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DEVELOPMENT OF SIGNIFICANTLY IMPROVED CATALYSTS FOR
COAL LIQUEFACTION AND UPGRADING OF COAL EXTRACTS

Quarterly Progress Report No. 4 for July 1–September 30, 1982

By

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Work Performed Under Contract No. AC22-81PC40091

American Cyanamid Company
Chemical Research Division
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Technical Information Center
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United States Department of Energy

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DEVELOPMENT OF SIGNIFICANTLY IMPROVED CATALYSTS
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QUARTERLY PROGRESS REPORT NO. 4

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CONTRACT NO. DE-AC22-81PC40091

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SUMMARY

This Quarterly Progress Report covers work on Tasks 3, 5, 6 and 8 during the period July-September 1982.

Task 3 - Cold flow ebullation tests to determine the ranges of operability of bead catalysts continued. Data reported show the effects of higher catalyst density, wider particle size distributions, and higher fluid viscosity on ebullation of bead catalysts. A relation for determining limiting diameters in a liquid-solid fluidized bed was developed. Correlation of the three-phase data is being investigated. An explosion-proof cold flow test unit was constructed.

Tasks 5 and 6 - The CSTR Catalyst Aging Test Unit is described. The system operates under computer control. High pressure hydrogen and coal slurry are fed continuously to a one-liter stirred autoclave. High pressure nitrogen for inerting and flush oil are used in the event of an interlock shutdown. The product is passed to a 3-gallon collection vessel or to a smaller sampling vessel during material balance periods. The gas disentrains from the mixture and is scrubbed.

Analytical techniques to be used in the course of the catalyst testing program were evaluated. H-COAL[®] PDU products and SRC-II Heavy distillate were analyzed. Standard deviations for many of the procedures were determined. Specific methods are summarized.

Task 8 - Development of surface analytical techniques to study coal liquefaction catalysts focused on used Amocat 1A catalysts from coal run 10 at the H-COAL Pilot Plant. Preliminary results of analysis by ESCA and X-ray microprobe are given. Further work planned is described.

TABLE OF CONTENTS

	<u>Page</u>
1. Introduction	1
2. Objectives and Scope	2
3. TASK 3: COLD FLOW EBULLATION TESTS	
3.1 Introduction	3
3.2 Equipment	3
3.3 Experimental Work	6
3.3.1 Data Collection	6
3.3.2 Tabulation and Collected Data	6
3.4 Results and Discussion	6
3.5 Data Analysis	20
3.5.1 Richardson-Zaki Type Correlation	20
3.5.2 Calculations for Polydisperse Systems using the Correlations of Richardson and Zaki . .	20
3.5.3 Limiting Diameters for Polydisperse Systems	22
3.5.4 Correlation of Three-Phase Data	24
3.6 Explosion-Proof Cold Flow Test Unit	32
3.7 Conclusions	32
3.8 References	34
4. TASKS 5 and 6: CATALYST BATCH SCREENING AND CONTINUOUS AGING TESTS	
4.1 CSTR Catalyst Aging Test Unit	35
4.2 Analytical Techniques	41
4.2.1 H-COAL® Products	42
4.2.2 SRC-II Heavy Distillate	46
4.2.3 D1160 Distillations	46
4.2.4 Conclusions	52
4.2.5 Summary of Analytical Procedures	52
4.3 References	55

Table of Contents (Cont'd)

	<u>Page</u>
5. TASK 8: ANALYTICAL METHODS REVIEW	
5.1 Introduction	57
5.2 Experimental	57
5.3 Results and Discussion	60
5.4 Future Work	70
5.5 References	78
APPENDIX I: Tabulation of Collected Data - TASK 3	79-90

LIST OF TABLES

	<u>Page</u>
3. TASK 3: COLD FLOW EBULLATION TESTS	
Table 3.1 Bead-Fluid Combinations	4
3.2 Calculated Bed Heights, Liquid + Gas Fractions and Bed Expansions for -6 +6 Mesh HD Beads, Kerosene, Nitrogen	8
3.3 Calculated Bed Heights, Liquid + Gas Fractions and Bed Expansions for -5 +18 Mesh Beads, 5 cp Fluid, Nitrogen	9
3.4 Calculated Bed Heights, Liquid + Gas Fractions and Bed Expansions for -12 +20 Mesh Beads, Kerosene, Nitrogen	10
3.5 Calculated Bed Heights, Liquid + Gas Fractions and Bed Expansions for -10 +20 Mesh Beads, Kerosene, Nitrogen	11
3.6 Calculated Bed Heights, Liquid + Gas Fractions and Bed Expansions for -8 +25 Mesh Beads, Kerosene, Nitrogen	12
3.7 Calculated Bed Heights, Liquid + Gas Fractions and Bed Expansions for -6 +25 Mesh Beads, Kerosene, Nitrogen	13
3.8 Calculated Bed Heights, Liquid + Gas Fractions and Bed Expansions for -6 +30 Mesh Beads, Kerosene, Nitrogen	14
3.9 Calculated Bed Heights, Liquid + Gas Fractions and Bed Expansions for -6 +40 Mesh Beads, Kerosene, Nitrogen	15
3.10 Mesh Size Composition of 1.3mm Beads	17
3.11 Exponents and Terminal Velocities for 1.3mm Beads from Fits of Richardson-Zaki Type Correlation	21
4. TASKS 5 and 6: CATALYST BATCH SCREENING AND CONTINUOUS AGING TESTS	
4.1 Analysis of H-COAL® PDU Products	43
4.2 Properties of SRC II Heavy Distillate (FSN-12)	47

