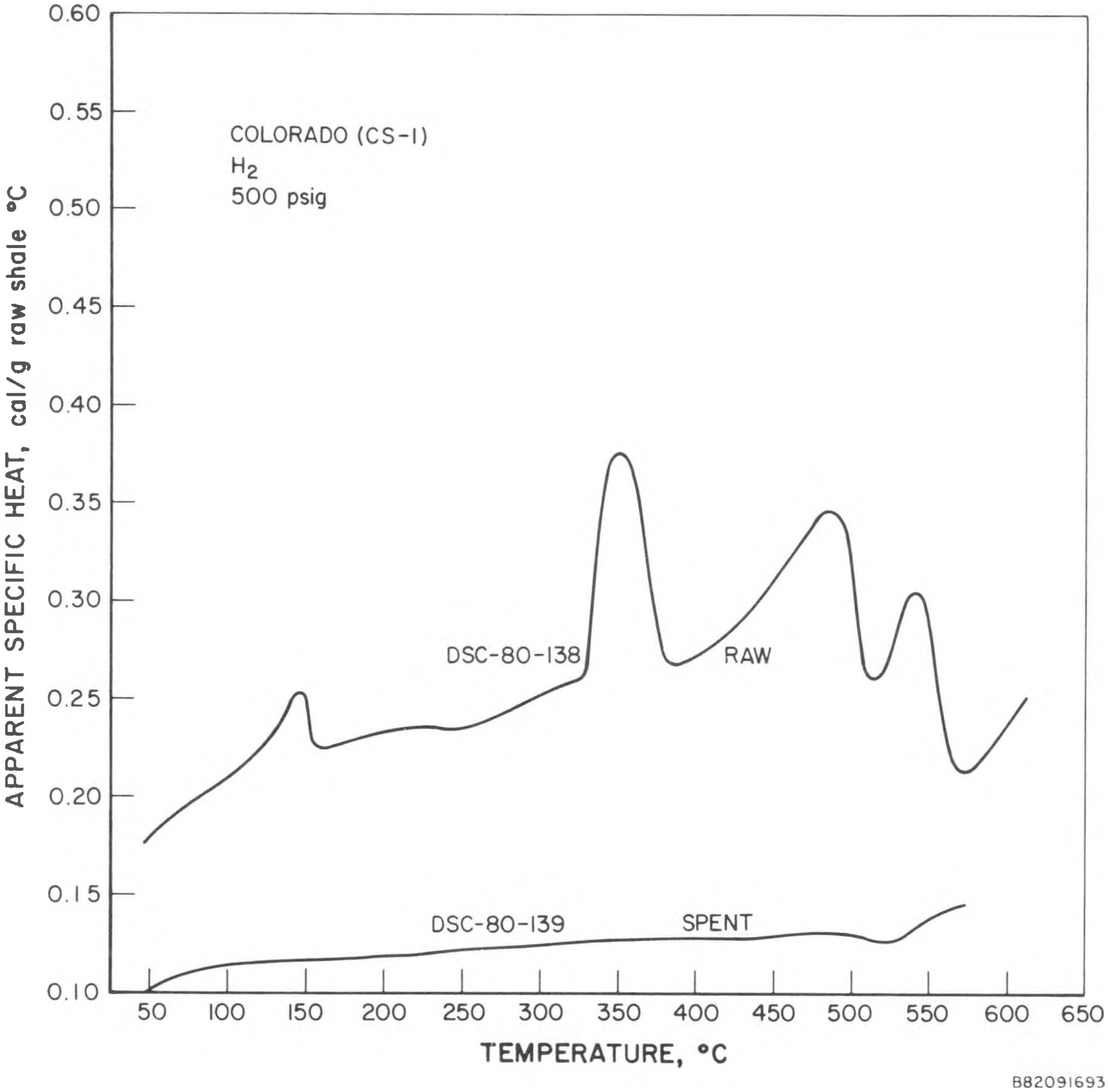

Figure 17. APPARENT SPECIFIC HEATS OF COLORADO SHALE IN 500 psig H_2

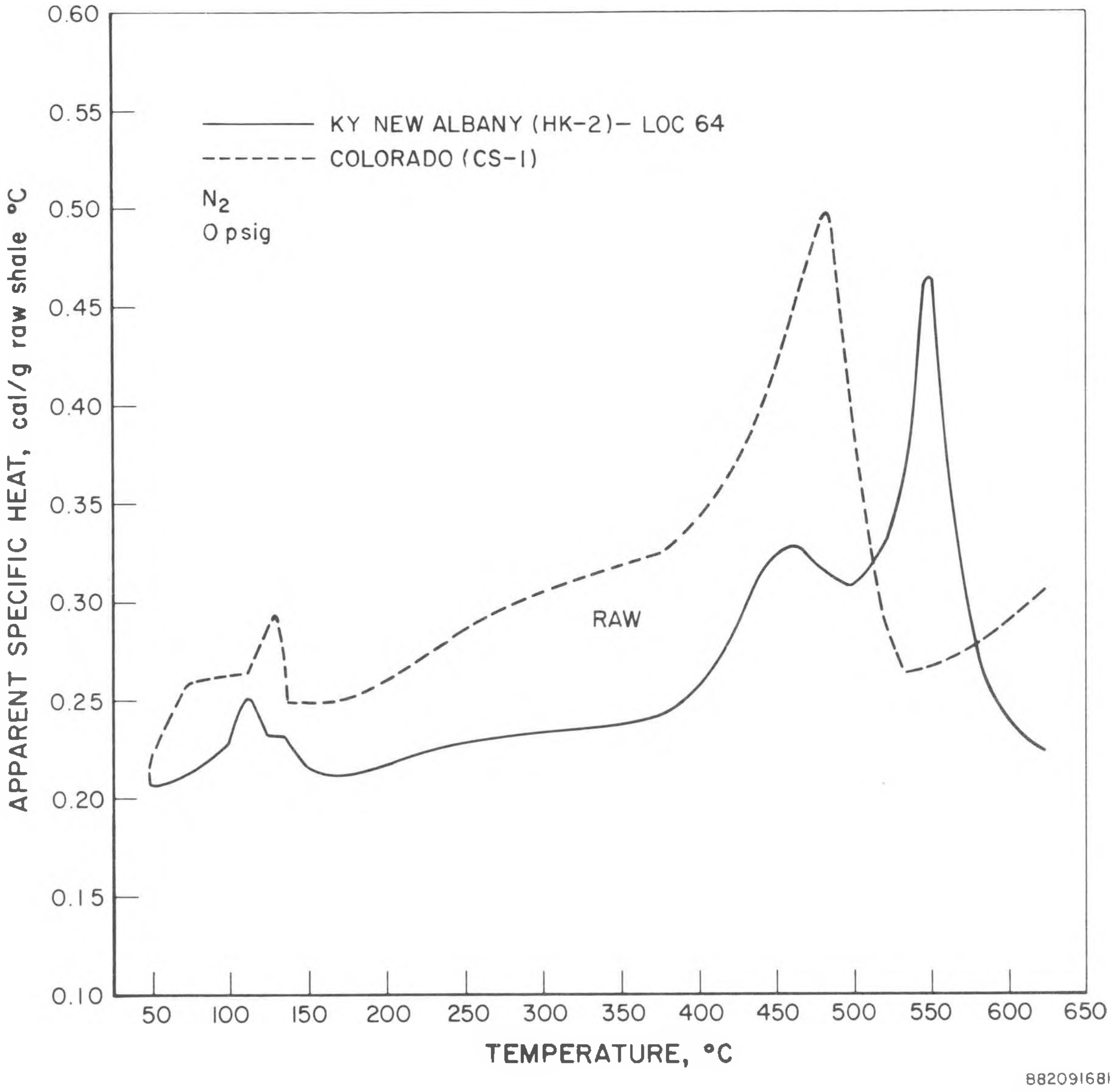

Figure 18. APPARENT SPECIFIC HEATS IN NITROGEN

Table 9. SULFUR CONTENTS OF SHALES

Shale	Sulfur, wt % Dry Basis
Kentucky New Albany (HK-2)	5.38
Colorado (CS-1)	0.86
Sunbury (SS-1)	2.69
Cleveland (CL-2)	2.41
Kentucky New Albany (NA-1A)	5.66
Gassaway (GA-2)	6.61
Gassaway (GA-3)	2.38
Lower Huron (HU-2)	4.41

Figure 18 shows the apparent specific heats of Kentucky New Albany (HK-2) and Colorado (CS-1) shales in 0 psig N_2. Both shales appear to have a lower apparent specific heat at 0 psig in N_2 than in H_2. In 0 psig N_2 both shales showed an endotherm between 400° and 525°C, and the Kentucky New Albany shale also still had a sharp peak near 540°C. The Colorado shale endothermic peak near 350°C observed in H_2 atmospheres was not observed in N_2.

Task 2. Bench-Scale Program

Subtask 2.1. Bench-Scale Reactor Tests

In this subtask tests were conducted to determine the effect of initial scale-up from laboratory- to bench-scale equipment. With the 4-inch-diameter electrically heated reactor, sufficient oil shale is processed that complete material and elemental (carbon, hydrogen, and oxygen) balances can be performed, and sufficient oil is produced so that its many properties may be determined and it can be used for further testing.

This quarter, two successful bench-scale runs using two Eastern shales were performed. Runs 81BSU-2 and 81BSU-4 were performed on a sample of the Dowelltown member and a sample of the Gassaway member of the Chattanooga shale, respectively. Run 81BSU-4 was voluntarily terminated after 4 hours of steady-state operation. The results of three runs (80BSU-15, 80BSU-16, and 80BSU-18) made during the previous quarter along with the results of the runs made during this quarter are reported here.

Approximate common operating conditions for the five runs were —

Shale Feed Rate, lb/h	83
Reactor Pressure, psig	500
Maximum Bed Temperature, °F	1420
Shale Residence Time, min	42
Feed Gas/Oil Shale Ratio, SCF/lb	7.4

Detailed summaries of the results for each of the runs are given in Tables 10 through 14.

A comparison of the oil yields and organic carbon conversions for all the Eastern shales studied thus far is given in Table 15. Detailed summaries of the results for runs made on the New Albany, Cleveland, and Sunbury shales were reported earlier. As all the runs listed below were made under similar conditons, a comparison of the test results for different shale types may be made.

Considering the oil yields on an absolute basis, the Sunbury shale has the highest yield: 0.076 lb/lb shale fed. The oil yield per pound of organic carbon in the feed shale is also given so that a comparison among different types of shales having different organic carbon content can be made. Based on the tabulated results, again the Sunbury had the highest yield: 0.56 lb/lb of organic carbon. The Michigan Antrim shale, despite its low organic carbon content (only 6.66%) also has a yield of 0.56 lb/lb of organic carbon fed.

Although Runs 79BSU-2, 80BSU-2, 80BSU-3, and 80BSU-11 were all made at a maximum temperature of 1200°F, the oil yields were not significantly different from similar tests performed at a maximum temperature of 1400°F.

The product oil properties of the shales tested in the five runs reported have had very similar physical properties, for example, viscosity and C/H ratio. The average viscosity and C/H ratio were 35.8 SSU and 8.06, respectively.

A comparison of the temperature profiles of the five runs (Figures 19 through 23) shows that they all had a fairly linear temperature gradient throughout the bed. The average maximum temperature reached was about 1420°F. Run 80BSU-16, which used Gassaway shale feed, had a nearly isothermal temperature profile which, when combined with the elevated temperature level, probably caused the higher gas yield for this run. Run 80BSU-16 was made with a maximum bed temperature of 1450°F — higher than the detailed results for the other four runs reported here. This higher maximum temperature probably resulted in a lower carbon selectivity to oil (higher selectivity to gas).

Table 10. BENCH-SCALE (80BSU-15) TESTS RESULTS — COUNTERCURRENT MOVING-BED OPERATION WITH KENTUCKY (Lower Huron) SHALE

Operating Conditions and Test Results

Oil Shale Location (State)	154 (Kentucky)
Oil Shale Particle Size (U.S. Sieve)	-6+10
Test Duration, hr	5.25
Steady-State Operating Period, hr	4.00
Bed Height, ft	9.0
Pressure, psig	513
Average Bed Temperature, °F	1153
Maximum Bed Temperature, °F	1432
Oil Shale Feed Rate, lb/hr	70.9
Oil Shale Space Velocity, lb/ft^3-hr	89
Oil Shale Mass Rate, lb/ft^2-hr	802
Feed Gas*/Shale Ratio, SCF/lb	7.0
Feed Gas* Superficial Velocity, ft/s	0.14
Product Gas Yield, SCF/lb Shale Fed†	8.0
Residue Shale Yield, lb/lb Shale Fed	
By Direct Weight Measurement	0.737
By Ash Balance Calculation	0.793
Liquid Hydrocarbon Yield, lb/lb Shale Fed	0.061
Water Yield, lb/lb Shale Fed	0.133
Material Balance, %**	106.6
Organic Carbon Distribution in Products, %**	
Gaseous Hydrocarbons	30.2
Liquid Hydrocarbons	44.1
Residue Shale	29.2

Feed and Product Compositions

	Oil Shale	
Sample	Feed	Residue
Lab No.	124283	124284
Moisture, Wt %	3.5	0.0
Ultimate Analysis, Wt % (Dry Basis)		
Organic Carbon	12.21	4.50
Mineral Carbon	1.69	0.08
Hydrogen	1.57	0.23
Nitrogen	0.58	0.18
Oxygen (by HTW)	3.07	0.65
Sulfur	0.80	0.75
Ash	75.45	95.14
Total	101.56	101.82

Shale Oil	
Lab No.	44598
C/H Weight Ratio	7.99
Sulfur, Wt %	1.32
Nitrogen, Wt %	1.66
Viscosity, SSU at 100°F	38.2
Specific Gravity, (60/60°F)	0.921
Distillation	
IBP	144
50%	544
EP	760
Pour Point, °F	-65

Gas Composition, Mol %†	Feed	Product†
Hydrogen Sulfide		0.0
Nitrogen & Argon		5.09
Carbon Monoxide		2.94
Carbon Dioxide		1.98
Hydrogen	100.00	80.28
Methane		7.19
Ethane +		2.10
Ethylene +		0.37
Benzene +		0.05
Total	100.00	100.00

* Hydrogen

** By ash balance calculations.

† Purge-free basis.

Table 11. BENCH-SCALE (80BSU-16) TEST RESULTS — COUNTERCURRENT MOVING-BED OPERATION WITH TENNESSEE (Chattanooga) SHALE

Operating Conditions and Test Results

Oil Shale Location (State)	180 (Tennessee)
Oil Shale Particle Size (U.S. Sieve)	-6+10
Test Duration, hr	8.00
Steady-State Operating Period, hr	6.00
Bed Height, ft	9.0
Pressure, psig	526
Average Bed Temperature, °F	1170
Maximum Bed Temperature, °F	1450
Oil Shale Feed Rate, lb/hr	89.9
Oil Shale Space Velocity, lb/ft^3-hr	113
Oil Shale Mass Rate, lb/ft^2-hr	1017
Feed Gas[*]/Shale Ratio, SCF/lb	5.56
Feed Gas[*] Superficial Velocity, ft/s	0.14
Product Gas Yield, SCF/lb Shale Fed[+]	5.21
Residue Shale Yield, lb/lb Shale Fed	
By Direct Weight Measurement	0.848
By Ash Balance Calculation	0.865
Liquid Hydrocarbon Yield, lb/lb Shale Fed	0.031
Water Yield, lb/lb Shale Fed	0.043
Material Balance, %[**]	102.1
Organic Carbon Distribution in Products, %[**]	
Gaseous Hydrocarbons	32.8
Liquid Hydrocarbons	27.1
Residue Shale	39.1

Feed and Product Compositions

Oil Shale		
Sample	Feed	Residue
Lab No.	124425	124546
Moisture, Wt %	0.9	0.0
Ultimate Analysis, Wt % (Dry Basis)		
Organic Carbon	10.05	4.50
Mineral Carbon	0.10	0.07
Hydrogen	1.06	0.16
Nitrogen	0.30	0.15
Oxygen	2.73	0.56
Sulfur	6.21	3.13
Ash	82.36	94.40
Total	102.81	102.97

Shale Oil	
Lab No.	44702
C/H Weight Ratio	7.92
Sulfur, Wt %	1.08
Nitrogen, Wt %	0.64
Viscosity, SSU at 100°F	31.7
Specific Gravity, (60/60°F)	0.890
Distillation	
IBP	172
50%	486
EP	749
Pour Point, °F	0

Gas Composition, Mol %[+]	Feed	Product[+]
Hydrogen Sulfide		4.09
Nitrogen & Argon		7.38
Carbon Monoxide		0.37
Carbon Dioxide		0.18
Hydrogen	100.00	73.78
Methane		11.03
Ethane +		2.78
Ethylene +		0.27
Benzene +		0.12
Total	100.00	100.00

[*] Hydrogen

[**] By ash balance calculations.

[+] Purge-free basis.

Table 12. BENCH-SCALE (80BSU-18) TEST RESULTS — COUNTERCURRENT MOVING-BED OPERATION WITH MICHIGAN (Antrim) SHALE

Operating Conditions and Test Results

Oil Shale Location (State)	Antrim (Michigan)
Oil Shale Particle Size (U.S. Sieve)	-6+20
Test Duration, hr	3.80
Steady-State Operating Period, hr	2.00
Bed Height, ft	9.0
Pressure, psig	472
Average Bed Temperature, °F	1108
Maximum Bed Temperature, °F	1410
Oil Shale Feed Rate, lb/hr	88.4
Oil Shale Space Velocity, lb/ft^3-hr	111
Oil Shale Mass Rate, lb/ft^2-hr	1000
Feed Gas*/Shale Ratio, SCF/lb	7.53
Feed Gas* Superficial Velocity, ft/s	0.23
Product Gas Yield, SCF/lb Shale Fed+	6.28
Residue Shale Yield, lb/lb Shale Fed	
By Direct Weight Measurement	0.783
By Ash Balance Calculation	0.871
Liquid Hydrocarbon Yield, lb/lb Shale Fed	0.037
Water Yield, lb/lb Shale Fed	0.103
Material Balance, %**	104.5
Organic Carbon Distribution in Products, %	
Gaseous Hydrocarbons	31.6
Liquid Hydrocarbons	49.0
Residue Shale**	23.5

Feed and Product Compositions

Oil Shale		
Sample	Feed	Residue
Lab No.	124780	124781
Moisture, Wt %	1.8	0.0
Ultimate Analysis, Wt % (Dry Basis)		
Organic Carbon	6.66	1.76
Mineral Carbon	0.29	0.04
Hydrogen	1.00	0.18
Nitrogen	0.18	0.00
Oxygen	3.90	1.02
Sulfur	2.59	1.75
Ash	86.65	97.67
Total	101.27	102.42

Shale Oil	
Lab No.	44911
C/H Weight Ratio	7.92
Sulfur, Wt %	1.32
Nitrogen, Wt %	1.21
Viscosity, SSU at 100°F	36.1
Specific Gravity, (60/60°F)	0.916
Distillation	
IBP	148
50%	520
EP	760
Pour Point, °F	0

Gas Composition, Mol %	Feed	Product
Hydrogen Sulfide		0.42
Nitrogen & Argon		4.98
Carbon Monoxide		0.74
Carbon Dioxide		0.81
Hydrogen	100.00	87.02
Methane		3.83
Ethane +		1.79
Ethylene +		0.33
Benzene		0.08
Total	100.00	100.00

* Hydrogen

\+ Purge Free

**By ash balance calculations.

Table 13. BENCH-SCALE (81BSU-2) TEST RESULTS — COUNTERCURRENT MOVING-BED OPERATION WITH KENTUCKY (Dowelltown) SHALE

Operating Conditions and Test Results

Oil Shale Location (State)	148 (Tennessee)
Oil Shale Particle Size (U.S. Sieve)	-6+20
Test Duration, hr	4.25
Steady-State Operating Period, hr	3.00
Bed Height, ft	9.0
Pressure, psig	486
Average Bed Temperature, °F	1152
Maximum Bed Temperature, °F	1405
Oil Shale Feed Rate, lb/hr	84.9
Oil Shale Space Velocity, lb/ft^3-hr	107
Oil Shale Mass Rate, lb/ft^2-hr	960
Feed Gas*/Shale Ratio, SCF/lb	8.13
Feed Gas* Superficial Velocity, ft/s	0.23
Product Gas Yield, SCF/lb Shale Fed+	7.56
Residue Shale Yield, lb/lb Shale Fed	
By Direct Weight Measurement	0.754
By Ash Balance Calculation	0.816
Liquid Hydrocarbon Yield, lb/lb Shale Fed	0.059
Water Yield, lb/lb Shale Fed	0.059
Material Balance, %**	100.8
Organic Carbon Distribution in Products, %	
Gaseous Hydrocarbons	28.2
Liquid Hydrocarbons	37.5
Residue Shale**	29.6

Feed and Product Compositions

Oil Shale		
Sample	Feed	Residue
Lab No.	125120	124928, 124929
Moisture, Wt %	1.0	0.0
Ultimate Analysis, Wt % (Dry Basis)		
Organic Carbon	13.60	4.88
Mineral Carbon	0.28	0.03
Hydrogen	1.45	0.18
Nitrogen	0.30	0.12
Oxygen	3.67	0.57
Sulfur	6.19	3.30
Ash	77.38	93.81
Total	102.87	102.89

Shale Oil	
Lab No.	45036
C/H Weight Ratio	8.24
Sulfur, Wt %	2.22
Nitrogen, Wt %	1.12
Viscosity, SSU at 100°F	37.9
Specific Gravity, (60/60°F)	0.920
Distillation	
IBP	140
50%	544
EP	760
Pour Point, °F	-70

Gas Composition, Mol %	Feed	Product+
Hydrogen Sulfide		0.85
Nitrogen & Argon		4.22
Carbon Monoxide		0.69
Carbon Dioxide		0.34
Hydrogen	100.00	83.19
Methane		7.89
Ethane +		2.39
Ethylene +		0.35
Benzene+		0.08
Total	100.00	100.00

* Hydrogen
\+ Purge Free
** By Ash Balance Calculations

Table 14. BENCH-SCALE (81BSU-4) TEST RESULTS — COUNTERCURRENT MOVING-BED OPERATION WITH TENNESSEE (Chattanooga) SHALE

Operating Conditions and Test Results

Oil Shale Location (State)	Gassaway (Tennessee)
Oil Shale Particle Size (U.S. Sieve)	-6+20
Test Duration, hr	5.00
Steady-State Operating Period, hr	4.00
Bed Height, ft	9.0
Pressure, psig	505
Average Bed Temperature, °F	1130
Maximum Bed Temperature, °F	1415
Oil Shale Feed Rate. lb/hr	81.6
Oil Shale Space Velocity, lb/ft^3-hr	103
Oil Shale Mass Rate, lb/ft^2-hr	923
Feed Gas*/Shale Ratio, SCF/lb	8.68
Feed Gas* Superficial Velocity, ft/s	0.23
Product Gas Yield, SCF/lb Shale Fed†	7.55
Residue Shale Yield, lb/lb Shale Fed	
By Direct Weight Measurement	0.804
By Ash Balance Calculation	0.820
Liquid Hydrocarbon Yield, lb/lb Shale Fed	0.062
Water Yield, lb/lb Shale Fed	0.063
Material Balance, %**	101.2
Organic Carbon Distribution in Products, %	
Gaseous Hydrocarbons	28.3
Liquid Hydrocarbons	41.0
Residue Shale**	33.1

Feed and Product Compositions

Oil Shale

Sample	Feed	Residue
Lab No.	125068	125027,125028
Mositure, Wt %	1.5	0.0
Ultimate Analysis, Wt % (Dry Basis)	13.34	5.30
Organic Carbon	13.34	5.30
Mineral Carbon	0.16	0.11
Hydrogen	1.42	0.27
Nitrogen	0.41	0.12
Oxygen	3.55	1.07
Sulfur	6.46	3.39
Ash	77.53	93.10
Total	102.87	103.36

Shale Oil

Lab No.	45153
C/H Weight Ratio	8.24
Sulfur, Wt %	1.12
Nitrogen, Wt %	1.01
Viscoisty, SSU at 100°F	35.0
Specific Gravity, (60/60°F)	0.916
Distillation	
IBP	136
50%	526
EP	760
Pour Point, °F	-75

Gas Composition, Mol %	Feed	Product
Hydrogen Sulfide		1.02
Nitorgen & Argon		3.11
Carbon Monoxide		0.60
Carbon Dioxide		0.42
Hydrogen	100.00	85.08
Methane		6.76
Ethane †		2.47
Ethylene †		0.43
Benzene †		0.11
Total	100.00	100.00

* Hydrogen

† Purge Free

** By Ash Balance Calculations

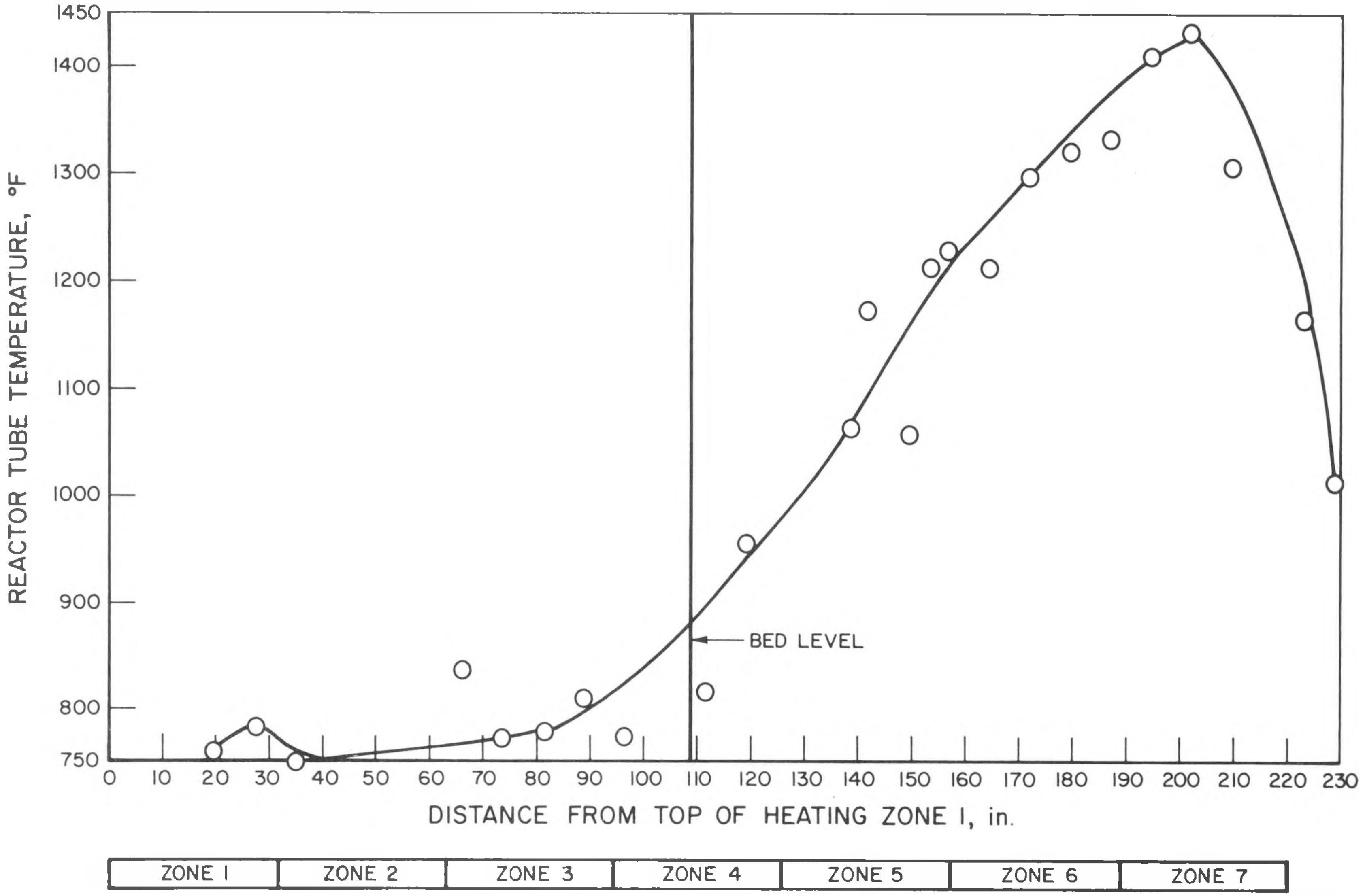

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Figure 19. TEMPERATURE PROFILE FOR RUN 80BSU-15

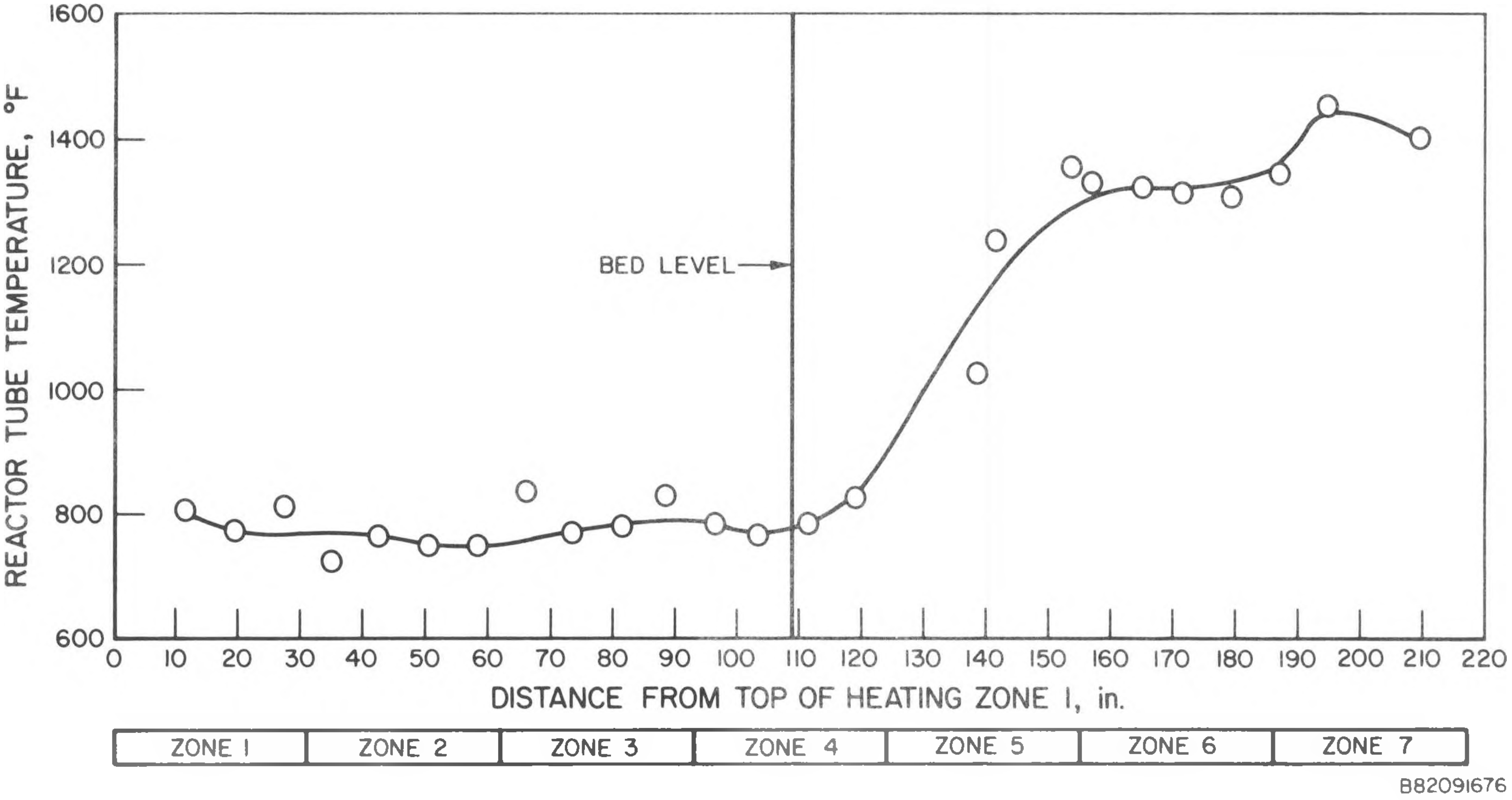

Figure 20. TEMPERATURE PROFILE FOR RUN 80BSU-16

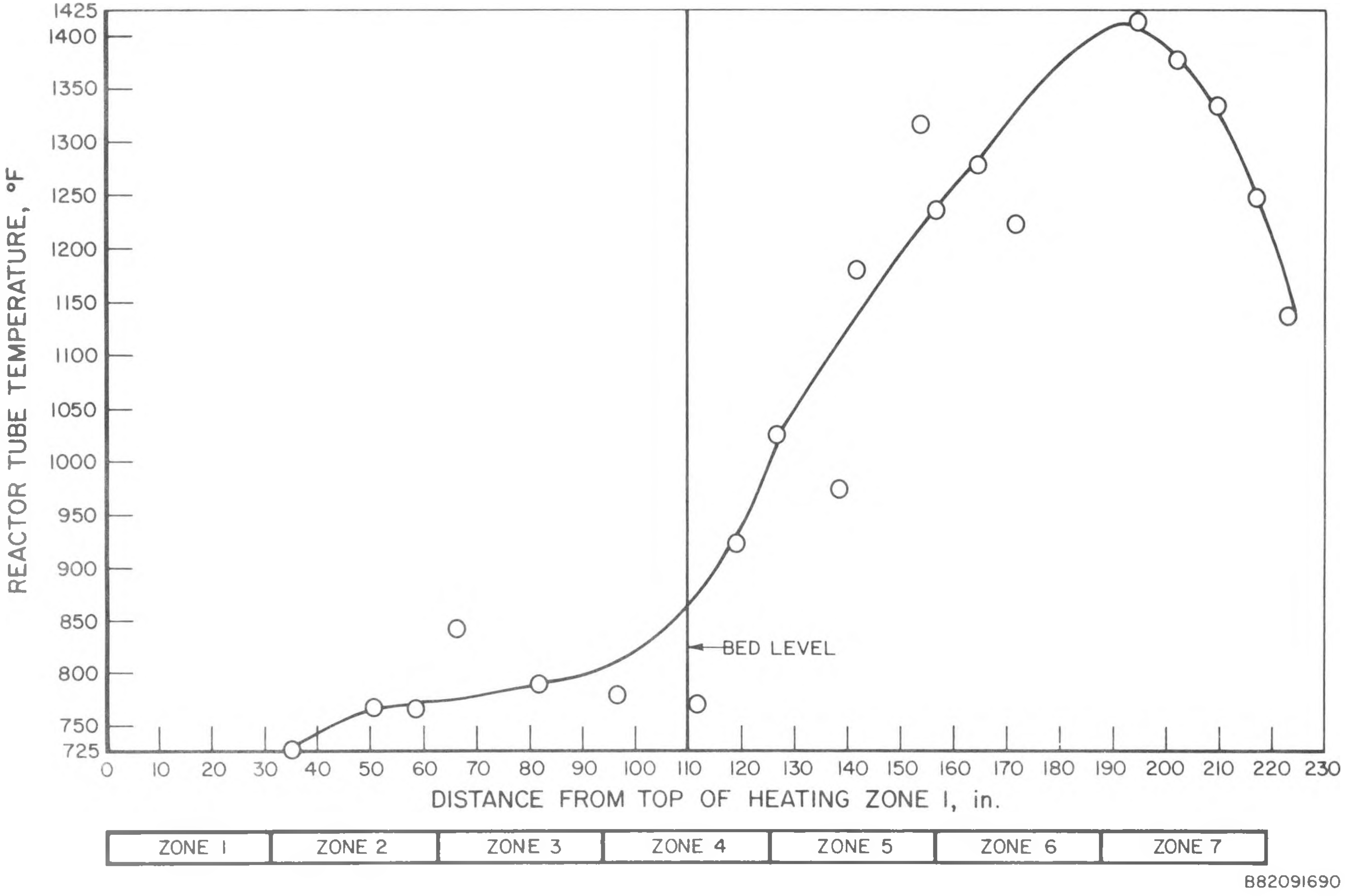

Figure 21. TEMPERATURE PROFILE FOR RUN 80BSU-18

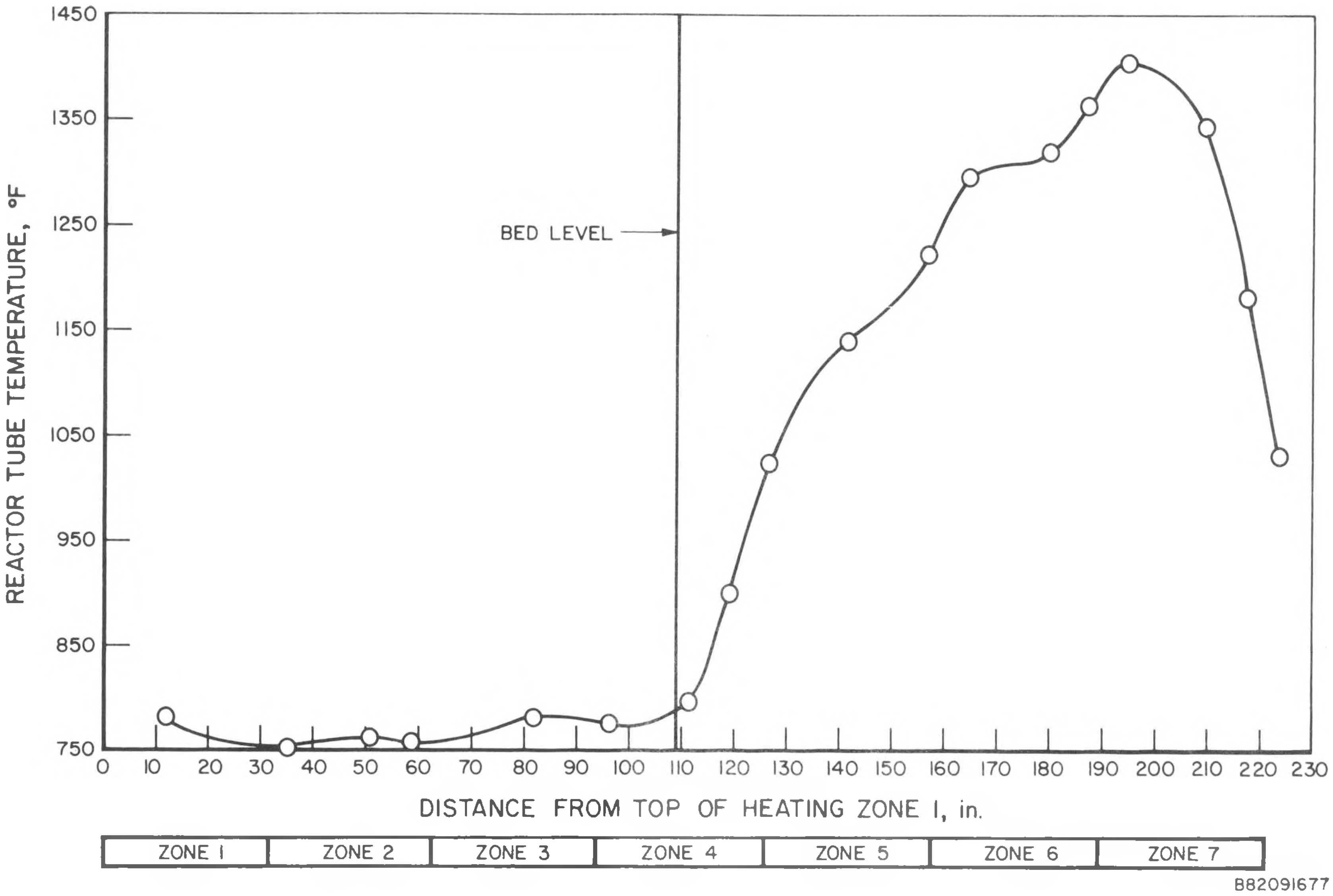

Figure 22. TEMPERATURE PROFILE FOR RUN 81BSU-2

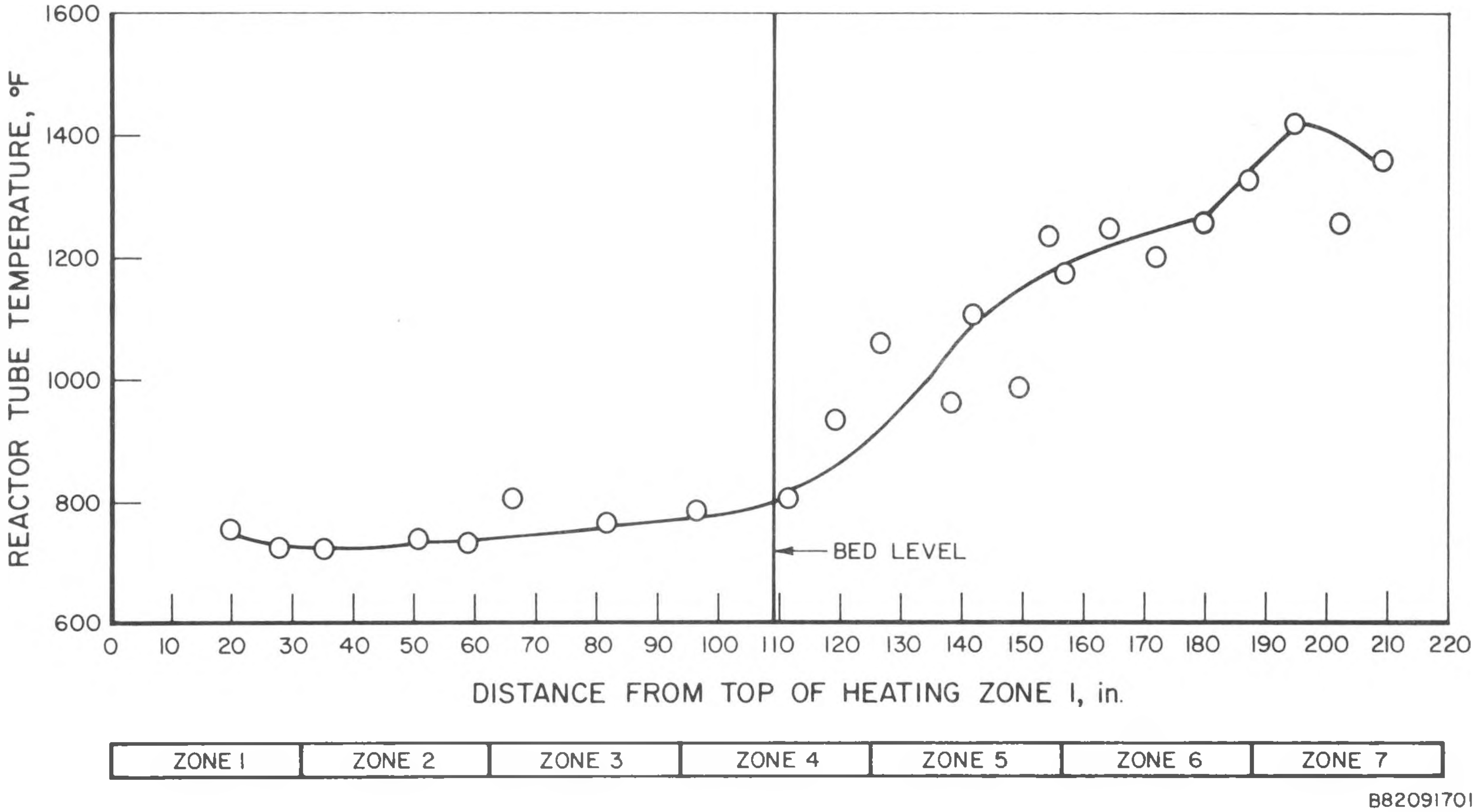

Figure 23. TEMPERATURE PROFILE FOR RUN 81BSU-4

Table 15. COMPARISON OF OIL YIELDS FROM DIFFERENT SHALES

Run	Shale Identification	Organic Carbon Conversion	Oil Yield lb/lb Shale	lb/lb of Feed Shale Org. C
79BSU-2	New Albany	70.9	0.061	0.46
80BSU-2	Cleveland	56.1	0.042	0.42
80BSU-3	Cleveland	49.0	0.017	0.17
80BSU-11	Sunbury	72.6	0.076	0.56
80BSU-15	Lower Huron	70.8	0.061	0.50
80BSU-16	Gassaway Loc. 80	60.9	0.031	0.31
80BSU-18	Antrim	76.5	0.037	0.56
81BSU-2	Dowelltown	70.4	0.059	0.43
81BSU-4	Gassaway Loc. 148	66.9	0.062	0.46

Subtask 2.2. Hydrogen Production Studies

In this subtask tests were conducted to determine the technical feasibility of producing at least a portion of the hydrogen requirement of the HYTORT process using either the spent shale, which contains residual organic carbon from the process, or the raw shale fines from the crushing plant. Steam and oxygen were used to supply the hydrogen produced and the heat duty of the process, respectively. During this quarter, one raw shale fines gasification test was conducted.