List of Tables (Cont'd)

Page

5. TASK 8: ANALYTICAL METHODS REVIEW

Table 5.1	Bulk Concentration of Catalyst Powders (weight %) from EDX Work	61
Table 5.2	Atomic Concentrations (%) from ESCA Data of Catalyst Extrudates	63

LIST OF FIGURES

	<u>Page</u>
3. TASK 3: COLD FLOW EBULLATION TESTS	
Figure 3.1 Schematic Diagram of Cold Flow Equipment . .	5
3.2 Incremental Effect of Gas Flow on Liquid + Gas Porosity, -8 +12 Mesh	26
3.3 Incremental Effect of Gas Flow on Liquid + Gas Porosity, -5 +12 Mesh	27
3.4 Incremental Effect of Gas Flow on Liquid + Gas Porosity, -5 +18 Mesh	28
3.5 Incremental Effect of Gas Flow on Liquid + Gas Porosity, -8 +25 Mesh	29
3.6 Incremental Effect of Gas Flow on Liquid + Gas Porosity, -6 +30 Mesh	30
3.7 Incremental Effect of Gas Flow on Liquid + Gas Porosity, -6 +40 Mesh	31
4. TASKS 5 and 6: CATALYST BATCH SCREENING AND CONTINUOUS AGING TESTS	
4.1 CSTR Coal Liquefaction Unit	36
4.2 D1160 Distillation Vacuum Still Overheads . .	49
4.3 D1160 Distillation Atmospheric Still Bottoms.	50
4.4 D1160 Distillation SRC-II Heavy Distillate	51
5. TASK 8: ANALYTICAL METHODS REVIEW	
5.1 ESCA Survey Scan of Used Amocat 1A	62
5.2 Secondary Electron Images of Crossections of Amocat 1A	64
5.3 Co, Mo, and Al X-ray Intensities Relative to Distance Across a Catalyst Pellet	66
5.4 Energy Dispersive X-ray Spectra of Fresh Amocat 1A Catalyst	67
5.5 Energy Dispersive X-ray Spectra of Amocat 1A Catalysts	68

LIST OF FIGURES (Cont'd)

	<u>Page</u>
5. TASK 8: (Cont'd)	
5.6 Wavelength Dispersive Linescans of Used Catalyst	71
5.7 X-ray Maps of Fresh Catalyst Taken with the Wavelength Dispersive Detector	72
5.8 X-ray Maps of Used Catalyst Taken with the Wavelength Dispersive Detector (Co, Mo, S). .	73
5.9 X-ray Maps of Used Catalyst Taken with the Wavelength Dispersive Detector (Fe, Ca, Ti) .	75

1. INTRODUCTION

During 1979-80, a new generation of very active long-lived catalysts for hydrotreating was discovered at the Stamford Research Laboratories of the American Cyanamid Company. The catalysts are based on a unique substrate prepared in bead form from a rehydratable alumina. Their spherical shape, crush strength, and abrasion resistance make them ideal candidates for use in ebullated bed reactors. The beads have internal pore structures that are controllable over a wider range than conventional alumina supports, leading to active catalysts that are resistant to poisoning.

In September, 1981, the Department of Energy granted a 3-year contract to the American Cyanamid Company for the development of significantly improved catalysts for coal liquefaction and for upgrading coal extracts, utilizing bead substrates.

2. OBJECTIVES AND SCOPE

The primary objective of this project is to develop one or more catalysts for coal liquefaction and/or for upgrading coal extracts which will be superior to the best currently known catalysts. The superiority will be judged by considering both the activity and the life of the catalysts. Catalysts will first be screened in a batch reactor to identify promising candidates. The latter will be tested in a continuous aging reactor to evaluate their resistance to deactivation under coal-liquefaction and extract upgrading conditions. In addition, a program of cold flow ebullation tests is being carried out to determine the median size and size range of beads compatible with operation of ebullated bed reactors. Furthermore, special analytical methods will be used to characterize catalysts before and after performance testing to elucidate the mechanism of catalyst deactivation.

The work of the contract has been divided into the following eight tasks:

- Task 1: Project Work Plan - Completed
- Task 2: State-of-the-Art Assessment - Completed
- Task 3: Cold Flow-Ebullation Tests
- Task 4: Bead Catalyst Characterization
- Task 5: Catalyst Batch Screening
- Task 6: Continuous Aging Tests
- Task 7: Catalyst Regenerability
- Task 8: Analytical Methods Review

This Quarterly Progress Report covers work on Tasks 3, 5, 6 and 8 during the period July - September, 1982.

3. Task 3: COLD FLOW EBULLATION TESTS

3.1 Introduction

Cold flow ebullation tests to determine the ranges of operability of bead catalysts of various particle sizes and particle-size distributions have continued. Table 3.1 shows the bead-fluid combinations which have been tested and those that remain. The listing in this table differs from that in the previous Quarterly Report. The changes are that some of the runs with the 5cp fluid have been dropped and many runs with kerosene have been added to explore the effect of changing the width of a distribution from narrow to broad.

3.2 Equipment

The equipment is shown schematically in Figure 3.1 and has remained unchanged since March, 1982. The fluidizing liquid is pumped to the bottom of the column where it passes through a section filled with glass beads before entering the test-section through a catalyst retaining screen. Nitrogen is introduced through a sparger immediately above the screen. The sparger has six radial arms with 1/64-inch holes in each arm. The pressure along the column is measured as the differential pressure between any tap and a pressure tap located 9 inches above the screen. The 10 pressure

Table 3.1 Bead-Fluid Combinations

<u>Mesh Size of Beads</u>	<u>Kerosene 1.4 cp</u>	<u>Kerosene Mineral Oil</u>	<u>Heptane (0.4cp)</u>
-25 +45	1 * ✓	-	2
-20 +50	3 ✓	-	4
-18 +60	5 ✓	-	6
-16 +70	-	-	7
-7 +14	8 ✓	9 ✓	-
-6 +16	10 ✓	11 ✓	-
-8 +12	12 ✓	-	-
-7 +12	13 ✓	-	-
-6 +12	14 ✓	-	-
-5 +12	15 ✓	-	-
-5 +14	16 ✓	-	-
-5 +18	17 ✓	18 ✓	-
-7 +14 HD	19 ✓	-	-
-6 +16 HD	20 ✓	-	-
-12 +20	21 ✓	-	-
-10 +20	22 ✓	-	-
-8 +25	23 ✓	-	-
-6 +25	24 ✓	-	-
-6 +30	25 ✓	-	-
-6 +40	26 ✓	27 ✓	28

* Combination number

Figure 3.1

SCHEMATIC DIAGRAM OF COLD FLOW EQUIPMENT

taps are at 6-inch intervals starting with the first tap 3 inches above the screen. The taps are attached to 9 individual monometers. When water was the fluidizing liquid, trichloroethane was used as the monometric fluid; with kerosene and kerosene + mineral oil, water is the manometric fluid.

3.3 Experimental Work

3.3.1 Data Collection

The primary data collected consists of the differential manometric readings which are used to define and locate the fluidized bed interface as explained in Appendix I of Reference 1. Segregation and mixing in the solid phase were recorded photographically as before, and the observation that stratification occurs with the liquid phase alone and mixing on the introduction of gas remains unchanged.

3.3.2 Tabulation of Collected Data

The new manometric data collected from 7/1/82 through 9/30/82 are included as Appendix I to this report. They comprise runs 812 through 1097.

3.4 Results and Discussion

The data of runs 812-1097 studies the effects of higher catalyst density (Runs 812-841), progressively wider particle size distributions (Runs 877-1062), and higher fluid viscosity (Runs 842-876, 1063-1097) on the ebullation characteristics of the bead catalysts. Bed heights, liquid

and gas fractions and bed expansions calculated from the data of Appendix I are presented in Tables 3.2-3.9.

Higher Catalyst Density: Runs 812-841 were made with a catalyst batch of median diameter 2mm and nominal size range -6 +16 mesh. The catalyst was virtually the same as that used for Runs 362-406 and Runs 589-622 (Ref. 1) except that its kerosene-filled density had been adjusted to 1.76 gm/cm^3 versus the earlier 1.44 gm/cm^3 representing a 20% increase in density simulating the deposition of metals and coke during use in a coal liquefaction reactor. As might be expected, the bed expansion was lower for the higher density catalyst at the same gas and liquid flow rates than for the lower density catalyst. For example, at a kerosene rate of 0.16 fps the low density catalyst showed a bed expansion of 83% (ϵ_t of 0.70) while at 0.17 fps kerosene rate the bed expansion for the high density catalyst was 64% (ϵ_t of 0.68). Similarly, the increase in expansion due to the gas phase was also lower for the higher density catalyst. For example, the increases in bed expansions were 18% and 10%, respectively, for the lower and the higher density catalysts at the liquid rates mentioned above when nitrogen flow at 0.15fps was introduced. The bed interface was correspondingly sharper for the higher density catalyst.

Wider Particle Size Distributions: Runs 877-1062 were made with a catalyst batch of median diameter 1.3mm and

Table 3.2 Calculated Bed Heights, Liquid + Gas Fractions and Bed Expansions for -6 +16 Mesh HD Beads, Kerosene, Nitrogen

Run No.	Liquid Vel. (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Settled Bed Height In.	Measured Bed Height In.	Calcd Bed Height In.	Liq. + Gas Frctn $E_L + E_G$	Bed Expsn %
RN	UL	UG	HO	HM	HC	EFSLN	BEDEX
812	.086	.000	18.00	20.0	19.00	.539	5.6
813	.086	.012	18.00	20.0	19.70	.555	9.4
814	.086	.025	18.00	21.0	20.00	.562	11.1
815	.086	.050	18.00	21.0	20.60	.574	14.4
816	.086	.100	18.00	21.0	20.70	.576	15.0
817	.086	.150	18.00	22.0	21.20	.586	17.8
818	.127	.000	18.00	23.0	22.00	.602	22.2
819	.127	.012	18.00	23.0	22.60	.612	25.6
820	.127	.025	18.00	23.0	22.80	.615	26.7
821	.127	.050	18.00	23.0	23.40	.625	30.0
822	.127	.100	18.00	23.0	24.00	.635	33.3
823	.127	.150	18.00	23.0	24.40	.641	35.6
824	.168	.000	16.50	27.0	27.00	.675	63.6
825	.168	.012	16.50	27.0	27.10	.676	64.2
826	.168	.025	16.50	27.0	27.60	.682	67.3
827	.168	.050	16.50	26.0	28.00	.687	69.7
828	.168	.100	16.50	26.0	28.50	.692	72.7
829	.168	.150	16.50	26.0	28.70	.695	73.9
830	.209	.000	16.00	31.0	29.60	.704	85.0
831	.209	.012	16.00	30.0	29.60	.704	85.0
832	.209	.025	16.00	30.0	30.20	.710	88.8
833	.209	.050	16.00	30.0	30.10	.709	88.1
834	.209	.100	15.50	30.0	30.20	.710	94.8
835	.209	.150	15.50	30.0	30.20	.710	94.8
836	.250	.000	15.50	38.0	36.30	.758	134.2
837	.250	.012	15.50	37.0	35.10	.750	126.5
838	.250	.025	15.50	37.0	35.80	.755	131.0
839	.250	.050	15.50	37.0	36.30	.758	134.2
840	.250	.100	15.50	36.0	35.80	.755	131.0
841	.250	.150	15.50	35.0	35.90	.756	131.6