Equipment Description

The same bench-scale equipment was used for the oil shale fines steam-oxygen gasification test as was used in earlier spent shale gasification tests. The 6-inch-ID reactor was modified, however, for fluidized-bed operation and for the feeding of oil shale fines to the bottom of the reactor. A flow diagram of the bench-scale gasification unit is shown in Figure 24. Figure 25 is a schematic sectional drawing showing the reactor, the reactor discharge housing, the bottom of the feed hopper, the feed hopper extension, and the feed screw housing. The oil shale fines are injected horizontally into a tee housing below the reactor shell and then transferred

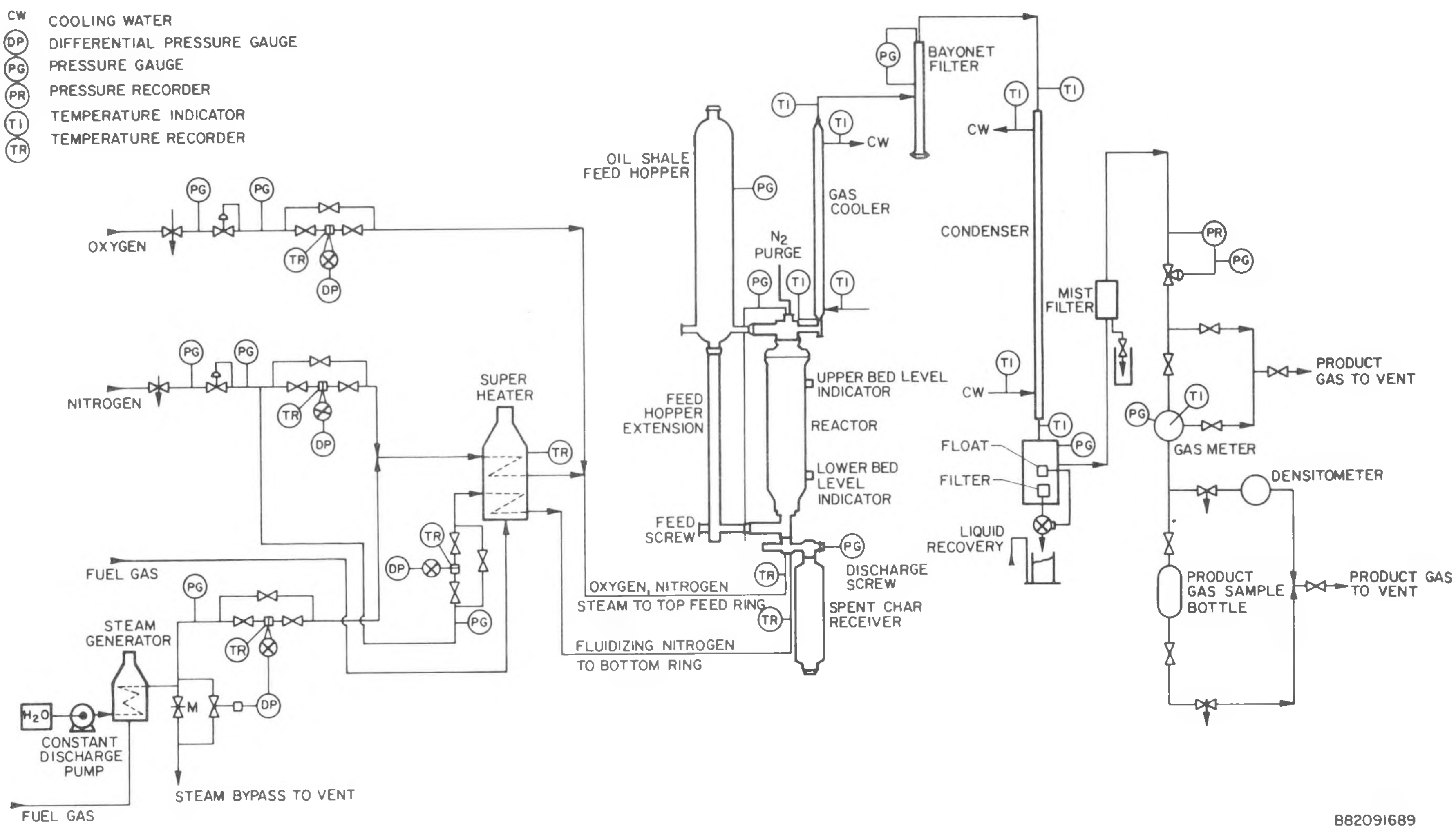

Figure 24. BENCH-SCALE STEAM-OXYGEN GASIFICATION UNIT

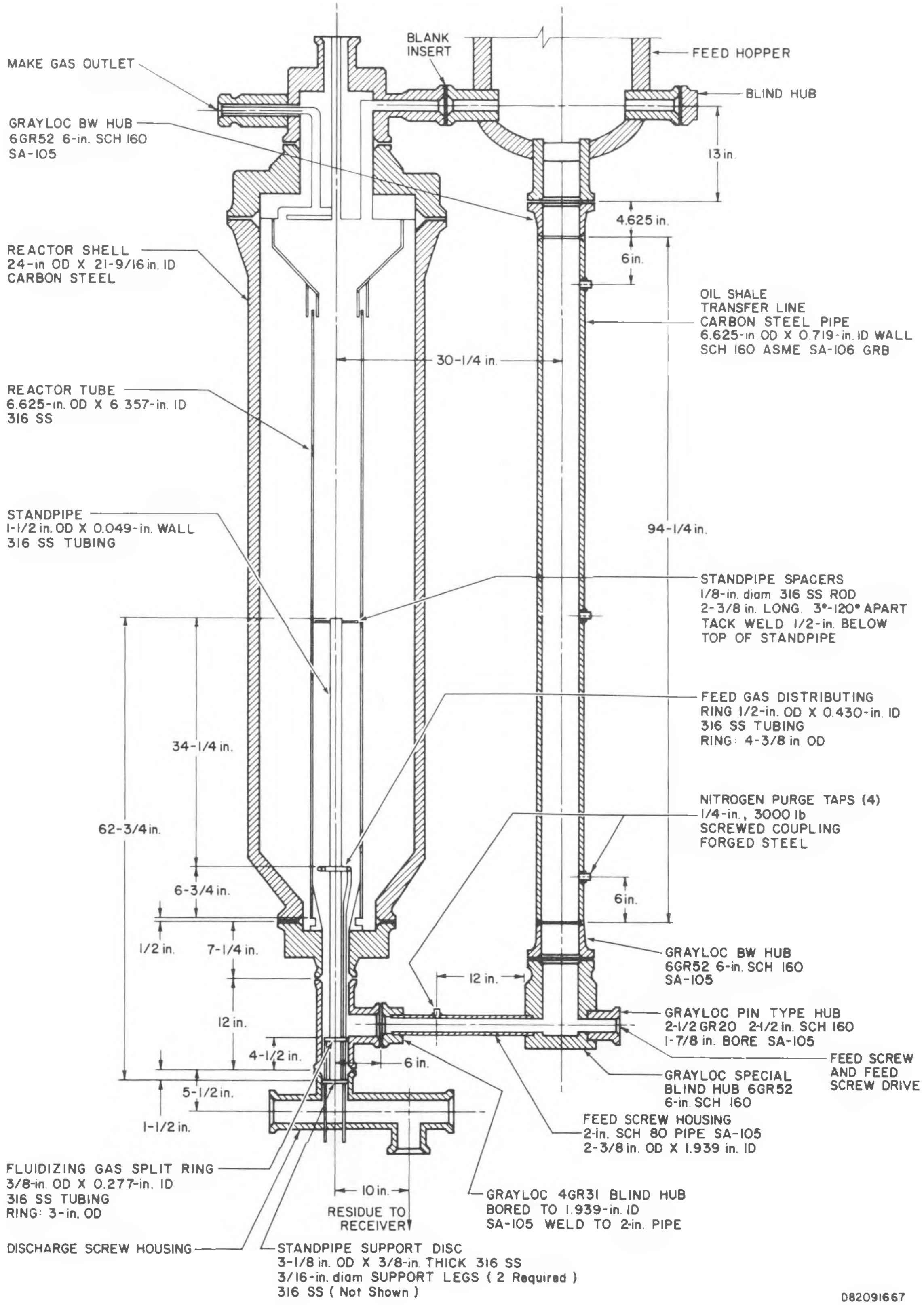

Figure 25. SCHEMATIC DIAGRAM OF MODIFIED 6-INCH-DIAMETER STEAM-OXYGEN GASIFIER

vertically with fluidizing nitrogen into the gasification zone. The oil shale fines are gasified with steam and oxygen in an annulus formed by the inside walls of the 6.3-inch-ID reactor tube and a 1.5-inch OD standpipe. The standpipe serves to remove gasified oil shale from the reactor and transfer them by gravity to the discharge screw. It also extends 34 inches above the steam-oxygen feed gas distributing ring and controls the height of the fluidized bed. Product gases are withdrawn at the top of the reactor. Reactor tube and inside bed thermocouple locations are shown in Figure 26. Details of the ring distributor for introducing fluidized nitrogen to lift the oil shale fines into the gasification zone are given in Figure 27. The steam-oxygen feed gas distributing ring detail are shown in Figure 28.

Test Conditions and Results

Test conditions for the steam-oxygen gasification of oil shale fines were based on a process concept in which the fines would be fed to the bottom of the reactor where they would be contacted with steam and oxygen, fluidized, and gasified in cocurrent gas-solids flow. Feeding of the oil shale fines to the bottom of the reactor allows for the rapid gasification of the hydrocarbon vapors with oxygen after they are evolved in the preheat zone, thus maximizing the production of gaseous products. At the same time the exothermic heat release by the carbon-oxygen reactions provides the heat requirements for the endothermic hydrocarbon-steam reaction. With fluidized-bed operation there is a relatively high rate of heat transfer between the oil shale particles and the fluidizing gas, and the heat distribution up through the fluidized bed should be adequate to prevent sintering of the oil shale particles.

Test conditions for Run FS-1 were selected so as to achieve a relatively high rate of carbon gasification at comparatively moderate gasification conditions. Conditions desired were reactor pressure, 350 psig; gasification temperature, 1600°F; oil shale feed rate, 100 lb/h; oxygen feed rate, 300 SCF/h (0.65 moles/mole C); steam feed rate, 35 lb/h (1.60 moles/mole C); and fluidization velocity, 1.0 ft/s. Test conditions and results for Run FS-1 are given in Table 16.

The Eastern oil shale used in Run FS-1 was a Kentucky New Albany oil shale in the -40+100 U.S.S. sieve size range. Fines smaller than 100 U.S.S. sieve size) were screened out to minimize elutriation. Chemical and screen analyses of the oil shale feed are presented in Table 17.

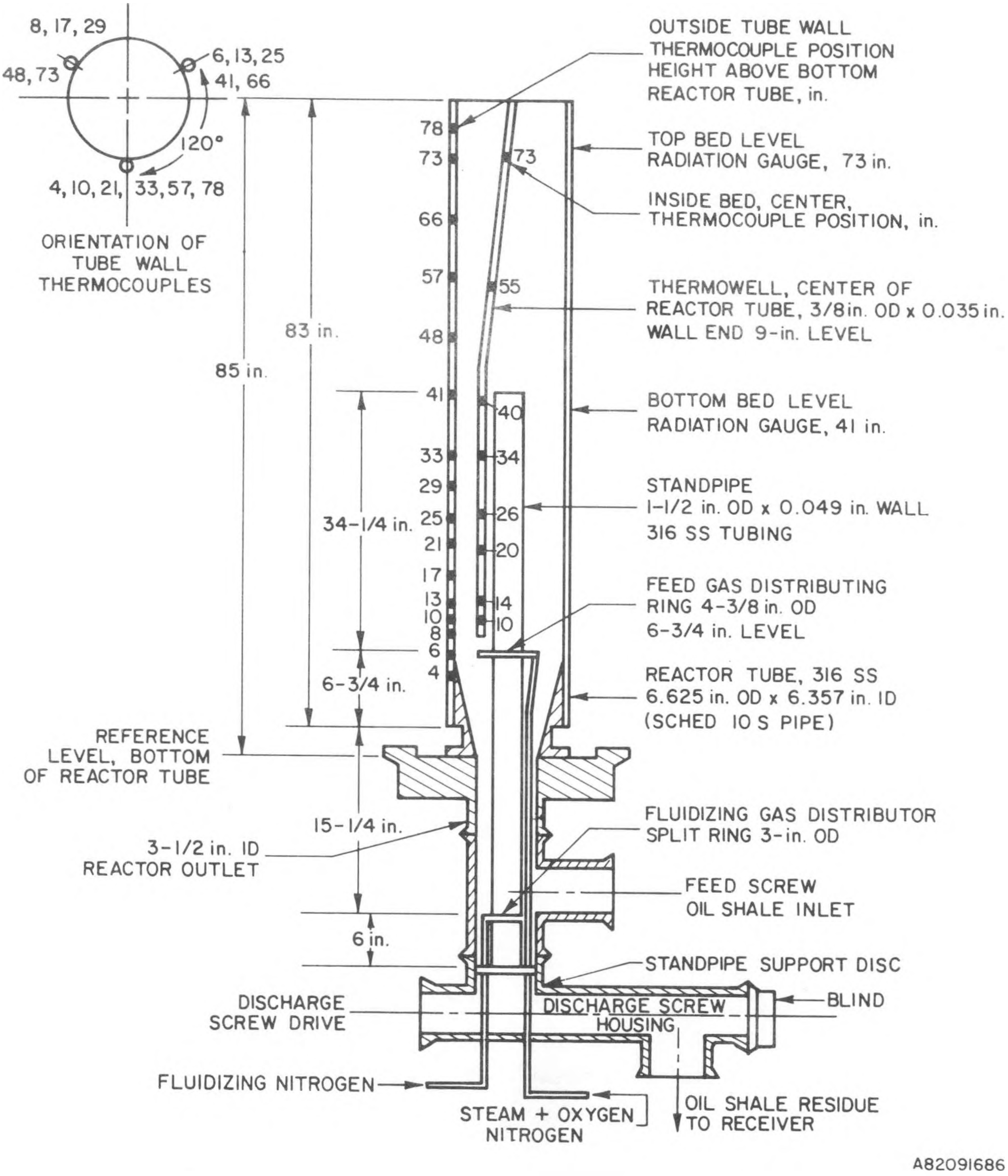

Figure 26. THERMOCOUPLE, GAS DISTRIBUTOR, STANDPIPE, AND RADIATION GAUGE LOCATIONS

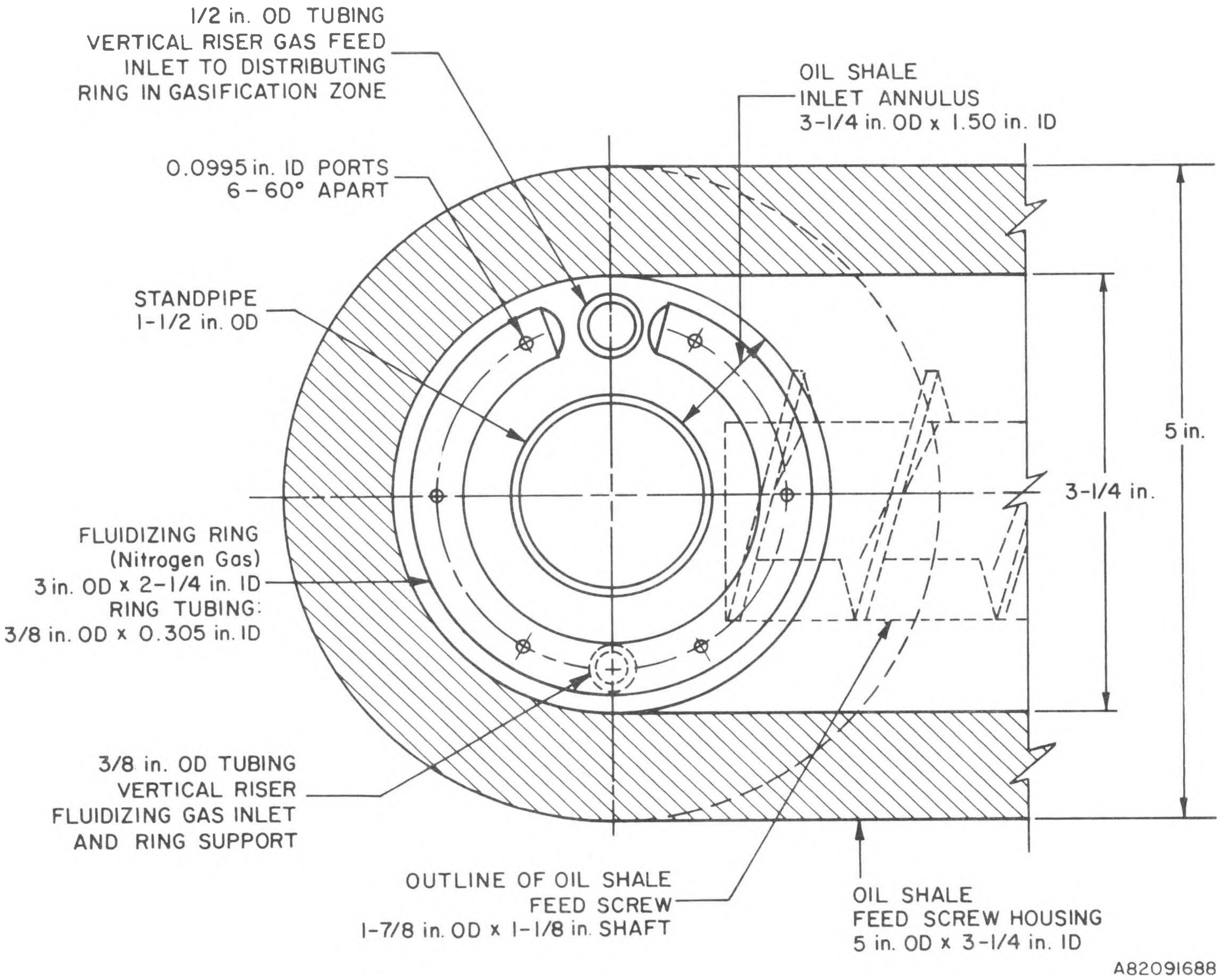

Figure 27. FLUIDIZING NITROGEN FEED RING

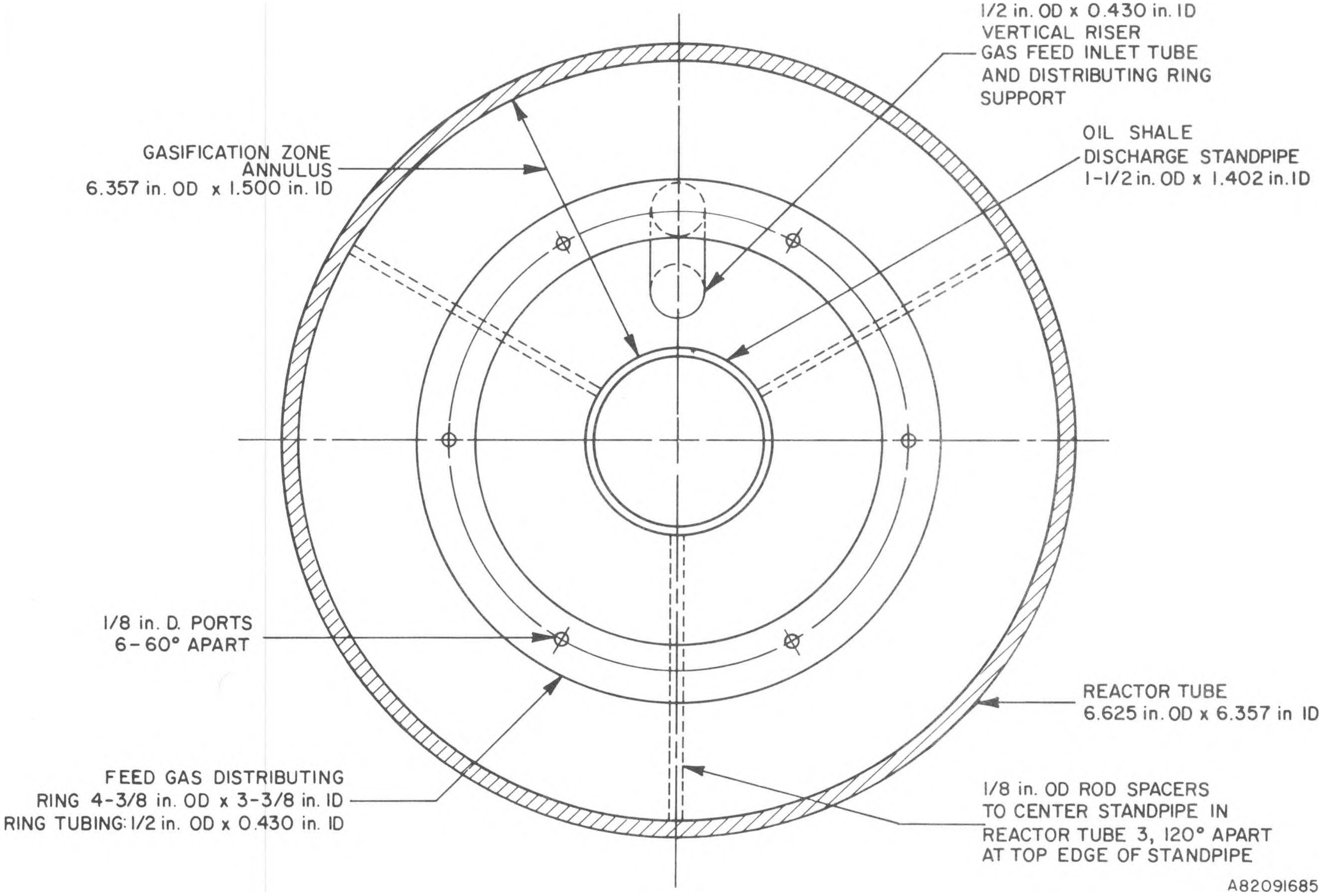

Figure 28. STEAM-OXYGEN FEED GAS DISTRIBUTION

Table 16. OPERATING CONDITIONS AND RESULTS OF OIL SHALE FINES STEAM-OXYGEN GASIFICATION IN A FLUIDIZED BED

Run No.	FS-1
Feed Material	New Albany Oil Shale Fines
Feed Source	Kentucky
Sieve Size, U.S.S.	-40+100
Duration of Test, h	2-1/2
Steady-State Operating Period, min	60
Operating Conditions	
Standpipe and Oil Shale Bed Height, in.	34
Standpipe Outside Diameter, in.	1-1/2
Reactor Pressure, psia	390
Reactor Temperature, °F	
Tube Wall, in. from tube bottom	
4	1190
6	840
8	425
10	975
13	1135
17	--
21	875
25	895
29	--
33	1070
41	--
48	--
57	1270
66	920
73	--
78	845
Internal, in. from tube bottom	
10	1505
14	1465
20	1425
26	--
34	1340
40	1350
55	1316
73	1290
Average Reaction Zone Temperature	1415
Feed Gas Distributor,[a] no. of feed ports	6
Fludizing Gas Distributor,[b] no. of feed ports	6
Oil Shale Feed Rate (Dry),[c] lb/h	122.2
Nitrogen Rate (Feed Gas), SCF/h	1536
Oxygen Rate, SCF/h	316
Oxygen Concentration at Feed Gas Ports, mole %	16.7
Oxygen/Carbon Ratio, mol/mol	0.587
Steam Rate, lb/h	36.4
Steam/Carbon Ratio, mol/mol	1.483
Nitrogen Rate (Fluidizing Gas Distributor),[d] SCF/h	1250
Purge Nitrogen (Feed Screw), SCF/h	259
Purge Nitrogen (Feed Hopper), SCF/h	218
Purge Nitrogen (Shell), SCF/h	290
Oil Shale Fluidized-Bed Density, lb/ft^3	30.3
Oil Shale Residence Time,[e] min	10.6
Superficial Fluidizing Velocity, ft/s	
Reactor Bed[f]	0.791
Oil Shale Feed Inlet[g]	0.895

Table 16, Cont. OPERATING CONDITIONS AND RESULTS OF OIL SHALE FINES STEAM-OXYGEN GASIFICATION IN A FLUIDIZED BED

Operating Results	
Product Gas Rate (Shell Purge Nitrogen-Free)[h] SCF/h	3594
Product Gas Yield, SCF/lb dry oil shale (Nitrogen-Free)	2.70
Hydrogen Yield, SCF/lb	0.059
Carbon Monoxide Yield, SCF/lb	0.00
Carbon Dioxide Yield, SCF/lb	2.53
Hydrocarbon Gas Yield, SCF/lb	0.044
Residue Oil Shale Rate,[i] lb/h	96.32
Bayonet Filter Fines Rate, lb/h	5.81
Sintered Ash, Total, lb	0.32
Condensed Liquid Rate,[j] lb/h	
Water	45.0
Oils	0.181
Net MAF Oil Shale Gasified, %	
Based on Product Gas Analysis	61.6
Based on Oil Shale Feed and Residue Analyses	67.9
Carbon Gasified, %	
Based on Product Gas Analysis	58.6
Based on Oil Shale Feed and Residue Analyses	68.4
Steam Reacted, % of steam fed	nil
Steam Concentration in Product Gas, mole %	20.8
Material Balances, %	
Overall	99.2
Carbon	90.6
Hydrogen	85.2
Oxygen	100.6
Product Gas Composition (Shell Purge Nitrogen-Free), mole %	
Nitrogen	90.8
Carbon Monoxide	0.0
Carbon Dioxide	8.6
Oxygen	0.0
Hydrogen	0.2
Argon	0.01
Methane	0.02
Ethane	0.03
Hydrogen Sulfide	0.06
Sulfur Dioxide	0.20
Total	99.92
Heating Value[k] Btu/SCF	2.4
Specific Gravity, (Air = 1.00)	1.020

[a] Consists of a 4-3/8-inch-diameter torus made of 1/2-inch-diameter tubing. Each port diameter is 1/8 inch. Located 6-3/4 inches above the reactor tube bottom.

[b] Consists of a 3-inch-diameter torus made of 3/8-inch-diameter tubing. Each port diameter is 0.0995 inch. Located 22 inches below the feed gas distributor.

[c] Operating conditions and results are based on weight of dry feed.

[d] To convey oil shale fines from the feed screw discharge up into the reactor bottom.

[e] (Fluidized-bed density)(oil shale bed volume)/(residue oil shale plus bayonet filter fines rate from reactor).

[f] Based on feed gas flow rates: (CF/s at reactor temperature and pressure)/(reactor annulus cross-sectional area).

[g] Based on fluidizing nitrogen plus feed screw and feed hopper extension nitrogen.

[h] Dry gas volume in SCF at 60°F, 30-inch Hg pressure.

[i] By ash balance.

[j] Based on weight of liquid recovered.

[k] Gross: Gas saturated at 60°F, 30-inch Hg pressure.

Table 17. CHEMICAL AND SCREEN ANALYSES OF NEW ALBANY OIL SHALE FEED AND GASIFIED RESIDUES

Run No.	FS-1		
Sample	Feed	Residue	Filter Fines
Proximate Analysis, wt %			
Moisture	2.8	0.1	1.8
High-Temperature Water	4.6	1.8	2.1
Ash	73.4	90.5	85.0
Ultimate Analysis (dry), wt %			
Ash	75.53	90.59	86.58
Carbon (Total)	14.15	5.43	4.56
Organic Carbon	13.98	5.29	4.49
Mineral Carbon	0.17	0.14	0.07
Hydrogen	1.58	0.29	0.33
Sulfur	5.43	3.01	4.25
Nitrogen	0.50	0.21	0.21
Carbon Dixoide	0.63	0.51	0.25
Oxygen (by HTW*)	4.20	1.56	1.90
Total	101.85	101.46	98.01
Screen Analysis, U.S.S. sieve size, wt %			
20	0.0	0.2	6.1
40	0.8	10.0	0.4
60	11.4	20.8	0.4
80	19.1	18.8	0.2
100	16.5	13.6	0.6
120	9.1	8.0	0.4
140	7.9	6.8	0.2
170	4.8	4.8	0.4
200	4.5	4.1	0.4
325	19.3	8.6	25.7
-325	6.6	4.3	65.2
Total	100.0	100.0	100.0
Bulk Density, lb/ft^3	76.4	69.9	47.3
Helium Density, g/cc	2.29	2.61	2.65

* High-temperature water.

The total duration of Run FS-1 was 2-1/2 hours, with nearly 1 hour of steady-state operation during which time the reaction zone temperatures were in the range of 1400° to 1600°F. The test was terminated when all the oil shale in the feed hopper had been fed (342 lb). Actual operating conditions for the test were as follows: a reactor pressure of 390 psia, a maximum average reactor bed temperature of 1505°F, an oil shale feed rate of 122 lb/h during the steady-state part of the test, and a fluidization velocity of 0.79 ft/s. Oxygen was fed at a rate of 316 SCF/h, resulting in an oxygen-to-carbon ratio of 0.59 mole/mole. The oxygen concentration at the feed gas inlet ports was 16.7%. The steam feed rate was 36.4 lb/h giving a steam-to-carbon ratio of 1.48 moles/mole.

We experienced difficulty in getting the oil shale to flow uniformly out of the feed hopper, which caused the reactor temperatures to cycle. A peak temperature of 1840°F was reached at one time for a period of about 30 seconds. The average temperature profile during the steady-state portion of the test indicated that the highest temperature was at the bottom of the fluidized bed (1505°F), with a decrease in temperature upward through the bed to 1350°F at the top of the bed.

After shutdown of the test a small amount of sintered ash was found at the bottom of the fluidized oil shale bed just above the steam-oxygen feed ring. The small amount of ash sinter formed indicates it was probably formed at the shutdown of the test when the oil shale feed was turned off and there was still oxygen present in the gas at the bottom of the reactor.

In Run FS-1, 58.6% of the total carbon in the oil shale was converted to gaseous products, based on the product gas analysis and the product gas rate. Total carbon conversion (based on the carbon in the oil shale feed and in the residue) was 68.4%. There was no net decomposition of the steam fed to the reactor. Condensed steam recovered as water from the product gas amounted to 45 lb/h, compared with 36.4 lb/h of steam fed to the reactor. This indicates that a significant portion of the hydrogen in the oil shale reacted with feed oxygen to form water.

The product gas analysis (Table 16) shows that the major gaseous component, other than the nitrogen used for fluidization, is carbon dioxide. Other components, in smaller concentrations are hydrogen, methane, ethane, hydrogen sulfide, and sulfur dioxide. No carbon monoxide was present in the product gas.

An analysis of the condensed liquids recovered in Run FS-1 is presented in Table 18. The oil produced was only 0.4% of the water-oil emulsion. Carbon in the oil represents 0.85% of the carbon in the oil shale fed.

The results of this fluidized bed gasification test do not indicate that it is feasible to generate hydrogen by steam-oxygen gasification of raw shale fines in a single-stage fluidized-bed reactor. Because of the large amounts of oxygen required to supply the sensible heat for raw shale preheating, the reaction products are primarily carbon dioxide and water. However, by use of staged fluid beds, for example, for raw shale preheat, for gasification and for spent shale sensible heat recovery it should be possible to generate a significant fraction of the HYTORT process hydrogen requirement. Testing of this approach is beyond the scope of this study, however.

Subtask 2.3. Shale Oil Upgrading

In this subtask the hydrogen consumption and yield distributions were determined for hydrotreating the raw whole shale oils produced in the other subtasks (PDU and bench-scale units). Although it is possible to reduce the nitrogen level to very low values, 500 to 1000 ppm nitrogen was the level used as a target value.

Two upgrading runs were made during this quarter with Tennessee shale oil produced in Run 80BSU-16. The new (1-1/2 inch, Schedule 160 pipe) hydrotreating reactor was used, and 1830 cc of Shell 324 Ni/Mo catalyst which was used during previous hydrotreating tests occupied the 7-foot-long heated bed.

A summary of the two Tennessee shale oil upgrading tests is presented in Table 19. A comparison between the raw and hydrotreated product shale oil is presented in Table 20. As in our previous tests, the sulfur removal is above 95%. However, the nitrogen removal was 100% with the Chattanooga shale oil. The properties of the product oil were unusual for the shale oil in that the API gravity was very high (43.7°) and 95% of the oil boiled below 605°F.

The hydrogen consumption, calculated from the feed and product oil analyses, can be compared for the four shale oils used in these tests. For the Eastern-shale-derived oils (Kentucky Sunbury, Kentucky New Albany, and Tennessee Chattanooga), the hydrogen consumption ranged from 1650 to 1770 SCF/bbl. For the only Western-shale-derived oil (Colorado), the

Table 18. ANALYSIS OF CONDENSED LIQUIDS IN STEAM-OXYGEN GASIFICATION OF NEW ALBANY OIL SHALE FINES IN RUN FS-1

Phase Separation of Condensed Liquids	
Water Recovered, wt %	99.5
Oil Recovered, wt %	0.4
Solids in Liquid Recovered, wt %	0.1
Total Condensed Liquids, lb/h	45.181
Water; lb/h	45.0
Oil, lb/h	0.181
Oil Analysis (dry basis), wt %	
Carbon	81.56
Hydrogen	9.84
Ash	0.00
Total	91.40
Carbon/Hydrogen Weight Ratio	8.29
Specific Gravity, 60/60 F	0.978

Table 19. TENNESSEE SHALE OIL UPGRADING TESTS
(Feed Oil: Nitrogen - 0.64 wt %, Sulfur - 1.08 wt %)
(Catalyst: 1830 cc, Shell 324 Ni/Mo)

Run No.	HTT-1	HTT-2
Duration, h	2.5	2.5
Pressure, psig	2008	2003
Average Catalyst Temperature, °F	823	797
Oil Feed Rate, g/h	639	647
H_2 Feed Rate, SCF/h	22.6	23.4
Product Gas Rate, SCF/h	14.9	15.6
H_2/Oil Ratio, SCF/bbl	5003	5119
Liquid Hourly Space Velocity, h^{-1}	0.4	0.4
Product Oil		
Sulfur, wt %	0.16	--
Nitrogen, wt %	0.00	0.00
Water-Washed Oil		
Sulfur, wt %	0.03	0.03
Nitrogen, wt %	0.00	0.00
Acid-Washed Oil		
Sulfur, wt %	0.03	0.02
Nitrogen, wt %	0.00	0.00
Hydrogen Consumption, SCF/bbl		
By Oil Analysis	1708	1713
By Gas Meter	1806	1779

Table 20. LIQUID PROPERTIES, RAW WHOLE SHALE OIL AND HYDROTREATED PRODUCT

Type of Shale Oil	Tennessee	
Run Source	BSU-16 (Feed)	HTT-1 (Product)
Ultimate Analysis, wt %		
Carbon	87.04	86.24
Hydrogen	10.99	13.60
Sulfur	1.08	0.16
Nitrogen	0.64	0.00
C/H Weight Ratio	7.92	6.34
Specific Gravity	0.890	0.808
Gravity, °API	27.5	43.7
Distillation, °F		
IBP	172	120
5%	200	176
10%	238	204
20%	296	241
30%	364	278
40%	426	326
50%	486	369
60%	540	410
70%	596	442
80%	650	486
90%	710	544
95%	748	605
End Point	749	649
% Recovery	96.5	97.5
% Residue	2.0	1.5

calculated hydrogen consumption was a substantially lower 980 SCF/bbl. This was to be expected in light of the lower C/H ratio and sulfur content of this oil.

Subtask 2.4. Mist Size Control Studies

In this subtask a laboratory test system was designed, constructed, and operated to evaluate techniques of enhancing fog formation. A schematic diagram of the unit is shown in Figure 29. The unit consists of an inert solid bed (1/4-inch quartz particles) with equipment for feeding and measuring the flow rate of gas (nitrogen) and oil (decane). Simple controls are used to maintain temperature, pressures, and flow rates at the desired values.

The liquid oil is vaporized by being pumped into the hot main gas stream that has passed through a preheater. The vaporized oil and main stream gas mixture then enter the bottom of the reactor. Band heaters provide enough heat to keep the bottom of the reactor at the desired temperature. Another gas stream enters the ionization chamber, which is connected to the side of the reactor 1 foot above the bottom of the bed. The ionization chamber consists of two electrodes (spark plugs) screwed into opposite sides of a 1-inch-ID pipe. Foreign nuclei are generated inside the ionization chamber by applying high-voltage electricity to the electrodes. Oil mist coming out of the reactor is collected by two coalescing filters in series where 99.9% of the mist is collected; gas and vapor then pass through a condenser for further condensation of vapors. The oil-saturated cold nitrogen is then vented.

In this quarter, the apparatus was modified to minimize absorption of the oil by the reactor insulation material. Different oil feed rates and the effects of different side stream gases and different gas flow rates through the arc discharge were studied.

Several experimental runs were made. Oil feed rates of 9.5 and 20 cc/min were studied. The amount of oil collected by the filters in the runs with the electric discharge was consistently and considerably higher than in similar runs without the discharge for both low and high oil feed rates. Because of the limitations of this apparatus we cannot study higher oil feed rates. Gas flow rates of 2 and 5 SCF/h were studied in the side steam. The same amount of oil was collected in both cases. No further effort was made to determine the optimum gas flow rate. Nitrogen and carbon dioxide were used in the side

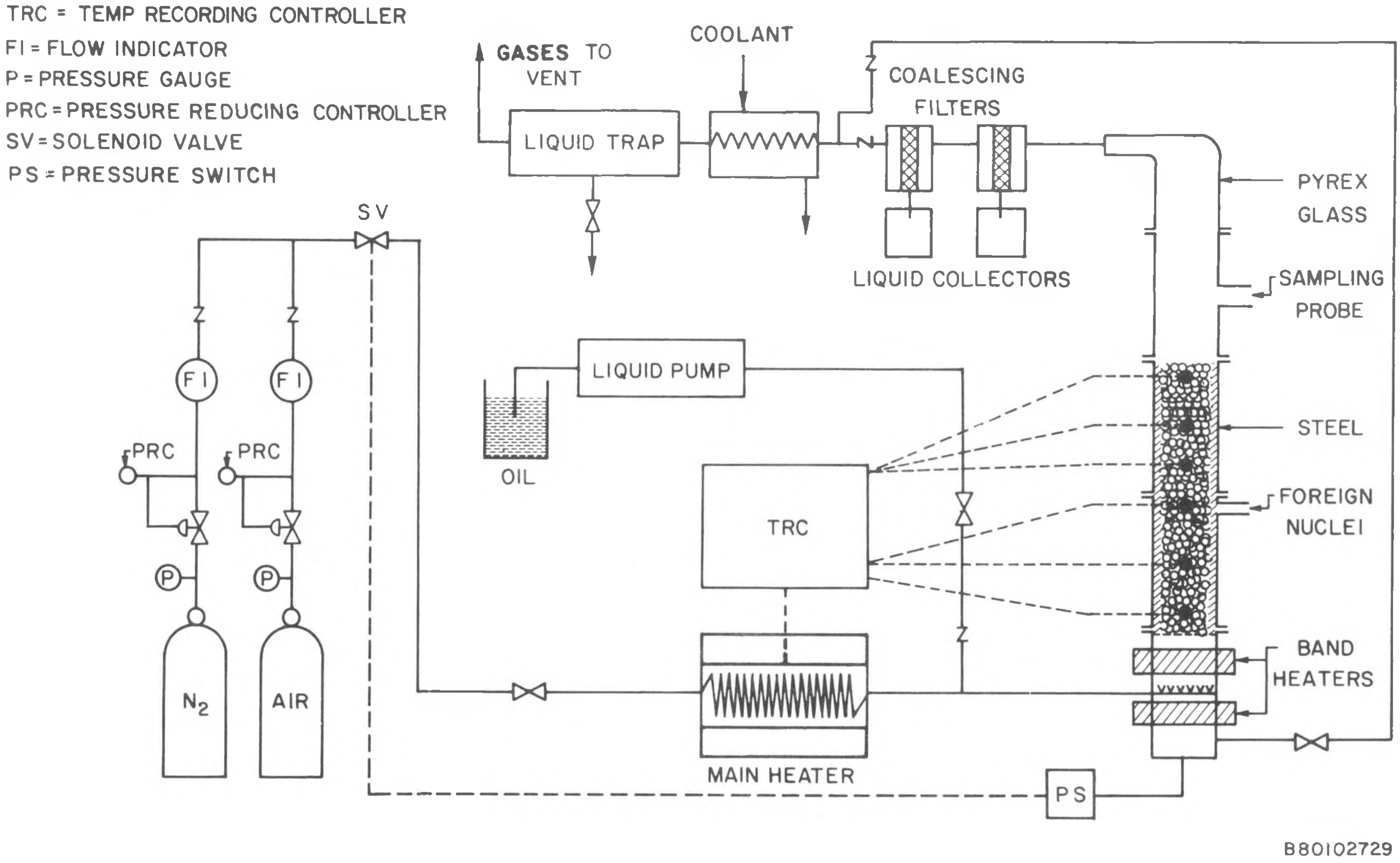

Figure 29. SCHEMATIC DIAGRAM OF LABORATORY TEST SYSTEM

stream. Although the results are not conclusive, they suggest that nitrogen and CO_2 are both effective in enhancing mist formation.

Figure 30 shows the oil collected by the first mist filter in Runs 22 and 23. The oil feed rate for both runs was 9.5 cc/min. Electric power was supplied to the electrodes only in Run 23. Figure 31 shows the oil collected in Runs 25 and 26 with an oil feed rate of 20 cc/min. Run 26 was conducted with electric discharge and Run 25 without discharge.

Each figure shows two regimes of operation. The first regime (no temperature breakthrough) is the period from the start of the oil feed during which time the temperature of the vapor leaving the bed remained relatively constant (ambient temperature). The second regime is after the start of the temperature rise. A temperature breakthrough occurs during this period. The temperature of the vapor leaving the bed increases drastically, which leads to a significant increase in the rate of oil collection by the filter regardless of nuclei introduction. Because a major fraction of the oil collected in the second regime is either condensed vapor or mist formed in the gas phase, our basis of comparison is the total amount of oil collected by the mist filter before the temperature breakthrough occurs.

Two runs were made (Run 20 with and Run 21 without the arc discharge) feeding previously collected oil (the oil obtained by coalescing the mist that was formed in earlier runs with arc discharge) instead of fresh oil. The same amount of oil was collected by mist filters in both runs and it was greater than the amount of oil collected in a similar run feeding fresh oil and supplying power to the electrodes. Another run was made (Run 24) feeding used oil (oil collected from runs without arc discharge) to study the effect on mist formation of whatever may have been carried by the mist in those runs. The rate of collection of oil mist was greater than that of Run 22 (similar run made with fresh feed oil), but it was much less than the rate of collection in Run 21 (similar run made with collected oil from runs with discharge as feed oil). Power was not supplied to the electrodes in Run 24. This may suggest that the foreign nuclei that enhance the fog formation stay in the oil, and that the amount of oil collected feeding the used oil is the maximum amount of oil mist one could collect in a similar run because per volume of oil fed into the system the collected oil contains the maximum number of nuclei.