Table 3.3 Calculated Bed Heights, Liquid + Gas
Fractions and Bed Expansions for -5 +18
Mesh Beads, 5 cp Fluid, Nitrogen

RN	UL	UG	HO	HM	HC	EPSLN	BEDEX
842	.129	.000	19.00	48.0	48.00	.775	152.6
843	.103	.000	19.00	39.0	36.80	.707	93.7
844	.076	.000	19.00	31.5	30.10	.642	58.4
845	.049	.000	19.00	26.0	24.90	.567	31.1
846	.022	.000	19.00	21.5	21.40	.496	12.6
847	.129	.200	19.00	46.0	46.60	.769	145.3
848	.103	.200	19.00	36.0	38.60	.721	103.2
849	.076	.200	19.00	31.0	34.50	.688	81.6
850	.049	.200	19.00	30.0	30.00	.641	57.9
851	.022	.200	19.00	24.0	27.60	.609	45.3
852	.129	.150	19.00	40.0	46.50	.768	144.7
853	.103	.150	19.00	36.0	37.50	.713	97.4
854	.076	.150	19.00	33.0	33.00	.673	73.7
855	.049	.150	18.50	29.0	29.60	.636	60.0
856	.022	.150	18.50	26.0	26.70	.596	44.3
857	.129	.100	18.50	46.0	48.30	.777	161.1
858	.103	.100	18.50	40.0	37.10	.709	100.5
859	.076	.100	18.50	33.0	32.10	.664	73.5
860	.049	.100	18.50	28.0	26.90	.599	45.4
861	.022	.100	18.50	23.0	24.50	.560	32.4
862	.129	.050	18.50	43.0	47.70	.774	157.8
863	.103	.050	18.50	36.0	36.60	.705	97.8
864	.076	.050	18.50	31.0	31.00	.652	67.6
865	.049	.050	18.50	28.0	26.70	.596	44.3
866	.022	.050	18.50	21.0	23.00	.531	24.3
867	.129	.025	18.50	48.0	48.30	.777	161.1
868	.103	.025	18.50	35.0	36.10	.701	95.1
869	.076	.025	18.50	31.0	30.50	.647	64.9
870	.049	.025	18.50	26.0	26.60	.595	43.8
871	.022	.025	18.50	20.0	22.50	.521	21.6
872	.129	.012	18.00	44.0	48.80	.779	171.1
873	.103	.012	18.00	36.0	35.40	.695	96.7
874	.076	.012	18.00	32.0	30.40	.645	68.9
875	.049	.012	18.00	26.0	26.50	.593	47.2
876	.022	.012	18.00	20.0	22.00	.510	22.2

Table 3.4 Calculated Bed Heights, Liquid + Gas
Fractions and Bed Expansions for -12 +20
Mesh Beads, Kerosene, Nitrogen

RN	III	UG	HO	HM	HC	EPSLN	BEDEX
877	.023	.000	10.00	11.0	16.50	.666	65.0
878	.023	.012	10.00	10.0	17.30	.682	73.0
879	.023	.025	10.00	10.0	19.10	.712	91.0
880	.023	.050	10.00	12.0	20.10	.726	101.0
881	.023	.100	10.00	13.0	19.20	.713	92.0
002	.023	.150	10.00	16.0	19.70	.720	97.0
883	.023	.200	10.00	16.0	20.10	.726	101.0
884	.043	.000	10.00	13.5	15.50	.645	55.0
885	.043	.012	10.00	13.0	16.80	.672	68.0
886	.043	.025	10.00	14.0	17.40	.683	74.0
887	.043	.050	10.00	16.0	18.20	.697	82.0
888	.043	.100	10.00	18.0	19.40	.716	94.0
889	.043	.150	10.00	19.0	20.00	.725	100.0
890	.043	.200	10.00	19.0	19.80	.722	98.0
891	.064	.000	10.00	14.5	15.30	.640	53.0
892	.064	.012	10.00	15.0	16.20	.660	62.0
893	.064	.025	10.00	15.0	17.00	.676	70.0
894	.064	.050	10.00	16.0	18.30	.699	83.0
895	.064	.100	10.00	18.0	19.40	.716	94.0
896	.064	.150	10.00	19.0	20.20	.727	102.0
897	.064	.200	10.00	19.0	20.40	.730	104.0
898	.084	.000	10.00	17.5	16.90	.674	69.0
899	.084	.012	10.00	16.0	17.40	.683	74.0
900	.084	.025	10.00	17.0	18.00	.694	80.0
901	.084	.050	10.00	18.0	20.00	.725	100.0
902	.084	.100	10.00	19.0	20.60	.733	106.0
903	.084	.150	10.00	19.0	22.10	.751	121.0
904	.084	.200	10.00	19.0	22.60	.756	126.0
905	.104	.000	10.00	22.5	22.50	.755	125.0
906	.104	.012	10.00	21.0	22.90	.759	129.0
907	.104	.025	10.00	22.0	23.60	.767	136.0
908	.104	.050	10.00	23.0	24.60	.776	146.0
909	.104	.100	10.00	23.0	25.40	.783	154.0
910	.104	.150	10.00	23.0	27.70	.801	177.0
911	.104	.200	10.00	23.0	28.50	.807	185.0