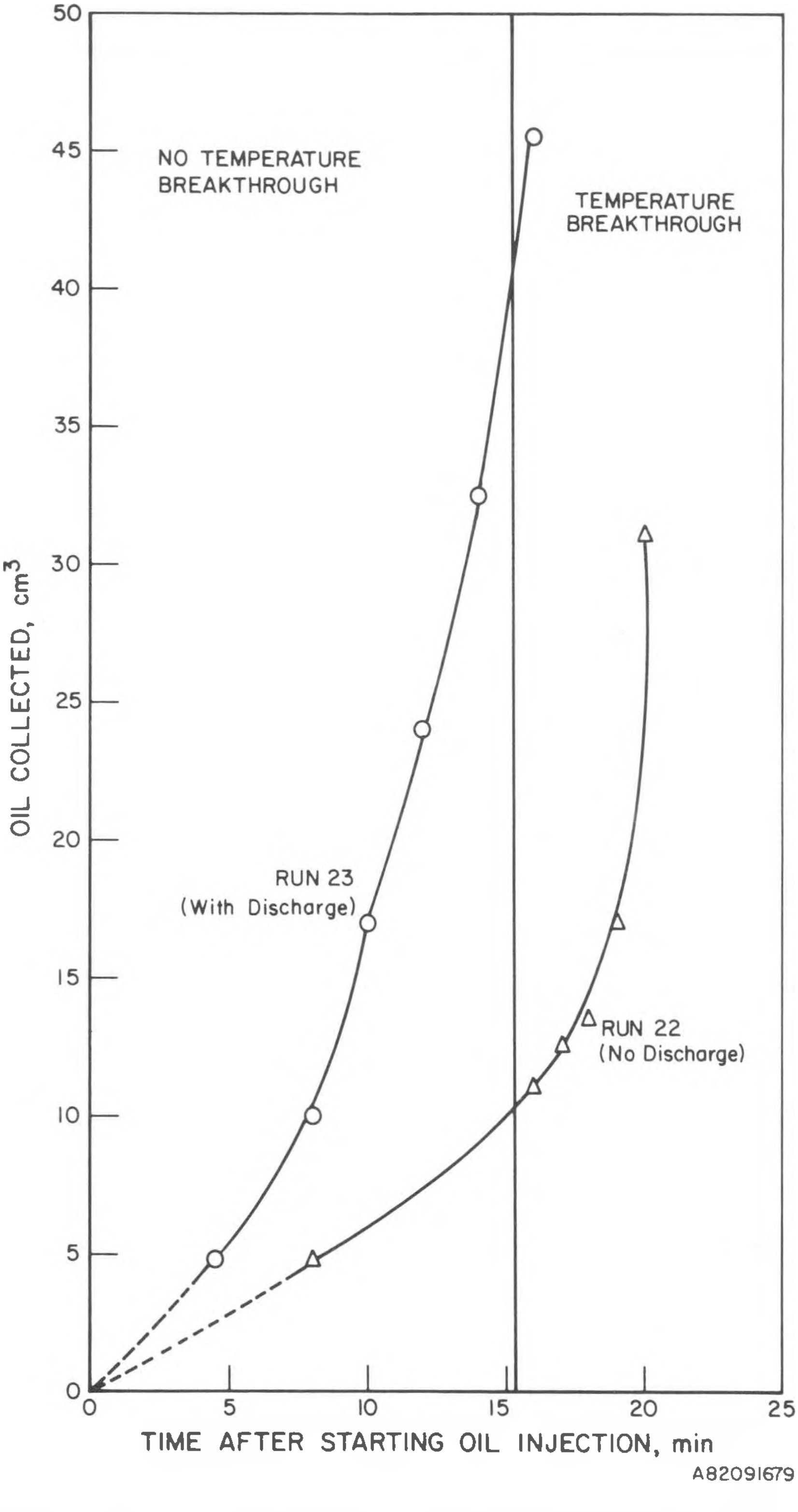

Figure 30. OIL COLLECTED BY MIST FILTER, RUNS 22 AND 23

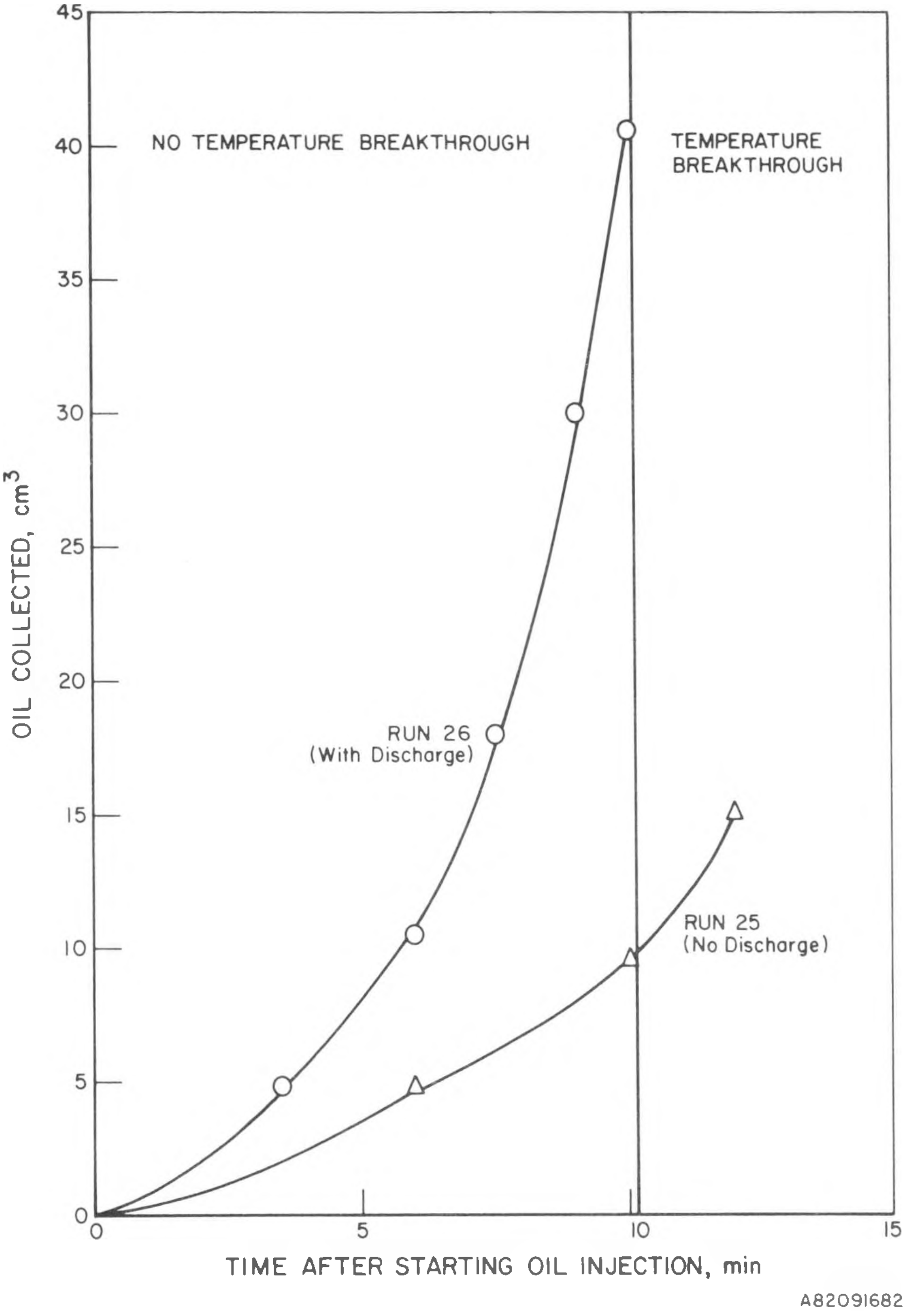

Figure 31. OIL COLLECTED BY MIST FILTER, RUNS 25 AND 26

Figure 32 compares oil mist collection in Runs 20, 21, 23, and 24. The oil feed rate in these runs was 9.5 cc/min. Collected oil from the previous run with discharge was fed in Runs 20 and 21, collected oil from prevous runs without the discharge was fed in Run 24, and fresh oil was used in Run 23. Power was supplied to the electrodes in Runs 21 and 23. Gas flow rates in the main and side stream were 60 SCF/h and 2 SCF/h, respectively.

A sample of collected oil and another sample of fresh oil were sent to our laboratory for analysis. Visible spectroscopy, infrared spectroscopy, and X-ray fluorescence spectroscopy analyses did not detect any difference between the two samples.

Summaries of all the runs made are presented in Table 21. However, as the apparatus and operating procedure were changed several times, the oil collected from runs with similar operating conditions cannot be directly compared unless experiments were conducted using the same apparatus configuration. Time of collection is defined as the time required for the exit gas temperature to begin to rise after the start of the oil feed. Total oil collection is the cumulative oil mist collected by the defined time of collection.

Based on the results of these tests, it can be concluded that injecting a stream of gas, which has first been passed through an electric arc discharge, into a moving-bed retort may result in enhanced fog formation. However, this approach would have to be tested in an operating moving-bed retort before firm conclusions can be drawn.

Subtask 2.5. Liquid-Sealed Lockhopper Tests

In this subtask the effect of a) the raw shale feed rate, b) the particle size distribution, and c) the discharge pressure on the amount of water carry-over to the retort were determined. This amount of water must be known accurately in order to effectively design a continuously operating retort using this feed mechanism. That is, the heat duty caused by this excess surface water must be accounted for in the upper portion of the retorting zone.

Data for the three runs conducted this quarter are reported here. Three different shales, a Kentucky New Albany, a Kentucky Sunbury, and a Tennessee Gassaway were tested in the liquid-sealed lockhopper in Runs 80PF-8, 80PF-9, and 80PF-10, respectively.

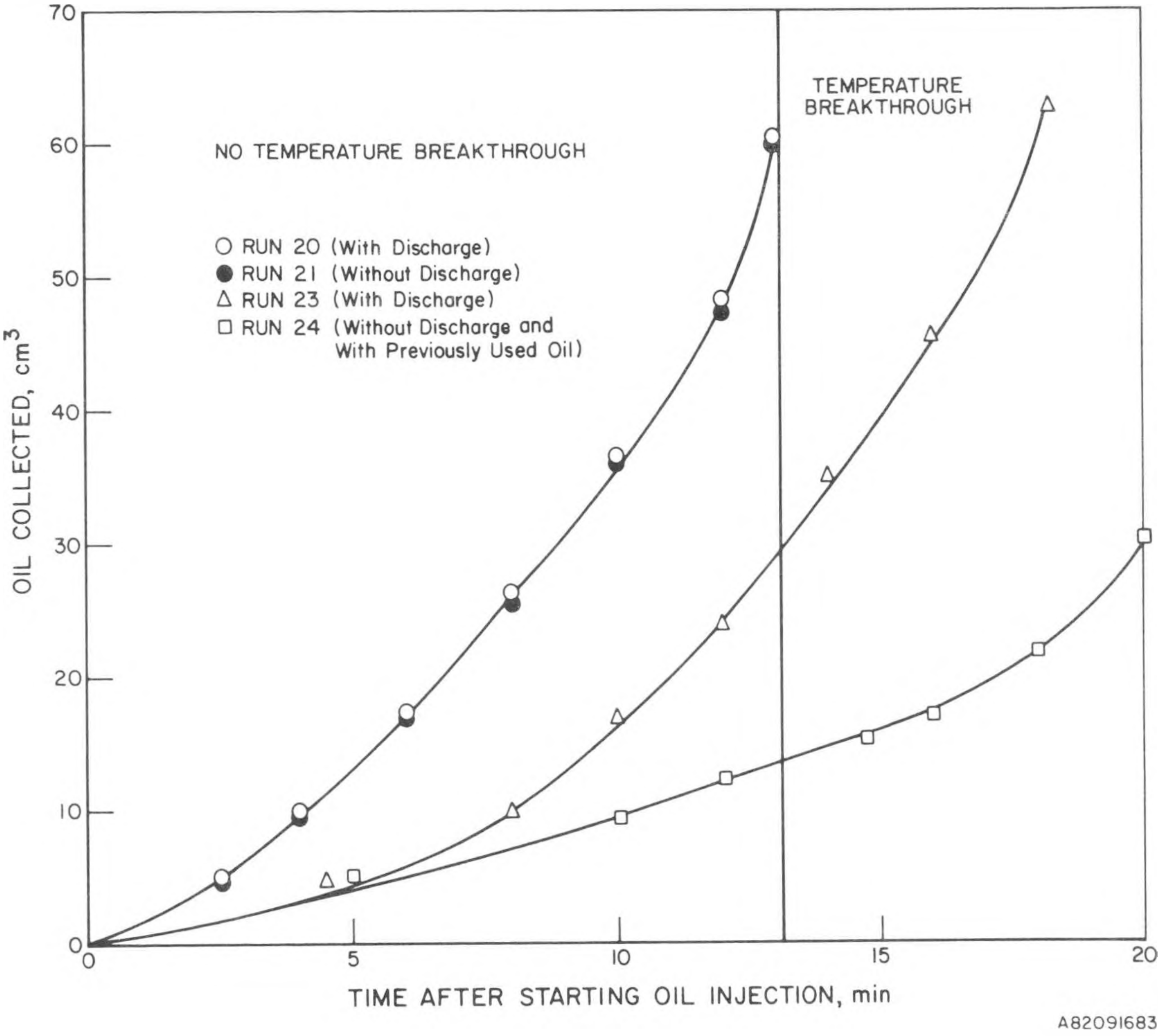

Figure 32. OIL COLLECTED BY MIST FILTER FEEDING FRESH AND USED OIL

Table 21. SUMMARIES OF RUNS IN MIST-SIZE-CONTROL STUDIES

Run No.	Main Stream Gas Flow Rate, SCF/h	Gas Flow Rate Through the Arc, SCF/h	Oil Feed Rate, cc/min	Electric Arc Discharge	Time of Collection, min	Total oil Collection, cc	Remarks
1-7	60	2	2.25	On & Off	-	-	Visual observation of mist formation by turning on the electric discharge.
8	60	2	2.25	Off	40	5	
9	60	2	2.25	On	40	14	
10	60	2	5.25	Off	20	14	Apparatus was modified after Run 11.
11	60	2	5.25	On	20	25.5	
12	60	2	5.25	On	-	-	Mechanical failure of equipment.
13	60	2	5.25	Off	20	1	
14	60	2	5.25	On	20	8	
15	60	2	9.5	Off	15	7	
16	60	2	9.5	On	15	30	
17 & 18	-	-	-	-	-	-	Mechanical failure of equipment.
19	60	-	10.5	On	15	40	Apparatus modified after this run.
20	60	2	9.5	Off	13	60	Oil collected from Runs 9, 11, 14 and 16 was fed instead of fresh oil fed.
21	60	2	9.5	On	13	61	
22	60	2	9.5	Off	15	10	Fresh oil was fed.
23	60	2	9.5	On	15	39	
24	60	2	9.5	Off	15	15	Oil collected from Runs 8, 10, 13 and 15 was fed into the system.
25	60	2	20	Off	10	10	Fresh oil.
26	60	2	20	On	10	41	
27	60	5	9.5	On	15	37	Increase in flow rate through the arc discharge.
28	60	2	9.5	On	14	40	CO_2 was passed through the arc discharge instead of N_2

Times for various steps in the feed cycles for these runs are presented in Table 22. The Sunbury and New Albany shales, which had a smaller particle size distribution (PSD) than the Gassaway, required more time to fill the liquid-sealed lockhopper than the Gassaway. This would be expected as smaller particles have lower terminal settling velocities. These results agree qualitatively with atmospheric pressure Plexiglas modeling tests performed in an earlier test program.[4]

The nominal PSD for the Kentucky New Albany and Sunbury shales was -1/4 inch +10 mesh, whereas the nominal PSD for the Tennessee Gassaway was -1/2 inch +6 mesh. The actual PSD for each of the shales before and after testing is presented in Table 23. The PSD data show that the discharged material is slightly larger for the Kentucky Sunbury and Kentucky New Albany shales. The apparent increase in the average particle size (APS) of the discharge material may be due to incomplete drying prior to sieve analysis and wet shale particles "sticking" to each other. The PSD of the Tennessee Gassaway shale indicated that there was a small amount of particle breakdown occurring. The APS of the discharged shale was also somewhat smaller than the original feed material.

The net water gain for the smaller particle size range (-1/4 inch +10 mesh) is appreciably larger than for the larger particle size range (-1/2 inch +6 mesh). In a previous test, Run 80PF-7, Kentucky New Albany shale (-1/2 inch +6 mesh) gained 4.2 weight percent moisture (dry basis) whereas for Run 80PF-8, using a smaller particle size (-1/4 inch +6 mesh), the water gain was 9.2 weight percent (dry basis). As expected, the smaller shale particle sizes gain more moisture than large shale particles of the same shale under similar operating conditions because of the greater specific surface area of the smaller shale particles.

A new 8-inch ball valve (made by a different manufacturer) was received and installed to replace the defective ball valve that had limited operating pressures to 200 psig because of seal leakages. The liquid-sealed lockhopper, after installation of the new ball valve, was successfully pressure-tested to 1000 psig. There were no feeding tests performed with the new ball valve since its delivery was delayed by the manufacturer.

Table 22. TIMES FOR VARIOUS STEPS IN FEEDING CYCLES

Run No.	80PF-8	80PF-9	80PF-10
Shale Type	Kentucky New Albany	Kentucky Sunbury	Gassaway (Tennessee)
Operating Pressure, psig	200	200	200
Number of Feed Cycles	8	8	8
Shale Feed/Cycle, lb	200	200	212
Operating Step	Time, s		
1. Open Feed Hopper Slide Valve	9.8	9.7	9.0
2. Open Top Lockhopper Ball Valve	2	2	2
3. Drop Shale From Feed Hopper Into Lockhopper	47	60	26
4. Close Top Lockhopper Ball Valve	1	1	1
5. Pressurize Lockhopper with Water	4.6	1.8	2.6
6. Open Bottom Lockhopper Ball Valve	2	2	2
7. Discharge Oil Shale From Lockhopper With Screw Feeder	360	480	300
8. Close Bottom Lockhopper Ball Valve	1.5	1	1
9. Depressurize Lockhopper	9.7	5.9	9.3

Table 23. RESULTS OF HIGH-PRESSURE FEEDING TESTS

Run No.	80PF-8		80PF-9		80PF-10	
Shale Type	Kentucky New Albany		Kentucky Sunbury		Gassaway (Tennessee)	
Nominal Feed Size	-1/4 inch +10 mesh		-1/4 inch +10 mesh		-1/2 inch +6 mesh	
Pressure, psig	200		200		200	
Lab No.	124512	124513	124514	124515	124611	124612
Material	Feed	Discharged	Feed	Discharged	Feed	Discharged
Moisture, wt %	2.0	10.1	3.8	9.3	1.2	4.7
Net Water Gain (dry basis), wt %	--	9.2	--	6.3	--	3.7
Screen Analysis (U.S.Sieve), wt %						
+1/2 inch	0.0	0.0	0.0	0.0	0.0	0.0
+3/8 inch	0.0	0.0	0.0	0.0	30.5	27.1
+1/4 inch	0.0	0.1	0.5	0.2	59.0	56.2
+6	74.8	78.2	63.4	75.8	10.1	16.5
+8	--	--	--	--	0.1	0.1
+12	24.8	21.2	33.5	23.2	0.0	0.0
+14	--	--	--	--	0.0	0.0
Pan	0.4	0.5	2.6	0.8	0.3	0.1
Total	100.0	100.0	100.0	100.0	100.0	100.0
Bulk Density, lb/ft^3	70.4	69.9	66.8	66.5	70.5	73.1

It can be seen, from the tests conducted on a number of different shale types, that water carryover is a strong function of the shale type. Also, water carryover decreases with increases in shale particle size.

Task 3. Process Development Unit (PDU) Tests

In this task the technical feasibility of the hydroretorting process with various Eastern and Western shales under the adiabatic (thermally balanced) operating conditions that are encountered in large-scale equipment was determined. During this quarter, the PDU was repiped to convert the unit back to two-stage operation. Run 81PDU-1 was subsequently made using a sample of the Gassaway member of the Chattanooga shale from Tennessee. The run was voluntarily terminated after 8.0 hours of total shale feeding time including 6.0 hours of steady-state operation.

During the previous quarter the PDU was modified to operate using a single-stage (Stage 2) reactor instead of the two-stage reactor system used in previous tests. Two long successful tests were subsequently made, Runs 80PDU-6 and 80PDU-7. In both runs, a Kentucky New Albany shale was fed at a rate of approximately 1000 lb/h. During Run 80PDU-6, 8.5 hours of shale feed time, including 5.5 hours of steady-state operation were achieved. In Run 80PDU-7, shale was fed for 8.0 hours with 6.5 hours of steady-state operation. Both runs were voluntarily terminated.

Detailed test results for Runs 80PDU-6 and 80PDU-7 are presented in Tables 24 and 25, respectively. The poor selectivity to oil (31.7%) in Run 80PDU-6 was possibly due to refluxing, in which hot product oil vapors condense on cold incoming shale and the condensed oil is carried by the shale down into the hotter zone where revaporization and cracking occur and the cycle repeats itself. The superficial gas velocity (0.37 ft/s) may not have been high enough in this run to entrain the oil and carry it out as liquid droplets.

The higher selectivity to oil observed in Run 80PDU-7 compared with that observed in Run 80PDU-6 would tend to support the deleterious effects of the low superficial gas velocity observed in Run 80PDU-6 compared with the higher superficial velocity seen in Run 80PDU-7 (0.37 ft/s compared with 0.82 ft/s, respectively). The high-quality oil that was collected during Run 80PDU-6

Table 24. PDU (80PDU-6) TEST RESULTS — SINGLE-STAGE OPERATION WITH KENTUCKY NEW ALBANY SHALE

Operating Conditions and Test Results	
Oil Shale Location (State)	Kentucky New Albany
Oil Shale Particle Size (U.S. Sieve)	-1/2 +6
Test Duration, hr	8.5
Steady-State Operating Period, hr	5.5
Bed Height, ft	10
Pressure, psig	403
Average Bed Temperature, °F	740
Maximum Bed Temperature, °F	1400
Oil Shale Feed Rate, lb/hr	924.4
Oil Shale Space Velocity, lb/ft^3-hr	106.0
Oil Shale Mass Rate, lb/ft^2-hr	1060.0
Feed Gas*/Shale Ratio, SCF/lb	14.95
Feed Gas* Superficial Velocity, ft/s	0.37
Air/Shale Ratio, SCF/lb	1.06
Product Gas Yield, SCF/lb Shale Fed	14.22
Residue Shale Yield, lb/lb Shale Fed	
By Direct Weight Measurement	0.797
By Ash Balance Calculation	0.835
Liquid Hydrocarbon Yield, lb/lb Shale Fed	0.032
Water Yield, lb/lb Shale Fed	0.083
Material Balance, %	101
Organic Carbon Distribution in Products, %	
Gaseous Hydrocarbons	43.5
Liquid Hydrocarbons	20.2
Residue Shale	41.1

Feed and Product Compositions

Oil Shale		
Sample	Feed	Stage 2 Residue
Lab No.	124141	124142
Moisture, Wt % (Dry Basis)	1.8	0.2
Ultimate Analysis, Wt %		
Organic Carbon	13.83	6.69
Mineral Carbon	0.24	0.18
Hydrogen	1.67	0.40
Nitrogen	0.52	0.26
Oxygen (by HTW)**	2.45	0.89
Sulfur	5.16	3.53
Ash	76.93	90.61
Total	100.80	102.56

Shale Oil	
Sample	Stage 2
Lab No.	44486
C/H Weight Ratio	7.93
Sulfur, Wt %	1.41
Nitrogen, Wt %	1.06
Viscosity, SSU at 100°F	31.8
Specific Gravity (60/60°F)	0.90
Distillation	
IBP	215
50%	409
EP	728
Pour Point, °F	-75

Gas Composition, Mol %	Feed	Stage 2 Product†
Hydrogen Sulfide	--	0.89
Nitrogen & Argon	0.52	2.92
Carbon Monoxide	2.70	1.20
Carbon Dioxide	--	0.29
Hydrogen	95.83	84.89
Methane	0.95	7.61
Ethane +	--	1.74
Ethylene +	--	0.38
Benzene	--	0.08
Total	100.00	100.00

* Hydrogen

† Purge and Stage 2 Combustion Air-Free

** High Temperature Water

Table 25. PDU (80PDU-7) TEST RESULTS — SINGLE-STAGE OPERATION WITH KENTUCKY NEW ALBANY SHALE

Operating Conditions and Test Results

Oil Shale Location (State)	Kentucky New Albany
Oil Shale Particle Size (U.S. Sieve)	-1/2 +6
Test Duration, hr	8.0
Steady-State Operating Period, hr	6.5
Bed Height, ft	10
Pressure, psig	403
Average Bed Temperature, °F	1470
Maximum Bed Temperature, °F	1525
Oil Shale Feed Rate, lb/hr	1055.9
Oil Shale Space Velocity, lb/ft^3-hr	121.0
Oil Shale Mass Rate, lb/ft^2-hr	1210
Feed Gas*/Shale Ratio, SCF/lb	18.28
Feed Gas* Superficial Velocity, ft/s	0.82
Air/Shale Ratio, SCF/lb	0.82
Product Gas Yield, SCF/lb Shale Fed	16.73
Residue Shale Yield, lb/lb Shale Fed	
By Direct Weight Measurement	0.781
By Ash Balance Calculation	0.821
Liquid Hydrocarbon Yield, lb/lb Shale Fed	0.050
Water Yield, lb/lb Shale Fed	0.085
Material Balance, %	104
Organic Carbon Distribution in Products, %	
Gaseous Hydrocarbons	43.8
Liquid Hydrocarbons	31.9
Residue Shale	34.5

Feed and Product Compositions

	Oil Shale	
Sample	Feed	Stage 2 Residue
Lab No.	124510	124511
Moisture, Wt % (Dry Basis)	1.60	0.1
Ultimate Analysis, Wt %		
Organic Carbon	13.60	5.63
Mineral Carbon	0.28	0.10
Hydrogen	1.56	0.23
Nitrogen	0.50	0.14
Oxygen (by HTW)**	2.28	1.04
Sulfur	5.87	3.25
Ash	77.35	92.80
Total	101.44	103.19

Shale Oil	
Sample	Stage 2
Lab No.	44880
C/H Weight Ratio	8.39
Sulfur, Wt %	1.48
Nitrogen, Wt %	1.21
Viscosity, SSU at 100°F	39.6
Specific Gravity (60/60°F)	0.94
Distillation	
IBP	218
50%	514
EP	741
Pour Point, °F	-50

Gas Composition, Mol %	Feed	Stage 2 Product†
Hydrogen Sulfide	--	0.59
Nitrogen & Argon	0.35	5.21
Carbon Monoxide	3.48	2.80
Carbon Dioxide	--	0.32
Hydrogen	95.74	83.16
Methane	0.43	6.18
Ethane +	--	1.30
Ethylene +	--	0.30
Benzene	--	0.14
Total	100.00	100.00

* Hydrogen

† Purge and Stage 2 Combustion Air-Free

**High Temperature Water

would also support the hypothesis that refluxing and cracking had occurred in Run 80PDU-6.

The time-averaged shale bed temperature profile for Run 80PDU-6 for the entire run period is shown in Figure 33. The profile shows that the bottom of the shale bed was only approximately 1000°F, despite the fact that the incoming gas temperature was approximately 1400°F. The time-averaged shale bed temperature profile for Run 80PDU-6 during the steady-state run period is depicted in Figure 34. The two temperature plots are very similar. After the feed screw was shut off, a third time-averaged shale bed temperature profile was drawn (Figure 35), which shows that the feed gas temperature for Run 80PDU-6 was considerably higher than the temperatures indicated in Figures 33 and 34. It is possible that the feed gas/shale ratio was too low to provide a balance of gas and shale enthalpies for this run.

The time-averaged shale bed temperature profile for Run 80PDU-7 for the steady-state operating period is shown in Figure 36. Reactor wall skin temperatures were measured with a contact pyrometer during the steady-state operating period. These skin temperatures are also shown in Figure 36 at the reactor location (height) at which they were taken. These temperature measurements give an indication of the effectiveness of the Fiberfrax insulation used at the temperature indicated and provide an indication of the radial heat loss from the reactor.

The bed temperature profiles for Run 80PDU-7 at various times are shown in Figures 37 through 43. It is instructive to note that the external reactor skin temperatures changed throughout the "steady-state" portion of Run 80PDU-7. The thermocouples indicated that the reactor wall temperature increased throughout the entire run. This temperature rise would indicate that the PDU was continously heating up the insulation and reactor wall. Near the end of the run the rate of temperature rise of the reactor wall decreased markedly; this temperature rate decrease indicated that the PDU heat losses had reached a nearly constant value.

Runs 80PDU-6 and 80PDU-7 were both made using a Kentucky New Albany shale at total reactor pressures of 400 psig with shale residence times of approximately 38 minutes. Organic carbon conversion to gaseous and liquid products for Run 80PDU-6 was only 58.9%, calculated by an ash balance. Because there were indications that refluxing occurred in Run 80PDU-6, the

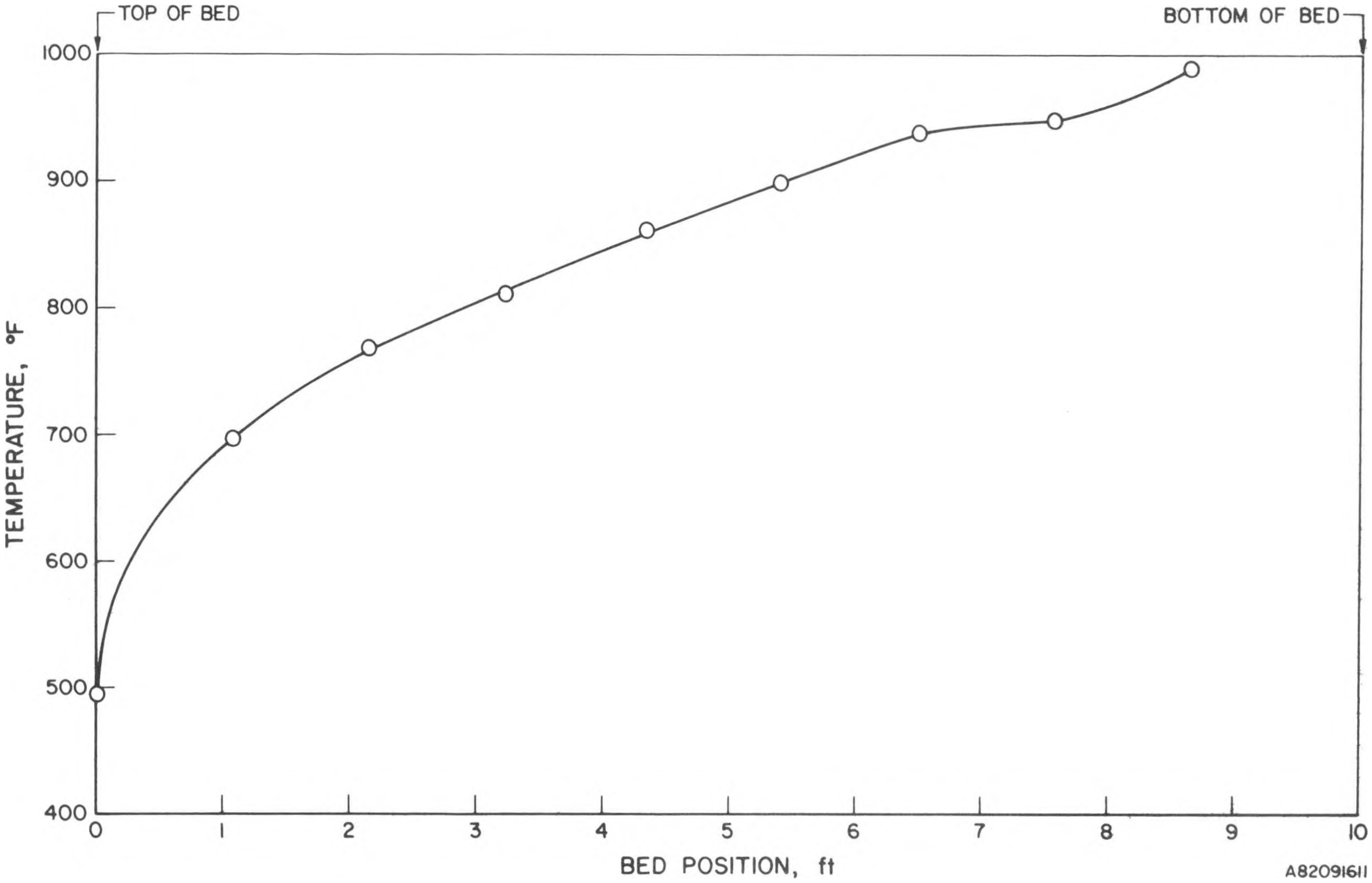

Figure 33. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-6
(Average for 1130 to 1930 Hours)

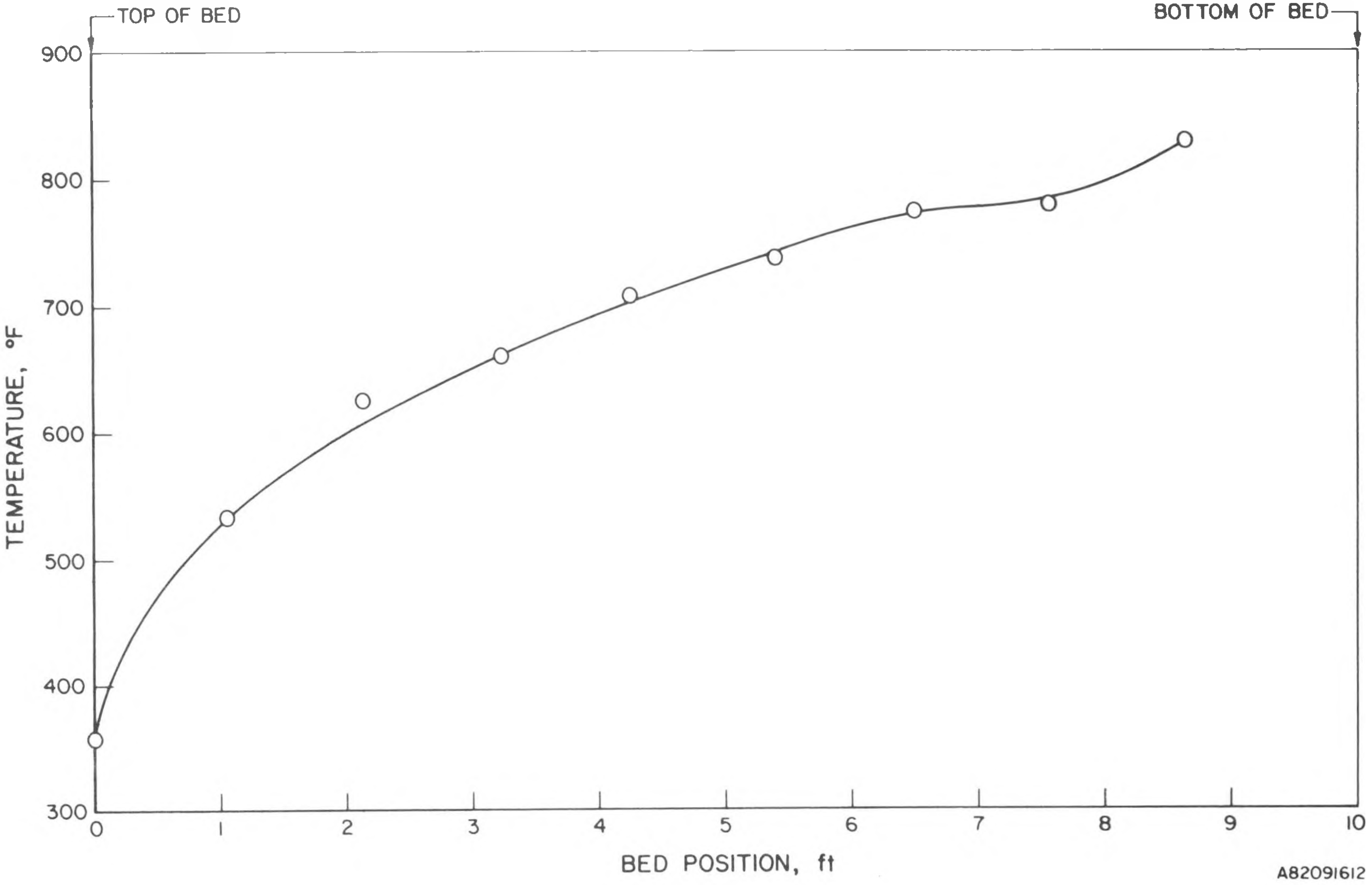

Figure 34. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-6
(Average for 1400 to 1930 Hours)

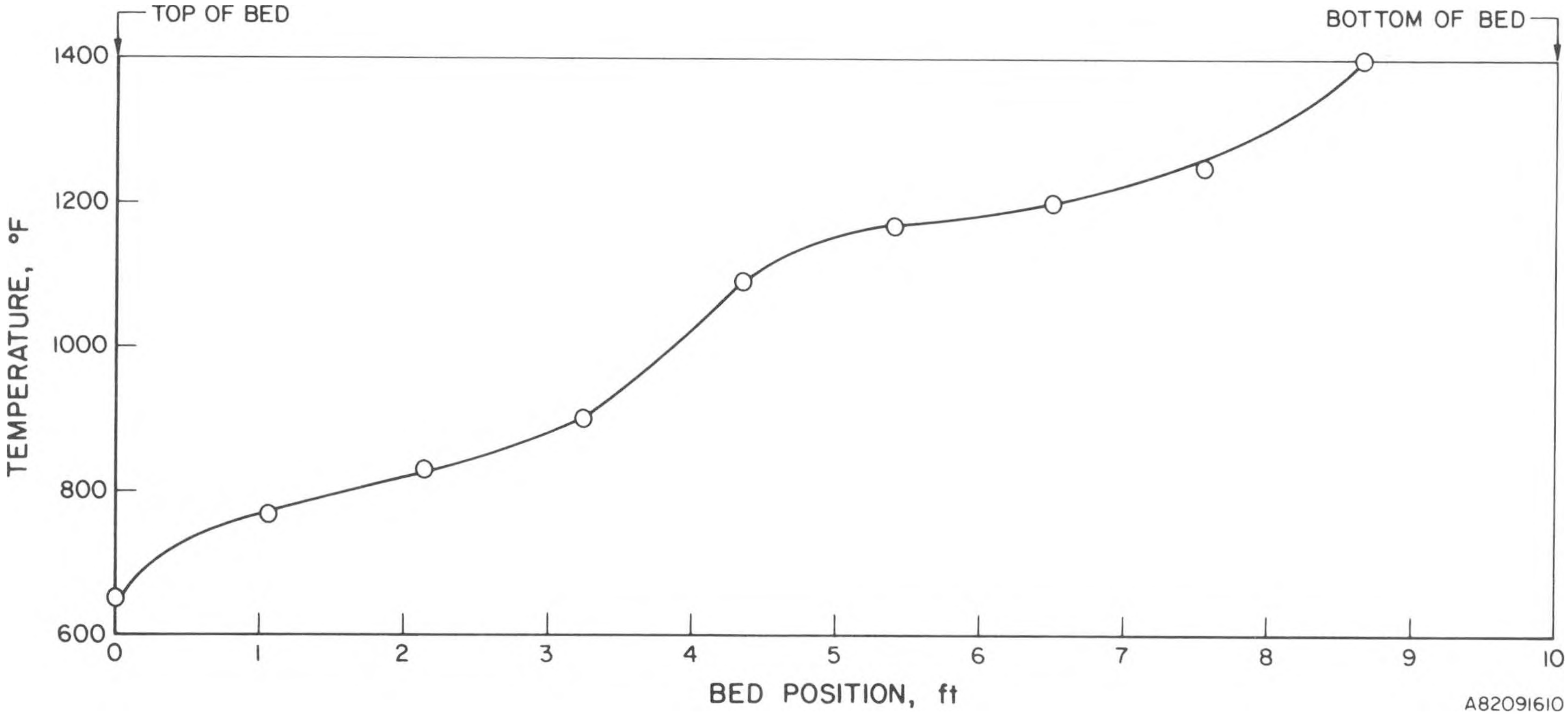

Figure 35. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-6 (Average for 1935 to 2030 Hours)

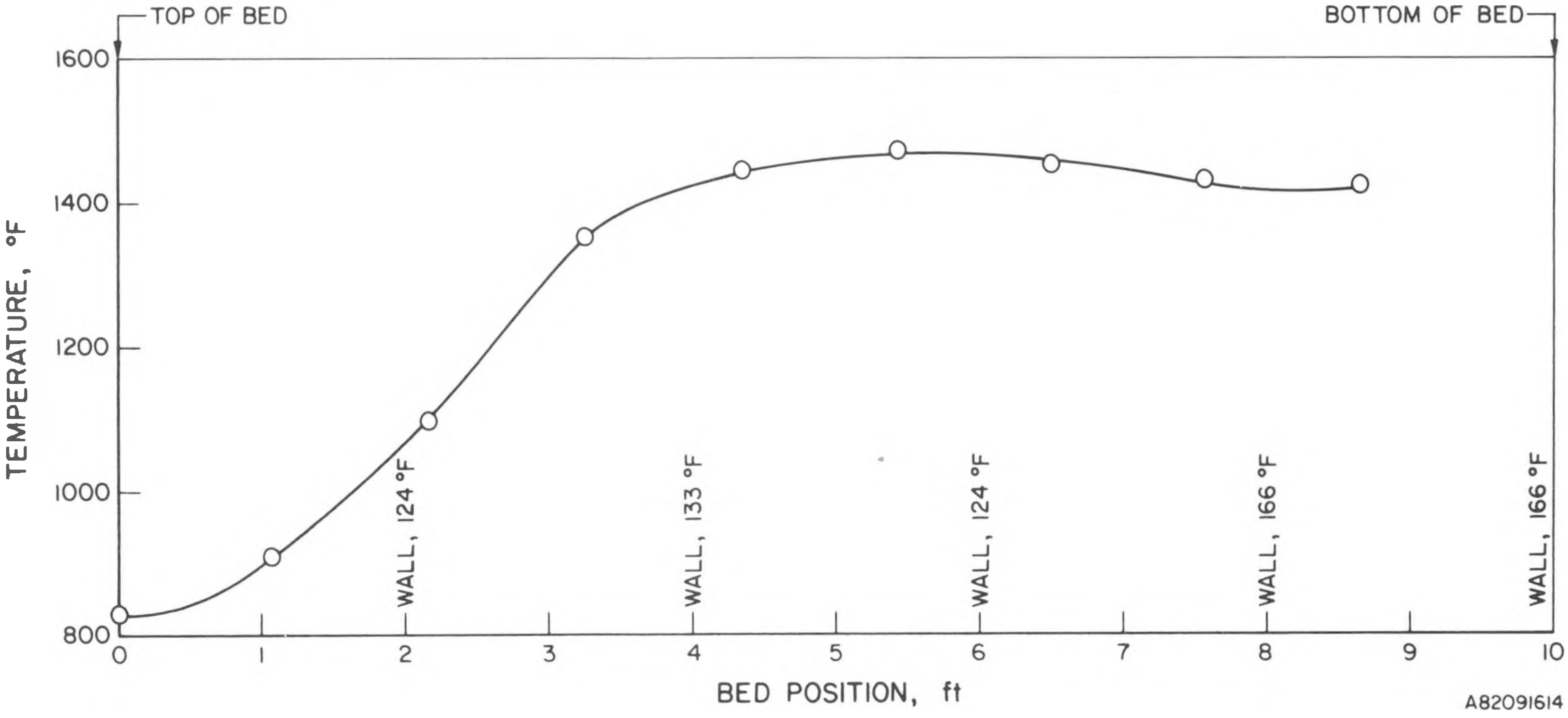

Figure 36. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-7
(Time: Steady-State Average)

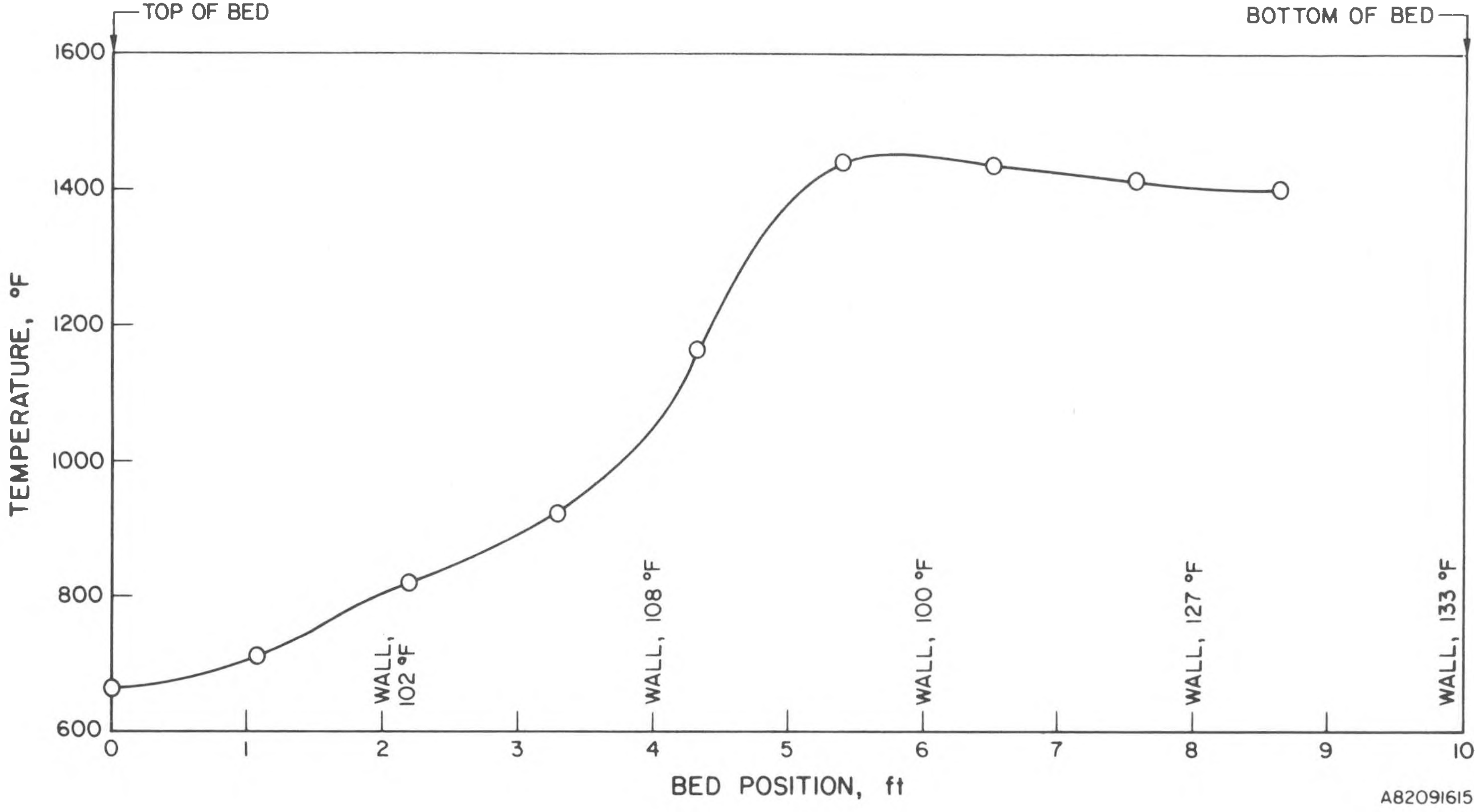

Figure 37. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-7
(Time: 1230 Hours)

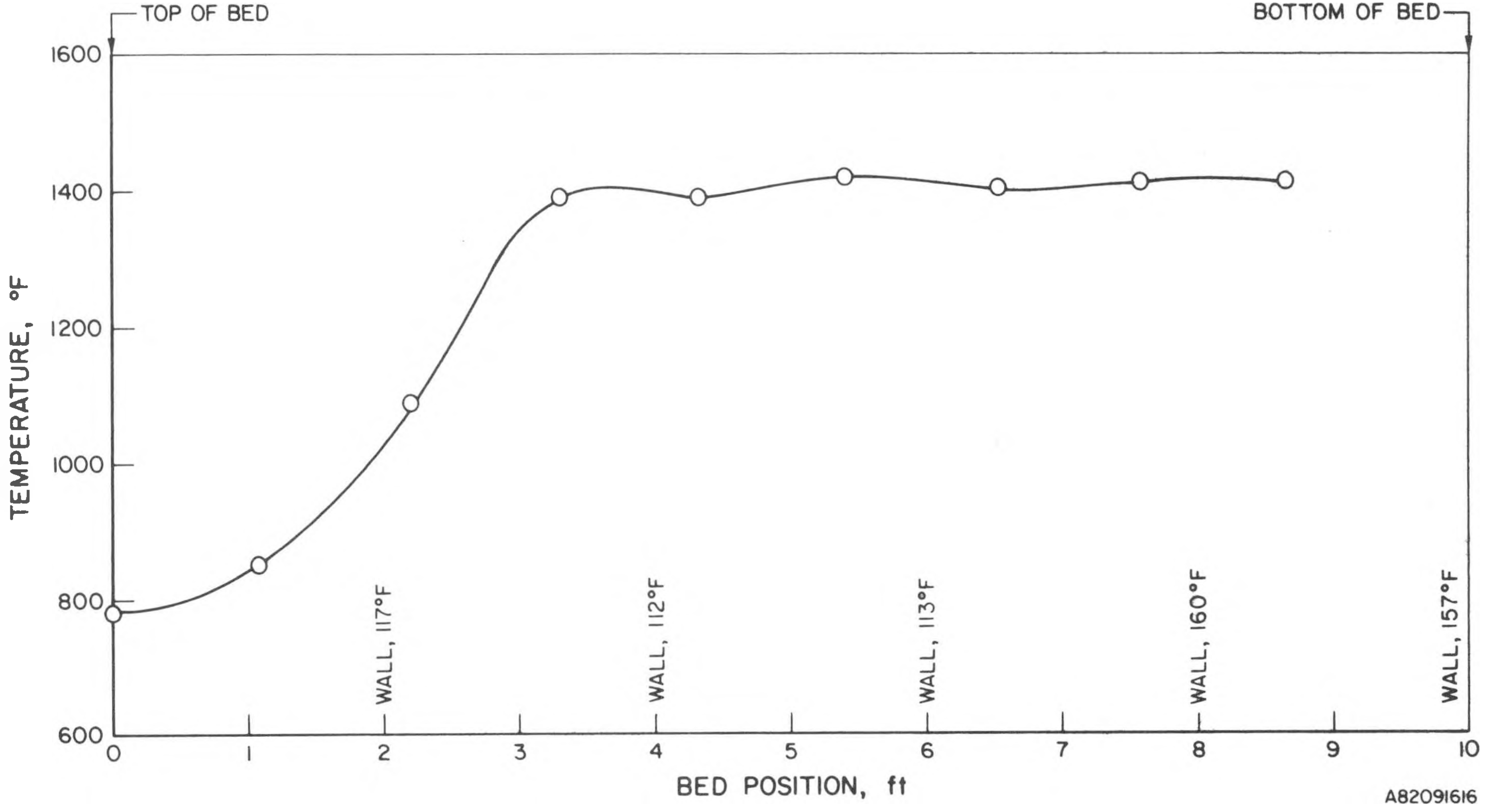

Figure 38. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-7
(Time: 1300 Hours)

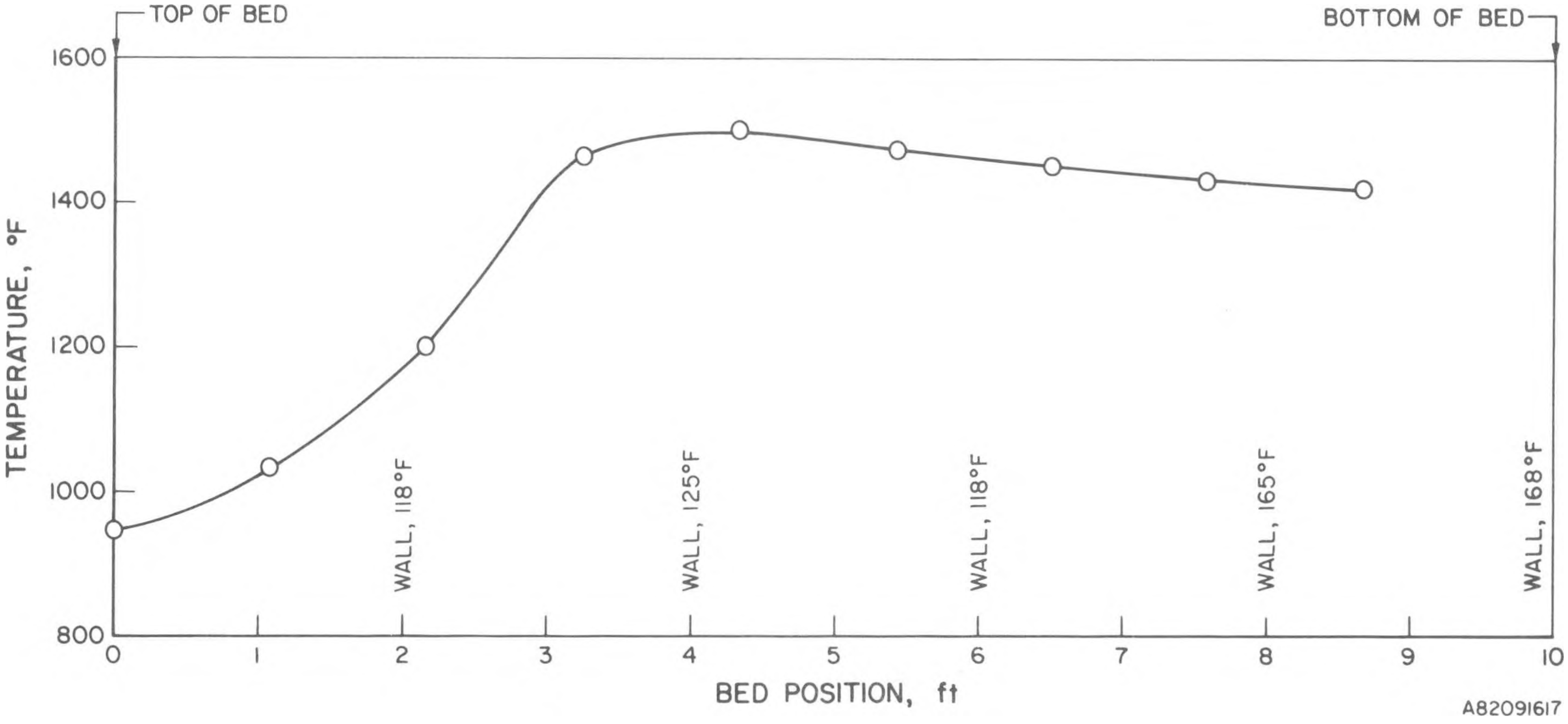

Figure 39. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-7
(Time: 1400 Hours)

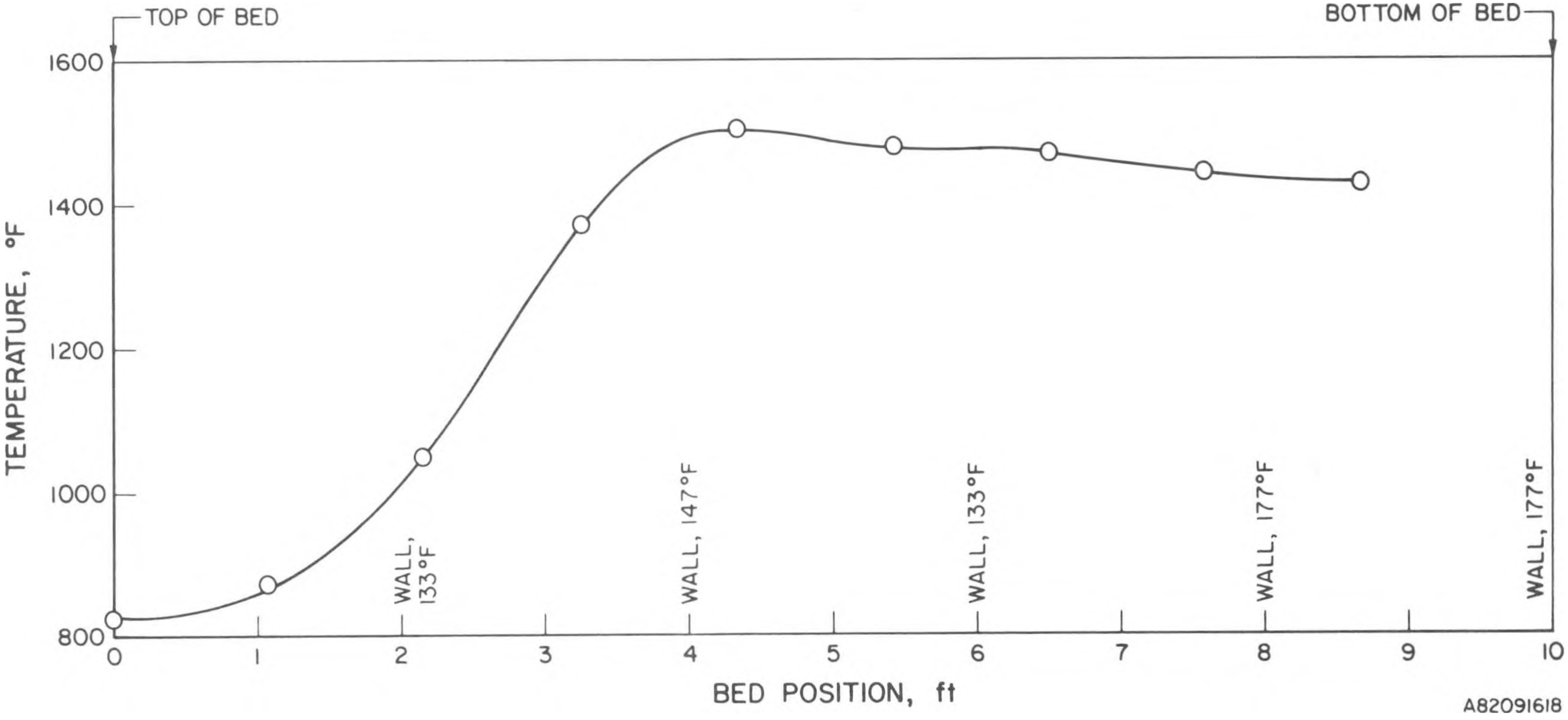

Figure 40. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-7
(Time: 1500 Hours)

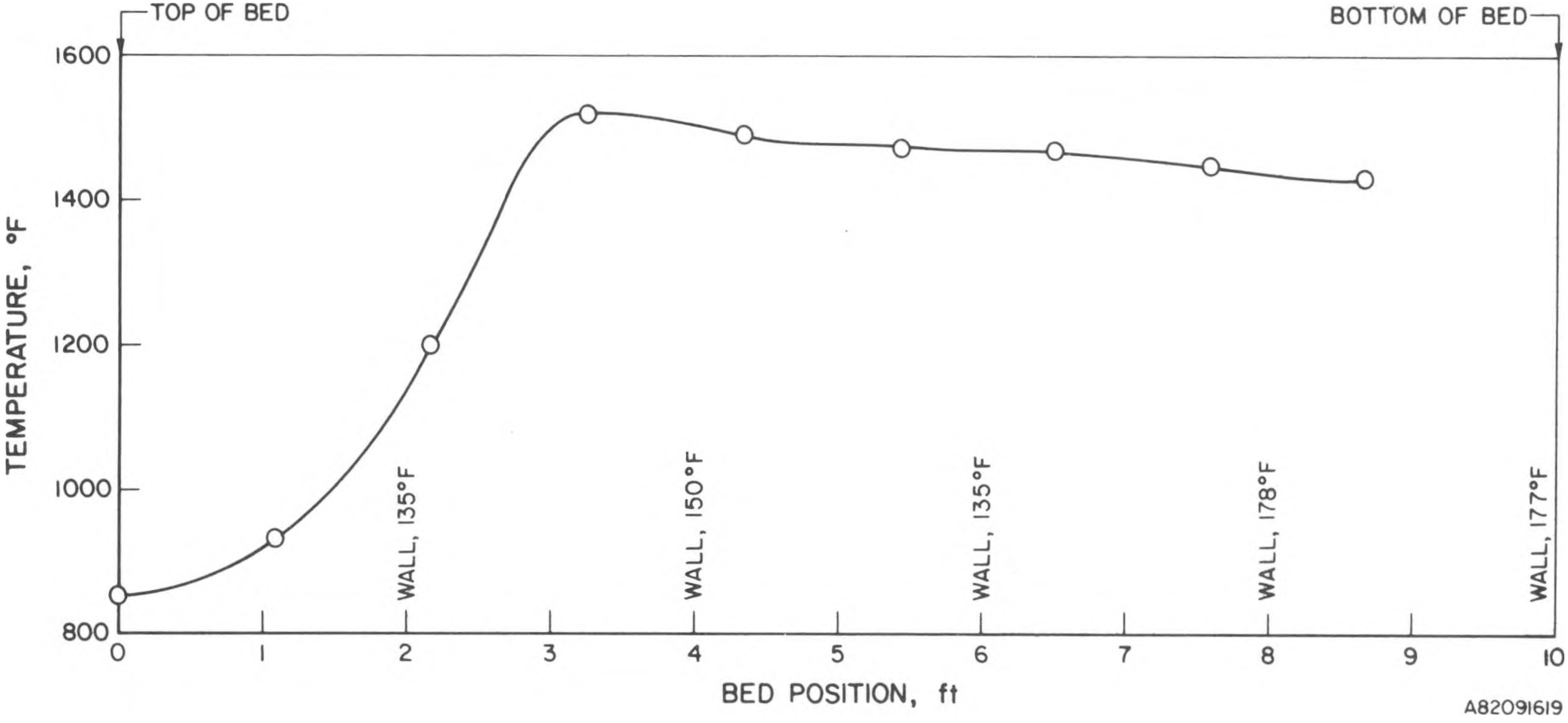

Figure 41. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-7
(Time: 1600 Hours)

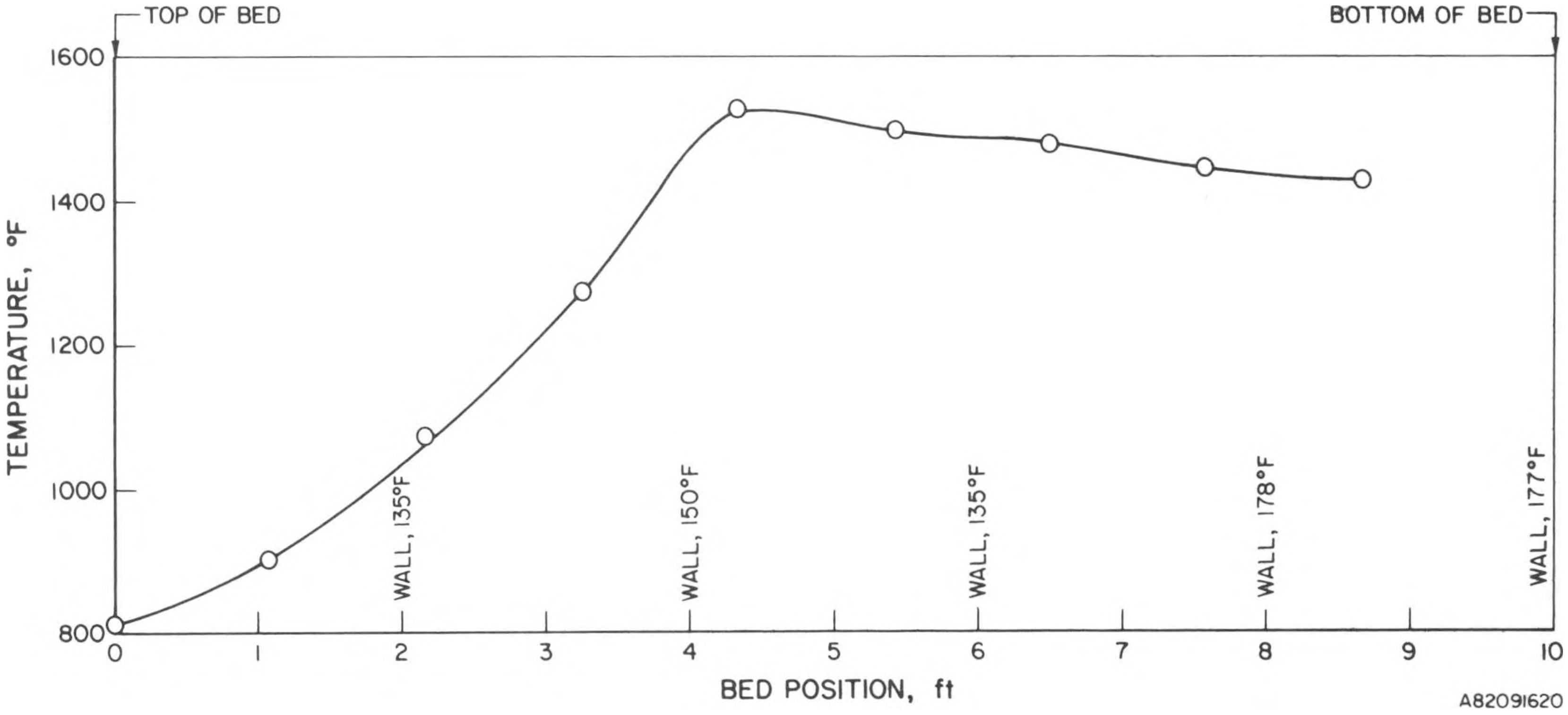

Figure 42. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-7
(Time: 1700 Hours)

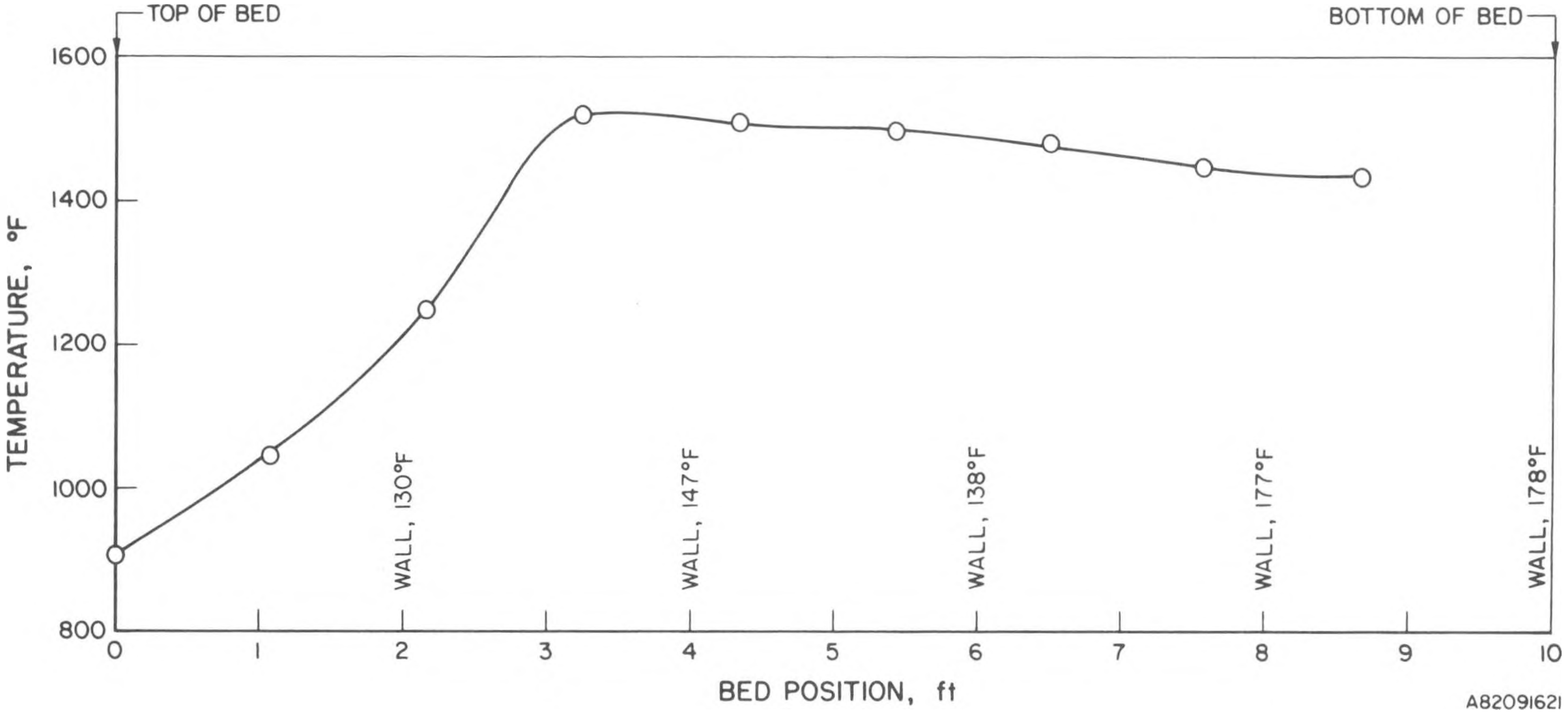

Figure 43. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 80PDU-7
(Time: 1800 Hours)

hydrogen-to-shale ratio was increased for Run 80PDU-7 in order to increase the superficial gas velocity and improve organic carbon conversion. The organic carbon conversion for Run 80PDU-7 did increase approximately 6.5% to 65.4%. The increased organic carbon conversion seen in Run 80PDU-7 exhibited itself as a 50% improvement in oil yield with gas yields remaining approximately constant.

A summary of the test results and operating conditions for Run 81PDU-1, a test with the Gassaway member of the Chattanooga shale, is shown in Table 26. Run 81PDU-1 was made at a shale feed rate of approximately 1100 lb/h, a reactor pressure of 405 psig, and shale bed heights of 10.0 feet for Stage 1 and Stage 2. The hydrogen-to-shale ratio for this run was chosen so that the enthalpy of the shale would nearly equal the enthalpy of the feed gas for each respective stage. The Stage 2 feed-hydrogen-to-shale ratio is lower than the ratio for Stage 1 because a small amount of air was added to Stage 2, which increased the total enthalpy of the feed gas. Some of the feed hydrogen was burned (with injected air) to increase the enthalpy of the Stage 2 gas.

The organic carbon conversion to gaseous and liquid products was approximately 41%, calculated by an ash balance. The organic carbon conversion for this run using a Gassaway member of the Chattanooga shale was somewhat lower than the conversions achieved using a Kentucky New Albany shale under similar operating conditions.

The steady-stage time-averaged bed temperature profiles for Stage 1 and Stage 2 for Run 81PDU-1 are shown in Figures 44 and 45, respectively. The Stage 1 profile shows a nearly constant slope, indicating that the feed gas and feed shale enthalpies were properly matched (nearly equal) to achieve a uniform slow heat-up rate. The Stage 2 profile indicates a nearly horizontal profile for the top of the shale bed (at 700°F) followed by a relatively rapid heat-up, followed by another horizontal profile (at 1420°F) for the bottom of the shale bed. Therefore, only 20% (approximately 2.0 feet) of the shale bed was required for heat transfer and to achieve operating temperatures.

Task 4. Process Environmental Assessment

In this task the PDU was used to establish a data base in order to answer process-specific questions about the environmental characterization of the HYTORT process, for scale-up to a commercial-scale HYTORT plant. The Pace

Table 26. PDU (81PDU-1) TEST RESULTS — COUNTERCURRENT OPERATION WITH TENNESSEE (Chattanooga) SHALE

Operating Conditions and Test Results

Oil Shale Location (State)
Oil Shale Particle Size (U.S. Sieve)
Test Duration, hr
Steady-State Operating Period, hr

	Stage 1	Stage 2
Bed Height, ft	10.0	10.0
Pressure, psig	405	
Average Bed Temperature, °F	581	981
Maximum Bed Temperature, °F	798	1420
Oil Shale Feed Rate, lb/hr	1109	--
Oil Shale Space Velocity, lb/ft^3-hr	129	127
Oil Shale Mass Rate, lb/ft^2-hr	1290	1270
Feed Gas*/Shale Ratio, SCF/lb	17.7	11.6
Feed Gas* Superficial Velocity, ft/s	0.45	0.36
Air/Shale Ratio, SCF/lb	--	0.59
Product Gas Yield, SCF/lb Shale Fed	15.9	12.0
Residue Shale Yield, lb/lb Shale Fed		
By Direct Weight Measurement	--	0.838
By Ash Balance Calculation	--	0.915
Liquid Hydrocarbon Yield, lb/lb Shale Fed	0.002	0.021
Water Yield, lb/lb Shale Fed	0.015	0.030
Material Balance, %	99.4	
Organic Carbon Distribution in Products, %		
Gaseous Hydrocarbons	5.8	21.4
Liquid Hydrocarbons	2.1	18.1
Residue Shale	59.1	

Feed and Product Compositions

Oil Shale

Sample	Feed	Stage 2 Residue
Lab No.	124888	124887
Moisture, Wt % (Dry Basis)	0.8	--
Ultimate Analysis, Wt %		
Organic Carbon	9.86	6.32
Mineral Carbon	0.04	0.09
Hydrogen	1.08	0.42
Nitrogen	0.28	0.15
Oxygen (by HTW)**	2.72	1.83
Sulfur	6.49	3.56
Ash	83.50	90.56
Total	103.97	102.93

Shale Oil

Sample	Stage 1	Stage 2
Lab No.	45051	45052
C/H Weight Ratio	7.38	8.39
Sulfur, Wt %	1.86	1.01
Nitrogen, Wt %	0.13	0.56
Viscosity, SSU at 100°F	36.2	41.5
Specific Gravity (60/60°F)	0.893	0.937
Distillation		
IBP	272	200
50%	502	580
EP	686	748
Pour Point, °F	-35	-40

Gas Composition, Mol %	Feed	Stage 1 Product†	Stage 2 Product†
Hydrogen Sulfide	0.0	0.34	0.74
Nitrogen & Argon	0.8	0.00	1.88
Carbon Monoxide	2.7	2.60	1.95
Carbon Dioxide	0.0	0.00	0.12
Hydrogen	95.3	95.48	90.38
Methane	1.2	1.38	4.07
Ethane +	0.0	0.14	0.63
Ethylene +	0.0	0.05	0.17
Benzene	0.0	0.01	0.06
Total	100.0	100.00	100.00

* Hydrogen
† Purge and Stage 2 Combustion Air-Free
**High Temperature Water

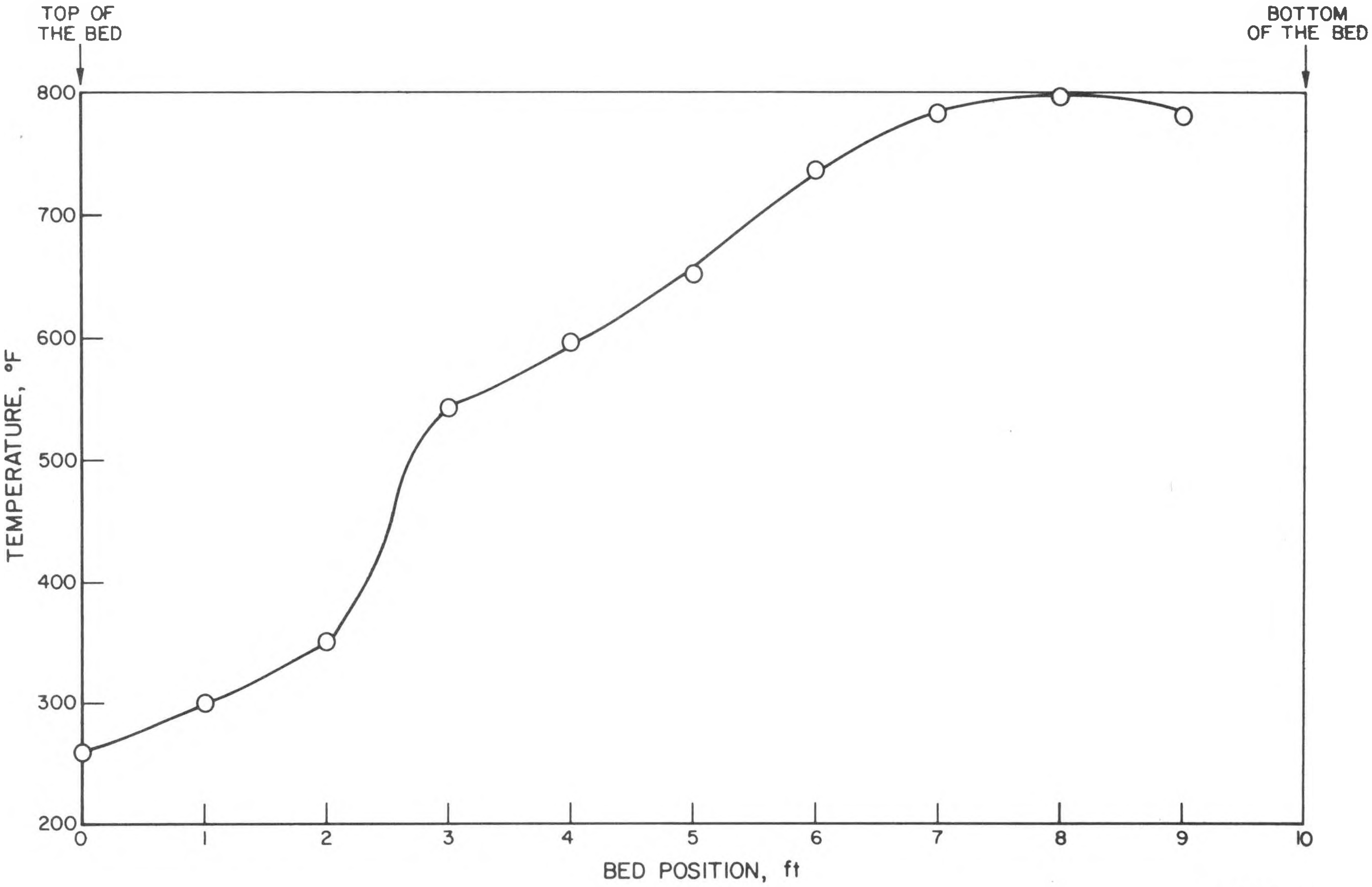

Figure 44. STAGE 1 BED TEMPERATURE PROFILE FOR RUN 81PDU-1
(Time: Steady-State Average)

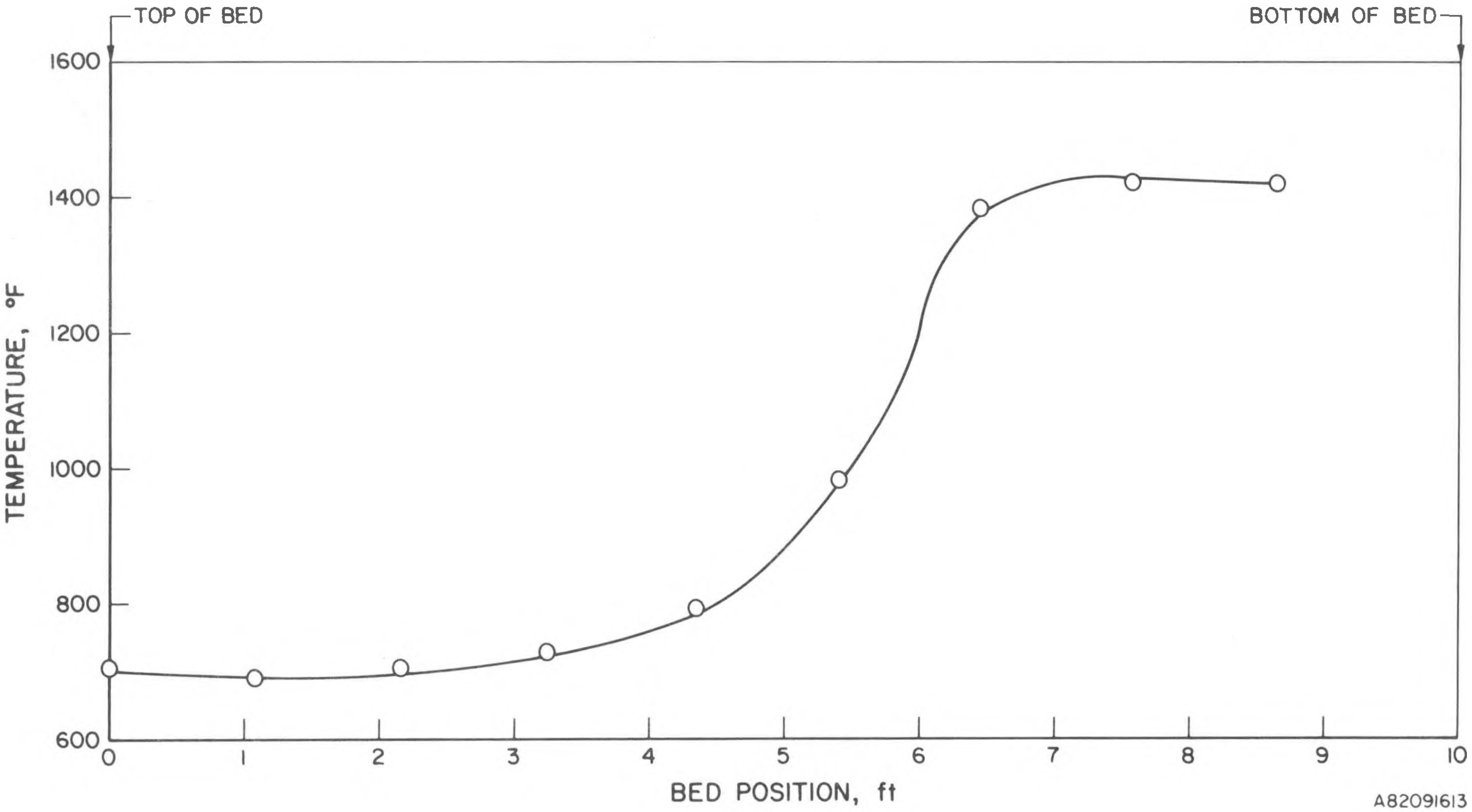

Figure 45. STAGE 2 BED TEMPERATURE PROFILE FOR RUN 81PDU-1
(Time: Steady-State Average)

Company from the Jacobs Engineering Group, Inc., was retained as a subcontractor to assist IGT in the compilation and interpretation of the data base.

The Phase I final report has been revised by Pace and is included in Appendix B. The Pace subcontract originally involved three work phases. However, to minimize program costs, the Phase II and Phase III work was not completed.

In the Pace Phase I report, the HYTORT process concept is reviewed for use on both Eastern and Western shales and questions about major environmental effects are formulated for commercial HYTORT plant operations. The recommended PDU sampling and analysis program includes the raw and spent shales, retort gases, process water, and the shale oil. In general, the origin and fate of pollutants regulated by the Federal Water Pollution Control Administration (FWPCA) and the U.S. Environmental Protection Agency (EPA) "priority pollutants" are given the highest priorities. Additionally, pollutants on the EPA's A, B, and C pollutant lists are also considered because their ambient levels can affect process and run-off water treatment, usage, or disposal. Another important category includes pollutants that may be covered in proposed regulations for prevention of significant deterioration (PSD) to a plant's geographic air space. The PDU environmental program focuses on as many of these pollutants as possible within experimental and budgetary constraints.

A sampling probe has been placed into the second stage of the PDU, to collect samples for accurate environmental characterization of the HYTORT process gases and condensates. This system provides sample removal at the operating temperature and pressure of the reactor. The sample passes through an electrostatic precipitator and, after pressure reduction, is introduced into a condenser and then through a series of sample bombs, each containing scrubbants to recover specific organic and inorganic species. To promote high sampling efficiencies, two or three sampling bombs containing the same preservative solution are used in series. Major environmental species are captured in these bombs, and the liquid samples are preserved for chemical analysis. The exit-gas flow rate is measured to provide a means of calculating the actual species loadings in the gas stream. A schematic diagram of the sampling train and a compilation of environmental data have been reported previously.

Table 27 reports the completed trace element analyses for feed and residue oil shales from the PDU test program. If no reaction of a specific trace element occurs during hydroretorting then the element concentration in the residue shale should proportionately increase by the weight ratio of the feed shale to residue shale as shown in Equation 4. By mass balance —

$$(C_i)_F \, W_F = (C_i)_R \, W_R \qquad (3)$$

and —

$$\frac{(C_i)_R}{(C_i)_F} = \frac{W_F}{W_R} \qquad (4)$$

where —

W_F = shale fed, lb

W_R = residue shale, lb

C_i = concentration of element i, wt %

Trace element concentration ratios are compared to feed and residue weight ratios in Table 28. In general, many of the trace element ratios are in good agreement (within 10%) with the shale-feed-to-residue ratios. However, as evidenced by good agreement for only four trace element concentrations (F, Mn, Ni, and V) in two samples of Kentucky New Albany feed shale (Runs 80PDU-2 and 80PDU-7, Table 26), this data base needs to be enlarged so that sampling errors can be estimated and firm conclusions drawn on the fate of trace elements during oil shale hydroretorting.

Task 5. Process Design and Economics

In this task process design and economics studies for the conversion of Western Eocene shale and Eastern Devonian shale were made to evaluate the potential of various oil shales as feedstocks for oil and to help guide the experimental program. A mathematical model to predict gas and shale temperature profiles in moving-bed-type retorts was also developed.

Process Design and Cost Estimate for Conversion of Eastern Devonian (Sunbury) Oil Shale to 51,915 bbl/SD of Syncrude by the HYTORT Process

Introduction

This study presents a design for the manufacture of syncrude from Eastern (Kentucky Sunbury) oil shale as a companion study to that based on Western (Colorado) oil shale. In an earlier program[4] process designs and economic studies were performed for producing syncrude and SNG from Kentucky New Albany

Table 27. TRACE ELEMENT ANALYSES FOR OIL SHALES USED IN PDU TESTS

Run No.	80PDU-1		80PDU-2		80PDU-3		80PDU-4	80PDU-5	80PDU-6	80PDU-7		81PDU-1	
Shale	Ohio-Cleveland Member		Kentucky New Albany		Ohio-Cleveland Member		Kentucky New Albany	Kentucky Sunbury	Kentucky New Albany	Kentucky New Albany		Chattanooga Gassaway Member	
Sample	Feed	Residue	Feed	Residue	Feed	Residue	Residue	Residue	Residue	Feed	Residue	Feed	Residue
	ppm												
Al*	–**	–	–	6.2	–	8.5	6.3	9.6	5.8	5.1	6.4	6.7	5.8
Sb	–	–	–	<0.2	–	<0.2	<0.2	23.3	<0.2	<0.2	<0.2	0.60	1.43
As	32	55	56	65	–	48	70	52	61	48	70	105	106
Ba	61	54	53	74	43	96	103	107	68	24	75	23	30
Be	2.8	3.1	5.5	6.0	5.0	10.0	3.3	3.3	3.2	2.3	3.2	–	–
B	<100	<100	<100	<100	<100	< 100	<80	<80	<80	<80	<80	–	–
Cd	5.2	5.0	–	–	–	–	–	–	–	–	–	–	–
Cl	–	–	–	220	–	88	248	74	171	208	178	–	–
Co	5.5	6.6	33	37	–	–	–	–	–	–	–	–	–
Cr	130	150	60	75	140	150	87	150	123	–	125	–	–
Cu	140	110	–	180	–	181	118	202	113	95	121	–	–
F	240	300	240	240	–	220	280	250	350	250	350	302	358
Fe*	2.2	2.6	–	7.0	–	4.0	7.6	4.8	7.0	5.3	6.9	–	–
Pb	15	18	–	96	–	46	98	78	183	109	120	–	–
Li	45	48	30	30	57	50	–	–	–	–	–	–	–
Mn	48	57	220	290	51	81	295	193	260	212	260	–	–
Mo	74	56	–	–	–	–	–	–	–	–	–	–	–
Hg*	140	26	–	–	–	–	–	50	–	–	–	–	–
Ni	104	100	110	140	120	110	130	350	148	115	140	–	–
Se	10	5.6	–	0.81	1.0	1.6	0.7	11.9	0.9	1.11	1.82	1.26	0.81
Ag	–	–	–	2.3	–	1.6	0.35	0.44	0.23	0.83	0.43	–	–
Sr	–	–	–	82	–	74	80	72	73	72	78	–	–
Tl	0.38	0.73	–	–	–	–	–	–	–	–	–	–	–
V	130	140	170	210	–	660	220	2800	211	171	366	–	–
Zn	210	244	72	92	230	280	73	1890	71	43	288	–	–

* Al and Fe in wt %, Hg in ppb.
** Analysis not performed.

Table 28. RATIO OF TRACE ELEMENT CONCENTRATIONS IN RESIDUE SHALE TO THAT IN FEED SHALE — PDU TESTS

Run No.	80PDU-1	80PDU-2	80PDU-3	80PDU-7	81PDU-1
Shale Type	Ohio-Cleveland Member	Kentucky New Albany	Ohio-Cleveland Member	Kentucky New Albany	Chattanooga Gassaway Member
R_2*	1.26	1.25	1.24	1.25	1.14
Al	-**	-	-	1.25	0.87
Sb	-	-	-	-	2.38
As	1.72	-	-	1.46	1.01
Ba	0.88	1.16	2.23	3.13	1.30
Be	1.11	1.40	2.00	1.39	-
Cd	0.96	-	-	-	-
Cl	-	-	-	0.86	-
Co	1.20	1.12	-	-	-
Cr	1.15	1.25	1.07	-	-
Cu	0.79	-	-	1.27	-
F	1.25	1.00	-	1.40	1.18
Fe	1.18	-	-	1.30	-
Pb	1.20	-	-	1.10	-
Li	1.07	1.00	0.88	-	-
Mn	1.19	1.32	1.59	1.23	-
Mo	0.77	-	-	-	-
Hg	0.19	-	-	-	-
Ni	0.96	1.27	0.92	1.22	-
Se	0.56	-	1.60	1.64	0.64
Ag	-	-	-	0.52	-
Sr	-	-	-	1.08	-
Tl	1.92	-	-	-	-
V	1.08	1.24	-	2.14	-
Zm	1.17	1.28	1.22	6.70	-

---

* R_2 = ratio of shale feed to residue shale, lb/lb.

** Indicates ratio not available.

oil shale by hydroretorting. An existing process design for the manufacture of raw oil from Sunbury oil shale was utilized here that gives a reasonable appraisal of the process. A raw oil hydrotreating operation was added for consistency with the Colorado oil shale case. Sizing and estimation of costs for the different sections of the plant are based on the Colorado case wherever applicable. This was cost- and time-effective and also made for better comparability between the two studies. In keeping with this objective, costs are based on January 1, 1980, as in the Colorado oil shale case.

Process Description

Figure 46 is the flow diagram for the process. Compositions and flow rates for the process streams are given in Table 29. The size of this plant is based on the production of the same mass quantity of raw shale oil as in the Colorado oil shale case — 715,714 lb/h. Because the oil from the Sunbury shale has a higher density, the volumetric output is 9% less. While it is, of course, understood that the petroleum industry rates plants, production, and refining in volumetric terms (as in this plant), sizing the two plants for equal pounds of raw oil makes for a closer comparison of energy output from the shale hydroretorting operation.

Because of the lower oil yield from the Sunbury shale, more of this shale must be mined and handled than in the Colorado design — 100,500 tons/SD to give 91,365 tons/SD of shale feed (3% moisture) fed to the reactor proper after passing through the lockhopper feed system.

The reactor pressure is substantially higher than for the Colorado shale design — 575 versus 415 psia. This design is also based on a high hydrogen recycle through the reactor with hydrogen recovery by cryogenic separation. The input hydrogen/shale ratio is 15.2 SCF/lb dry shale. The average of inlet and outlet hydrogen pressures for the Eastern shale design is 518 psia. Conversion of Sunbury shale organic carbon, based on a specific test, is 75.1%. Most of the hydrogen entering the HYTORT reactor passes through unreacted and the recycle hydrogen stream is recovered by low-temperature separation. Twenty-seven percent of the reactor feed gas is preheated in a gas-fired furnace. The hot, spent shale is cooled by heat exchange with the other 73%. All the methane and higher hydrocarbons produced in the reactor are used in generating hydrogen and for plant fuel. The amount of heat input required from the fired heater depends on the heat of reaction of the shale.