Table 3.5 Calculated Bed Heights, Liquid + Gas
Fractions and Bed Expansions for -10 +20
Mesh Beads, Kerosene, Nitrogen

RN	UL	UG	HO	HM	HC	EPSLN	BEDEX
912	.024	.000	12.00	13.0	15.20	.560	26.7
913	.024	.025	12.00	13.0	17.50	.618	45.8
914	.024	.050	12.00	14.0	18.10	.631	50.8
915	.024	.100	12.00	16.0	19.60	.659	63.3
916	.024	.150	12.00	17.0	20.50	.674	70.8
917	.024	.200	12.00	17.0	21.80	.693	81.7
918	.051	.000	12.00	17.5	17.10	.609	42.5
919	.051	.025	12.00	17.0	18.40	.637	53.3
920	.051	.050	12.00	18.0	19.20	.652	60.0
921	.051	.100	12.00	19.0	20.90	.680	74.2
922	.051	.150	12.00	20.0	21.30	.686	77.5
923	.051	.200	12.00	20.0	22.80	.707	90.0
924	.077	.000	12.00	21.0	21.00	.682	75.0
925	.077	.025	12.00	20.0	23.50	.715	95.8
926	.077	.050	12.00	21.0	24.20	.724	101.7
927	.077	.100	12.00	22.0	25.40	.737	111.7
928	.077	.150	12.00	22.0	26.30	.746	119.2
929	.077	.200	12.00	22.0	27.30	.755	127.5
930	.104	.000	12.00	27.0	27.00	.752	125.0
931	.104	.025	12.00	24.0	29.00	.769	141.7
932	.104	.050	12.00	25.0	31.00	.784	158.3
933	.104	.100	12.00	26.0	30.00	.777	150.0
934	.104	.150	12.00	27.0	31.80	.790	165.0
935	.104	.200	12.00	27.0	32.70	.796	172.5
936	.132	.000	12.00	35.0	33.50	.800	179.2
937	.132	.025	12.00	30.0	30.10	.778	150.8
938	.132	.050	12.00	30.0	31.00	.784	158.3
939	.132	.100	12.00	31.0	31.50	.788	162.5
940	.132	.150	12.00	32.0	31.80	.790	165.0
941	.132	.200	12.00	32.0	32.70	.796	172.5

Table 3.6 Calculated Bed Heights, Liquid + Gas
Fractions and Bed Expansions for -8 +25
Mesh Beads, Kerosene, Nitrogen

RN	UL	UG	HO	HM	HC	EP/SLN	BEDEX
942	.016	.000	16.00	17.0	16.30	.471	1.9
943	.016	.025	16.00	15.5	17.10	.496	6.9
944	.016	.050	16.00	16.0	18.30	.529	14.4
945	.016	.100	16.00	19.0	20.60	.582	28.7
946	.016	.150	16.00	21.0	22.10	.610	38.1
947	.016	.200	16.00	22.0	23.00	.625	43.8
948	.039	.000	16.00	20.0	19.30	.553	20.6
949	.039	.025	16.00	21.0	20.60	.582	28.7
950	.039	.050	16.00	21.0	21.20	.593	32.5
951	.039	.100	16.00	24.0	22.70	.620	41.9
952	.039	.150	16.00	24.0	23.90	.639	49.4
953	.039	.200	16.00	25.0	24.00	.641	50.0
954	.063	.000	16.00	24.0	23.70	.636	48.1
955	.063	.025	15.50	24.5	24.40	.647	57.4
956	.063	.050	15.50	25.0	24.50	.648	58.1
957	.063	.100	15.50	26.0	26.00	.668	67.7
958	.063	.150	15.50	27.0	27.90	.691	80.0
959	.063	.200	15.50	28.5	28.00	.692	80.6
960	.086	.000	15.50	31.5	30.00	.713	93.5
961	.086	.025	15.50	26.5	29.60	.709	91.0
962	.086	.050	15.50	28.5	29.80	.711	92.3
963	.086	.100	15.50	29.5	30.50	.717	96.8
964	.086	.150	15.50	31.0	32.60	.736	110.3
965	.086	.200	15.50	33.0	32.00	.731	106.5
966	.109	.000	15.50	35.5	32.70	.736	111.0
967	.109	.025	15.50	29.0	31.20	.724	101.3
968	.109	.050	15.50	30.0	31.40	.725	102.6
969	.109	.100	15.50	30.5	32.00	.731	106.5
970	.109	.150	15.50	31.5	32.50	.735	109.7
971	.109	.200	15.50	32.5	34.70	.752	123.9