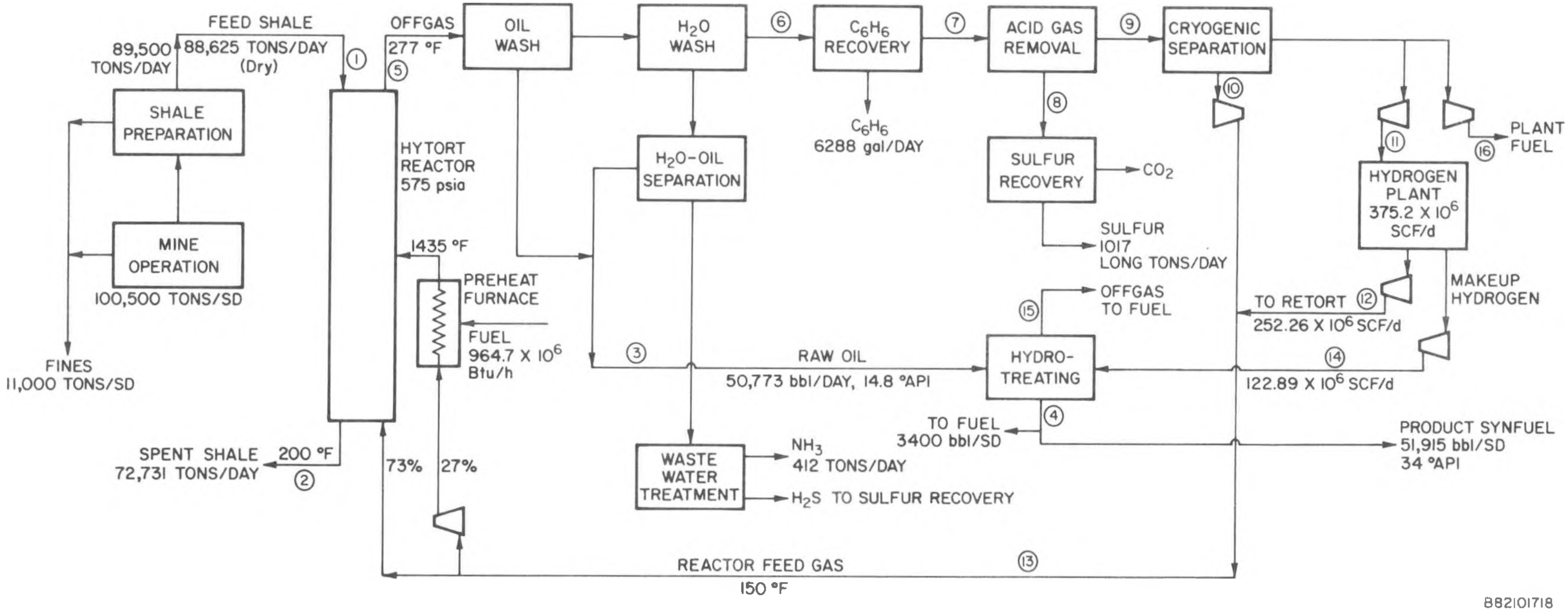

Figure 46. BASIC FLOW DIAGRAM FOR SYNCRUDE FROM EASTERN (Sunbury) OIL SHALE BY THE HYTORT PROCESS

Table 29. COMPOSITIONS AND FLOW RATES FOR PROCESS STREAMS FOR PRODUCTION OF 51,915 bbl/SD OF SYNFUEL FROM EASTERN (Sunbury) OIL SHALE BY THE HYTORT PROCESS

Stream No.	1		2		3		4	
Name	Shale to Retort		Spent Shale		Raw Shale Oil		Hydrotreated Oil	
Temperature, °F	80		200		100		100	
Pressure, psia	575		580		565		75	
Component	lb/h	wt %	lb/h	wt %	lb/h	wt %	lb/h	wt %
Organic C	1,110,760	15.04	276,378	4.56	593,684	82.95	586,273	85.07
H	126,290	1.71	11,515	0.19	66,132	9.24	84,967	12.33
O	277,690	3.76	29,092	0.48	31,563	4.41	17,211	2.50
N	40,620	0.55	9,093	0.15	14,744	2.06	482	0.07
S	199,405	2.70	104,246	1.72	9,591	1.34	207	0.03
Mineral Matter	5,630,610	76.24	5,630,610	92.90	--	--	--	--
Subtotal (Dry)	7,385,375	100.00	6,060,934	100.00	715,714	100.00	689,140	100.00
Free H_2O	228,415	--	--	--	--	--	--	--
Total	7,613,790	100.00	6,060,934	100.00	715,714	100.00	689,140	100.00

Stream No.	5		6		7		8		9		10	
Name	Retort Offgas		Scrubbed Retort Offgas		Feed to Acid Gas Removal		Acid Gas to Sulfur Recovery		Feed to Low Temp. Separation		Recycle Gas	
Temperature, °F	277		100		100		140		100		145	
Pressure, psia	575		565		545		20		540		595	
Component	mol/h	mol %	mol/h	mol %	mol/h	mol %	mol/h	mol %	mol/h	mol %	mol/h	mol %
H_2	272,458.3	81.84	272,458.3	90.31	272,458.3	90.32	--	--	272,458.3	91.04	269,734.6	95.71
CO	5,672.0	1.70	5,672.0	1.88	5,672.0	1.88	--	--	5,672.0	1.90	5,104.8	1.81
CO_2	302.5	0.09	273.0	0.09	273.0	0.09	302.5	9.25	--	--	--	--
CH_4	17,388.1	5.23	17,388.1	5.76	17,388.1	5.76	--	--	17,388.1	5.81	6,989.5	2.48
C_2H_6	2,142.4	0.64	2,142.4	0.71	2,142.4	0.71	--	--	2,142.4	0.72	--	--
C_3H_8	478.9	0.14	478.9	0.16	478.9	0.16	--	--	478.9	0.16	--	--
C_4H_{10}	315.2	0.09	315.2	0.10	315.2	0.10	--	--	315.2	0.11	--	--
C_2H_4	214.2	0.06	214.2	0.07	214.2	0.07	--	--	214.2	0.07	--	--
C_3H_6	264.7	0.08	264.7	0.09	264.7	0.09	--	--	264.7	0.09	--	--
C_4H_8	302.5	0.09	302.5	0.10	302.5	0.10	--	--	302.5	0.10	--	--
C_6H_6	25.1	0.01	25.1	0.01	--	--	--	--	--	--	--	--
NH_3	1,198.2	0.36	--	--	--	--	--	--	--	--	--	--
H_2S	2,631.6	0.79	2,132.9	0.71	2,132.9	0.71	2,629.2	89.49	--	--	--	--
COS	36.9	0.01	36.9	0.01	36.9	0.01	36.9	1.26	--	--	--	--
H_2O	25,034.1	7.53	--	--	--	--	--	--	--	--	--	--
Shale Oil	4,473.2	1.34	--	--	--	--	--	--	--	--	--	--
Total	332,937.9	100.00	301,704.2	100.00	301,679.1	100.00	2,968.6	100.00	299,236.3	100.00	281,828.9	100.00

Stream No.	11		12		13		14		15		16	
Name	Feed and Fuel To H_2 Plant		Makeup H_2 To Reactor		Reactor Feed Gas		Makeup H_2 to Hydrotreater		Hydrotreater Offgas		High-Btu Gas Plant Fuel	
Temperature, °F	230 (Feed)		320		150		100		200		200	
Pressure, psia	350 (Feed)		595		580		2500		40		40	
Component	mol/h	mol %	mol/h	mol %	mol/h	mol %	mol/h	mol %	mol/h	mol %	mol/h	mol %
H_2	2,427.9	15.64	26,937.0	97.0	296,671.7	95.82	13,122.0	97.0	160.1	35.00	295.8	15.64
CO	505.6	3.26	--	--	5,104.8	1.65	--	--	--	--	61.6	3.26
CO_2	--	--	--	--	--	--	--	--	--	--	--	--
CH_4	9,269.3	59.73	833.9	3.0	7,822.6	2.53	405.8	3.0	120.8	26.41	1,129.5	59.73
C_2H_6	1,909.7	12.31	--	--	--	--	--	--	80.1	17.51	232.7	12.31
C_3H_8	426.8	2.75	--	--	--	--	--	--	57.8	12.64	52.0	2.75
C_4H_{10}	280.9	1.81	--	--	--	--	--	--	38.6	8.44	34.2	1.81
C_2H_4	190.9	1.23	--	--	--	--	--	--	--	--	23.3	1.23
C_3H_6	255.9	1.53	--	--	--	--	--	--	--	--	28.7	1.53
C_4H_8	269.7	1.74	--	--	--	--	--	--	--	--	32.9	1.74
C_6H_6	--	--	--	--	--	--	--	--	--	--	--	--
NH_3	--	--	--	--	--	--	--	--	--	--	--	--
H_2S	--	--	--	--	--	--	--	--	--	--	--	--
COS	--	--	--	--	--	--	--	--	--	--	--	--
H_2O	--	--	--	--	--	--	--	--	--	--	--	--
Shale Oil	--	--	--	--	--	--	--	--	--	--	--	--
Total	15,516.7	100.00	27,770.1	100.0	309,599.1	100.00	13,527.8	100.0	457.4	100.00	1,890.7	100.00

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This design is based on an estimated endothermic heat of reaction of 40 Btu/lb of dry shale. Much of the heat input is incompletely recovered and is carried out of the reactor with the effluent gas and spent shale streams.

The effluent gas and oil leaves at 277°F after preheating the raw shale and vaporizing the water. At the exit conditions all the water and 8% of the oil are in the vapor state. Most of the oil is thus assumed present as a mist, which is extracted in the primary separator.

The gas is then sent to a direct contact washer-cooler where the water is condensed and the ammonia is dissolved. This ammonia plus small amounts of H_2S and CO_2 are recovered by the Chevron process. The scrubbed gas is sent to acid-gas removal and low-temperature separation. Ninety-four percent of the scrubbed gas is recycled to the reactor.

The Eastern oil shale produces about 10% as much CO_2 as was made from Colorado oil shale, about 6 times as much H_2S, and 3.5 times as much ammonia. Because of the low exit gas CO_2 concentration, a DGA scrubbing unit was selected instead of a Selexol unit as was used in the Western shale case. The H_2S/CO_2 ratio is very favorable for a Claus sulfur recovery unit. Total sulfur recovery is 1017 long tons/day. Ammonia by-product is 412 tons/day. Benzene at a rate of 6288 gallons/day is recovered as a by-product by activated carbon adsorption.

Reformer feed plus fuel for hydrogen manufacture is supplied by stream 11. This contains about 60% CH_4, 21% heavier hydrocarbons, and the rest H_2 and CO. A conventional natural-gas-fueled hydrogen plant is assumed here. This is considered to include a methanation unit, given a 97% H_2 plus 3% CH_4 product. Total hydrogen production is 375.2 million SCF/day, two-thirds of which is makeup for retorting and one-third for hydrotreating. This is 28% more hydrogen than for the Colorado oil shale case. For the Sunbury shale, 63% more makeup hydrogen is needed for retorting and 6% less is needed for hydrotreating the product oil.

Raw shale oil, 14.8°API, is manufactured at a rate of 50,773 bbl/SD or 715,714 lb/h. This oil is upgraded by conventional catalytic hydrotreating to reduce sulfur and nitrogen to low levels. The product characteristics and hydrotreater offgas yields are based on IGT test data (Subtask 2.3) with a nickel-molybdenum catalyst. The gas produced from hydrotreating Sunbury shale oil amounts to less than one-half (expressed as weight percent of the product oil) of that produced from Colorado shale oil. As a result, a higher proportion of the feed hydrogen appears in the product oil. The hydrogen consumption amounts to 2347 SCF/bbl of oil feed. The hydrotreating design pressure is 2000 psig. In lieu of a detailed design, costs and utilities for hydrotreating are based on the same source as the design for Colorado shale.

The heating value of the product oil, based on correlations, is 5.76 million Btu/bbl. Of total plant fuel requirements of 1841.6 million Btu/h, 56% is supplied by retort and hydrotreater gas. The remainder is supplied by 3400 bbl/SD of hydrotreated oil, leaving a net product of 51,915 bbl/SD. Table 30 shows a comparison of the properties of raw and hydrotreated shale oil.

Table 30. COMPARISON OF PROPERTIES OF CRUDE AND HYDROTREATED SUNBURY SHALE OIL

	Raw Oil	Hydrotreated Oil
°API	14.8	34
C/H Weight Ratio	8.98	6.90
	----------- wt %	------------
C	82.95	85.07
H	9.24	12.33
O	4.41	2.50
N	2.06	0.07
S	1.34	0.03
Total	100.00	100.00
lb/h	715,714	689.140
bbl/SD	50,773	55,315

Plant Utilities and Overall Efficiency

Tables 31, 32, and 33 summarize, respectively, the power, steam, and cooling water requirements. Steam requirements are rather modest for synfuels plants of this size, compared with an SNG-from-coal plant where all the power requirements are generated onsite and a large amount of CO_2 is made in the gasification step. The major steam usage is in regenerating the DGA solution used to absorb H_2S and CO_2.

Table 31. SUMMARY OF POWER REQUIREMENTS FOR CONVERSION OF EASTERN (Sunbury) OIL SHALE TO 51,915 bbl/SD OF SYNCRUDE

Service	hp
Shale Handling	2,100
Shale Preparation	9,400
Shale Feed System	9,430
Reactor Effluent Scrubber	1,630
Recycle Gas Compressor	52,316
Hydrogen Plant Reformer Feed Compressor	19,750
Hydrogen Plant	21,800
Retort Make-up Hydrogen Compressor	17,400
Hydrotreater Make-up Hydrogen Compressor	22,220
Hydrotreater	10,900
Preheated Retort Gas Booster Compressor	5,500
Fuel Gas Compressor	3,860
Acid Gas Removal	1,810
Sulfur Recovery	4,360
Low-Temperature Separation	2,420
Waste Water Treatment	1,415
Boiler Feed Water Pump	150
Cooling Water Pumps	8,460
Spent Shale Disposal	2,100
Miscellaneous	10,000
Total Power	207,021

154,375 kW Purchased

Table 32. SUMMARY OF STEAM REQUIREMENTS FOR CONVERSION OF EASTERN (Sunbury) OIL SHALE TO 51,915 bbl/SD OF SYNCRUDE

50 psig, 298°F	lb/h
Acid Gas Scrubbing	369,100
Waste Water Treatment	171,765
Activated Carbon Regeneration	10,045
	550,910
Less By-Product Steam From Sulfur Recovery	216,590
Net Required	334,320

Boiler Duty, 304.76 X 10^6 Btu/h

150 psig, 366°F	lb/h
Hydrotreating	73,000
Waste Water Treatment	56,580
	129,580

Boiler Duty, 111.05 X 10^6 Btu/h

Total Boiler Duty, 415.81 X 10^6 Btu/h

Table 33. SUMMARY OF COOLING WATER REQUIREMENTS FOR CONVERSION OF EASTERN (Sunbury) OIL SHALE TO 51,915 bbl/SD OF SYNCRUDE

Service	gal/min
Retort Effluent Cooling	54,680
Acid Gas Removal	22,399
Sulfur Recovery	3,343
Low-Temperature Separation	915
Hydrogen Plant	33,867
Hydrotreating	6,000
Waste Water Treatment	2,425
Benzene Recovery	649
Compressor Intercoolers	3,850
Total	128,128

Because of the high oil product quality and value, it does not seem sensible to generate electric power onsite by burning the oil. To do this would require about 6272 bbl/SD of product. Because of the proximity of coal-generated power in the Eastern United States, it would be better to import it from large plants that generate power for less cost. Total imported power is 15,375 kW, much more than for the Colorado shale design. The major reason for this is the much larger power need of the gas compressors. The recycle gas compressors above use 52,300 hp. The use of cryogenic separation results in a loss of pressure in the hydrocarbon-rich streams, requiring recompression.

Steam requirements are higher than for the Colorado shale design, requiring about 83 million Btu/h more boiler duty.

Cooling water requirements are 70% higher than for the Colorado shale case. Because all of the water in the shale reactor effluent gas is in the vapor state and the amount of gas to be cooled is much greater, the cooling water needs are correspondingly higher. Acid-gas removal requires more cooling water because of the higher steam usage.

Table 34 summarizes the overall plant efficiency of products as a percentage of the heating value of mined shale at 2642 Btu/lb. The product oil plus by-products heating values amount to 56.37% of the mined shale plus fuel to generate the imported electric power at 35% efficiency. The product oil yield is 24.6 gal/ton of dry oil shale fed to the reactor. This compares very well with the 25.2 gal/ton yield of the Colorado oil shale case. This shows the benefits of the HYTORT process in raising the resource potential of Eastern shale to a level comparable to the Western oil shale.

The biggest single factor in reducing overall efficiency relative to the Colorado shale case is the 14% higher Btu/lb H.H.V. for the Sunbury shale.

Process Economics

As noted earlier, costs have been based on January 1980, to be on a comparable basis with the Colorado oil shale case.

Estimates have been made of the plant investment, annual operating costs, revenue requirements, and product prices. Four different cases for private investor financing were developed using equations developed for the Colorado oil shale study.

Table 34. OVERALL PLANT EFFICIENCY FOR CONVERSION OF EASTERN (Sunbury) OIL SHALE TO 51,915 bbl/SD OF SYNCRUDE

IN	10^6 Btu/h	% of Input
Mixed Oil Shale H.H.V. 100,500 tons/SD, 1% H_2O, 2642 Btu/lb dry	21,905.48	93.57
Electric Power, 154,375/kW, 35% efficiency	1,505,38	6.43
	23,410.86	100.00
OUT		
Oil, Syncrude, Product H.H.V. 646,770 lb/h 19,250 Btu/lb (5.76 X 10^6 Btu/h)	12,450.32	53.18
Benzene, 6288 gal/day	34.92	0.15
Ammonia, 412.3 tons/day	332.17	1.42
Sulfur, 1017 long tons/day	378.10	1.62
	13,195.51	
Overall Efficiency		56.37

1. 100% equity financing with 12% DCF rate of return
2. A debt-to-equity ratio of 25/75 with 12% DCF rate of return for the equity portion and 9% interest rate on the debt portion
3. A debt-to-equity ratio of 50/50 with 12% DCF rate of return for the equity portion and 9% interest rate on the debt portion
4. 100% equity financing with a 15% DCF rate of return.

Plant Investment

The use of four different financial methods does not affect the basic investment cost. However, because of the additions due to interest during construction, shale cost, start-up, and working capital, there are four separate numbers for total investment corresponding to the basic plant investment (T.P.I).

This number is $1183 million; 23%, or $222 million, more than the Colorado case. The total installed plant cost ex contingencies, is higher by $174 million. Nearly all plant sections are higher in cost. Increased shale usage, higher process gas and hydrogen rates, and increased utilities all contribute to the increased cost of the plant, due, in general, to the required increases in capacities for the different sections. The cost of hydrotreating was not changed. The largest single cost item is the hydrogen plant, followed by hydrotreating. Together with feed compressors they represent, respectively, 15.4% and 13.7% of the total plant cost. The HYTORT reactors, per se, are a small percentage of the total, indicating (as in coal gasification) that we cannot focus attention solely on the reactor in appraising a process. Reactors and feed systems constitute 9.2% of the total cost. Shale storage and preparation costs are 11% of the total cost. Total capital ranges from $1525 to $1647 million (Table 35).

Shale Cost

Estimation of the cost of mining Eastern oil shale is beyond the scope of this work. Therefore, we have used the same basic costs as in the Colorado study. The mining and handling of large quantities of rock pose the same problems for Eastern and Western locations. Published studies indicate that costs probably should be similar. The effect of varying shale cost has been calculated and is discussed later.

The four different financing methods also give four different base shale costs.

Table 35. SUMMARY OF INVESTMENT COSTS FOR PRODUCTION OF 51,915 bbl/SD OF OIL FROM EASTERN (Sunbury) OIL SHALE BY THE HYTORT PROCESS
(January 1980 Cost Basis)

Plant Section	$\$10^6$
Shale Storage	15.9
Shale Preparation	85.7
Shale Retort Feed and Discharge	29.8
Hydroretorts	55.4
Retort Effluent Scrubbing, Cooling, Oil-Gas Separation	69.9
Acid Gas Removal	21.5
Sulfur Recovery, Tail Gas Treating	30.0
Retort Feed Gas Preheat	59.6
Hydrogen Plant	136.0
Hydrotreating Section	122.0
Benzene Recovery	2.4
Hydrogen Plant Feed Compressors	7.1
Retort Make-up Hydrogen Compressors	4.7
Recycle Gas Compressors	14.0
Hydrotreater Make-up Hydrogen Compressors	4.6
Fuel Gas Compressors	3.9
Preheated Retort Gas Booster Compresser	3.7
Low-Temperature Separation	34.0
Steam Generation	16.6
Cooling Water Supply	7.6
Electric Power Distribution	34.1
Make-up and Boiler Feed Water Treatment	13.3
Blowdown Water Treatment	28.0
Waste Water Treatment	27.5
Particulate Emission Control	5.0
General Facilities	94.3
Installed Plant Cost, Excluding Contingencies	926.6
Contingencies, 15%	139.0
Installed Plant Cost	1065.6
Contractor's Overhead and Profits, 11%	117.2
Total Plant Investment (T.P.I.)	1182.8

Debt/Equity Ratio	0/100	25/25	50/50	0/100
DCF Rate of Return	12			15
Interest on Debt	--	9	9	--
Return on Investment During Construction	266.1	249.6	232.9	332.7
Start up Cost, 20% of Gross Operating Cost	63.3	60.3	58.0	67.6
WCAP				
30 Days Raw Materials	22.0	20.6	19.5	23.9
Supplies (0.009 X T.P.I)	10.7	10.7	10.7	10.7
1/24 Revenue	24.6	22.2	20.2	29.2
	57.3	53.5	50.9	63.8
Total Capital	1569.5	1546.2	1524.6	1646.9

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Financial Factors	Shale Cost, $/ton
12% DCF, 100% Equity	4.60
15% DCF, 100% Equity	5.25
25/75 Debt/Equity, 12% DCF, 9% Interest	4.14
50/50 Debt/Equity, 12% DCF, 9% Interest	3.80

The original basis for shale costs is in published information. Further discussion of the background and derivation of these costs is given in Reference 6.

Catalysts and Chemicals

The major item in this category is the hydrotreating catalyst, as indicated in Table 36. This accounts for $1.29/bbl of product cost.

Table 36. ANNUAL COST OF CATALYSTS AND CHEMICALS

	$1000/Year
Hydrotreating Catalyst	22,000
Hydrogen Manufacture	940
Activated Carbon	10.4
DGA Chemical	150
Water Treatment	1,650
	24,750.4

Annual Operating Costs and Revenue

Table 37 summarizes the annual operating costs and required revenue for the annual production of 17.054 million bbl of syncrude.

Product prices are based on annual revenues calculated for private investor financing. The method used is based on a DCF return on equity for varying debt/equity ratios. The basic approach is adapted from the DCF method 100% equity financing of the Supply-Technical Advisory Task Force — Synthetic Gas Coal of the FPC. Financial factors are summarized below.

Project Life	20 years
Depreciation	16-year sum-of-the-years'-digits
Debt/Equity Ratio	0/100, 25/75, 50/50
% Return	Equity, 12% and 15% DCF; Interest, 9%
Federal Income Tax	48%
Return on Investment During Construction	Average return rate X 1.875* years X total plant investment

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*10% for 3 years, 90% for 1.75 years.

Plant Stream Factor	90%
Contingencies	15% of installed plant cost
Contractor's Overhead and Profit	11% of total plant cost
Start-up Cost[†]	20% of gross operating costs
Working Capital	Raw materials inventory (30-day feed at full rate) Material and supplies (0.9% of total plant investment) Net receivables (1/24 X annual revenue received)

Formulas for calculating annual revenues and the net income for the four financial methods are outlined in Reference 6.

The major operating cost is the cost of the oil shale, which ranges from 43% to 51% of gross operating costs, and varies from $7.3 to $10.1/bbl of product. Purchased electric power is the second most costly item, amounting to $3.57/bbl of product. Operating labor and maintenance are rather small — less than 10% of total revenue, and equivalent to about $2.66/bbl. Direct operating labor is the same as the Colorado shale case: 55 men/shift at $9.25/hr. Capital charges — depreciation, net income, and Federal income tax — are the largest component of product price. The total of these ranges from 44% to 54% of required revenue for the four sets of financial factors.

The price of syncrude ranges from $28.35/bbl for the 50/50 debt-to-equity ratio case with 12% DCF and 9% interest to $41.11 for 100% equity with 15% DCF return. These prices are 14% to 15% higher than the corresponding ones for the Colorado oil shale design.

Sensitivities to Change in Shale Cost and Plant Investment

Figure 47 shows the effect of shale cost on syncrude price. There is only a slight variation among the four methods, the typical sensitivity being just over $2/bbl for a $1/ton change in shale cost.

The effect of variations in plant cost is shown in Figure 48. Here the variation is in basic installed plant cost before addition of factors for contingency, contractor charges, interest during construction, start-up, and

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[†] This cost is not depreciated but is expensed immediately before the first operating year as part of net cash flow.

Table 37. SUMMARY OF ANNUAL OPERATING COSTS AND REVENUE REQUIREMENTS FOR PRODUCTION OF 51,915 bbl/SD OF SYNCRUDE FROM EASTERN (Sunbury) OIL SHALE BY THE HYTORT PROCESS (90% Stream Factor, January 1980 Cost Basis)

Financing Method	---- Private Financing, Discounted Cash Flow ----			
Debt/Equity Ratio	0/100	25/75	50/50	0/100
Discounted Cash Flow Rate of Return, %	--------------- 12 ---------------			15
Interest On Debt, %	--	9	9	--
Raw Shale Cost, $/Ton	4.6	4.14	3.80	5.25
Annual Operating Costs	------------------ $$10^6$/Year ------------------			
Raw Materials				
Oil Shale, 100,500 Tons/Day	151.86	136.67	125.45	173.32
Catalysts and Chemicals	24.75			
Make-Up Water at $1/1000 gal	3.02			
Electric Power, 154,375 kW at 5¢/kWh	60.86			
Labor				
Direct Operating Labor, 55 Men/Shift at $9.25/h	4.46			
Supervision, 20% of Direct Operating Labor	0.89			
Administrative and General Overhead, 60% of Direct Operating Labor and Supervision	3.21			
Operating Supplies, 30% of Direct Operating Labor	1.34			
Maintenance, 3% of Total Plant Investment	35.48			
Local Taxes and Insurance, 1.5% of Total Plant Investment	17.74			
Shale Fines Disposal at $4.25	13.00			
Gross Operating Costs	316.61	301.42	290.20	338.07
Less By-Product Credits				
Benzene, 6288 gal/Day at $1/gal	-2.07			
Sulfur, 1079 Long Tons/Day at $20/Long Ton	-6.68			
Ammonia, 412 Long Tons/Day at $100/Ton	-13.53			
Net Operating Costs (N)	294.33	279.14	267.92	315.79
Depreciation (20-Year Equivalent Average)*	72.45	71.62	70.79	75.78
Net Income	114.43	93.81	73.81	159.17
Federal Income Tax	105.63	86.59	68.13	146.92
1/20 of Initial Start-Up Expense	3.17	3.00	2.90	3.38
Total Revenue Required	590.01	534.16	483.55	701.04
Annual Syncrude Production, 10^6 bbl	-------------------- 17,054,077 --------------------			
Syncrude Price, $/bbl	34.60	31.32	28.35	41.11

* Total plant investment and return on investment during construction depreciated over 20 years.

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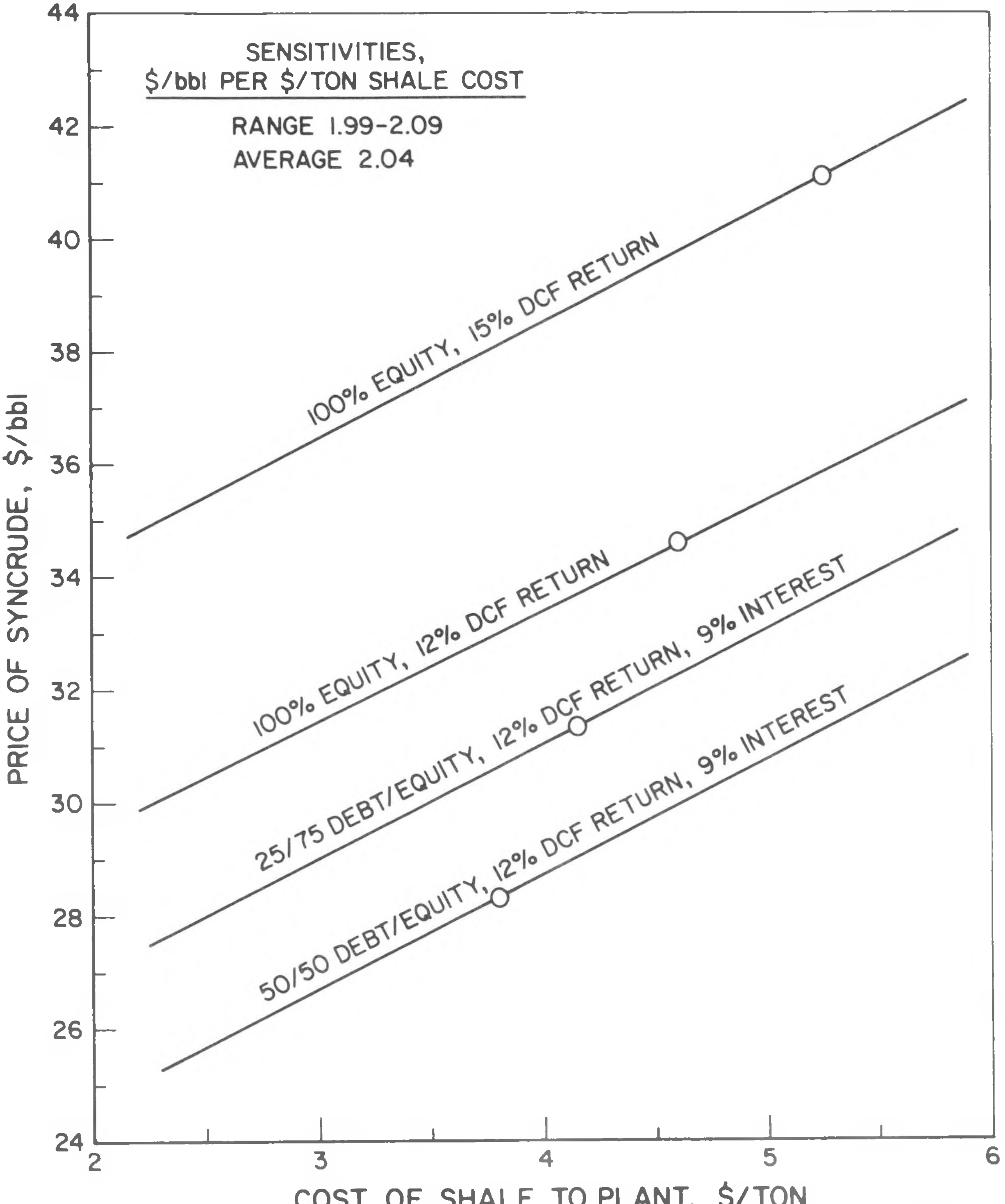

Figure 47. EFFECT OF SHALE COST ON SYNCRUDE PRICE WITH DIFFERENT FINANCIAL FACTORS

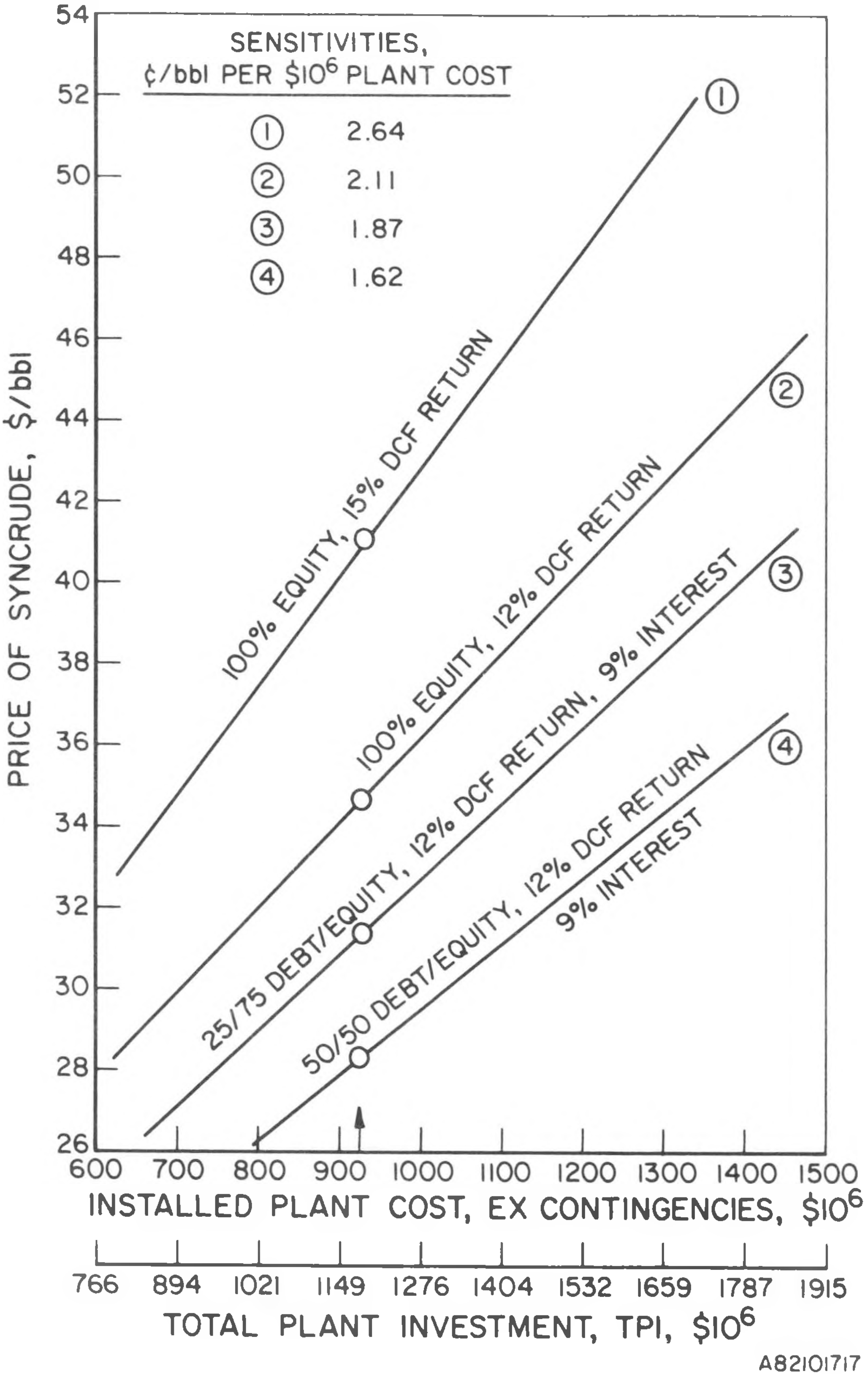

Figure 48. EFFECT OF PLANT COST ON SYNCRUDE PRICE WITH DIFFERENT FINANCIAL FACTORS

working capital. These factors add from 65% to 78% to the installed plant cost. Relating sensitivity to the latter provides an estimate of the effect of changes in the cost of specific installed equipment on the price of syncrude. A $1 million change in installed cost affects the product cost by only a small amount. As both the percentage equity and DCF return increase, the sensitivity increases, as can be seen by the increasing slopes of the lines in Figure 48. The range is 1.62¢ to 2.64¢/bbl per $1 million change in installed equipment cost.

This economic study shows that the HYTORT process can produce oil from Eastern Sunbury oil shale in nearly the same yield and at only a small increase in cost over oil from Colorado oil shale. The higher cost for oil from Eastern shale is mitigated by the closeness to markets, the greater availability of water, and the less fragile ecology of the Eastern United States. Thus, the HYTORT process makes both Eastern and Western oil shales economically attractive sources of oil.

Updated Costs

As has been stated earlier, for comparability with the earlier study, costs are based on January 1980. The Chemical Engineering Plant Cost Index shows an increase from January 1980 to mid-1982 of 26% and the Marshall-Swift Equipment Index shows an increase of 20%. On the basis of a 25% increase overall for the 2-1/2 year period, the range of synfuel prices would rise to approximately $35 to $51/bbl for the four cases.

Moving-Bed Retort Model

No further work was done this quarter on development of this model.

REFERENCES CITED

1. Leffert, C. B. and Schroeder, R. R., "Fischer Assay Analyses of the Antrim Oil Shale of the Michigan Basin," DOE Report FE-2346-83, Midland, Michigan: Dow Chemical Co., August 1980.

2. Snyder, R. H. and Maness, M., "Pyrochromatographic Study of Michigan Antrim Shale," DOE Report FE-2346-66, Midland, Michigan: Dow Chemical Co., May 1980.

3. Ells, G. D., "Stratigraphic Cross Sections Extending From Devonian Antrim Shale to Mississippian Sunbury Shale in the Michigan Basin," DOE Report FE-2346-30, Midland, Michigan: Dow Chemical Co., November 1978.

4. Institute of Gas Technology, "Hydrogasification of Oil Shale," Final Report for the period 1972-1979, GRI (A.G.A.) Project IU-4-7. Chicago, October 1980.

5. Institute of Gas Technology, "Synthetic Fuels From U.S. Oil Shales, a Technical and Economic Verification of the HYTORT Process," Project 61040 Quarterly Report for the Period October 1 through December 31, 1980, Report No. DOE/ET/14102-3. Chicago, July 1981.

6. Institute of Gas Technology, "Synthetic Fuels From U.S. Oil Shales, a Technical and Economic Verification of the HYTORT Process," Project 61040 Annual Report for the Period October 1, 1979, through September 30, 1980, Report No. DOE/ET/14102-2. Chicago, March 1981.

143/RPP/61040qr

APPENDIX A. Summaries of Thermobalance Test Results

Table A-1. SUMMARY OF THERMOBALANCE RUNS USING SINK-FLOAT SEPARATED CLEVELAND (Location 139) SHALE

Run	Density, g/cm^3	Gas	Pressure, atm	α, °F/min	Time at T_{max}, min	T_{max}, °F	Total C Conv, %	Organic C Conv, %	Mineral C Conv, %	Total C Conversion			
										C → LHC	C → CO	C → CO_2	C →liq
79-40	-2.05	H_2	35.3	20	10.3	1400	78.3	NA	NA	NA	NA	NA	NA
79-41	2.05-2.1	H_2	35.0	20	10.3	1400	82.2	NA	NA	NA	NA	NA	NA
79-42	2.1-2.15	H_2	35.0	20	10.2	1400	81.2	NA	NA	NA	NA	NA	NA
79-43	2.15-2.2	H_2	35.0	20	10.1	1400	38.6	NA	NA	28.2	NA	NA	10.4
79-44	2.2-2.25	H_2	35.0	20	10.2	1400	76.5	NA	NA	26.9	NA	NA	49.5
79-45	2.25-2.3	H_2	35.3	20	10.2	1400	75.6	NA	NA	19.7	NA	NA	55.8
79-46	2.3+	H_2	35.0	20	10.2	1400	73.9	NA	NA	20.5	NA	NA	53.4
80-1	-2.05	H_2	35.8	20	10.3	1400	74.8	NA	NA	21.1	0.1	NA	53.7
80-2	2.05-2.1	H_2	35.0	20	10.2	1400	78.5	NA	NA	23.7	0.1	NA	54.8
80-3	2.1-2.15	H_2	35.0	20	10.2	1400	72.6	NA	NA	23.5	0	NA	49.1
80-4	2.15-2.2	H_2	35.0	20	10.1	1400	38.0	NA	NA	25.0	0	NA	13.0
80-5	2.2-2.25	H_2	35.0	20	10.1	1400	68.8	NA	NA	23.7	0	NA	45.1
80-6	2.25-2.3	H_2	35.0	20	10.1	1400	67.8	NA	NA	22.1	0.5	NA	45.7
80-7	2.15-2.2	N_2	35.0	20	10.2	1400	15.5	NA	NA	9.8	2.0	NA	3.7
80-8	2.3+	H_2	35.0	20	10.2	1400	70.5	NA	NA	21.5	0	NA	39.1
	Shale Source (Using New Albany Shale at Different Depths)												
79-37	Loc 64 (22.0 ft)*	H_2	35.2	17.9	10.0	1400	87.9	NA	NA	24.4	NA	NA	63.5
79-38	Loc 64 (4.5 ft)	H_2	35.0	18.0	9.9	1400	87.8	NA	NA	26.5	NA	NA	61.3
79-39	Loc 64 (7.5 ft)	H_2	35.1	19.2	10.1	1400	88.2	NA	NA	21.7	NA	NA	66.5

*Distance from top of rock unit.

Table A-2. SUMMARY OF THERMOBALANCE RUNS USING VARIOUS SHALES

Run	Shale	Loc	Gas	Pressure, atm	α, °F/min	Time at T_{max}, min	T_{max}, °F	Total C Conv, %	Organic C Conv, %	Mineral C Conv, %	Total C Conversion C → LHC	 C → CO	 C → CO_2	 C → liq
80-9	Cleveland	139	H_2	35.0	20.8	9.7	1400	68.7	68.7	69.5	27.2	0.5	NA	40.9
80-10	Cleveland	139	N_2	35.0	17.2	10.2	1400	27.9	26.7	83.8	6.2	2.2	NA	19.5
80-15	Antrim	147	H_2	34.7	21.6	9.3	1400	69.6	68.6	90.8	32.7	4.3	NA	32.5
80-16	Antrim	147	N_2	34.9	16.7	10.1	1400	48.9	46.7	88.0	5.4	2.8	NA	40.7
80-17	Utah	142A	H_2	34.8	20.9	10.0	1400	95.1	93.7	98.6	NA	15.8	NA	NA
80-19	Utah	142A	N_2	35.0	14.9	10.1	1400	75.7	83.3	56.9	NA	3.5	4.3	NA
80-36	Utah	142A	H_2	34.6	20.0	13.9	1400	96.2	96.3	95.5	16.3	11.7	1.6	66.6
80-37	Utah	142A	N_2	35.0	16.7	15.5	1400	82.8	89.9	51.0	3.8	7.7	7.9	63.4
80-46	Lower Huron	154	H_2	35.4	20.4	10.1	1200	78.0	77.7	81.4	13.9	3.8	NA	58.7*
80-47	Lower Huron	154	N_2	35.0	15.3	11.8	1200	34.1	34.7	28.6	5.0	2.0	NA	27.4*
80-71	Utah	168	H_2	35.7	20.5	11.3	1400	92.6	93.6	88.1	18.4	9.1	2.2	62.9
80-72	Utah	168	N_2	35.0	17.9	11.0	1400	67.0	75.4	29.2	7.0	6.5	4.3	49.2
80-73	Wy Green River	176	H_2	35.4	18.6	11.6	1400	96.3	96.2	96.8	14.4	2.1	0.5	79.3
80-74	Gassaway	180	H_2	35.0	20.7	11.1	1400	68.7	69.0	14.2	28.6	0.4	<0.1	39.6
80-75	Wy Washakie	177	H_2	35.7	18.2	9.9	1400	95.5	95.8	92.8	14.3	5.4	3.4	72.4
81-1	Gassaway	148/4	H_2	35.9	22.3	11.0	1400	78.8	78.9	75.1	28.1	0.7	1.1	48.9
81-2	Gassaway	148/4	N_2	35.7	17.2	11.8	1400	43.7	43.9	33.8	4.9	2.2	2.4	34.2
81-11	Dowelltown	148/5	H_2	35.6	20.3	13.0	1400	75.9	76.0	74.5	30.0	0.6	1.1	44.2
81-12	Dowelltown	148/5	N_2	36.4	12.9	11.0	1400	36.5	33.2	80.4	3.8	3.3	4.3	25.1

[*] Based on organic C assuming organic C forms only LHC and liquids.

Table A-3. SUMMARY OF THERMOBALANCE RUNS USING KENTUCKY NEW ALBANY (HK-1) SHALE

Run	Gas	Pressure, atm	α, °F/min	Time at T_{max}, min	T_{max}, °F	Total C Conv, %	Organic C Conv, %	Mineral C Conv, %	Total C Conversion			
									C → LHC	C → CO	C → CO_2	C → liq
80-20	N_2	35.2	15.7	10.3	1400	41.3	39.9	72.3	7.9	1.8	3.0	28.6
80-21	N_2	3.6	22.0	10.0	1400	44.9	44.9	44.5	4.9	1.0	1.9	37.1
80-22	N_2	3.6	∞	43.4	1410	52.7	51.6	76.8	12.5	5.1	4.0	31.1
80-23	N_2	34.9	∞	22.7	1410	46.2	45.0	76.6	14.1	4.2	2.5	25.4
80-24	H_2	34.9	19.3	10.3	1400	87.6	87.7	68.1	19.2	0.9	1.0	66.5
80-25	H_2	14.5	19.3	22.7	1400	72.0	71.4	81.6	10.0	0.6	0.5	60.9
80-26	H_2	7.3	22.3	20.4	1400	59.9	59.0	77.3	9.3	0.8	0.5	49.3
80-27	H_2	3.0	23.2	21.3	1400	54.5	53.1	96.9	8.5	0.8	0.5	44.7
80-28	H_2	34.6	∞	17.6	1500	70.0	69.1	100.0	15.4	1.4	0.6	52.6
80-29	H_2/N_2-65/35	35.1	15.6	12.7	1200	73.8	73.4	74.2	16.6	0.6	0.8	55.8
80-30	H_2/N_2-65/35	22.2	19.2	10.3	1200	61.9	62.3	28.2	14.4	0.6	0.7	46.2
80-31	H_2/N_2-65/35	11.9	20.9	11.2	1200	56.0	56.4	26.9	12.4	0.5	0.7	42.4
80-32	H_2/N_2-65/35	6.7	22.4	10.5	1200	52.5	53.1	18.6	11.4	0.6	0.9	39.6
80-33	H_2/N_2-35/65	34.9	15.9	10.6	1200	62.9	63.2	43.5	13.5	0.6	0.6	48.2
80-34	H_2/N_2-35/65	23.4	19.0	10.4	1200	56.3	56.6	51.0	11.7	0.5	0.7	43.4
80-35	H_2/N_2-35/65	13.9	20.4	11.2	1200	51.0	51.0	45.8	10.1	0.7	0.8	39.4
80-38	H_2	35.1	8.8	11.8	1300	88.4	88.5	82.3	16.6	1.2	0.7	69.9
80-39	N_2	35.1	8.3	21.5	1200	41.5	41.0	76.2	10.5	0.3	1.9	28.8
80-40	H_2	35.0	30.7	11.2	1200	79.7	80.0	59.7	14.6	0.8	0.8	63.5
80-41	N_2	35.0	27.2	10.1	1200	40.2	39.5	80.1	7.0	0.8	1.9	30.5
80-42	H_2	35.0	4.9	31.6	1200	78.9	78.6	96.5	15.3	0.4	0.9	62.3

Table A-4. SUMMARY OF THERMOBALANCE RUNS USING KENTUCKY NEW ALBANY (HK-2) SHALE

Run	**Gas**	Pressure, atm	α, °F/min	Time at T_{max}, min	T_{max}, °F	**Total C Conv, %**	**Organic C Conv, %**	**Mineral C Conv, %**	**Total C Conversion**			
									C → LHC	**C → CO**	**C → CO_2**	**C → liq**
80-48	H_2	35.7	18.0	10.6	1200	84.0	84.9	19.5	12.0	0.4	NA	70.8
80-49	N_2	35.0	18.6	11.9	1200	42.3	42.5	21.8	5.4	0.6	NA	34.4
80-50	H_2/N_2-65/35	35.0	24.9	9.8	1200	73.8	73.8	65.6	16.5	0.8	NA	55.8
80-51	H_2/N_2-65/35	35.0	8.1	11.0	1200	78.5	79.0	44.4	17.0	0.2	NA	60.6
80-54	N_2	36.0	4.7	14.3	1200	39.5	40.1	0	7.8	NA	NA	29.6
80-55	H_2/N_2-65/35	36.0	4.6	12.6	1200	83.8	84.3	45.3	18.2	0.1	NA	64.8
80-56	H_2/N_2-80/20	35.8	17.4	10.5	1400	82.3	82.5	56.2	21.6	0.8	NA	59.1+
80-57	H_2/N_2-80/20	25.4	18.6	10.8	1400	77.3	77.3	74.3	19.3	1.0	NA	56.2+
80-58	H_2/N_2-80/20	15.2	21.6	12.8	1400	66.0	66.3	43.2	16.5	1.0	NA	47.7+
80-59	H_2/N_2-80/20	5.8	18.8	9.5	1400	55.9	56.0	37.2	16.2	1.0	NA	37.9+
80-60	H_2/N_2-90/10	35.0	17.4	11.8	1400	86.8	87.3	49.9	18.6	0.8	NA	66.6+
80-61	H_2/N_2-90/10	25.0	19.0	12.0	1400	80.2	80.7	32.0	19.1	0.8	NA	59.5+
80-62	H_2/N_2-90/10	15.1	20.6	10.3	1400	69.4	69.6	47.8	19.3	0.5	NA	48.8+
80-63	H_2/N_2-90/10	5.8	19.8	11.0	1400	57.9	58.3	24.5	16.2	0.6	NA	40.3+
80-64	H_2	36.0	∞	54.2	825	69.9	70.9	0	2.5	0.2	0.1	67.1
80-65	H_2	35.5	21.3	0	718	4.9	5.0	0	0.75	0.05	0	4.1
80-66	H_2	35.0	19.8	0	1018	76.1	77.1	3.5	10.1	0.2	0.3	65.5
81-14	H_2/N_2-90/10	35.4	∞ < 600° 14.9 > 600°	0	1290	81.8	81.9	74.4	19.8	0.7	0.5	60.8
81-15	H_2/N_2-80/20	35.2	∞ < 600° 14.4 > 600°	15.9	1400	86.3	86.3	91.6	23.4	0.9	0.6	61.4
81-17	H_2	35.0	21.6	10.0	1400	86.2	86.6	54.3	13.9	0.4	0.9	71.0

Table A-5. SUMMARY OF THERMOBALANCE RUNS USING LARGE PIECES OF KENTUCKY NEW ALBANY (HK-2) SHALE

Run	Piece	ρ, g/cm^3	Dimensions, in.	Gas	Pressure, atm	α, °F/min	Time at T_{max}, min	T_{max}, °F	Total C Conv, %	Organic C Conv, %	Mineral C Conv, %	Total C Conversion			
												C → LHC	C → CO	C → CO_2	C → liq
81-3	2	2.19	1.75 x .45 x .35	H_2	35.2	18.1	11.2	1400	81.4	81.9	4.2	26.1	0.4	0.5	54.4
81-4	23	1.81	1.94 x .50 x .44	H_2	35.0	21.7	10.9	1400	79.4	79.4	80.1	28.9	0.6	0.5	49.4
81-5	9	2.20	1.87 x .45 x .30	H_2	36.2	18.1	11.0	1400	79.6	80.2	46.0	28.6	0.2	0.4	50.4
81-6	4	2.25	1.41 x .28 x .25	H_2	35.6	20.5	10.7	1400	83.4	83.4	42.5	24.8	0.2	0.5	57.3
81-7	5	2.17	1.60 x .50 x .41	H_2	35.7	20.6	10.5	1400	80.2	80.8	46.7	26.1	0.5	0.6	53.0
81-8	1	2.29	.78 x .44 x .19	H_2	35.0	20.5	6.0	1400	81.5	82.0	50.6	24.4	0.8	0.7	55.6
81-9	20A	2.26	.88 x .36 x .36	H_2	35.0	20.8	8.5	1400	80.0	80.1	74.7	28.2	0.7	0.7	50.4
81-10	16A	2.25	1.62 x .44 x .20	H_2	35.0	21.5	11.5	1400	81.0	81.5	47.3	22.6	0.5	0.8	57.1
81-13	6B	2.21	1.66 x .44 x .41	H_2/N_2-80/20	35.0	14.2	11.4	1400	77.9	78.0	92.7	29.2	0.9	0.6	47.2
81-16	6C	2.21	1.66 x .44 x .41	H_2/N_2-80/20	35.4	11.2	11.2	1400	79.3	79.3	70.9	20.9	0.3	0.5	57.6

APPENDIX B. The Pace Company Phase I Final Report to IGT: "Problem Definition and Recommended Environmental Sampling Program," October 31, 1980

THE PACE COMPANY CONSULTANTS & ENGINEERS, INC.
Rocky Mountain Division *(Formerly Cameron Engineers)*
CHERRY CREEK PLAZA II, 650 SOUTH CHERRY STREET • SUITE 400 • DENVER, COLORADO 80222 • 303/321-3919

PROBLEM DEFINITION AND RECOMMENDED ENVIRONMENTAL SAMPLING PROGRAM

Prepared For

Institute of Gas Technology

FOR PACE

C. D. Kelley
C. D. Kelley

L. A. Eckstein
L. A. Eckstein

Cary B. Jackson
Cary B. Jackson

Ted C. Borer
Ted C. Borer

Jerry E. Sinor
Jerry E. Sinor

~~July 1980~~
10/31/80

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A

INTRODUCTION

The Institute of Gas Technology has subcontracted the Pace Company Consultants & Engineers, Inc. (IGT Subcontract No. 514368) to provide environmental engineering and consulting under IGT's HYTORT program (DOE Contract No. DE-AC01-79ET14102). This is the report for Phase 1 of our contract, Problem Definition.

The two broad objectives of the phase were:

- To review outstanding environmental concerns and unanswered question pertaining to the IGT HYTORT Process.
- To propose specific experimental test programs designed to answer those questions within the overall equipment and funding limitations of the project.

B

EXECUTIVE SUMMARY

A sampling program has been prepared for the IGT Hytort process based upon a review of all applicable regulations, areas of concern to the Hytort process and unanswered questions pertaining to oil shale processes and development. Applicable statutes and regulations reviewed include the National Environmental Policy Act, The Clean Air Act, the Federal Water Pollution Control Act, the Toxic Substance Control Act, the Resource Conservation and Recovery Act, the Consolidated Permit Regulations, the Surface Mining Act, the Occupational Safety and Health Act and the Noise Control Act. A list of regulated pollutants was developed from this review. This list was divided into pollutants with regulatory requirements of primary interest and further subdivided into trace elements of interest to western and eastern shale. Elements of concern to the Hytort process were divided into air monitoring, water monitoring, and solids monitoring.

Section F presents a recommended sampling program for the Hytort process. Analyses are broken up into four areas; raw and spent shale, shale oil, process water, and off-gas analyses. Also included in this section is the recommended location and type of samples needed. A cost summary is included in the Appendix.

C

APPLICABLE STATUTES AND REGULATIONS

This section includes a short overview of the statutes and regulatory program including permits issued and substances regulated. The National Environmental Policy Act, the The Clean Air Act, the Federal Water Pollution Control Act, The Toxic Substances Control Act, the Resource Conservation and Recovery Act, the EPA Consolidated Permit Program, Surface Mining Regulation, the Occupational Safety and Health Act and the Noise Control Act are included.

NATIONAL ENVIRONMENTAL POLICY ACT OF 1969

The purpose of the National Environmental Policy Act of 1969 (NEPA) is to set forth a national environmental policy, promote efforts to prevent or eliminate environmental damage, enrich our understanding of ecological systems, and to create a Council of Environmental Quality (CEQ) to oversee Federal efforts to comply with NEPA. In form, NEPA is a statute; in spirit, a constitution. It is one of the few laws in our history that has dramatically affected the decision-making process of the Federal agencies.

The practical importance of NEPA today stems almost entirely from its environmental impact statement (EIS) requirement. All Federal agencies are required to prepare an EIS detailing the environmental impact of and alternatives to every proposal for a major Federal action significantly affecting the quality of the human environment.

New CEQ final regulations went into effect on July 30, 1979 and establish binding uniform procedures for implementing procedural provisions of NEPA. These regulations (43 FR 55978-56007, November 29, 1978), required by Executive Order 11991 issued May 24, 1977, are intended to reduce paperwork, reduce delays and produce a better decision-making product. The need for new regulations arose from an evolution of inconsistent agency practices and interpretations in the law. The lack of a uniform, government wide approach for implementing NEPA has impeded Federal coordination and has made it more difficult for those outside government to understand and participate in the environmental review process, and has caused unnecessary duplication, delay and paperwork.

However, caution must be exercised in the use of the guidelines as they are of a general nature and can be extremely limited in specific cases.

Of primary concern is Section 1507.3 of the new regulations which requires that each agency of the Federal Government adopt procedures to implement the CEQ regulations. At the present time, many agencies' regulations are still in the preparation state, although most agencies have published proposed regulations. The lead agency for the environmental impact statement is determined by the first "major" federally-approved permit. The procedural regulations of that agency are then followed for preparation of the environmental impact statement.

Contents of the EIS

The EIS must be a self-contained document, written in language than is understandable to the layman yet allows for meaningful consideration by decision-makers and scientists, which responds to opposing opinions and has suffcient depth to permit a reasoned choice. Section 1502 of the CEQ regulations set forth the following requirements for content of the EIS:

1. A cover sheet and summary statement
2. A statement of the purpose and need for an EIS
3. A comparison of the porposed action and alternatives to the action
4. A description of the affected environment. This section is the statuatory basis for the baseline studies on vegetation, soils, air and water quality.

5. A description of the environmental consequences. This section includes direct and indirect effects of the action, possible conflicts, effects of the alternatives and means to mitigate adverse environmental impacts.

6. A cost-benefit analysis of the alternatives if relevant.

Existing Oil Shale EIS's

Four environmental impact statements on western oil shale development have been prepared or are in the draft stage. These are:

1. The Final Environmental Statement for the Prototype Oil Shale Leasing Program, 1973,

2. The Final Environmental Statement on the Proposed Development of Oil Shale Resources by Colony Development Corporation, 1977,

3. The Final Environmental Statement on the Proposed Superior Oil Company Land Exchange and Oil Shale Resources Development, 1980, and

4. The Draft Environmental Statement on the Paraho Anvil Point Project. The final statement on this project may never be issued due to termination of this project at its present site.

THE CLEAN AIR ACT

The Clean Air Act was originally passed in 1963 with subsequent amendments broadening its authority in 1965, 1967, 1979, and 1977. To implement the air quality program, 247 Air Quality Control Regions (AQCR) were set up in the United States to enforce the National Ambient Air Quality Standards (NAAQS). Prior to 1977, ambient air quality standards were promulgated for five pollutants, sulfur dioxides, particulate matter, carbon monoxide, hydrocarbons, nitrogen dioxide, and photochemical oxidants. Lead was added in 1978 and ozone was subsequently added. Currently, carbon monoxide, sulfur oxides and particulates are undergoing review in the form of criteria documents. Nitrogen oxides have also been added to the list as a result of the 1977 amendments. Pollutants that are under consideration as possible candidates for addition to NAAQS include cadmium, arsenic, POM, and radioactive pollutants. Table C-1 shows current ambient air quality regulated pollutants.

The regulatory framework set up to implement the NAAQS requires the states to submit State Implementation Plans (SIP's) for enforcement of the NAAQS. Specific industrial source categories were addressed under standards of performance for new stationary sources (NSPS). National Emissions Standards for Hazardous Air Pollutants (NESHAP) were also promulgated. In 1977, two major additions were made in the SIP program. These were SIP revisions for areas not meeting NAAQS (non-attainment areas) and for Prevention of Significant Deterioration (PSD) of air quality where the NAAQS are being met.

The 1979 Clean Air Act requires EPA to list new stationary sources which contribute significantly to air pollution and which may be reasonably anticipated to endanger the public health or welfare. New Source Performance Standards have been promulgated for 26 industries. Performance standards generally include the category of the source, the facilities in that source which are affected by the standard, the pollutants emitted by each facility which are covered by the standard and the type of monitoring required by EPA to ensure the standards are met. Existing standards must be reviewed every 4 years to determine if new developments indicate that the standard requires updating. NSPS have not been defined for the oil shale industry, nor have guidance documents been published. However, the best available control technology has been defined by PSD permits granted to five western oil shale facilities.

Very hazardous pollutants from specific source catagories are regulated by the EPA under the National Emission Standards for Hazardous Air Pollutants (NESHAP). Under this program EPA has that authority to promulgate designs, equipment, work practices, operational standards, or a combination thereof, to protect the public health. Asbestos, beryllium, mercury, vinyl chloride,

TABLE C-1

NATIONAL AMBIENT AIR QUALITY STANDARDS, μG/M^3***

Pollutant	Averaging time	Primary standard	Secondary standard
SO_2	Annual	80	—
	24 hour	365	—
	3 hour	—	1,300
Particulate matter	Annual	75	60
	24 hour	260	150
NO_x (as NO_2)	Annual	100	100
O_3	1 hour	240	240
CO	8 hour	10,000	10,000
	1 hour	40,000	40,000
Lead	Quarterly	1.5	1.5
HC (non CH_4)	3 hour	160***	160***

* 40 CFR Part 50

** Reference conditions = 760 mm Hg and 25 °C

*** Not a standard; a guide to show achievement of the O_3 standard hydrogen sulfide & fluorine are currently designated as hazardous pollutants. Radiation may be so designated in the future.

Under the Clean Air Act Amendments of 1977 an Air Quality Control Region (AQCR) was divided into areas of Prevention of Significant Deterioration (PSD) where NAAQS were being met and Non-Attainment areas (NA) where the air quality does not meet NAAQS. It is possible for an AQCR to have PSD regulations regarding one pollutant and NA regulations regarding another pollutant. New construction of certain major stationary sources is subject to PSD review in areas regulated by PSD regulations. Before construction it must be shown that emissions from the new facility will not cause an increase in the ambient air concentrations greater than allowed increments and that the ambient concentration will remain below the NAAQS. PSD regions are assigned one of three clean air classifications. Class I areas include national parks, national monuments, and wilderness areas. Class III areas may be allowed to deteriorate up to the secondary standard. Class II areas include the rest of the United States. Class II areas were designed to allow for moderate, controlled growth. Redesignation to Class I or Class III by the state is possible. Table C-2 shows PSD standards for all three classes. Construction or expansion of major stationary sources in non-attainment areas are subject to the EPA's emission offset policy. This requires expanding industrial facilities to reduce emissions at their own facilities to the lowest level possible and to obtain emission reductions from other plants in the affected AQCR so that the net ambient pollution levels are lowered. New facilities must also attain an emission level that is the Lowest Achievable Emission Rate (LAER).

Litigation under the Alabama Power vs EPA case changed some of EPA's PSD regulations. These regulations were proposed in September 1979 and incorporate the following changes:

1. A Best Available Control Technology assessment for all pollutants must be performed under the Act, except where controlled emissions will be below de minimis levels. Table C-3 shows Pollutant de Minimis Levels.

TABLE C-2

PREVENTION OF SIGNIFICANT DETERIORATION OF AIR QUALITY (PSD) STANDARDS*

Pollutant	Averaging time	Maximum Allowable Increase, mg/m^3		
		Class I	Class II	Class III
Particulate matter	Annual	5	19	37
	24 hour	10	37	75
SO_2	Annual	2	20	40
	24 hour	5	91	182
	3 hour	25	512	700

* 40 CFR 52.21 and 42 USC 7401 et seq section 163.

Notes:

1. Variances to the Class I increments are allowed under certain conditions as specified at Section 165 (d) (c) (ii) and (iii) and at 165 (d) (D) (i) of the Clean Air Act of 1977.

2. EPA was to have promulgated similar increments for HC, CO, O_3 and NO_x by August 7, 1979; they are under development. Increments for Pb are due to be promulgated by October 5, 1980.

TABLE C-3

POLLUTANT DE MINIMIS LEVELS

Pollutant	Guideline for BACT (tons per year)	Guideline for Detailed Impact Analysis ($\mu g/m^3$)	
CO	100	500	8 hour
NO_2	10	1	Annual
TSP	10	5	24 hour
SO_2	10	5	24 hour
O_3(Voc)	10	-	-
Pb	1	0.03	3 month
Hg	0.2	0.1	24 hour
Be	0.004	0.005	24 hour
Asbestos	1	1	1 hour
F	0.02	0.01	24 hour
H_2SO_4	1	1	24 hour
Vinyl Chloride	1	1	Max
TRS			
H_2S	1	1	1 hour
methyl mercaptan	1	0.5	1 hour
diemthyl sulfide	1	0.5	1 hour
dimethyl disulfide	1	2	1 hour
CS_2	10	200	1 hour
COS	10	200	1 hour

Source September 5, 1979 Federal Register, 40 CFR 51 and 52

2. An analysis of baseline air quality concentrations for all pollutants listed under the Act must be performed.

3. Air quality impact analyses on fugitive dust emissions is eliminated.

4. New guidelines on modeling, phased construction and stack heights is presented. The analyses for baseline pollutant concentrations may include one year of continous monitoring.

Final new PSD regulations will not be published until June 2, 1980.

In addition to the new PSD regulations, the EPA is implementing an alternative emissions reduction concept known as the "bubble concept." This concept enables states to revise their SIPs to permit facilities to place a greater burden of control on sources where the marginal cost of control is low, and a lesser burden where cost is high, thus employing a more economically efficient use of controls to meet air pollution standards.

Under the alternative emmissions reduction concept, a facility with multiple process-related emission sources (stacks, vents, ports, etc.,) each of which is subject to specific emission limitations requirements under an approved SIP, may propose to meet the total emission control requirements of the SIP for a given pollutant through a different mix of controls that are mandated by the existing or proposed regulations. The entire plant will be covered by a theoretical "bubble" so that all of the hydrocarbons, sulfur compounds, and other pollutants could be measured on a facility-wide basis, rather than by individual stacks or vents. The concept is generally similar to the offset policy, except that, to avoid overburdening the program, the alternative approach outlined is restricted to use only within a single facility.

It is the regulatee's responsibility to come forward with the alternative control approach. The implementation will differ depending on whether the alternative approach is being applied to existing SIP requirements or new ones. Where existing SIP requirements are concerned, overall emissions limits and compliance deadlines are known. Once a plant comes forward with a promising alternative proposal that seems to achieve the goals of the current compliance schedule, then the control agency must decide upon a test to verify the equivalency of the proposed trade. If the source is able to present sufficient evidence, and the control agency agrees, then a provision would be drawn up as an addition to the existing SIP requirement.

When a State is revising a SIP, plants may, in anticipation of overall emissions limits, or in response to limits being proposed, present a counterproposal. The plant would then have to show that its alternative mix of controls would be environmentally equivalent to the process-specific standards. If the demonstration is successful, the counter-proposal can be adopted as part of the SIP.

As part of the PSD process, Federal Land Managers are given an affirmative responsibility to protect the air quality including visibility of lands within a Class I area. The EPA is involved in litigation over visibility regulations which were to have been promulgated by August 1979. The EPA is currently under court order to propose regulations by May 18 and promulgate regulations concerning visibility by November 18, 1980. Based on limited visibility analyses performed via PSD reviews on oil shale projects to date, visibility protection could provide additional constraints beyond the Class I PSD increments to the size of an industry in the western oil shale region.

Monitoring Requirements

At the present time, air quality monitoring guidelines do not exist for oil shale. Best Available Control Technology has been defined for any proposed oil shale facility with the potential for emitting 100 tons or more per year, using two criteria. These are on a case-by-case basis for granting Prevention of Significant Deterioration permits and the BACT for oil shale-related facilities. In addition, any state requirements will apply. Table C-4 gives a list of additional pollutants that the EPA will investigate in the future for possible regulatory requirements. Additional emphasis is also expected to be placed on trace metals and trace organics.

TABLE C-4

EPA "ADVOCATED CHARACTERIZATION" POLLUTANTS

Inhalable particulate (<15 micron)
Fine particulate (<2.5 micron)
COS
CS_2
RSH
NH_3
AsH_3
Se
SO_4
NO_3
Cd
Radioactivity
POM

FEDERAL WATER POLLUTION CONTROL ACT

The stated goal of the Federal Water Pollution Control Act (FWPCA) is to "restore and maintain the chemical, physical, and biological integrity of the nation's waters." The regulatory program established by the Act and its amendments has ten major components. These are:

1. Technology-based effluent limitations for existing industrial facilities discharging directly into the U.S. waters,

2. Technology-based effluent limitations for new industrial facilities discharging directly in U.S. waters,

3. Effluent limitations for publicly-owned treatment works; related requirements for pretreatment by industrial users of such facilities and for user charges and cost recovery from such users,

4. Water quality related effluent limitations applicable to all dischargers into certain waterways,

5. Continuation, upgrading and extension of federal-state and water quality standards,

6. A nationwide discharge permit program designated as the National Pollutant Discharge Elimination System (NPDES),

7. Detailed inspection and monitoring provisions,

8. Streamlined enforcement provisions,

9. Extensive requirements for interstate, state and local planning and implementation programs, and

10. A program for regulation of toxic pollutants.

An NPDES permit must be obtained under requirements of Section 402 of the Act if water is discharged in to a navigable stream. Specific New Source Performance Standards (NSPS) have not yet been promulgated for the oil shale industry. However, in their absence NPDES effluent limits are established on a best engineering basis. NSPS for the oil shale industry are scheduled to be promulgated 30 months after completion of all EPA guidance documents on the industry. Currently this is scheduled for April 1981, putting the promulgation of the NSPS in 1983-1984. It should also be noted that through litigation, the definition of an NPDES permit has been changed from an operations permit to a construction permit in the case of a new source. However, many permit conditions are either negotiable or suceptible to legal attack and should not be accepted if either burdensome or unjustified.

Under monitoring regulations, monitoring must be performed on any discharge which is not a minor discharge or which contains toxic pollutants. To date, NPDES permits have specified four or five pollutants to be monitored. This will be increased with the inclusion of the EPA's new toxic pollutant strategy. The EPA can require monitoring for the following parameters:

1. Flow,

2. Pollutants subject to reduction or elimination under the permit terms,

3. Pollutants specified by the monitoring authority as having a significant impact on water quality, and

4. Any other pollutant which the monitoring authority feels should be monitored.

In situations where a permit is issued prior to the publication of the effluent guidelines, such as in oil shale, there is considerable flexibility in the determination of the precise effluent limitations which will be mandated by the permit. At the present time, petroleum refining industry guidelines give an indication of potential oil shale guidelines. Even after promulgation of guidelines, the applicant may seek modification of limits in the permit by means of a variance clause. A careful engineering analysis of proposed permit limits is definitely required. "Conventional" pollutants regulated by effluent standards pursuant to Section 304 of the FWPCA are contained in Table C-5.

In late April or early May the EPA will publish final regulations for the implementation of the Consolidated Permit Program (See Consolidated Permit Section). Procedural requirements for the NPDES permit program will be affected. Parts of the EPA toxic pollutant strategy will be implemented under the NPDES Consolidated Permit regulations. Refer to the section on consolidated permit regulations for a list of monitoring requirements. In addition, the EPA is considering the application of the "bubble" concept similar to the one used to control air quality for water pollution control. If implemented the "bubble" concept would enable industries to control effluents on a plant-wide basis instead of having to comply with limits for each separate discharge point. A decision will be made after the consolidated permit regulations become effective.

TABLE C-5

POLLUTANTS AND WATER QUALITY CHARACTERISTICS ADDRESSED BY EFFLUENT STANDARDS

Aluminum
Ammonia
Ammonia (as N)
Arsenic
Asbestos
Barium
Cadmium
Chlorine (free Available)
Chrome
Chromium; total
Chromium; dissolved
Copper
Copper; dissolved
Cyanide (total)
Cyanide (Amenable to chlorination)

Fluorides
Fluoride (as F)
Gold
Iridium
Iron
Iron; dissolved
Iron; filtrable
Lead
Manganese
Mercury
Nickel
Nickel; dissolved
Nitrate (as N)
Organic nitrogen (as N)
Osmium
Palladium

PCB compounds
Phenol
Phenolic compounds
Phosphorus
Phosphorus (as P)
Phosphorus, elemental
Platium
Radium^{226}; total
Radium^{226}; dissolved
Rhodium
Ruthenium
Selenium
Sulfate
Uranium
Zinc

BOD$_5$
COD
pH
TKN
TSS

Color
Debris
Fecal coliform
Oil and grease
Settleable solids

Suspended solids
Drilling muds & cuttings
Water
Sand
Deck drainings

Section 307 of the FWPCA requires implementation of a toxic substances control policy. This is one of the most controversial aspects of FWPCA. Originally, in 1973, the EPA published proposed regulations for control of toxic substances. As a result of a lawsuit over these regulations, the EPA was directed to approach toxic substance control on a chemical-by-chemical basis. This was incorporated into FWPCA by the 1977 amendments. The industry-by-industry technology-based effluent limitations approach was then transformed to very specific limitations on 129 or more different pollutants for each industry catagory. Table C-6 gives a listing of the 129 toxic substances currently designated by the EPA as "priority" pollutants.

Section 311 of the Act also applies to the oil shale industry in that it designates a hazardous polluting substances control plan. This pertains to accidental or unanticipated discharge of harmful quantities of hazardous polluting substances defined by EPA. Currently 271 substances have been designated as harmful. These substances are subject to a Spill Prevention Control and Countermeasure Plan, and are listed in 40 CFR 116.

Section 208 of the FWPCA required States to designate area wide waste treatment planning agencies. The Colorado West Area Council of Governments (CWACOG) was so designated for the oil shale area in Colorado. The Uinta Basin Association of Governments (UBAG) was designated for Utah oil shale country. These 208 agencies are to plan, promulgate, and implement a program designed to protect surface water quality. Stream classifications and water quality standards are to be developed. The Colorado Council and UBAG have submitted plans to their respective states. The UBAG 208 plan was certified by the State on November 22, 1978, and approved by EPA on October 2, 1979. The CWACOG 208 plan is anticipated to be certified by the State by April 1980 and approved by EPA by May 1980.

Section 404 of the Clean Water Act authorizes the Corps of Engineers to issue permits for the discharge of dredged or fill material into the navigable waters at specified disposal sites. Although this most obviously applies to the disposal of spoil from dredging navigation channels, the authority also extends to every situation where a pile is driven or any other structure installed in the navigable

TABLE C-6

LIST OF 129 SPECIFIC POLLUTANTS COMPOUND NAME

1. acenaphthene.
2. acrolein.
3. acrylonitrile.
4. benzene.
5. benzidine
6. carbon tetrachloride (tetrachlormethane).

Chlorinated benzenes (other than dichlorobenzenes):

7. chlorobenzene.
8. 1, 2, 4-trichlorobenzene.
9. hexachlorobenzene.

Chlorinated ethanes (including 1, 2-dichloroethane, 1, 1, 1-trichloroethane and hexachloroethane):

10. 1, 2-dichloroethane.
11. 1, 1, 1-trichloroethane.
12. hexachloroethane.
13. 1, 1-dichloroethane.
14. 1, 1, 2-trichloroethane.
15. 1, 1, 2, 2-tetrachloroethane.
16. chloroethane.

Chloroalkyl ethers (chloromethyl, chloroethyl and mixed ethers):

17. bis (choloromethyl) ether.
18. bis (2-chloroethyl) ether.
19. 2-chloroethyl vinyl ether (mixed).

Chlorinated naphthalenes:

20. 2-chloronaphthalene.

Chlorinated phenols (other than those listed elsewhere; includes trichlorophenols and chlorinated cresols):

21. 2, 4, 6-trichlorophenol.
22. parachlorometa cresol.
23. chloroform (trichloromethane).
24. 2-chlorophenol.

Dichlorobenzenes:

25. 1, 2-dichlorobenzene.
26. 1, 3-dichlorobenzene
27. 1, 4-dichlorobenzene

Dichlorobenzidine:

28. 3, 3-dichlorobenzidine.

Dichloroethylenes (1, 1-dichloroethylene and 1, 2-dichloroethylene):

29. 1, 1-dichloroethylene.
30. 1, 2-trans-dichloroethylene.
31. 2, 4-dichlorophenol.

TABLE C-6 (continued)

Dichloropropane and dichloropropene:

32. 1, 2-dichloropropane.
33. 1, 2-dichloropropylene (1, 3-dichloropropene).
34. 2, 4-dimethylphenol.

Dinitrotoluene:

35. 2, 4-dinitrotoluene.
36. 2, 6-dinitrotoluene.
37. 1, 2-diphenylhydrazine.
38. ethylbenzene.
39. fluoranthene.

Haloethers (other than those listed elsewhere):

40. 4-chorophenyl phenyl ether.
41. 4-bromophenyl phenyl ether.
42. bis (2-chlorosopropyl) ether.
43. bis (2-chloroethoxy) methane.

Halomethanes (other than those listed elsewhere):

44. methylene chloride (dichloromethane).
45. methyl chloride (chloromethane).
46. methyl bromide (bromomethane).
47. bromoform (tribromomethane).
48. dichlorobromomethane.
49. trichlorofluoromethane.
50. dichlorodifluoromethane.
51. chlorodibromomethane.
52. hexachlorobutadiene.
53. hexachlorocyclopentadiene.
54. isophorone.
55. naphthalene.
56. nitrobenzene.

Nitrophenols (including 2, 4,-dinitrophenol and dinitrocresol):

57. 2-nitrophenol.
58. 4-nitrophenol
59. 2, 4-dinitrophenol.
60. 4, 6-dinitro-o-cresol.

Nitrosamines:

61. N-nitrosodimethylamine.
62. N-nitrosodiphenylamine.
63. N-nitrosodi-n-propylamine.
64. pentachlorophenol.
65. phenol.

Phthalate esters:

66. bis (2-ethylhexyl) phthalate.
67. butyl benzyl phthalate.
68. di-n-butyl phthalate.
69. di-n-octyl phthalate.
70. diethyl phthalate.
71. dimethyl phthalate.

Polynuclear aromatic hydracarbons:

72. benzo (a) anthracene (1, 2-benzanthracene).
73. benzo (a) pyrene (3, 4-benzopyrene).
74. 3, 4-benzofluoranthene.
75. benzo (k) fluoranthane (11, 12-benzofluoranthene).
76. chrysene.
77. acenaphthylene.
78. anthracene.

TABLE C-6 (continued)

79. benzo (ghi) perylene (1, 2-benzoperylene.
80. fluroene.
81. phenathrene.
82. dibenzo (a-h) anthracene (1, 2, 5, 6-dibenzanthracene).
83. indeno (1, 2, 3-cd) pyrene (2, 3-o-phenylenepyrene).
84. pyrene.
85. tetrachlorethylene.
86. toluene.
87. trichloroethylene.
88. vinyl chloride (chloroethylene).

Pesticides and metabolites:

89. aldrin.
90. dieldrin.
91. chlordane (technical mixture & metabolites).

DDT and metabolites:

92. 4, 4'-DDT.
93. 4, 4'-DDE (p-p'-DDX).
94. 4, 4'-DDD (p, p'-TDE).

Endosulfan and metabolites.

95. a-endosulfan-Alpha.
96. b-endosulfan-Beta.
97. endosulfan sulfate.

Endrin and metabolites:

98. endrin.
99. endrin aldehyde.

Heptachlor and metabolites:

100. heptachlor.
101. heptachlor epoxide.

Hexachlorocyclohexane (all isomers):

102. a-BHC-Alpha.
103. b-BHC-Beta.
104. r-BHC (lindane)-Gamma.
105. g-BHC-Delta.

Polychlorinated biphenyls (PCB's):

106. PCB-1242 (Arochlor 1242).
107. PCB-1254 (Arochlor 1254).
108. PCB-1221 (Arochlor 1221).
109. PCB-1232 (Arochlor 1232).
110. PCB-1248 (Arochlor 1248).
111. PCB-1260 (Arochlor 1260).
112. PCB-1016 (Arochlor 1016).
113. toxaphene.
114. antimony (Total).
115. arsenic (Total).
116. asbestos (Fibrous)
117. beryllium (Total).
118. cadmium (Total).
119. chromium (Total).
120. copper (Total).
121. cyanide (Total).
122. lead (Total).
123. mercury (Total).
124. nickel (Total).
125. selenium (Total).
126. silver (Total).
127. thallium (Total).
128. zinc (Total).
129. 2, 3, 7, 8-tetrachlorodibenzo-p-dioxin (TCDD).

waters. Statutory responsibility for the total program is divided between the Corps and EPA. Sections 404(b) and (c) of the Clean Water Act empower EPA to issue criteria for the selection of environmentally acceptable disposal sites and to prohibit the selection of any individual area if it is environmentally not acceptable.

Specific activities are exempt from permit requirements, including normal agricultural, silvicultural and ranching operations, maintenance of dams and levees, and construction or maintenance of farm ponds and forestry roads. With respect to permits for industrial projects, however, the Corps of Engineers clearly plays the dominant role.

Permits may be individual permits authorizing a single specified discharge, or general permits authorizing specified types of discharges in a specified waterway or group of waterways. Nationwide permits have been issued for discharges of dredged or fill material into certain smaller or minor waters of the United States. Nationwide permits have also been issued for certain types of activities in all waters of the United States. These permits and their conditions are published in 33 CFR, Sections 322.4 and 323.4. If an activity is covered by a nationwide permit and the applicable conditions will be met, there is no need to apply for an individual permit. In effect, activities authorized by the nationwide permits in the regulation are permitted in advance. No paperwork or delay is required.

Water Quality Monitoring

Suggested parameters for water quality monitoring are contained within three EPA guidance documents on the oil shale industry. These are Surface Water Quality Parameters for Monitoring Oil Shale Development, Groundwater Quality; Monitoring Methodology, and Groundwater Quality Monitoring of Western Oil Shale Development: Monitoring Program Development. The reports contain a prioritized listing of chemical, physical, and biological parameters recommended for inclusion in the monitoring program and will probably lead to promulgation of NSPS for oil shale. Water quality parameters should be selected based on an analysis of the possible pollutants and their toxicity, persistance, concentration, ease of analysis and identification, release potential and present level in receiving waters. Knowledge of organic compounds associated with oil shale development is incomplete due to the extreme difficulty in conducting organic analyses. Analyses of trace elements should be designed in accordance with possible sources that can be defined in the area. Of particular concern in the western oil shale are the elements fluoride and arsenic.

Surface Water Quality

Criteria for surface water quality parameters are divided into three priorities which are based on projected water quality in the Colorado River System for western oil shale or on specific beneficial water uses. The following definitions for priority A, B, and C parameters and the listing of these parameters in Tables C-7, C-8, and C-9 is included here from the EPA report Surface Water Quality Parameters for Monitoring Oil Shale Development.

Criteria for Priority A parameters:

1. The pollutant has (a) been reported in surface waters of the Colorado River Basin at levels equaling or exceeding acceptable limits for beneficial water uses and (b) is likely to have altered ambient levels in surface waters as a result of activities associated with the development and operation of an oil shale industry, to the point where further impairment of beneficial water uses will result; or

2. The pollutant is (a) one for which water quality criteria must be established for particular receiving waters based on tolerance levels of important, sensitive species in those waters, and (b) one that is likely to have as a result of oil shale development and operation activities, altered ambient levels in receiving waters to the point where the biota may be adversely impacted; or

TABLE C-7 PRIORITY "A" CHEMICAL AND PHYSICAL PARAMETERS TO BE MEASURED IN SURFACE WATERS

Parameters	Constituent Reported	Units
Aluminum	Aluminum (Al), dissolved	ug/liter
	Aluminum (Al), total recoverable[a]	ug/liter
Ammonia	Nitrogen Ammonia (NH_3 - N)	mg/liter
Bicarbonates	Bicarbonate ion (HCO_3)$^-$	mg/liter
Boron, dissolved	Boron (B), dissolved	ug/liter
Chlorides, dissolved	Chloride ion (Cl), dissolved	mg/liter
Conductivity	Specific Conductance	umhos/cm at 25°C
Copper	Copper ion (Cu), dissolved	ug/liter
	Copper (Cu), total recoverable[a]	ug/liter
Cyanides	Cyanide (CN), total recoverable	mg/liter
Fluorides	Fluoride (F), dissolved	mg/liter
Hardness	Hardness, total as $CaCo_3$	mg/liter
Iron	Iron ion (Fe), dissolved	ug/liter
	Iron (Fe), total recoverable[a]	ug/liter
Lead	Lead ion (Pb), dissolved	ug/liter
	Lead (Pb), total recoverable[a]	ug/liter
Magnesium	Magnesium ion (Mg), dissolved	mg/liter
	Magnesium (Mg), total recoverable	mg/liter
Manganese	Manganese ion (Mn), dissolved	ug/liter
	Manganese (Mn), total recoverable	ug/liter
Mercury, total	Mercury (Hg), total recoverable[a]	ug/liter
Molybdenum, dissolved	Molybdenum ion (Mo), dissolved	ug/liter
Nickel, dissolved	Nickel ion (Ni), dissolved	ug/liter
Nitrogen, Nitrate	Nitrate Nitrogen (No_3 - N)	mg/liter
Oil and Grease	Visable Oil	Severity
	Emulsified Oils	mg/liter
Oxygen	Dissolved Oxygen	mg/liter
Oxygen demand, Chemical	Chemical Oxygen demand (COD)	mg/liter
Pesticides	Organochlorine Pesticides[a,b]	ug/liter
Phenols	Phenolic Compounds	ug/liter
Potassium dissolved	Potassium ion (K), dissolved	mg/liter
Sodium, dissolved	Sodium ion (Na), dissolved	mg/liter
Silica	Silica, dissolved (SiO_2)	mg/liter
	Silica, total (SiO_2)	mg/liter
Solids, dissolved	Total dissolved (filtrable) residue	mg/liter
	Fixed dissolved (filtrable) residue	mg/liter
Solids, suspended	Total suspended (non-filtrable) residue	mg/liter
	Fixed suspended (non-filtrable) residue	mg/liter
Sulfates, dissolved	Sulfate ion (SO_4), dissolved	mg/liter
Temperature	Temperature	°C
Turbidity	Turbidity	nephelometric turbidity units (NTU)
Water Volume	Discharge	m^3/sec
Zinc	Zinc ion (Zn), dissolved	ug/liter
	Zinc (Zn), total recoverable	ug/liter

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[a] To be measured in streambed sediments also (ug/kg).

[b] Applies to consent decree protocol pesticides.

TABLE C-8 PRIORITY "B" CHEMICAL AND PHYSICAL PARAMETERS TO BE MEASURED IN SURFACE WATERS

Parameters	Constituent Reported	Units
Acidity, total	Acidity, total as $CaCo_3$	mg/liter
Alkalinity, total	Alkalinity, total as $CaCO_3$	mg/liter
Alpha, gross	Total Alpha Activity	pCi/liter
Arsenic	Arsenic ion (As), dissolved	ug/liter
	Arsenic (As), total recoverable	ug/liter
Barium, dissolved	Barium ion (Ba), dissolved	ug/liter
Beryllium, dissolved	Beryllium ion (Be), dissolved	ug/liter
Beta, gross	Total Beta Activity	pCi/liter
Cadmium	Cadmium ion (Cd), dissolved	ug/liter
	Cadmium (Cd), total recoverable[a]	ug/liter
Calcium	Calcium ion (Ca), dissolved	mg/liter
	Calcium (Ca), total recoverable	mg/liter
Carbonates	Carbonate ion (CO_3)	mg/liter
Carbon dioxide	Carbon dioxide (CO_2), dissolved	ug/liter
Chromium	Chromium ion (Cr)[b], dissolved	ug/liter
	Chromium (Cr), total recoverable[a]	ug/liter
Cobalt, dissolved	Cobalt ion (Co), dissolved	ug/liter
Lithium, dissolved	Lithium ion (Li), dissolved	ug/liter
Nitrogen, Nitrite	Nitrite Nitrogen (NO_2 - N)	mg/liter
Nitrogen, total	Total Nitrogen (N)	mg/liter
Oxygen demand, biochemical	Biochemical oxygen demand (BOD)	mg/liter
Pesticides	Organophosphate pesticides[a,c]	ug/liter
Phosphorus, total	Total phosphorus (P-Total)	mg/liter
Phthalate esters	Total phthalate esters	ug/liter
pH	pH	Standard Units
Polychlorinated Biphenyls	Total plychlorinated biphenyls	ug/liter
Radium 226, 228	Radium 226, 228, dissolved	pCi/liter
	Radium 226, 228, total	pCi/liter
Sediments, streambed	Streambed sediments	-
Selenium	Selenium ion (Se), dissolved	ug/liter
	Selenium (Se), total recoverable	ug/liter
Silver	Silver ion (Ag), dissolved	ug/liter
	Silver (Ag), total recoverable	ug/liter
Strontium 89, 90	Strontium 89, 90, dissolved	pCi/liter
	Strontium 89, 90, total	pCi/liter
Tin	Tin (Sn), total	ug/liter
Titanium	Titanium (Ti), total	ug/liter
Tungsten	Tungsten ion (W), dissolved	ug/liter
	Tungsten (W), total	ug/liter
Tritium	Tritium in water molecules	$Hydrogen^3$ units
	Tritium, dissolved	pCi/liter
	Tritium, total	pCi/liter
Vanadium, total	Vanadium (V), total recoverable	ug/liter

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[a] To be measured in streambed sediments; also (ug/kg).

[b] Both trivalent and trivalent plus hexavalent forms should be measured.

[c] Applies only to specific organophosphate pesticides.

TABLE C-9 PRIORITY "C" CHEMICAL AND PHYSICAL PARAMETERS TO BE MEASURED IN SURFACE WATERS

Parameters	Constituent Reported	Units
Acetone	Acetone Compounds	ug/liter
Antimony	Antimony ion (Sb), dissolved	mg/liter
	Antimony (Sb), total	mg/liter
Benzene	Benzene Compounds	ug/liter
Bismuth	Bismuth ion (Bi), dissolved	ug/liter
	Bismuth (Bi), total	ug/liter
Bromine	Bromine (Br)	mg/liter
	Bromide ion (Br), dissolved	mg/liter
Carbon	Organic Carbon, dissolved	mg/liter
	Organic Carbon, total	mg/liter
Cerium	Cerium ion (Ce), dissolved	ug/liter
	Cerium (Ce), total	ug/liter
Cerium 144	Cerium 144, total	pCi/liter
Cesium	Cesium ion (Cs), dissolved	ug/liter
	Cesium (Cs), total	ug/liter
Cesium 137	Cesium 137, dissolved	pCi/liter
	Cesium 137, total	pCi/liter
Chlorine	Residual chlorine (Cl), total	mg/liter
Color	True Color (Platinum Cobalt Units)	PCU
Cresole	Cresole Compounds	ug/liter
Detergent builders linear alkylate sulfonates	LAS, total	mg/liter
Dysprosium	Dysprosium (Dy), total	ug/liter
Erbium	Erbium (Er), total	ug/liter
Europium	Europium (Eu), total	ug/liter
Gadolinium	Gadolinium (Gd), total	ug/liter
Gallium	Gallium ion (Ga), dissolved	ug/liter
	Gallium (Ga), total	ug/liter
Germanium	Germanium ion (Ge), dissolved	ug/liter
	Germanium (Ge), total	ug/liter
Gold	Gold (Au), total	ug/liter
Hafnium	Hafnium (Ha), total	ug/liter
Holmium	Holmium (Ho), total	ug/liter
Iodine	Iodine ion (I), dissolved	mg/liter
Iridium	Iridium (Ir), total	ug/liter
Lanthanum	Lanthanum ion (La), dissolved	ug/liter
	Lanthanum (La), total	ug/liter
Lutetium	Lutetium (Lu), total	ug/liter
Neodymium	Neodymium (Nd), total	ug/liter
Niobium	Niobium (Nb), total	ug/liter
Nitrogen Kjeldahl	Kjeldahl Nitrogen, total	mg/liter
Nitrogen, Organic	Organic Nitrogen, dissolved as N	mg/liter
	Organic Nitrogen, total as N	mg/liter
Oils and Grease	Hexane Extractable Substances[a]	mg/kg
Osmium	Osmium (Os), total	ug/liter
Palladium	Palladium (Pd), total	ug/liter
Pesticides	Miscellaneous pesticides[b]	ug/liter
Phosphorus	Phosphorus, total dissolved	ug/liter

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[a] To be measured in streambed sediments only.

[b] Applies only to specific pesticides.

(Continued)

TABLE C-9 PRIORITY "C" CHEMICAL AND PHYSICAL PARAMETERS TO BE MEASURED IN SURFACE WATERS (continued)

Parameters	Constituent Reported	Units
Platinum	Platinum (Pt), total	ug/liter
Plutonium 238, 239	Plutonium 238, 239, dissolved	pCi/liter
Praseodymium	Praseodymium (Pr), total	ug/liter
Rhenium	Rhenium (Re), total	ug/liter
Rhodium	Rhodium (Rh), total	ug/liter
Rubidium	Rubidium ion (Rb), dissolved	ug/liter
	Rubidium (Rb), total	ug/liter
Ruthenium	Ruthenium (Ru), total	ug/liter
Ruthenium 106	Ruthenium 106, total	pCi/liter
Samarium	Samarium (Sm), total	ug/liter
Scandium	Scandium ion (Sc), dissolved	ug/liter
	Scandium (Sc), total	ug/liter
Strontium	Strontium ion (Sr), dissolved	ug/liter
Tantalum	Tantalum (Ta), total	ug/liter
Technetium	Technetium (Tc), total	ug/liter
Tellurium	Tellurium (Te), total	ug/liter
Terbium	Terbium (Tb), total	ug/liter
Thallium	Thallium ion (Tl), dissolved	ug/liter
	Thallium (Tl), total	ug/liter
Thorium	Thorium (Th), total	ug/liter
Toulene	Toulene Compounds	ug/liter
Uranium	Uranium ion (U), dissolved	ug/liter
	Uranium (U), total	ug/liter
Ytterbium	Ytterbium ion (Yb), dissolved	ug/liter
	Ytterbium (Yb), total	ug/liter
Yttrium	Yttrium ion (Y), dissolved	ug/liter
	Yttrium (Y), total	ug/liter
Zirconium	Zirconium ion (Zr), dissolved	ug/liter
	Zirconium (Zr), total	ug/liter

3. The pollutant is one whose measurement is essential for interpreting other water quality data.

Priority A parameters require intensive monitoring because: (1) very slight changes in their ambient levels would render water unacceptable for specified designated beneficial water uses, (2) changes in ambient levels would be indicative of potentially deleterious changes in water quality characteristics, (3) and data are required for the interpretation of other water quality data.