Table 3.7 Calculated Bed Heights, Liquid + Gas
Fractions and Bed Expansions for -6 +25
Mesh Beads, Kerosene, Nitrogen

RN	UL	UG	HO	HM	HC	EFSLN	REDEX
972	.030	.000	18.00	21.0	21.00	.517	16.7
973	.030	.025	18.00	21.5	23.30	.565	29.4
974	.030	.050	18.00	23.0	23.40	.566	30.0
975	.030	.100	18.00	24.5	25.50	.602	41.7
976	.030	.150	18.00	26.5	26.80	.621	48.9
977	.030	.200	18.00	26.5	27.90	.636	55.0
978	.050	.000	18.00	25.5	25.00	.594	38.9
979	.050	.025	18.00	24.0	24.40	.584	35.6
980	.050	.050	18.00	25.0	25.00	.594	38.9
981	.050	.100	18.00	26.0	26.60	.619	47.8
982	.050	.150	18.00	27.0	27.90	.636	55.0
984	.068	.000	18.00	28.0	28.20	.640	56.7
985	.068	.025	18.00	26.5	28.50	.644	58.3
986	.068	.050	18.00	28.0	29.10	.651	61.7
987	.068	.100	18.00	29.0	30.30	.665	68.3
988	.068	.150	18.00	30.5	31.50	.678	75.0
989	.068	.200	18.00	31.5	31.70	.680	76.1
990	.090	.000	18.50	34.5	34.00	.702	83.8
991	.090	.025	18.50	31.5	36.40	.721	96.8
992	.090	.050	18.50	31.0	37.70	.731	103.8
993	.090	.100	18.50	32.0	36.00	.718	94.6
994	.090	.150	18.50	33.0	35.70	.716	93.0
995	.090	.200	18.50	34.0	36.90	.725	99.5
996	.109	.000	18.50	45.0	40.00	.746	116.2
997	.109	.025	18.50	35.0	35.70	.716	93.0
998	.109	.050	18.50	33.0	35.60	.715	92.4
999	.109	.100	18.50	34.0	36.80	.724	98.9
1000	.109	.150	18.50	35.5	37.20	.727	101.1
1001	.109	.200	18.50	36.5	38.00	.733	105.4

Table 3.8 Calculated Bed Heights, Liquid + Gas
Fractions and Bed Expansions for -6 +30
Mesh Beads, Kerosene, Nitrogen

RN	UL	UG	HO	HM	HC	EPSLN	BEDEX
1002	.026	.000	18.50	21.5	21.00	.483	13.5
1003	.026	.025	18.50	20.5	22.80	.524	23.2
1004	.026	.050	18.50	22.0	24.30	.553	31.4
1005	.026	.100	18.50	24.5	25.40	.573	37.3
1006	.026	.150	18.50	25.5	26.60	.592	43.8
1007	.026	.200	18.50	26.5	28.20	.615	52.4
1008	.045	.000	18.50	24.5	23.40	.536	26.5
1009	.045	.025	18.50	24.5	23.70	.542	28.1
1010	.045	.050	18.50	25.5	24.60	.559	33.0
1011	.045	.100	18.50	26.0	26.40	.589	42.7
1012	.045	.150	18.50	27.5	26.90	.597	45.4
1013	.045	.200	18.50	28.5	28.10	.614	51.9
1014	.065	.000	18.50	28.5	28.10	.614	51.9
1015	.065	.025	18.50	27.5	28.60	.621	54.6
1016	.065	.050	18.50	28.0	26.30	.587	42.2
1017	.065	.100	18.50	29.0	30.40	.643	64.3
1018	.065	.150	18.50	30.5	31.30	.653	69.2
1019	.065	.200	18.00	31.5	32.70	.668	81.7
1020	.084	.000	18.50	33.5	33.00	.671	78.4
1021	.084	.025	18.50	29.0	35.50	.694	91.9
1022	.084	.050	18.50	30.3	35.20	.692	90.3
1023	.084	.100	18.50	31.5	35.50	.694	91.9
1024	.084	.150	18.50	32.5	36.90	.706	99.5
1025	.084	.200	18.50	33.8	36.60	.704	97.8
1026	.104	.000	18.50	41.0	40.80	.734	120.5
1027	.104	.025	18.50	32.5	41.10	.736	122.2
1028	.104	.050	18.50	33.5	42.30	.743	128.6
1029	.104	.100	18.50	34.3	41.20	.737	122.7
1030	.104	.150	18.50	35.5	41.10	.736	122.2
1031	.104	.200	18.50	36.5	40.50	.732	118.9

Table 3.9 Calculated Bed Heights, Liquid + Gas
Fractions and Bed Expansions for -6 +40
Mesh Beads, Kerosene, Nitrogen