Criteria for priority B parameters:

1. The pollutant has been reported at levels in the Colorado River Basin that are generally within acceptable limits with respect to beneficial water uses, but its ambient levels in surface water could be altered by activities associated with the development and operation of the oil shale industry, to the point where impairment of beneficial water uses could result; or

2. The pollutant is (a) one of recognized significance in the aquatic environment, but one for which water quality criteria have been established with respect to ambient levels and beneficial water uses, and (b) it is one whose ambient levels in surface waters could be altered by activities associated with the development and operation of an oil shale industry, to the point where impairment of beneficial water uses could result.

Priority B parameters require routine monitoring of a lower intensity than do those in the priority A category because slight changes in ambient levels can be tolerated without exceeding established limits for designated beneficial water uses. The measurement of parameters in this category should be in addition to those in the priority A category, but at reduced frequencies.

Criteria for priority C parameters:

1. The pollutant is one for which no water quality criteria are established and whose significance in terms of beneficial use criteria is largely unknown; but its ambient levels in surface waters could be altered by activities associated with the development and operation of an oil shale industry, with unknown consequences for beneficial water uses; or

2. The pollutant is one for which adequate surface water quality data are unavailable to characterize ambient levels in the Colorado River Basin, but it has been identified as a potential pollutant subject to release to surface waters by activities associated with the development and operation of an oil shale industry.

Priority C parameters require periodic monitoring in addition to those in the A and B categories to characterize water quality with respect to ambient levels of particular constituents and designated beneficial water uses.

Groundwater Quality

A groundwater monitoring methodology should be designed for an oil shale project to identify potential pollutants, define existing groundwater quality and hydrogeology and evaluate the infiltration potential of wastes at the land surface along with the mobility and attentuation of pollutants in the unsaturated and saturated zones. Recommendations for monitoring well placement and sampling of groundwater is contained in the EPA report Groundwater Quality Monitoring of Western Oil Shale Development: Monitoring Program Development. This strategy is used for monitoring well placement for all liquid-holding reservoirs, and storage ponds as well as for spent shale disposal sites. Selection of chemical parameters to monitor should be based on hydrological and geochemical considerations as well as the nature of the potential pollution. A table of suggested chemical monitoring parameters for spent shale disposal is presented in Table C-10.

TABLE C-10 OUTLINE OF PRELIMINARY CHEMICAL ANALYSIS PROGRAM FOR MONITORING PROCESSED SHALE DISPOSAL AREA

Analysis Category Priority	Monitoring Priority for Constituents			
	Analysis Category	Highest	Intermediate	Lowest
Highest	General parameters	pH, e.c., Eh	TDS	—
	Major inorganics	Na, SO_4, Cl	Ca, Mg, K, HCO_3, CO_3, F, Sulfides NH_3	NO_3
	Trace elements	As, Se, Mo	Zn, Cd, Hg, B, Ni	Pb, Cu, Fe
	Organics	DOC		DOC fractionation, phenolics, specific compounds (BAP)
Intermediate	Radiological	gross α Activity gross β activity	Ra-226, 228	U, Th
Lowest	Bacteriological	TPC	TC	FC

Spent shale disposal is currently regulated on a haphazard basis by federal agencies. Contamination to the groundwater by leaching of spent shale may be regulated by the NPDES-Consolidated Permit Regulations. See the section on Consolidated Permit Regulations for further discussion. The Resource Conservation and Recovery Act will cover spent shale disposal when regulations are finally promulgated. If surface mining regulations for the oil shale industry are recommended by the President, this program would also apply. Currently, spent shale disposal and retort water pollution are regulated most effectively by the affected state. This is already being done in Colorado. However, for eastern shale, no state regulatory framework exists. For the purpose of this discussion, information on spent shale disposal can be found under the sections on the Federal Water Pollution Control Act, the Resource Conservation and Recovery Act and the Consolidated Permit Regulations.

TOXIC SUBSTANCES CONTROL ACT

The Toxic Substances Control Act (TSCA), enacted on October 11, 1976, has two main regulatory features: first, acquisition of sufficient information by EPA to identify and evaluate potential hazards from chemical substances, and second, to regulate the production, use, distribution, and disposal of such substances where necessary. The Act is codified as 40 CFR Part 720.

In order to carry out the mandate of TSCA, the interagency Testing Committee was established representing EPA, OSHA, Council on Environmental Quality, National Institute of Environmental Health and Safety, National Cancer Institute, National Science Foundation and the Department of Commerce. It is the responsibility of this committee to make recommendations to EPA on substances for which further testing is needed.

One of the major provisions under TSCA is the requirement for premarket notification. Under Section 5, a manufacturer must notify EPA 90 days before producing a new chemical substance, defined under Section 3(9) as any chemical not listed on a specially compiled inventory list which was published by EPA under Section 8(b) in May 1979. Oil shale is on this Inventory List. Being on the list, however, does not protect a product from possible future EPA control requirements, it only exempts developers from the testing requirements of premarket notification.

The Initial Inventory of all commercially-produced chemical compounds, published in May 1979, lists over 44,000 chemical substances identified by a preferred name and a unique Chemical Abstracts Service Registry number. Each preferred name identifies a chemical substance or category of chemical substances reported to EPA by manufacturers and importers during the Initial Inventory reporting period. A revised list will be published after a 210 day period for reporting of any additional chemicals. The 210 day period began on June 1, 1979 (Federal Register, Tuesday, May 15, 1979, pages 28557-28572).

Ten synthetic fuels were identified on this list, including shale oil. Being on the list does not "protect" a product from possible control requirements. If a material is found to be a hazard, certain restrictions including labeling, precautionary handling requirements or even a ban on its production may be imposed by EPA.

From this Inventory List, a Priority List of not more than 50 compounds or groups of compounds may be selected to be thoroughly tested in the 12 months following their selection. The selection of the compounds eligible for inclusion in this Priority List began with the Interagency Testing Committee studying an initial list of approximately 3500 compounds. This list was prioritized according to human and environmental exposure levels, and 300 compounds were selected as a Preliminary List, which was subsequently reduced to 100 entries from which the 50 compounds may be selected.

Thus far, only two substances have been regulated under TSCA. They are polychlorinated biphenyls or PCBs (40 CFR, Part 761) and fully halogenated chlorofluoroalkanes (40 CFR, Parts 712, 162). The other chemicals under consideration for regulation are:

Alkyl Epoxides
Alkyl Phthalates

Chlorinated Benzenes, (Mono-and Di-)
Chlorinated Paraffins
Chloromethane
Cresols
Hexachloro- 1,3-butadine
NBitrobenzene
Toluene
Xylene

New substances are also required to provide certain types of information, including studies of health and environmental risks under Section 5(d). Section 5(e) provides the authority to limit or prohibit the manufacture of a new chemical substance pending the development of adequate information for risk assessment, and Section 5(f) provides for the immediate regulation of a new substance under certain circumstances.

Existing chemicals can be regulated under several sections of TSCA, depending on the type and degree of risk to health or the environment. Regulatory options range from seizure of imminently hazardous substances, to prohibition of limitation of manufacture, use or disposal, to hazard warning labeling requirements.

In the May 9, 1979 Federal Register, EPA proposed under Section 4 of TSCA, standards for the development of data on chronic health effects. With the proposal, EPA initiated the process to codify health effects standards for incorporation into later issued TSCA Section 4 test rules. On July 26, 1979, EPA continued the process of codifying testing standards by proposing standards for testing chemicals for acute and subchronic toxicity, mutagenic effects, teratogenic effects, reproductive effects, and metabolism studies. These testing standards are similar to those proposed under the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA).

When EPA determines that chemical substances satisfy the testing prerequisites in Section 4(a), then EPA will begin finalization of the rules, with revisions and modifications, as appropriate.

Because toxicological testing is so expensive, TSCA borrowed from the Pesticide Act a provision for sharing of testing costs (FIFRA, Section 3). The reimbursement period is generally five years from date of submission but may be modified by the Administrator to conform to the time necessary to develop such data (TSCA, Section 4c). Sharing such data exempts subsequent producers from having to conduct or submit duplicative test results.

RESOURCE CONSERVATION AND RECOVERY ACT

RCRA, the hazardous waste portion of the Solid Waste Disposal Act, establishes a federal program to provide comprehensive regulation of hazardous waste. When fully implemented, this program will provide "cradle-to-grave" regulation of hazardous wastes.

Section 3001 (40 CFR Part 261) of RCRA requires EPA, after developing appropriate criteria, to promulgate a generic definition of hazardous waste and to compile a list of specific wastes. Hazardous wastes are defined by specific characteristics. A proposed list of four identifying characteristics and their procedure for determination of waste was published in the Federal Register on December 18, 1978. The list was supplemented on August 22, 1979. The initial characteristics include: ignitability, corrosivity, reactivity, and toxicity. The additional characteristics include: radioactivity, infectiousness, phytotoxicity, and teratogenicity and mutagenicity. EPA is currently assessing how oil shale wastes, particularly processed shale, should be categorized within the hazardous/solid waste system.

Promulgation of Section 3001 regulations triggers a requirement that generators file within 90 days a notification which identifies each hazardous waste generated, states its location, and provides a general description of the activity by which it is produced. This form, used for notification of both generation and transport activities was published in the Federal Register on February 26, 1980, pg. 12746 as Section 3010.

The final regulations promulgating Sections 3002 and 3003 (40 CFR Parts 262 and 263) of RCRA requiring EPA to regulate generators and transporters of hazardous wastes by establishing standards which govern temporary storage, record keeping, labeling, and transportation to authorized waste management facilities, were published in the Tuesday, February 26, 1980, Federal Register, page 12722. These regulations will take effect in August 1980.

Under Section 3002 a person who generates a solid waste must determine whether that waste is hazardous. This is accomplished by: (1) determining whether the waste fits the definition of hazardous waste as defined in 3001; (2) review of the hazardous waste list; and, (3) if the waste is not listed as hazardous, a determination must be made whether the waste is hazardous by identifying characteristics and testing procedures. Oil shale fits into this third category.

Even if the waste is listed as hazardous, the person has an opportunity to demonstrate that his waste is in fact not hazardous under Section 3001. After a waste is determined to be hazardous under Section 3002, the generator must obtain an EPA Identification Number through filing the notification form (Section 3010), and is then subject to the other provisions of 3002 including: specifying the facility and, if applicable, an alternate facility which is to receive the hazardous waste, termed "manifest system", protecting against such practices as roadside dumping; pre-transport requirements; record keeping and reports.

The final regulation under Section 3002 also requires a generator who ships wastes off-site to comply with all of the requirements of Section 3002. Generators who treat, store, or dispose of their wastes on-site are required only to comply with those sections for determination of whether or not a waste is hazardous. Generators who engage in on-site treatment, storage, or disposal will be required to comply with the treatment, storage, disposal standards, record keeping and reporting, promulgated pursuant to Section 3004 and to obtain a permit pursuant to Section 3005. "On-site" is defined as the same or geographically contiguous property which may be divided by public or private right(s)-of-way. Non-contiguous property owned by the generator but connected by a right-of-way which he controls and to which the public does not have access is also considered on-site property.

Transportation of hazardous waste off-site is subject to regulations under Section 3003. A transporter must also apply for an Identification Number by complying with Section 3010, thus assisting EPA in tracking waste movement and making transporters aware of EPA and DOT regulations. The transporter must also comply with the Manifest System in Section 3002, the placarding of vehicles, labeling and marking containers (to be joint EPA/DOT requirements), record keeping and reporting, and be responsible for cleaning up any hazardous waste discharge which occurs during transportation. In the event of an emergency, the requirement for an EPA Identification Number and compliance with the Manifest System may be temporarily suspended.

It appears that a concept of Best Engineering Judgement (BEJ) will be heavily relied upon in defining disposal standards on a case-by-case basis (Section 3004, 40 CFR Parts 264, 265 & 266). The BEJ concept is consistent with BACT, BAT, and BMP concepts for air and water. Oil shale developers will have to obtain a permit (Section 3005, 40 CFR, Parts 122, 123 & 124) for disposal of any hazardous waste from EPA until the State assumes the program (Section 3006, CFR Part 123).

Final Resource Conservation and Recovery Act (RCRA) regulations appeared in the Federal Register on May 19, 1980. At this time toxicity testing requirements were altered by adopting a proposed attenuation factor of 100 times the safe drinking water standard. This level was orginially set at ten times the safe drinking water standard. The toxicity test procedure, itself, was not altered, although there were many objections to the use of acetic acid as the leachate medium instead of distilled water. In evaluating the test procedure, the Environmental Protection Agency decided to stay with the acetic acid medium and change the attenuation factor, instead. It is thought that a ten-fold increase in the leachate toxicity levels will allow for any inaccuracies in the testing procedure.

Representative sampling methods, test procedures and chemical analysis methods for performing the toxicity test are contained within the regulations on page 33127 of the May 19, 1980 Federal Register. These pages are reproduced in the Appendix. ASTM Standards are followed for sample preparation. The extraction and chemical analyses procedures are detailed in the Environmental Protection Agency's publication, "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods," SW-846.

Testing of Antrim shale for heavy metal pollution by the RCRA methods has revealed that Antrim shale does not exceed RCRA limits for any of the parameters. If Eastern spent shale does not exceed these standards, it will be classified as a solid waste which is not currently regulated under the RCRA regulations. It is, however, regulated under by Section 4004 as proposed as 40 CFR, Part 257 (Solid Waste Disposal Act). After much more testing, the mutagenicity test may affect oil shale, but this is well into the future as this has been deferred indefinitely under the new regulations.

CONSOLIDATED PERMIT REGULATIONS

On June 14, 1979, the Environmental Protection Agency published proposed regulations for a consolidated permit program along with a draft consolidated permit application form and proposed National Pollutant Discharge Elimination System Regulations. These proposed regulations will be finalized during late April or early May and are expected to have a major impact on the permitting process.

EPA administers several major permit programs to control the disposal of various waste materials into the environment. Each is established by a separate statute. When administered separately, these programs overlap in jurisdiction over the same facility, leave gaps, or duplicate coverage of regulated activities. EPA has concluded that consolidation of the permit regulations will result in both management and environmental benefits. The "Proposed Consolidated Permit Regulations" cover five programs:

- The Hazardous Waste Management Program (HWM), under the Resource Conservation and Recovery Act (RCRA), regulates the generation, transportation, and treatment, storage, or disposal of hazardous waste.

- The Underground Injection Control Program (UIC), under the Safe Drinking Water Act (SDWA), regulates underground injections into wells which could affect potable water supplies.

- The National Pollutant Discharge Elimination System (NPDES), under the Clean Water Act (CWA), regulates the discharge of wastewater into surface waters of the United States.

- The Dredge and Fill Program (404 Program), under Section 404 of the CWA, regulates the discharge of dredged or fill material into waters of the United States.

- The Prevention of Significant Deterioration Program (PSD), under the Clean Air Act (CAA), regulates discharges of air pollutants and requires permits for new sources located in "attainment" areas.

The proposed regulations are the first of EPA's efforts to consolidate permit programs and focus on consistency and unification of program definitions and descriptions, state program requirements, and permit issuance procedures. The NPDES permit program as revised June 7, 1979 has been incorporated in the "Proposed Consolidated Permit Regulations."

Proposed NPDES permit regulations which have been incorporated into the Consolidated Permit Regulations are mainly procedural revisions. Duration of permits has been affected by providing for review of all permits when one permit expires. Standards will be changed by incorporating new standards as they become applicable, such as for toxic pollutants. Best Management Practices may be imposed as permit conditions to regulate toxic or hazardous pollutants. This may involve control of plant runoff, spillage, sludge disposal, control of leaching from raw material storage, etc. If these conditions are implemented spent shale disposal may be regulated under the NPDES system.

As the draft Consolidated Permit application form has been designed, the following description of effluent characteristics is required:

- Quantitative data on BOD, temperature, COD, TOC, pH, TSS, total CN, phenols, total kjeldahl nitrogen for all outflows.

- For 36 industries, quantitive data on process water outflows for the 129 toxic pollutants. Oil shale is not named in these industries but oil shale-related industries such as refining, etc. are named.

- Analyses or best estimates for NH_3-N, asbestos, bromide, residual chlorine, color, fecal coliform, flouride, NO_3-N, NO_2-N oil and grease, total P, radioactivity, sulfate, sulfide, sulfite, surfactants, dioxin, and ten additional metals.

In addition, the discharge of any reported pollutant which is not regulated by guidelines or other means shall be limited to five times the average concentration reported in the application, or if the pollutant was not detected or reported as required, five times the highest of 10 parts per billion or the detection limit specified in the analytical method.

Since these are proposed regulations final regulations, to be published in late April or early May, may differ. In any case, it is probably safe to predict that many of the features of the EPA approach to toxic pollutant control will be challenged in the courts.

SURFACE MINING REGULATION

The Committee on Surface Mining and Reclamation (COSMAR) has now published its report on whether surface mining regulation via the Surface Mining Control and Reclamation Act of 1977 is applicable to minerals other than coal. The report entitled, "Surface Mining of Non-Coal Minerals," surprisingly concluded that a regulatory approach similar to the Surface Mining Act is not applicable to non-coal minerals including oil shale. This report will be given to the Council on Environmental Quality who will, in turn, prepare recommendations to the President and Congress for formulating specific legislation for regulating the conduct and effects of surface mining for non-coal minerals. If this recommendation is accepted, it appears that surface mining regulations for oil shale will be left in the hands of the states where it occurs.

Through a working paper on oil shale and tar sands several areas of potential environmental impact were pointed out for oil shale. These impacts apply mainly to western oil shale although eastern shale was taken into consideration. The panel sees adverse or unknown impacts of:

- CO_2 emissions to the atmosphere
- disposal of spent shale
- leachate mitigation from spent shale
- air quality degradation
- unregulated emissions, particularly mercury, arsenic, selenium, and trace metals.
- uncontrolled releases of emissions from various operations
- dust
- surface disturbances
- human activities
- instability of waste piles
- destruction of wildlife and plants
- poor land use
- retort water disposal problems
- water runoff and percolation
- conspicuous nature of land impacts
- increased salinity of Colorado River system

- destruction of aquifers
- increased population

One area of great concern in regulating surface mining of oil shale is spent shale disposal. Several studies have been done on spent shale disposal of western shale. Spent shale disposal is expected to have a major impact due to the large volume of material and potential for leaching. The high salt content of western shales is expected to affect revegetation success, even though spent shale disposal piles will be sealed prior to revegetation.

Spent shale disposal is expected to be regulated under several sources. Groundwater contamination will be regulated under the Clean Water Act and the EPA. Disposal of the solids wastes will be regulated under the Resource Conservation and Recovery Act as well as any surface mining regulations developed for oil shale, on either a federal or state basis.

OCCUPATIONAL SAFETY AND HEALTH ACT

The Occupational Safety and Health Act of 1970 intends to assure safe and healthful working conditions by authorizing enforcement of the standards developed under the Act, by assisting and encouraging the States in their efforts to assure safe and healthful working conditions and by providing for research, information, education, and training in the field of occupational safety and health. The Occupational Safety and Health Act created the Occupational Safety and Health Administration and charged the agency with the regulatory repsonsibility to protect workers from hazards of the workplace. The Act also created the National Institute of Occupational Safety and Health (NIOSH), which has among its responsibilities the duty to make recommendations for the OSHA regulatory standards.

Both safety and health regulations have been promulgated by OSHA under the Occupational Safety and Health Act. Safety regulations include handling and labeling requirements, and safety precautions (such as protective clothing or equipment). Health regulations are exemplified by employee health record requirements, and the setting of contaminant exposure limits and maximum contaminant levels for workplace air.

In 1974, OSHA promulgated the first regulations establishing levels of pollutants that would be acceptable for workplace atmosphere. The levels adopted are time weighted averages (TWA) based on 8 hours per day or 40 hours per week exposure. The levels specified reflect the recommended Threshold Limit Values (TLV^R) published by the American Conference of Governmental Industrial Hygienists (ACGIH) in 1968 and the then current American National Standards Institute (ANSI). These regulations are found as Subpart 2, 29 CFR Part 1910, "Toxic and Hazardous Substances" and shows values based upon ANSI recommendations, and Mineral Dust Concentration, also from the ACGIH standard.

Besides these regulations, occupational standards requiring special precautions and/or protective devices have been adopted or proposed for 20 additional workplace air contaminants. Also, numerical standards have been set or proposed for seven workplace air contaminants. Chemical contaminants have also been regulated as potential carcinogens by OSHA. Special handling, protective devices, and minimum contact regulations have been adopted to address these compounds.

The Occupational Safety and Health Administration has amended Title 29, CFR 1911 by adding a new Part 1990, to deal with the specific identification, classification and regulation of toxic substances for which there is reported to be evidence of a carcinogenic potential to humans. These rules and regulations were issued in the Federal Register, Tuesday, January 22, 1980, pages 5001-5296.

By utilizing the best available scientific knowledge, OSHA will determine whether a substance poses a carcinogenic risk to humans by relying upon scientifically evaluated evidence from human epidemiological studies, animal studies or both. The degree of conclusiveness of such data may permit regulation of the substance as either a Category I or II potential carcinogen.

Substances which on the basis of a brief scientific review of available data are considered candidates for further scientific review and possible regulation under this Part will be placed on the "Candidate List of Potential Occupational Carcinogens". The Candidate list and the priority lists (Categories I and II) have no regulatory effect and do not represent a determination that any of the substances on these lists are actual occupational carcinogens. Rather, these lists are necessary steps in setting priorities and in allowing for public input. Further review of the evidence may result in the removal of a substance from the lists or may trigger regulatory activity through the issuance of a proposal, or in appropriate circumstances, an emergency temporary standard to control exposure to the potential occupational carcinogen.

In general, Category I toxic substances are those whose carcinogenicity has been determined in humans or in two or more mammalian species of test animals or in one species if the results of that study have been replicated. Category II toxic substances are those whose carcinogenicity has been reported but the evidence for some reason is only suggestive or positive in one species, but not yet replicated. A third category (Category III) are those for which the evidence is inadequate to raise any concern regarding carcinogenicity or where the evidence consists of animal data in a single species that is less than suggestive (42 FR 54165).

OSHA is presently preparing the first Candidate List and will make it public by July 1980, updating a preliminary version prepared in July 1978. The number of substances that OSHA anticipates will be identified as candidates for Category I or II is expected to be in the range of 200 to 500 entries.

Mine Safety and Health Administration

If the operations fall under MSHA jurisdiction at the mine site, respirable dust standards are also enforced. These are identical to OSHA standards in that they are based upon the TLV's recommended by ACGIH. Sampling procedures and regulations are contained in 30 CFR 55.5. In addition, MSHA publishes a list of toxic substances which may not be used or stored except under laboratory conditions where approved by a national agency.

National Institute for Occupational Safety and Health

The Occupational Safety and Health Act in addition to creating the Occupational Safety and Health Administration in 1970, also created the National Institute for Occupational Safety and Health (NIOSH). NIOSH has among its responsibilities the duty to make recommendations and to provide research and background information to OSHA. Since 1971 NIOSH has published a yearly update of its listing of toxic effects of chemical substances. There are approximately 26,000 entries currently in this listing, and is used only as a research and information source — there are no standards associated with or resulting from the list. Varying levels of information are provided for each entry, depending on current knowledge, including data on animal and/or human test levels and source, such as air, water or solids. This list is similar to and is often confused with the Toxic Substances Initial Inventory listing used by EPA, however, they were originated for different reasons.

NOISE CONTROL ACT

Under the Noise Control Act of 1972, the Administrator of the EPA is required to develop and publish information about permissible levels of noise, and then to set noise standards for products that have been identified as major sources of noise. This exempts aircraft noise which remains under the control of the FAA. The law requires standards to be set for the catagories of construction equipment, transportation equipment, all motors and engines and electrial and electronic equipment. Manufacturers of noise-generating products must label their products and issue a warranty that their product falls within noise guidelines at the time of sale.

Regulations limiting noise levels (in dBA) emitted from a variety of sources have been promulgated and are located in 40 CFR, Parts 203 and 204. Effective dates of the regulations vary. Noise standards which may effect oil shale mining activities are mainly in the construction equipment catagory. Table C-11 shows the equipment regulated, the noise level and the effective date of compliance.

TABLE C-11

NOISE LEVELS RECOMMENDED FOR EQUIPMENT

Noise Source		Noise Level (dBA)	Effective Date
Medium and Heavy Trucks		83	January 1, 1978
		80	January 1, 1982
Crawler Tractors	20-199 HP	77	March 1, 1981
		74	1984
	20-450 HP	83	1981
		80	1984
Wheel Loaders	20-249 HP	79	1981
		76	1984
	250-500 HP	84	1981
		80	1984
Wheel Tractors	20 HP	74	March 1, 1981
Portable Air Compressors			
	<250 ft^3/min	76	January 1, 1978
	<250 ft^3/min	76	July 1, 1978

TABLE C-12

PERMISSIBLE NOISE EXPOSURES

Duration (Hours)	Sound Level (dBA), Slow Response
8	90
6	92
4	95
3	97
2	100
1-1/2	102
1	105
1/2	110
1/4 or less	115

Noise within a plant such as conveyors, crushers, etc. is regulated by the Mining Safety and Health Administration at a mine site and the Occupational Safety and Health Administration at an industrial site where monitoring requirements must be met. Noise exposure must be measured with approved equipment and meet standards shown in Table C-12.

No exposure shall exceed 115 dBA. Impact or impulsive noises shall not exceed 140 dBA peak sound pressure level.

ENVIRONMENTAL REGULATION - EASTERN STATES

The HYTORT process is particularily applicable to retorting of eastern oil shales. For this reason, this subsection has been included to provide some insight into how the eastern states of Kentucky, Indiana, Ohio and Tennessee will regulate oil shale development. These four states contain the largest potentially recoverable reserves of oil shale in the eastern United States.

Kentucky

General responsibility for environmental permitting is assumed by the Kentucky Department for Natural Resources and Environmental Protection. A regulatory program specific to oil shale has not yet been implemented. However, the 1979 legislature imposed a one year moratorium on leasing of oil shale so that standards may be developed. Currently, permitting is handled under the Division of Air Pollution Control, Division of Hazardous Material and Waste Managment, Division of Water Quality, Division of Water Resources, and the Bureau of Surface Mining Reclamation and Enforcement.

Kentucky has an approved State Implementation Plan for air quality regulation from the Environmental Protection Agency. This is in the process of being revised. Kentucky has also assumed state primacy in regulating water quality through the NPDES system. Oil shale operations would have to meet criteria developed under the aquatic life-secondary recreation classification for any discharges. Presently, there are no groundwater regulations as regulations under the underground injection control system are still being developed. Sedimentation structures are regulated under the Bureau of Surface Mining. The surface mining regulations in Kentucky are virtually a copy of the federal regulations. Comprehensive legislation was passed in the 1977-1979 period by Kentucky to implement the hazardous waste management system under the RCRA laws. Emergency regulations for hazardous waste are now in effect. Hearings were held on the proposed permanent regulations in April.

Indiana

General responsibility for environmental regulations is assumed in Indiana by the State Board of Health. Indiana does not have any regulations specific to oil shale at the present time and expects to follow a federal lead on implementation of such regulations. Permitting activities are carried out by the Division of Water Pollution Control Division of Air Pollution Control and Division of Sanitary Engineering.

Indiana submitted their State Implementation Plan in the summer of 1979 for air pollution control. The Plan varies somewhat from the federal guidelines in that there are several additions and deletions from the federal standards. Conditional approval was given to the SIP in March by the EPA. Further revisions must be made before the plan is accepted. Indiana has assumed state primacy for the federal NPDES program. Currently there are no oil shale specific water quality regulations. Underground injection control is handled by the Oil and Gas Division which only requires well and test hole permits. Additional permits may be required by counties. Regulations for hazardous waste have not yet been developed. Comprehensive legislation was passed earlier this year and only awaits implementation.

Ohio

General responsibility for environmental permitting is assumed by the Ohio Environmental Protection Agency. Ohio also does not have any specific regulations in regard to oil shale development. In this aspect they characterized themselves as "followers" of the federal government and do not plan to do any work in this area. Permitting activities are carried out by the Division of

Water Pollution Control, the Office of Air Pollution Control and the Office of Land Pollution Control.

Ohio has submitted a State Implementation Plan for air quality. This plan is currently being revised and new particulate matter standards were just issued in May. Ohio has also assumed state primacy in water pollution control under the NPDES system. These regulations are very close to federal standards. Ohio passed a hazardous waste law in 1979. These regulations are now under development with indications that the state will directly reference major sections of the federal regulations. Ohio submitted their surface mining plan to the Office of Surface Mining (OSM) in February 1980. In April the program was determined to be incomplete, Ohio has not submitted any further material to OSM since that time. After hearings in July OSM will rule on whether the program will be accepted in part, disapproved, or conditionally approved.

Tennessee

General responsibility for environmental regulations is assumed in Tennessee by the Bureau of Environmental Health Services. Tennessee does not have regulations specifically related to oil shale development. Tennessee will most likely follow a federal lead in developing such regulations. Permitting activities affecting oil shale development are carried out by the Division of Water Quality Control. The Division of Air Pollution Control and the Division of Solid Waste Management.

The state of Tennessee has submitted a State Implementation Plan to the EPA for air pollution control. Parts of this plan wre rejected by the EPA and are now undergoing revisions. Tennessee has also assumed state primacy under the NPDES program. One major difference between the state and federal program is that Tennessee regulates surface and subsurface waters under a discharge plan where the NPDES covers only surface waters. Water discharges by surface mining operations are covered under the authority of the Division of Surface Mining. This Division splits authority for water discharge with another agency, the Department of Conservation. Surface mining regulations generaly follow federal guidelines. Tennessee has passed comprehensive legislation dealing with hazardous waste guidelines. Hearings have been held on proposed regulations but no date has been set for making final rules.

A study is currently underway at the Tennessee Technological University on the commercialization of oil shale in the state. The study has a budget of one half million dollars and is mainly a feasibility study, but it may have an impact on future regulation of the industry.

D

CONTROLLED POLLUTANTS APPLICABLE TO OIL SHALE DEVELOPMENT

This section identifies pollutants of regulatory significance to oil shale operations and those of particular concern to the IGT Hytort process. Many regulatory programs which will affect oil shale developments are in a proposed stage with most final regulations expected to go into effect in the summer of 1980. Even so, many of these programs are sure to be challenged in the courts by industry or environmental groups. This section, then, gives an overview of regulations presently in effect with no attempt to foresee possible future regulatory requirements.

Table D-1 presents regulatory requirements of primary interest to oil shale operations as viewed by the Environmental Protection Agency. This material was taken from the final draft of "Environmental Perspective on the Emerging Oil Shale Industry" by the EPA Oil Shale Research Group. This list includes those regulatory requirements which are currently in effect plus those advocated in guidelines produced by the EPA.

Although many properties are the same or nearly the same for western and eastern oil shale, there are some differences in trace metal content and amount of sulfur. Generally, the eastern shales are much higher in sulfur content than the western shales. Also, the presence of radioactive elements in the trace metals of eastern shales necessitates a slightly different approach to monitoring. Therefore, Table D-2 will show the trace elements of interest to western oil shale monitoring and Table D-3 will represent eastern oil shale trace elements. The primary monitoring consideration has been retorting because a detailed commercial flowsheet is not yet available for other sections of a commercial plant. In addition, consideration of water treatment, spent shale disposal and mine design has been made in general.

TABLE D-1

REGULATORY REQUIREMENTS OF PRIMARY INTEREST

Air

TSP
SO_2
NO_x
O_3

Pb
H_2S
Hg
F

Inhalable Particulate (<15 Micron)
Fine particulate (<2.5 Micron)
COS
CS_2
RSH
NH_3
AsH_3
Se
SO_4
NO_3
Cd
Radioactivity
POM

Water

TSS
phenols
pH
Pb

As
Cd
TDS
SO_4
F
B
NH_3
Cl
COD
Temperature
Ba

Cu
Cr
Hg

Se
Ag

Al
Fe
Mo
oil and grease
Acid organics (as a class)
Neutral organics (as a Class)
Base organics (as a class)
DO
POM

Solid Waste

Toxicity
Ignitability
Corrosivity
Reactivity
Permeability
Leachability

POM
MO
B
F
As

TABLE D-2

TRACE ELEMENTS OF INTEREST-WESTERN SHALE

- Aluminum
- Antimony
- Arsenic
- Baruim
- Beryllium
- Boron
- Cadmium
- Chromium
- Cobalt
- Copper
- Flouride
- Iron
- Lead
- Magnesium
- Manganese
- Mercury
- Nickle
- Potassium
- Selenium
- Silver
- Sodium
- Vanadium
- Zinc
- Zirconium

TABLE D-3

TRACE ELEMENTS OF INTEREST-EASTERN SHALE

- Calcium
- Chloride
- Cobalt
- Copper
- Flouride
- Lead
- Magnesium
- Manganese
- Molybdenum
- Nickel
- Potassium
- Sodium
- Thorium
- Uranium
- Vanadium
- Zinc

E

SENSITIVE AREAS & UNANSWERED QUESTIONS

Since oil shale mining and retorting is a relatively new technology, many areas of uncertainty exist as to environmental impacts. A list of key unanswered questions is given below on the surface mining of oil shale. Although research is now taking place on many of these questions, most studies have been done on pilot operations and may not point out difficulties than could arise with a scale-up to commercial technology.

Air Pollution Control

- What are the concentrations of various sulfur species in retort off-gas streams?
- What is the impact of fugitive dust from mining, crushing, transporting, and stockpiling of raw oil shale?
- What is the concentration of trace metals and trace organics and how can their measurement in the field be improved?
- What is the quantity of CO_2 generated per unit of fuel product produced?
- What is the complete characterization of atmospheric fugitive emissions from the refining of crude shale oil?
- How can predictive techniques for air quality modelling in complex terrain be developed on a comparable basis?
- What will be the impacts of oil shale development on visability?

Water Pollution Control

- How much groundwater will be intercepted during mining and is reinjection an appropriate control measure?

- Can a groundwater monitoring technology be developed to detect early changes in groundwater quality?

- What are the expected trace element concentrations in process wastewaters and can trace organics and trace metals be removed from oil shale effluents and wastewaters?

- What are the problems associated with any impoundment of waste water?

Solid Waste Control

- Can the technology be developed to control leachate generation from spent shale disposal?

- How can impermeable barriers be constructed out of spent shale?

- How can erosion be controlled?

- How can vegetation be established on spent shale?

- Will re-established vegetation show uptake of trace elements and other toxic substances?

- How will solid wastes generated during the refining of crude shale oil such as bottom sludges from shale oil storage tanks, spent guard catalysts, etc. be disposed?

General

- What are the health effects of oil shale residuals?
- What are the health effects of shale oil and other by-products?
- What is the probable social and economic effects of the development of an oil shale industry?

F

RECOMMENDED SAMPLING PROGRAM

In addition to applicable analyses required by regulation and advocated by the Environmental Protection Agency, analyses are required for material balance calculations. Material balances provide a mechanism to assure that all pollutants are accounted for, to identify unforeseen environmental concerns, and to identify analytical deficiencies. We assume that ultimate analyses will be performed on all raw shale so that the results of the environmental investigations can be normalized and scaled up based on this production parameter.

The recommended analyses are presented in the following section according to sample type. Sections then follow on the sampling program and quality assurance consideration.

RECOMMENDED ANALYSES

Both raw and spent shale will require the analyses presented in Table F-1 for the purpose of material balance of the elements of concern. Material balances on specific elements will be determined on the basis of concentrations during initial sampling. As these are solid samples, instrumental neutron activation or x-ray fluorescence analysis should be the most accurate and cost effective means of analysis. Those elements that are not amenable to activation or x-ray fluorescence analysis require sample pre-treatment (digestion or salt fusion) followed by atomic absorption spectrophotometric (AAS) or atomic emission spectrophotometric analysis (AES).

In addition, because of RCRA, spent shale must be analysed to determine if it is to be classified as a hazardous waste. This determination is made by a standard leaching procedure with chemical analyses and perhaps direct toxicity testing of the leachate. These procedures were released through the Federal Register

TABLE F-1

ANALYSES RECOMMENDED FOR RAW AND SPENT SHALE

Element	Proposed Method* (and Alternate Methods)
N	Combustion method
S	Combustion method
Ca	INAA, AAS, XRF
Mg	INAA (short), AAS
Na	INAA, AES
K	INAA, AES, XRF
Pb	AAS, XRF
F	INAA, Distillation/Colorimetry
Be	AAS
Hg	AAS (cold vapor)
As	INAA, AAS, XRF
Se	INAA, AAS, XRF
Al	INAA (short), AAS
Cd	AAS
Ba	INAA, AAS
Cl	INAA
Cr	INAA, AAS
Cu	INAA (short), AAS, XRF
Fe	INAA, AAS, XRF
Mn	INAA, AAS, XRF
Ni	INAA, AAS, XRF
P	INAA
Zn	INAA, AAS, XRF
Li	AES
Sb	INAA, AAS
Ag	INAA, AAS
Tl	AAS
B	AAS
Mo	INAA, AAS, XRF
Sr	INAA, AAS, XRF
V	INAA (short), AAS
U	INAA
Th	INAA
Zr	INAA
Non-Elemental	
Radioactivity	Gross α, β, γ
Ultimate Analysis	C, H, S, N, O and Ash

*Note: INAA = Instrumental Neutron activation analysis
AAS = Atomic absorption spectrophotometry
AES = Atomic emission flame spectrophotometry
XRF = X-ray fluorescence spectrometry

on May 19, 1980 and are described in Section C and the Appendix. If funds are available IGT should consider conducting this leaching procedure on spent shale samples.

Shale oil should be analyzed for the purpose of material balance calculations. In addition, the EPA priority pollutants of concern to the oil shale industry should be determined in the oil. Toxicity screening of the various shale oils produced could be done to compare the hazards of handling these products compared to other synfuels and petroleum products. Table F-2 presents the recommended analyses to be performed on shale oil.

The process water will require the full complement of analyses outlined in Table F-3. A standard toxicity test should be of some value for comparison purposes although it is not required. It should be cautioned that existing methods for many elements are less than satisfactory when working with process waters (Fruchter, et al., 1979).

The retort gas stream requires on-line or grab samples for the species in Table F-4.

SAMPLING PROGRAM

Sampling is a critical consideration in a program of this type because meaningful analytical results depend on the integrity and representativeness of the sample. Care must be taken to minimize contamination of the sample during the acquisition steps. Samples must also be preserved in a manner which is most appropriate for the analysis to be performed.

Solid Samples

A sample of the raw feed shale should be taken prior to charging the feed hopper and, if possible a sample of the retorted shale should be taken periodically when steady state conditions are reached. A 20-25 Kg sample should be collected when steady state conditions are reached to represent the entire period of the retorting run. This sample should be crushed, mixed, and split into smaller samples for analysis (Wildeman and Heistand, 1979).

TABLE F-2

RECOMMENDED ANALYSES FOR SHALE OIL

Analysis	Method
All Table F-1 Analyses	(See Table F-1)
Acenaphthene	EPA Priority Pollutant Protocol*
Benzene	"
Benzidine	"
2,4-Dinitrotoluene	"
2,6-Dinitrotoulene	"
1,2-Diphenylhydrazine	"
Ethylbenzene	"
Fluoranthene	"
Isophorone	"
Naphthalene	"
Nitrobenzene	"
2-Nitrophenol	"
4-Nitrophenol	"
2,4-Dinitrophenol	"
4,6-Dinitro-o-cresol	"
Phenol	"
Bis(2-ethylhexyl)phthalate	"
Butyl benzyl phthalate	"
Bi-n-butyl phthalate	"
Di-n-octyl phthalate	"
Diethyl phthtlate	"
Dimethyl phthalate	"
Benzo(a)anthracene	"
Benzo(a)pyrene	"
3,4-Benzofluoranthene	"
Benzo(k)fluoranthene	"
Crysene	"
Acenaphthalene	"
Anthracene	"
Benzo(ghi)perylene	"
Fluorene	"
Phenanthrene	"
Dibenzo(a-h)anthracene	"
Indeno(1,2,3-cd)pyrene	"
Pyrene	"
Toluene	"

* Sampling and Analysis Procedure For Screening of Industrial Effluents For Priority Pollutants. It is recommended that a 30 m fused quartz SE-54 capillary column be used.

TABLE F-3

RECOMMENDED ANALYSES FOR PROCESS WATER

Analysis	Method
NH_4^+	418A[1]
CN^-	413B[1]
SO_4^{-}	427C[1]
Total Kjeldahl Nitrogen	421[1]
$S^=$	Ion Selective Electrode
COD	508[1]
BOD	507[1]
Total Suspended Solids	160.1[2]
Conductivity	205[1]
Total Dissolved Solids	160.2[2]
Alkalinity	403[1]
pH	150.1[2]
PO_4^{-3}	425D[1]
Priority Pollutants in Table F-2	(See Table F-2)
All Table F-1 Analyses	(See Table F-1)
Total Aromatic Hydrocarbons	See Text Below
Total Organic Carbon	415.1[2]

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1) APHA - AWWA - WPCF, "Standard Methods for the Examination of Water and Wastewater" Amer. Public Health Assn., Wash., D.C., 1976, pp 1193.

2) EPA, "Methods for Chemical Analysis of Water and Wastes". EPA Document EPA-600 4-79-020, March, 1979.

TABLE F-4

RECOMMENDED ANALYSES FOR PRODUCT GAS

Analysis	Method
NO	Thermo Electron Model 14D Chemical Detection System
NO_2	
NOx	Gastec or Drager Indicator Tubes #5, 92, 10 CH30001, CH29401, CH31001, or 671901.
NH_4	Gastec 3L, 3M Drager 20501
SO_2	Thermo Electron Model 40 Pulsed UV Fluorescence Analyzer. (preceeded by SO_2 scrubber). Gas Indicator Tubes
Total S	Meloy FID
COS	GC with FPD
CS_2	"
Thiols	"
Thioethers	"
CO	In-line GC with TCD
CO_2	"
O_3	"
Hg	Collect on Au-coated glass wool, beads, warm, analyze by AAS (1).
As	Collect on Ag-coated glass wool, beads, alkali rinse, As H_3 generation, AAS (2).
HC	On-line GC with FID
Asbestos	In-line filter, microscopic examination
Pb	In-line filter, digest, AA
Be	"
Se	"
Cd	"
F	In-line filter, digest, 340.1(3)
Radiation (Radon, Gross α, β, λ)	Adsorbant Trap, count.
POM (vapor)	Tenax-GC, GC with FID
SO_4	In-line filter, 375.1^2.

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1) Braman and Johnson (1974).
2) Johnson and Braman (1975).
3) EPA, "Methods for Chemical Analysis of Water and Wastes" EPA document EPA-600 4-79-020, March, 1979.

Oil Samples

It is expected that the process oil will be collected in two containers over the course of the retorting run. These samples should be well mixed before aliquots are taken for analysis. One-liter samples should be collected in Teflon bottles and the headspace purged with nitrogen. Nitrogen should not actually be bubbled through the sample as this may cause some loss of volatiles. The samples should then be refrigerated (0-4° C).

Water Samples

It is expected that the process water will be collected in a manner similar to that for the oil. Care should be exercised in collection to minimize contamination. Storage techniques should be consistent with the analyses to be performed. One-liter samples should be taken and filtered through quartz wood or quartz fiber filters to remove oil and grease. A series of 250 ml aliquots of each one-liter sample should be:

- Transferred to acid-leached glass bottles and acidified for mercury analysis.
- Transferred to Teflon bottles and readjusted to pH = 12 with sodium hydroxide for boron and selenium analysis.
- The remainder left untreated for the other analyses and stored in Teflon bottles at 0-4° C.

Values for pH, $S^=$ and total dissolved mercury should be determined immediately on site.

Both oil and process waters will require acid predigestions if the samples are to be analyzed by AAS or AES. Care must be taken to ensure complete destruction of organic matter. The procedure described by Walker, Runels and Merryfield (1976) is recommended.

Gas Samples

Off-gas samples should be analyzed on line for those components which are amenable to such methods. Grab samples should be taken after steady-state conditions are achieved.

We recommend sampling the gas streams exiting the Stage 1 and Stage 2 reactors prior to any gas cleanup. This should provide useful data should the gas cleanup and liquid/solid separation scheme change significantly from the PDU.

Bulk gas samples may be taken using evacuated Teflon-line stainless steel bottles. Water- and NaOH solution-filled impingers may be used for sampling the gas stream for trace metals. Gas volumes between 50-300 liters are recommended. A 0.45 μ nucleopore filter should be placed before the impinger train to measure total particulate load. Gas samples for trace organic analysis may be collected in stainless steel cartridges filled with Tenax or XAD-2 resins. The two documents, IERL-RTP Procedures Manual; Level 1 Environmental Assessment, EPA-600/2-76-160A June 1976, USEPA, and IERL-RTP Interim Procedures for Level 2 Sampling and Analysis of Organic Materials, PB-279 212, February 1978 U.S. Department of Commerce, will be useful in setting up the retort gas sampling program. Another valuable document for the analysis of oil shale related samples is Fruchter, et al. (1979).

QUALITY ASSURANCE PROGRAM

"The Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", is designed to provide qualitative/semiquantitative determination of the priority pollutants. In order to apply these procedures to a more quantitative analysis, adequate quality assurance practices must be incorporated into the procedures.

The program must be well documented and be integrated into field and laboratory procedures. It must include field and laboratory blanks where appropriate, equipment calibration checks, replicate and spiked sample analysis. It is recommended that the quality assurance/quality control procedures outlined in "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", revised April, 1977 be followed with the following modifications: Oil and water samples should be spiked with stable-labeled surrogates prior to extraction and analysis. Table F-5 presents the recommended isotopically labeled compounds. These compounds are functionally similar to the priority pollutants and are used as indicators for monitoring matrix effects and gross sample processing errors.