RN	UL	UG	HO	HM	HC	EPSLN	BEDEX
1032	.027	.000	18.00	21.0	21.00	.453	16.7
1033	.027	.025	18.00	20.5	22.80	.496	26.7
1034	.027	.050	18.00	21.5	24.00	.521	33.3
1035	.027	.100	18.00	22.3	25.00	.540	38.9
1036	.027	.150	18.00	24.5	26.50	.566	47.2
1037	.027	.200	18.00	26.5	28.90	.602	60.6
1038	.027	.012	18.00	19.3	21.80	.473	21.1
1039	.039	.000	18.00	22.0	21.30	.460	18.3
1040	.039	.025	18.00	20.8	22.70	.494	26.1
1041	.039	.050	18.00	22.5	23.70	.515	31.7
1042	.039	.100	18.00	24.5	25.10	.542	39.4
1043	.039	.150	18.00	25.8	27.80	.587	54.4
1044	.039	.200	18.00	26.5	27.50	.582	52.8
1045	.050	.000	17.50	24.0	23.30	.507	33.1
1046	.050	.025	17.50	23.0	23.60	.513	34.9
1047	.050	.050	17.50	25.0	23.80	.517	36.0
1048	.050	.100	17.50	26.0	26.60	.568	52.0
1049	.050	.150	17.50	27.3	27.20	.577	55.4
1050	.050	.200	17.50	28.3	28.60	.598	63.4
1051	.060	.000	17.50	27.5	27.50	.582	57.1
1052	.060	.025	17.50	23.3	28.30	.594	61.7
1053	.060	.050	17.50	24.5	29.50	.610	68.6
1054	.060	.100	17.50	26.0	31.40	.634	79.4
1055	.060	.150	17.50	28.5	31.80	.639	81.7
1056	.060	.200	17.50	29.3	32.10	.642	83.4
1057	.073	.000	17.50	29.0	27.80	.587	58.9
1058	.073	.025	17.50	25.5	29.60	.612	69.1
1059	.073	.050	17.50	27.0	29.30	.608	67.4
1060	.073	.100	17.50	28.0	30.30	.621	73.1
1061	.073	.150	17.50	29.5	31.80	.639	81.7
1062	.073	.200	17.50	30.8	33.10	.653	89.1

nominal size range -6 +40 mesh. The shape of the untreated particle size distribution was essentially a straight line on a log normal-probability graph, and the slope corresponded to a value achievable in a production facility. The batch was sieved into its component mesh fractions. Table 3.10 lists the mesh size composition of the batch. Runs were made with the central -12 +20 mesh fraction. Subsequent additions of coarse and fine fractions maintained approximately the same median diameter but changed the width of the distribution to -10 +20, -8 +25, -6 +25, -6 +30 and -6 +40 mesh. The ratio of the limiting diameters for -12 +20 mesh is 2 and those for the other widths are 2.38, 3.35, 4.73, 5.7 and 8.0 in that order.

Results up to a ratio of 5.7 (-6 +30 mesh) appeared satisfactory: the interface was not excessively diffuse, with kerosene flow alone, up to $\sim 100\%$ bed expansion, and the disengagement zone extended to only about 15 inches above the bed interface with nitrogen at 0.15 fps. Although the entire catalyst bed did not appear fluidized without gas flow at 14% bed expansion (kerosene velocity 0.026 fps), at 26% bed expansion (kerosene velocity 0.045 fps) even the coarse fractions were fluidized. With gas flow, the minimum liquid velocity required for fluidizing the entire bed was considerably reduced. For example, as mentioned above, at 0.026 fps kerosene velocity, the coarse fractions of the bed showed no movement and the bed expansion

Table 3.10 - Mesh Size Composition of 1.3mm Beads

<u>Mesh Size</u>	<u>Weight %</u>
+6	4
-6 +8	9
-8 +10	9
-10+12	10
-12+14	12
-14+16	13
-16+18	11
-18+20	11
-20+25	7.5
-25+30	6
-30+35	3.5
-35+40	2
-40+45	1.15
-45	0.85

was 14%, but on introduction of nitrogen at 0.025 fps at the same kerosene rate, the entire bed became fluidized with substantial axial mixing and the bed expansion increased to 23%. The mixing and turbulence increased further as the gas rate was increased to 0.05 fps and higher (to 0.2 fps). The disengagement zone remained less than about 15 inches with little carry-over of fines.

When the ratio of limiting diameters was increased to 8 (-6 +40 mesh), the interface became diffuse at liquid rates too low to fluidize the entire bed (Run 1032). On introducing gas at 0.012 fps, the entire bed showed a slow turnover after about 15 minutes and the mixing was very slow (Run 1038). At a gas rate of 0.025 fps the mixing rate was still too low (Run 1033) but the interface had become diffuse. At a gas rate of 0.05 fps a visually well-mixed bed of 33% bed expansion was obtained, but the carry-over of fines caused a pressure build-up across the catalyst retention screen at the top of the reactor. At higher liquid rates, the entire bed appeared fluidized at lower gas rates but the interface tended to be diffuse and fines were carried over at bed expansions as low as 35%. In general, the behavior of the -6 +40 mesh batch in the ebullated bed was marginal. Satisfactory fluidization of the bed could not be obtained with liquid flow alone without reaching the terminal velocity of the fines. With gas flow at liquid rates lower than the minimum fluidization velocity of the coarse fractions, complete fluidization could

be achieved, but fines entrained in the wake of the coalesced gas bubbles tended to be carried-over from the reactor.