TABLE F-5

RECOMMENDED ISOTOPICALLY LABELED SURROGATES

Compound	Fraction
d6 Benzene	VOA
d8 Toluene	VOA
d8 Ethylbenzene	VOA
d6 Phenol	Acid
d5 Anilene	Base/Neutral
d5 Pyridine	Base/Neutral
d8 Naphthalene	Base/Neutral
d10 Phenanthrene	Base/Neutral
d10 Anthracene	Base/Neutral

SAMPLING COSTS

We have collected cost data from commercial laboratories for performing specific analyses and have included this information in the Appendix.

REFERENCES

Arbuckle. Gordon, et al., Environmental Law Handbook, Government Institutes, Inc. May 1978.

Braman, R. D. and D. L. Johnson, (1974), Environmental Science Technology., 8, 996.

Chase, Clement K., The Chattanooga and Other Devonian Shales -The United States' Largest Uranium Source, Mountain States Research and Development, November 1979.

De Nevers, Noel, et al, Analysis of the Environmental Control Technology for Oil Shale Development, U.S. Department of Commerce, February 1978.

Fruchter, J.S., et al., "Source Characterization Studies at the Paraho Semiworks Oil Shale Retort", U.S. DOE, PNL-2945, May 1979.

Greenwood, D.R., Kingsbury, G.L. and Cleland, J.G., A Handbook of Key Federal Regulations and Criteria for Multimedia Environmental Control, U.S. Environmental Protection Agency, August 1979.

Hill, Ronald D. and Auerbach, Janet L., Solid Waste Disposal in the Mining Industry, U.S. Environmental Protection Agency, February 1980.

Johnson, D. L., and R. S. Braman, (1975), Chemosphere 4, 333.

Kinney, W.L., Brecheisen, A.N. and Lanbau, V.W., Surface Water Quality Parameters for Monitoring Oil Shale Development, U.S. Environmental Protection Agency, March 1979.

National Academy of Sciences, Surface Mining of Non-Coal Minerals, National Academy of Sciences, 1979.

National Academy of Sciences, Mining and Processing of Oil Shale and Tar Sands, A working paper prepared for the Committee on Surface Mining Reclamation, National Academy of Sciences, 1979.

Slawson, G.C., Groundwater Quality Monitoring of Western Oil Shale Development, U.S. Environmental Protection Agency, January 1979.

Sullivan, Thomas, Resource Conservation and Recovery Act: A Compliance Analysis, Government Institutes, Inc., January 1979.

Thoem, Terry, et al., Status of EPA Regulatory and Research Activities Affecting Oil Shale Development, U.S. Environmental Protection Agency May 1980 (draft)

U.S. Department of Energy, Environmental Monitoring and Analysis in Support of Antrim Oil Shale Research, U.S. Department of Energy, May 1979.

U.S. Environmental Protection Agency, Environmental Perspective on the Emerging Oil Shale Industry, U.S. Environmental Protection Agency, March 1980.

U.S. Environmental Protection Agency, Monitoring Environmental Impacts of the coal and Oil Shale Development, U.S. Environmental Protection Agency, February 1977.

Federal Register, Code of Federal Regulations Citations:

29 CFR Part 1910
30 CFR, Part 55
40 CFR, Parts 122-124, Parts 203-204, Parts 264-266, Part 720, Part 1502
42 FR 54165
43 FR 58946
44 FR 5001
44 FR 12746
44 FR 28557
44 FR 34387
44 FR 49402

Walker, H. H., and J. H. Runnels, Merrygield, Anal. Chem. (1976) 48, 2056.

Wilderman, T.R., and R. N. Heistand, 1979, "Trace Element Variations in an Oil Shale Retorting Operation", Fuel Division Reprints, 24, American Chemical Society.

Hazardous Waste No.	Substance[1]
U223	Toluene diisocyanate
U224	Toxaphene
	2,4,5-TP see U233
U225	Tribromomethane
U226	1,1,1-Trichloroethane
U227	1,1,2-Trichloroethane
U228	Trichloroethene
	Trichloroethylene see U228
U229	Trichlorofluoromethane
U230	2,4,5-Trichlorophenol
U231	2,4,6-Trichlorophenol
U232	2,4,5-Trichlorophenoxyacetic acid
U233	2,4,5-Trichlorophenoxypropionic acid alpha, alpha, alpha- Trichlorotoluene see U023
	TRI-CLENE see U228
U234	Trinitrobenzene (R,T)
U235	Tris(2,3-dibromopropyl) phosphate
U236	Trypan blue
U237	Uracil mustard
U238	Urethane
	Vinyl chloride see U043
	Vinylidene chloride see U078
U239	Xylene

[1] The Agency included those trade names of which it was aware; an omission of a trade name does not imply that it is not hazardous. The material is hazardous if it is used under its generic name.

Appendix I—Representative Sampling Methods

The methods and equipment used for sampling waste materials will vary with the form and consistency of the waste materials to be sampled. Samples collected using the sampling protocols listed below, for sampling waste with properties similar to the indicated materials, will be considered by the Agency to be representative of the waste.

Extremely viscous liquid—ASTM Standard D140–70 Crushed or powdered material—ASTM Standard D346–75 Soil or rock-like material—ASTM Standard D420–69 Soil-like material—ASTM Standard D1452–65

Fly Ash-like material—ASTM Standard D2234–76 [ASTM Standards are available from ASTM, 1916 Race St., Philadelphia, PA 19103]

Containerized liquid wastes—"COLIWASA" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods," [1] U.S. Environmental Protection Agency, Office of Solid Waste, Washington, D.C. 20460. [Copies may be obtained from Solid Waste Information, U.S. Environmental Protection Agency, 26 W. St. Clair St., Cincinnati, Ohio 45268]

Liquid waste in pits, ponds, lagoons, and similar reservoirs.—"Pond Sampler" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods." [1]

This manual also contains additional information on application of these protocols.

Appendix II— EP Toxicity Test Procedure

A. Extraction Procedure (EP)

1. A representative sample of the waste to be tested (minimum size 100 grams) should be obtained using the methods specified in Appendix I or any other methods capable of yielding a representative sample within the meaning of Part 260. [For detailed guidance on conducting the various aspects of the EP see "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods," SW-846, U.S. Environmental Protection Agency Office of Solid Waste, Washington, D.C. 20460.[1]]

2. The sample should be separated into its component liquid and solid phases using the method described in "Separation Procedure" below. If the solid residue[2] obtained using this method totals less than 0.5% of the original weight of the waste, the residue can be discarded and the operator should treat the liquid phase as the extract and proceed immediately to Step 8.

3. The solid material obtained from the Separation Procedure should be evaluated for its particle size. If the solid material has a surface area per gram of material equal to, or greater than, 3.1 cm^2 or passes through a 9.5 mm (0.375 inch) standard sieve, the operator should proceed to Step 4. If the surface area is smaller or the particle size larger than specified above, the solid material should be prepared for extraction by crushing, cutting or grinding the material so that it passes through a 9.5 mm (0.375 inch) sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described below.

4. The solid material obtained in Step 3 should be weighed and placed in an extractor with 16 times its weight of deionized water. Do not allow the material to dry prior to weighing. For purposes of this test, an acceptable extractor is one which will impart sufficient agitation to the mixture to not only prevent stratification of the sample and extraction fluid but also insure that all sample surfaces are continously brought into contact with well mixed extraction fluid.

5. After the solid material and deionized water are placed in the extractor, the operator should begin agitation and measure the pH of the solution in the extractor. If the pH is greater than 5.0, the pH of the solution should be decreased to 5.0 ± 0.2 by adding 0.5 N acetic acid. If the pH is equal to or less than 5.0, no acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction and if the pH rises above 5.2, 0.5N acetic acid should be added to bring the pH down to 5.0 ± 0.2. However, in no event shall the aggregate amount of acid added to the solution exceed 4 ml of acid per gram of solid. The mixture should be agitated for 24 hours and maintained at 20°–40° C (68°–104° F) during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45–A pH Controller manufactured by Chemtrix, Inc., Hillsboro, Oregon 97123 or its equivalent, in conjunction with a metering pump and reservoir of 0.5N acetic acid. If such a system is not available, the following manual procedure shall be employed:

(a) A pH meter should be calibrated in accordance with the manufacturer's specifications.

(b) The pH of the solution should be checked and, if necessary, 0.5N acetic acid should be manually added to the extractor until the pH reaches 5.0 ± 0.2. The pH of the solution should be adjusted at 15, 30 and 60 minute intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5N pH units.

(c) The adjustment procedure should be continued for at least 6 hours.

(d) If at the end of the 24-hour extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH should be adjusted to 5.0 ± 0.2 and the extraction continued for an additional four hours, during which the pH should be adjusted at one hour intervals.

6. At the end of the 24 hour extraction period, deionized water should be added to the extractor in an amount determined by the following equation:

$V = (20)(W) - 16(W) - A$
V = ml deionized water to be added
W = weight in grams of solid charged to extractor
A = ml of 0.5N acetic acid added during extraction

7. The material in the extractor should be separated into its component liquid and solid phases as described under "Separation Procedure."

8. The liquids resulting from Steps 2 and 7 should be combined. This

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These methods are also described in "Samplers and Sampling Procedures for Hazardous Waste Streams," EPA 600/2–80–018, January 1980.

[1] Copies may be obtained from Solid Waste Information, U.S. Environmental Protection Agency, 26 W. St. Clair Street, Cincinnati, Ohio 45268.

[2] The percent solids is determined by drying the filter pad at 80° C until it reaches constant weight and then calculating the percent solids using the following equation:

$$\frac{(\text{weight of pad + solid}) - (\text{tare weight of pad})}{\text{initial weight of sample}} \times 100 = \%\ \text{solids}$$

combined liquid (or the waste itself if it has less than ½ percent solids, as noted in Step 2) is the extract and should be analyzed for the presence of any of the contaminants specified in Table I of § 261.24 using the Analytical Procedures designated below.

Separation Procedure

Equipment: A filter holder, designed for filtration media having a nominal pore size of 0.45 micrometers and capable of applying a 5.3 kg/cm² (75 psi) hydrostatic pressure to the solution being filtered shall be used. For mixtures containing nonabsorptive solids, where separation can be affected without imposing a 5.3 kg/cm² pressure differential, vacuum filters employing a 0.45 micrometers filter media can be used. (For further guidance on filtration equipment or procedures see "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.")

Procedure: [3]

(i) Following manufacturer's directions, the filter unit should be assembled with a filter bed consisting of a 0.45 micrometer filter membrane. For difficult or slow to filter mixtures a prefilter bed consisting of the following prefilters in increasing pore size (0.65 micrometer membrane, fine glass fiber prefilter, and coarse glass fiber prefilter) can be used.

(ii) The waste should be poured into the filtration unit.

(iii) The reservoir should be slowly pressurized until liquid begins to flow from the filtrate outlet at which point the pressure in the filter should be immediately lowered to 10–15 psig. Filtration should be continued until liquid flow ceases.

(iv) The pressure should be increased stepwise in 10 psi increments to 75 psig and filtration continued until flow ceases or the pressurizing gas begins to exit from the filtrate outlet.

(v) The filter unit should be depressurized, the solid material removed and weighed and then transferred to the extraction apparatus, or, in the case of final filtration prior to analysis, discarded. Do not allow the material retained on the filter pad to dry prior to weighing.

(vi) The liquid phase should be stored at 4°C for subsequent use in Step 8.

B. Structural Integrity Procedure

Equipment: A Structural Integrity Tester having a 3.18 cm (1.25 in.) diameter hammer weighing 0.33 kg (0.73 lbs.) and having a free fall of 15.24 cm (6 in.) shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA., 22314, as Part No. 125, or it may be fabricated to meet the specifications shown in Figure 1.

Procedure:

1. The sample holder should be filled with the material to be tested. If the sample of waste is a large monolithic block, a portion should be cut from the block having the dimensions of a 3.3 cm (1.3 in.) diameter x 7.1 cm (2.8 in.) cylinder. For a fixated waste, samples may be cast in the form of a 3.3 cm (1.3 in.) diameter x 7.1 cm (2.8 in.) cylinder for purposes of conducting this test. In such cases, the waste may be allowed to cure for 30 days prior to further testing.
2. The sample holder should be placed into the Structural Integrity Tester, then the hammer should be raised to its maximum height and dropped. This should be repeated fifteen times.
3. The material should be removed from the sample holder, weighed, and transferred to the extraction apparatus for extraction.

Analytical Procedures for Analyzing Extract Contaminants

The test methods for analyzing the extract are as follows:

(1) For arsenic, barium, cadmium, chromium, lead, mercury, selenium or silver: "Methods for Analysis of Water and Wastes," Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268 (EPA-600/4-79-020, March 1979).

(2) For Endrin; Lindane; Methoxychlor; Toxaphene; 2,4-D; 2,4,5-TP Silver: in "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," September 1978, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 42568,

as standardized in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods."

For all analyses, the method of standard addition shall be used for the quantification of species concentration. This method is described in "Test Methods for the Evaluation of Solid Waste." (It is also described in "Methods for Analysis of Water and Wastes.")

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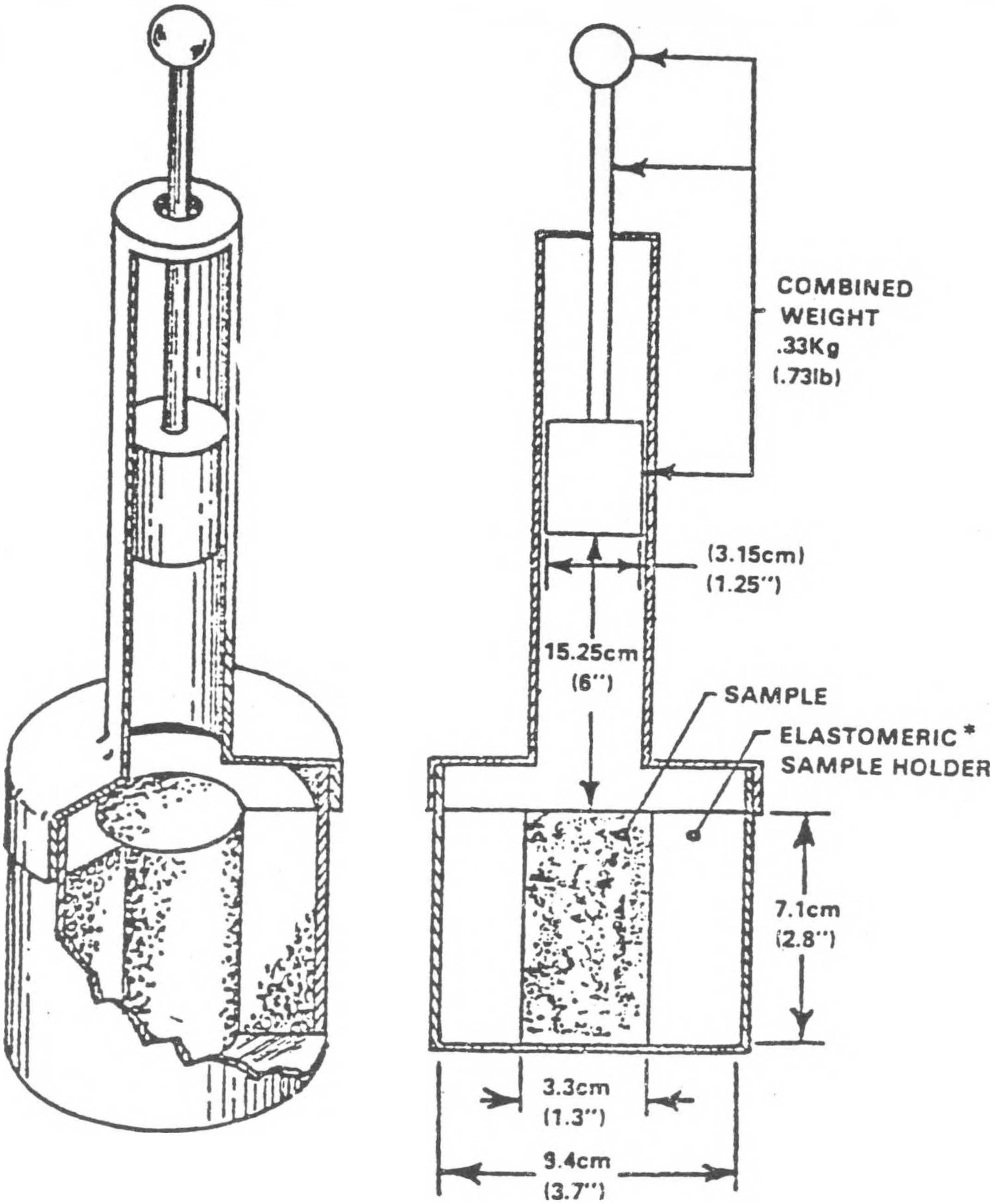
[3] This procedure is intended to result in separation of the "free" liquid portion of the waste from any solid matter having a particle size >0.45um. If the sample will not filter, various other separation techniques can be used to aid in the filtration. As described above, pressure filtration is employed to speed up the filtration process. This does not alter the nature of the separation. If liquid does not separate during filtration, the waste can be centrifuged. If separation occurs during centrifugation the liquid portion (centrifugate) is filtered through the 0.45um filter prior to becoming mixed with the liquid portion of the waste obtained from the initial filtration. Any material that will not pass through the filter after centrifugation is considered a solid and is extracted.



*ELASTOMERIC SAMPLE HOLDER FABRICATED OF MATERIAL FIRM ENOUGH TO SUPPORT THE SAMPLE

Figure 1

COMPACTION TESTER

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Appendix III—Chemical Analysis Test Methods

Tables 1, 2 and 3 specify the appropriate analytical procedures, described in "Test Methods for Evaluating Solid Waste" (SW–846), which should be used in determining whether the waste in question contains a given toxic constituent. Table 1 identifies the analytical class and the approved measurement techniques for each organic chemical listed in Appendix VII. Table 2 identifies the corresponding methods for the inorganic species. Table 3 identifies the specific sample preparation and measurement instrument introduction techniques which may be suitable for both the organic and inorganic species as well as the matrices of concern.

Prior to final selection of the analytical method the operator should consult the specific method descriptions in SW–846 for additional guidance on which of the approved methods should be employed for a specific waste analysis situation.

Table 1.—*Analytical Characteristics of Organic Chemicals*

Compound	Sample handling class/fraction	Non-GC methods	Measurement techniques		
				Conventional	
			GC/MS	GC	Detector
Acetonitrile	Volatile		8.24	8.03	NSD
Acrolein	Volatile		8.24	8.03	NSD
Acrylamide	Volatile		8.24	8.01	FID
Acrylonitrile	Volatile		8.24	8.03	NSD
Benzene	Volatile		8.24	8.02	PID
Benz(a)anthracene	Extractable/BN	8.10 (HPLC)	8.25	8.10	FID
Benzo(a)pyrene	Extractable/BN	8.10 (HPLC)	8.25	8.10	FID
Benzotrichloride	Extractable/BN		8.25	8.12	ECD
Benzyl chloride	Volatile or Extractable/BN		8.24	8.01	HSD
			8.25	8.12	ECD
Benz(b)fluoranthene	Extractable/BN	8.10 (HPLC)	8.25	8.10	FID
Bis(2-chloroethoxymethane)	Volatile		8.24	8.01	HSD
Bis(2-chloroethyl)ether	Volatile		8.24	8.01	HSD
Bis(2-chloroisopropyl)ether	Volatile		8.24	8.01	HSD
Carbon disulfide	Volatile		8.24	8.01	HSD
Carbon tetrachloride	Volatile		8.24	8.01	HSD
Chlordane	Extractable/BN		8.25	8.08	HSD
Chlorinated dibenzodioxins	Extractable/BN		8.25	8.08	ECD
Chlorinated biphenyls	Extractable/BN		8.25	8.08	HSD
Chloroacetaldehyde	Volatile		8.24	8.01	HSD
Chlorobenzene	Volatile		8.24	8.01	HSD
				8.02	PID
Chloroform	Volatile		8.24	8.01	HSD
Chloromethane	Volatile		8.24	8.01	HSD
2-Chlorophenol	Extractable/BN		8.25	8.04	FID, ECD
Chrysene	Extractable/BN	8.10 (HPLC)	8.25	8.10	FID
Creosote	Extractable/BN		8.25	8.10	ECD
Cresol(s)	Extractable/A		8.25	8.04	FID, ECD
Cresylic acid(s)	Extractable/A		8.25	8.04	FID, ECD
Dichlorobenzene(s)	Extractable/BN		8.25	8.01	HSD
				8.02	PID
				8.12	ECD
Dichloroethane(s)	Volatile		8.24	8.01	HSD
Dichloromethane	Volatile		8.24	8.01	HSD
Dichlorophenoxy-acetic acid	Extractable/A		8.25	8.40	HSD
Dichloropropanol	Extractable/BN		8.25	8.12	ECD
2,4-Dimethylphenol	Extractable/A		8.25	8.04	FID, ECD
Dinitrobenzene	Extractable/BN		8.25	8.09	FID, ECD
4,6-Dinitro-o-cresol	Extractable/A		8.25	8.04	FID, ECD
2,4-Dinitrotoluene	Extractable/BN		8.25	8.09	FID, ECD
Endrin	Extractable/P		8.25	8.08	HSD
Ethyl ether	Volatile		8.24	8.01	FID
				8.02	FID
Formaldehyde	Volatile		8.24	8.01	FID
Formic acid	Extractable/BN		8.25	8.06	FID
Heptachlor	Extractable/P		8.25	8.08	HSD
Hexachlorobenzene	Extractable/BN		8.25	8.12	ECD
Hexachlorobutadiene	Extractable/BN		8.25	8.12	ECD
Hexachloroethane	Extractable/BN		8.25	8.12	ECD
Hexachlorocyclopentadiene	Extractable/BN		8.25	8.12	ECD
Lindane	Extractable/P		8.25	8.08	HSD
Maleic anhydride	Extractable/BN		8.25	8.06	ECD, FID
Methanol	Volatile		8.24	8.01	FID
Methomyl	Extractable/BN	8.32 (HPLC)			
Methyl ethyl ketone	Volatile		8.25	8.01	FID
				8.02	FID
Methyl isobutyl ketone	Volatile		8.25	8.01	FID
				8.02	FID
Naphthalene	Extractable/BN		8.25	8.10	FID
Napthoquinone	Extractable/BN		8.25	8.06	ECD, FID
				8.09	FID
Nitrobenzene	Extractable/BN		8.25	8.09	ECD, FID
4-Nitrophenol	Extractable/A		8.24	8.04	ECD, FID
Paraldehyde (trimer of acetaldehyde)	Volatile		8.24	8.01	FID

Table 1.—*Analytical Characteristics of Organic Chemicals—Continued*

Compound	Sample handling class/fraction	Non-GC methods	Measurement techniques		
			GC/MS	Conventional	
				GC	Detector
Pentachlorophenol	Extractable/A		8.25	8.04	ECD
Phenol	Extractable/A		8.25	8.04	ECD, FID
Phorate	Extractable/BN			8.22	FPD
Phosphorodithioic acid esters	Extractable/BN			8.06 8.09 8.22	ECD, FID ECD, FID FPD
Phthalic anhydride	Extractable/BN		8.25	8.06 8.09	ECD, FID ECD, FID
2-Picoline	Extractable/BN		8.25	8.06 8.09	ECD, FID ECD, FID
Pyridine	Extractable/BN		8.25	8.06 8.09	ECD, FID ECD, FID
Tetrachlorobenzene(s)	Extractable/BN		8.25	8.12	ECD
Tetrachloroethane(s)	Volatile		8.24	8.01	HSD
Tetrachloroethene	Volatile		8.24	8.01	HSD
Tetrachlorophenol	Extractable/A		8.24	8.04	ECD
Toluene	Volatile		8.24	8.02	PID
Toluenediamine	Extractable/BN		8.25		
Toluene diisocyanate(s)	Extractable/nonaqueous		8.25	8.06	FID
Toxaphene	Extractable/P		8.25	8.06	HSD
Trichloroethane	Volatile		8.24	8.01	HSD
Trichloroethene(s)	Volatile		8.24	8.01	HSD
Trichlorofluoromethane	Volatile		8.24	8.01	HSD
Trichlorophenol(s)	Extractable/A		8.25	8.04	HSD
2,4,5-TP (Silvex)	Extractable/A		8.25	8.40	HSD
Trichloropropane	Volatile		8.24	8.01	HSD
Vinyl chloride	Volatile		8.24	8.01	HSD
Vinylidene chloride	Volatile		8.24	8.01	HSD
Xylene	Volatile		8.24	8.02	PID

[1] Analyze for phenanthrene and carbazole; if these are present in a ratio between 1.4:1 and 5:1, creosote should be considered present.

ECD = Electron capture detetor; FID = Flame ionization detector; FPD = Flame photometric detector; HSD = Halide specific detector; HPLC = High pressure liquid chromotography; NSD = Nitrogen-specific detector; PID = Photoionization detector.

Table 2—*Analytical Characteristics of Inorganic Species*

Species	Sample handling class	Measurement technique	Method number
Antimony	Digestion	Atomic absorbtion-furnace/flame	8.50
Arsenic	Hydride	Atomic absorbtion-flame	8.51
Barium	Digestion	Atomic absorbtion-furnace/flame	8.52
Cadmium	Digestion	Atomic absorbtion-furnace/flame	8.53
Chromium	Digestion	Atomic absorbtion-furnace/flame	8.54
Cyanides	Hydrolysis	Atomic absorbtion-spectroscopy	8.55
Lead	Digestion	Atomic absorbtion-furnace/flame	8.56
Mercury	Cold Vapor	Atomic absorbtion	8.57
Nickel	Digestion	Atomic absorbtion-furnace/flame	8.58
Selenium	Hydride digestion	Atomic absorbtion-furnace/flame	8.59
Silver	Digestion	Atomic absorbtion-furnace/flame	8.60

Table 3.—*Sample Prepartion/Sample Introduction Techniques*

Sample handling class	Physical characteristics of waste [1]		
	Fluid	Paste	Solid
Volatile	Purge and trap. Direct injection.	Purge and trap. Headspace	Headspace.
Semivolatile and nonvolatile.	Direct injection. Shake out	Shake out	Shake out. Soxhlet. Sonication.
Inorganic	Direct injection. Digestion Hydride	 Digestion Hydride	 Digestion. Hydride.

[1] For purposes of this Table, fluid refers to readily pourable liquids, which may or may not contain suspended particles. Paste-like materials, while fluid in the sense of flowability, can be thought of as being thixotropic or plastic in nature, e.g. paints. Solid materials are those wastes which can be handled without a container (i.e., can be piled up without appreciable sagging).

Procedure and Method Number(s)

Digestion—See appropriate procedure for element of interest.
Direct injection—8.80
Headspace—8.82
Hydride—See appropriate procedure for element of interest.
Purge & Trap—8.83
Shake out—8.84
Sonication—8.85
Soxhlet—8.86

Appendix VII.—*Basis for Listing Hazardous Wastes*

EPA hazardous waste No.	Hazardous constituents for which listed
F001	tetrachloroethylene, methylene chloride trichloroethylene, 1,1,1-trichloroethane chlorinated fluorocarbons, carbon tetrachloride
F002	tetrachloroethylene, methylene chloride, trichloroethylene, 1,1,1-trichloroethane, chlorobenzene, 1,1,2-trichloro-1,2,2-trifluoroethane, o-dichlorobenzene, trichlorofluoromethane
F003	N.A.
F004	cresols and cresylic acid, nitrobenzene
F005	methanol, toluene, methyl ethyl ketone, methyl isobutyl ketone, carbon disulfide, isobutanol, pyridine
F006	cadmium, chromium, nickel, cyanide (complexed)
F007	cyanide (salts)
F008	cyanide (salts)
F009	cyanide (salts)
F010	cyanide (salts)
F011	cyanide (salts)
F012	cyanide (complexed)
F013	cyanide (complexed)
F014	cyanide (complexed)
F015	cyanide (salts)
F016	cyanide (complexed)
K001	benzene, benz(a)anthracene, benzo(a)pyrene, chrysene, 4-nitrophenol, toluene, naphthalene phenol, 2-chlorophenol, 2,4-dimethyl phenol, 2,4,6-trichlorophenol, pentachlorophenol, 4,6-dinitro-o-cresol, tetrachlorophenol
K002	chromium, lead
K003	chromium, lead
K004	chromium
K005	chromium, lead
K006	chromium
K007	cyanide (complexed), chromium
K008	chromium
K009	chloroform, formaldehyde, methylene chloride, methyl chloride, paraldehyde, formic acid
K010	chloroform, formaldehyde, methylene chloride, methyl chloride, paraldehyde, formic acid, chloroacetaldehyde
K011	acrylonitrile, acetonitrile, hydrocyanic acid
K012	acrylonitrile, acetonitrile, acrolein, acrylamide
K013	hydrocyanic acid, acrylonitrile, acetonitrile
K014	acetonitrile, acrylamide
K015	benzyl chloride, chlorobenzene, toluene, benzotrichloride
K016	hexachlorobenzene, hexachlorobutadiene, carbon tetrachloride, hexachloroethane, perchloroethylene
K017	epichlorohydrin, chloroethers [bis(chloromethyl) ether and bis (2-chloroethyl) ethers], trichloropropane, dichloropropanols
K018	1,2-dichloroethane, trichloroethylene, hexachlorobutadiene, hexachlorobenzene
K019	ethylene dichloride, 1,1,1-trichloroethane, 1,1,2-trichloroethane, tetrachloroethanes (1,1,2,2-tetrachloroethane and 1,1,1,2-tetrachloroethane), trichloroethylene, tetrachloroethylene, carbon tetrachloride, chloroform, vinyl chloride, vinylidene chloride

Appendix VII.—*Basis for Listing Hazardous Wastes*—Continued

EPA hazardous waste No.	Hazardous constituents for which listed
K020	ethylene dichloride, 1,1,1-trichloroethane, 1,1,2-trichloroethane, tetrachloroethanes (1,1,2,2-tetrachloroethane and 1,1,1,2-tetrachloroethane), trichloroethylene, tetrachloroethylene, carbon tetrachloride, chloroform, vinyl chloride, vinylidene chloride
K021	antimony, carbon tetrachloride, chloroform
K022	phenol, tars (polycyclic aromatic hydrocarbons)
K023	phthalic anhydride, maleic anhydride
K024	phthalic anhydride, polynuclear tar-like materials, naphthoquinone
K025	meta-dinitrobenzene, 2,4-dinitrotoluene
K026	paraldehyde, pyridines, 2-picoline
K027	toulene diisocyanate, toluene-2,4-diamine, tars (benzdimidazapone)
K028	1,1,1-trichloroethane, vinyl chloride
K029	1,2-dichloroethane, 1,1,1-trichloroethane, vinyl chloride, vinylidene chloride, chloroform
K030	hexachlorobenzene, hexachlorobutadiene, hexachloroethane, 1,1,1,2-tetrachloroethane, 1,1,2,2-tetrachloroethane, ethylene dichloride
K031	arsenic
K032	hexachlorocyclopentadiene
K033	hexachlorocyclopentadiene
K034	hexachlorocyclopentadiene
K035	creosote, benz(a)anthracene, benz(b)fluoranthene, benzo(a)pyrene
K036	toulene, phosphorodithoic and phosphorothioic acid esters
K037	toulene, phosphorodithoic and phosphorothioic acid esters
K038	phorate, formaldehyde, phosphorodithioic and phosphorothioic acid esters
K039	phosphorodithioic and phosphorothioic acid esters
K040	phorate, formaldehyde, phosphorodithioic and phosphorothioic acid esters
K041	toxaphene
K042	hexachlorobenzene, ortho-dichlorobenzene
K043	2,4-dichlorophenol, 2,6-dichlorophenol, . 2,4,6-trichlorophenol
K044	N.A.
K045	N.A.
K046	lead
K047	N.A.
K048	chromium, lead
K049	chromium, lead
K050	chromium
K051	chromium, lead
K052	lead
K053	chromium
K054	chromium
K055	chromium, lead
K056	chromium, lead
K057	chromium, lead
K058	chromium, lead
K059	N.A.
K060	cyanide, naphthalene, phenolic compounds, arsenic
K061	chromium, lead, cadmium
K062	chromium, lead
K063	chromium, lead
K064	lead, cadmium
K065	lead, cadmium
K066	lead, cadmium
K067	lead, cadmium
K068	lead, cadmium
K069	chromium, lead, cadmium

N.A.—Waste is hazardous because it meets either the ignitability, corrosivity or reactivity characteristic.

Appendix VIII—Hazardous Constituents

Acetaldehyde
(Acetato)phenylmercury
Acetonitrile
3-(alpha-Acetonylbenzyl)-4-hydroxycoumarin and salts
2-Acetylaminofluorene
Acetyl chloride
1-Acetyl-2-thiourea
Acrolein
Acrylamide
Acrylonitrile
Aflatoxins
Aldrin
Allyl alcohol
Aluminum phosphide
4-Aminobiphenyl
6-Amino-1,1a,2,8,8a,8b-hexahydro-8-(hydroxymethyl)-8a-methoxy-5-methylcarbamate azirino(2',3':3,4) pyrrolo(1,2-a)indole-4,7-dione (ester) (Mitomycin C)
5-(Aminomethyl)-3-isoxazolol
4-Aminopyridine
Amitrole
Antimony and compounds, N.O.S.[1]
Aramite
Arsenic and compounds, N.O.S.
Arsenic acid
Arsenic pentoxide
Arsenic trioxide
Auramine
Azaserine
Barium and compounds, N.O.S.
Barium cyanide
Benz[c]acridine
Benz[a]anthracene
Benzene
Benzenearsonic acid
Benzenethiol
Benzidine
Benzo[a]anthracene
Benzo[b]fluoranthene
Benzo[j]fluoranthene
Benzo[a]pyrene
Benzotrichloride
Benzyl chloride
Beryllium and compounds, N.O.S.
Bis(2-chloroethoxy)methane
Bis(2-chloroethyl) ether
N,N-Bis(2-chloroethyl)-2-naphthylamine
Bis(2-chloroisopropyl) ether
Bis(chloromethyl) ether
Bis(2-ethylhexyl) phthalate
Bromoacetone
Bromomethane
4-Bromophenyl phenyl ether
Brucine
2-Butanone peroxide
Butyl benzyl phthalate
2-sec-Butyl-4,6-dinitrophenol (DNBP)
Cadmium and compounds, N.O.S.
Calcium chromate
Calcium cyanide
Carbon disulfide
Chlorambucil
Chlordane (alpha and gamma isomers)
Chlorinated benzenes, N.O.S.
Chlorinated ethane, N.O.S.
Chlorinated naphthalene, N.O.S.
Chlorinated phenol, N.O.S.
Chloroacetaldehyde
Chloroalkyl ethers
p-Chloroaniline
Chlorobenzene
Chlorobenzilate
1-(p-Chlorobenzoyl)-5-methoxy-2-methylindole-3-acetic acid
p-Chloro-m-cresol
1-Chloro-2,3-epoxybutane
2-Chloroethyl vinyl ether
Chloroform
Chloromethane
Chloromethyl methyl ether
2-Chloronaphthalene
2-Chlorophenol
1-(o-Chlorophenyl)thiourea
3-Chloropropionitrile
alpha-Chlorotoluene
Chlorotoluene, N.O.S.
Chromium and compounds, N.O.S.
Chrysene
Citrus red No. 2
Copper cyanide
Creosote
Crotonaldehyde
Cyanides (soluble salts and complexes), N.O.S.
Cyanogen
Cyanogen bromide
Cyanogen chloride
Cycasin
2-Cyclohexyl-4,6-dinitrophenol
Cyclophosphamide
Daunomycin
DDD
DDE
DDT
Diallate
Dibenz[a,h]acridine
Dibenz[a,j]acridine
Dibenz[a,h]anthracene(Dibenzo[a,h] anthracene)
7H-Dibenzo[c,g]carbazole
Dibenzo[a,e]pyrene
Dibenzo[a,h]pyrene
Dibenzo[a,i]pyrene
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
Dibromomethane
Di-n-butyl phthalate
Dichlorobenzene, N.O.S.
3,3'-Dichlorobenzidine
1,1-Dichloroethane
1,2-Dichloroethane
trans-1,2-Dichloroethane
Dichloroethylene, N.O.S.
1,1-Dichloroethylene
Dichloromethane
2,4-Dichlorophenol
2,6-Dichlorophenol
2,4-Dichlorophenoxyacetic acid (2,4-D)
Dichloropropane
Dichlorophenylarsine
1,2-Dichloropropane
Dichloropropanol, N.O.S.
Dichloropropene, N.O.S.
1,3-Dichloropropene
Dieldrin
Diepoxybutane
Diethylarsine
0,0-Diethyl-S-(2-ethylthio)ethyl ester of phosphorothioic acid
1,2-Diethylhydrazine
0,0-Diethyl-S-methylester phosphorodithioic acid
0,0-Diethylphosphoric acid, 0-p-nitrophenyl ester
Diethyl phthalate
0,0-Diethyl-0-(2-pyrazinyl)phosphorothioate
Diethylstilbestrol
Dihydrosafrole
3,4-Dihydroxy-alpha-(methylamino)-methyl benzyl alcohol
Di-isopropylfluorophosphate (DFP)
Dimethoate
3,3'-Dimethoxybenzidine
p-Dimethylaminoazobenzene
7,12-Dimethylbenz[a]anthracene
3,3'-Dimethylbenzidine
Dimethylcarbamoyl chloride

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[1] The abbreviation N.O.S. signifies those members of the general class "not otherwise specified" by name in this listing.

1,1-Dimethylhydrazine
1,2-Dimethylhydrazine
3,3-Dimethyl-1-(methylthio)-2-butanone-O-[(methylamino) carbonyl]oxime
Dimethylnitrosoamine
alpha,alpha-Dimethylphenethylamine
2,4-Dimethylphenol
Dimethyl phthalate
Dimethyl sulfate
Dinitrobenzene, N.O.S.
4,6-Dinitro-o-cresol and salts
2,4-Dinitrophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene Di-n-octyl phthalate
1,4-Dioxane
1,2-Diphenylhydrazine
Di-n-propylnitrosamine
Disulfoton
2,4-Dithiobiuret
Endosulfan
Endrin and metabolites
Epichlorohydrin
Ethyl cyanide
Ethylene diamine
Ethylenebisdithiocarbamate (EBDC)
Ethyleneimine
Ethylene oxide
Ethylenethiourea
Ethyl methanesulfonate
Fluoranthene
Fluorine
2-Fluoroacetamide
Fluoroacetic acid, sodium salt
Formaldehyde
Glycidylaldehyde
Halomethane, N.O.S.
Heptachlor
Heptachlor epoxide (alpha, beta, and gamma isomers)
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclohexane (all isomers)
Hexachlorocyclopentadiene
Hexachloroethane
1,2,3,4,10,10-Hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-endo,endo-dimethanonaphthalene
Hexachlorophene
Hexachloropropene
Hexaethyl tetraphosphate
Hydrazine
Hydrocyanic acid
Hydrogen sulfide
Indeno(1,2,3-c,d)pyrene
Iodomethane
Isocyanic acid, methyl ester
Isosafrole
Kepone
Lasiocarpine
Lead and compounds, N.O.S.
Lead acetate
Lead phosphate
Lead subacetate
Maleic anhydride
Malononitrile
Melphalan
Mercury and compounds, N.O.S.
Methapyrilene
Methomyl
2-Methylaziridine
3-Methylcholanthrene
4,4'-Methylene-bis-(2-chloroaniline)
Methyl ethyl ketone (MEK)
Methyl hydrazine
2-Methyllactonitrile
Methyl methacrylate
Methyl methanesulfonate
2-Methyl-2-(methylthio)propionaldehyde-o-(methylcarbonyl) oxime
N-Methyl-N'-nitro-N-nitrosoguanidine
Methyl parathion
Methylthiouracil
Mustard gas
Naphthalene
1,4-Naphthoquinone
1-Naphthylamine
2-Naphthylamine
1-Naphthyl-2-thiourea
Nickel and compounds, N.O.S.
Nickel carbonyl
Nickel cyanide
Nicotine and salts
Nitric oxide
p-Nitroaniline
Nitrobenzene
Nitrogen dioxide
Nitrogen mustard and hydrochloride salt
Nitrogen mustard N-oxide and hydrochloride salt
Nitrogen peroxide
Nitrogen tetroxide
Nitroglycerine
4-Nitrophenol
4-Nitroquinoline-1-oxide
Nitrosamine, N.O.S.
N-Nitrosodi-N-butylamine
N-Nitrosodiethanolamine
N-Nitrosodiethylamine
N-Nitrosodimethylamine
N-Nitrosodiphenylamine
N-Nitrosodi-N-propylamine
N-Nitroso-N-ethylurea
N-Nitrosomethylethylamine
N-Nitroso-N-methylurea
N-Nitroso-N-methylurethane
N-Nitrosomethylvinylamine
N-Nitrosomorpholine
N-Nitrosonornicotine
N-Nitrosopiperidine
N-Nitrosopyrrolidine
N-Nitrososarcosine
5-Nitro-o-toluidine
Octamethylpyrophosphoramide
Oleyl alcohol condensed with 2 moles ethylene oxide
Osmium tetroxide
7-Oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid
Parathion
Pentachlorobenzene
Pentachloroethane
Pentachloronitrobenzene (PCNB)
Pentacholorophenol
Phenacetin
Phenol
Phenyl dichloroarsine
Phenylmercury acetate
N-Phenylthiourea
Phosgene
Phosphine
Phosphorothioic acid, O,O-dimethyl ester, O-ester with N,N-dimethyl benzene sulfonamide
Phthalic acid esters, N.O.S.
Phthalic anhydride
Polychlorinated biphenyl, N.O.S.
Potassium cyanide
Potassium silver cyanide
Pronamide
1,2-Propanediol
1,3-Propane sultone
Propionitrile
Propylthiouracil
2-Propyn-1-ol
Pryidine
Reserpine
Saccharin
Safrole
Selenious acid
Selenium and compounds, N.O.S.
Selenium sulfide
Selenourea
Silver and compounds, N.O.S.
Silver cyanide
Sodium cyanide
Streptozotocin
Strontium sulfide
Strychnine and salts
1,2,4,5-Tetrachlorobenzene
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
Tetrachloroethane, N.O.S.
1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane
Tetrachloroethene (Tetrachloroethylene)
Tetrachloromethane
2,3,4,6-Tetrachlorophenol
Tetreethyldithiopyrophosphate
Tetraethyl lead
Tetraethylpyrophosphate
Thallium and compounds, N.O.S.
Thallic oxide
Thallium (I) acetate
Thallium (I) carbonate
Thallium (I) chloride
Thallium (I) nitrate
Thallium selenite
Thallium (I) sulfate
Thioacetamide
Thiosemicarbazide
Thiourea
Thiuram
Toluene
Toluene diamine
o-Toluidine hydrochloride
Tolylene diisocyanate
Toxaphene
Tribromomethane
1,2,4-Trichlorobenzene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene (Trichloroethylene)
Trichloromethanethiol
2,4,5-Trichlorophenol
2,4,6-Trichlorophenol
2,4,5-Trichlorophenoxyacetic acid (2,4,5-T)
2,4,5-Trichlorophenoxypropionic acid (2,4,5-TP) (Silvex)
Trichloropropane, N.O.S.
1,2,3-Trichloropropane
O,O,O-Triethyl phosphorothioate
Trinitrobenzene
Tris(1-azridinyl)phosphine sulfide
Tris(2,3-dibromopropyl) phosphate
Trypan blue
Uracil mustard
Urethane
Vanadic acid, ammonium salt
Vanadium pentoxide (dust)
Vinyl chloride
Vinylidene chloride
Zinc cyanide
Zinc phosphide

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APPENDIX B

COST OF RECOMMENDED SAMPLING PROGRAM

Cost estimates for the program were derived from commercial laboratories and do not reflect gas analyses performed on line. The costs are based on group analyses, therefore costing less than individual determinations. Listed below are the methods, groups of analyses associated with the method, and the estimated cost.

Method	Compound or Elements	Cost
Instrumental neutron activation analysis	Ca, Mg, Na, K, F, As, Se, Al, Ba, Cl, Cr, Cu, Fe, Mn, Ni, P, Zn, Sb, Ag, Mo, Sr, V, U, Th, Zr	\$200 - \$300
Combustion method	N, S	\$60
Atomic absorption - emission spectrophotometry	Fb, Be, Hg, Cd, Tl, B	\$203
Radioactivity	Gross α, β, γ	\$35
Priority pollutant analysis	acenaphthene benzene benzidine 2,4-dinitrotoluene 2,6-dinitrotoluene 1,2-diphenylhydrazine ethylbenzene fluoranthene isophorone naphthalene nitrobenzene 2-nitrophenol 4-nitrophenol 2,4-dinitrophenol 4,6-dinitro-o-cresol phenol bis (2-ethylhexyl) phthalate	\$648

	butyl benzyl phthalate di-n-butyl phthalate di-n-octyl phthalate diethyl phthalate benzo (a) anthracene benzo (a) pyrene 3,4-benzofluoranthene chrysene acenaphthalene anthracene benzo (ghi) perylene fluorene phenanthrene dibenzo (ah) anthracene indeno (1,2,3-cd) pyrene pyrene toluene	
Total extractables	Acids, bases, neutrals	$120
TOC	Total organic carbon	$35
Electron microscopy	Asbestos	$150 - $300
Gas Chromatography - FID	POM	$50
Standard Methods	NH_4^{-1}, CN^{-1}, SO_4^{-2}, TKN, S^{-2}, COD, BOD, TSS, Conductivity, TDS, Alkalinity, pH, PO_4^{-3}	$230