Higher Fluid Viscosity: Runs 842-876 were carried out with a 5cp fluid (mixture of kerosene and a mineral oil) and 2mm median diameter beads previously fluidized with kerosene (Runs 786-805). Runs 1063-1097 used a 3 cp kerosene-mineral oil mixture and the 1.3mm median diameter beads used in Runs 1032-1062. As expected, the liquid velocities required to reach the same bed expansions were considerably lower with the higher liquid viscosity than with kerosene. For example, at a kerosene velocity of 0.116 fps the bed expansion is only 32% (Run 791), while at a 5cp fluid velocity of 0.103 fps the bed expansion is 94%. Further comparison of bed expansions and liquid + gas fractions with and without gas flow shows that, in general, the effect of the gas phase is greater in the higher viscosity fluids. This may be due to the larger size of the coalesced bubbles in liquids of higher viscosity, their correspondingly larger wakes, and the lower terminal velocities of the solids. Apparently the larger bubble size permits the bubble to develop a rising velocity greater than the terminal velocity of the fines in spite of the increased drag due to the higher viscosity.

The data of Runs 1063-1097 are being analyzed. Observations during the runs were qualitatively similar to those of Runs 842-876.

3.5 Data Analysis

3.5.1 Richardson-Zaki Correlation

The two parameter Richardson-Zaki type correlation was used to correlate the liquid solid data obtained with 1.3mm beads (Runs 877-1062). Table 3.11 lists the exponent n , and the terminal velocity, U_t , calculated for the several distributions in this series. Both n , and U_t seem to decrease with increasing width of the distribution. The terminal velocity for a 1.3mm diameter alumina bead in kerosene can be calculated from the expression given by Kunii and Levenspiel(2) to be 0.36 ft/sec which is well within the range of U_t in Table 3.11.

3.5.2 Calculations for Polydisperse Systems Using the Correlations of Richardson & Zaki

The method of calculating bed expansions for liquid solid systems, in which Richardson and Zaki's correlations are used for each sieve fraction of a polydisperse system and the expanded volumes are considered additive (1), was used also for the 1.3mm median diameter beads (Runs 877-1062). 22 of the 30 calculated values of e_L agreed within 10% of the experimental values. Of the 8 values which did not, 3 were for beds 12 inches or less in depth with low liquid velocities, which did not permit accurate determination of the bed height, and the other 5 were with wide distribution batches at liquid velocities too low to achieve complete fluidization.

Table 3.11 Exponents and Terminal Velocities for
1.3mm Beads from Fits of Richardson-Zaki
Type Correlation, $e^n = \frac{U_L}{U_t}$

<u>System</u> <u>Mesh Size-Fluid</u>		<u>Exponent</u> <u>n</u>	<u>Terminal Velocity</u> <u>U_t, ft/sec</u>
-12+20	1.4 cp	4.86	0.38
-10+20	1.4 cp	4.47	0.38
-8+25	1.4 cp	4.0	0.36
-6+25	1.4 cp	3.55	0.32
-6+30	1.4 cp	3.26	0.31
-6+40	1.4 cp	2.93	0.32

For well-fluidized liquid-solid polydisperse systems the method outlined above appears to be satisfactory for the determination of liquid fractions and, therefore, bed expansions.

3.5.3. Limiting Diameters for Polydisperse Systems

Equating the correlations for the minimum fluidization velocity U_{mf} (3) and the terminal velocity, U_t (2) it is possible to develop a relation for the limiting diameters in a liquid-solid (two-phase) fluidized bed. If d_1 is the smallest diameter and d_2 is the largest diameter in the solid phase,

$$U_{mf} = 0.00114 g d_2^2 (\rho_s - \rho) / \mu \quad 3.1$$

$$U_t = \left[\frac{4 (\rho_s - \rho)^2 g^2}{225 \rho \mu} \right]^{1/3} d_1 \quad 3.2$$

Equating U_{mf} to U_t ,

$$\frac{d_2^2}{d_1} = \left[\frac{4 \mu^2}{(0.00114)^3 (225) g \rho (\rho_s - \rho)} \right]^{1/3} \quad 3.3$$

where ρ and μ are the density and the viscosity of the liquid, ρ_s is the density of the solid and g is the acceleration due to gravity. For kerosene as the liquid phase and alumina beads, such as used in Cyanamid's novel catalysts,

as the solid phase Equation 3.3 simplifies to

$$\frac{d_2^2}{d_1} = 1.673 \text{ cm.} \quad 3.4$$

Using this relation the following table can be prepared for the limiting diameters:

No.	If d_1		then d_2		d_2/d_1
	cm	mesh	cm	mesh	
1	0.119	16	0.445	-4+5	3.75
2	0.1	18	0.409	5	4.1
3	0.084	20	0.375	-5+6	4.46
4	0.071	25	0.345	-5+6	4.86
5	0.059	30	0.314	-6+7	5.32
6	0.05	35	0.289	7	5.78
7	0.042	40	0.265	-7+8	6.31
8	0.035	45	0.242	8	6.9
9	0.030	50	0.224	-8+10	7.47
10	0.025	60	0.204	10	8.16
11	0.021	70	0.187	-10+12	8.90

The observed behavior of the different batches generally agrees with this table. Line No. 5 suggests that a batch comprising -6+30 mesh particles would be at the limit of operability in a two-phase system. In a gas-solid-liquid (3 phase) system, however, since the presence of the gas phase

lowers the minimum fluidization velocity of the coarser particles, the bed can be operated at lower liquid velocities than necessary for two-phase systems. The width of the distribution could, therefore, be wider for three-phase systems than indicated in the table above. Runs with the -6+40 mesh batch, discussed earlier, agree with this conclusion, although it is also obvious from the discussion that the range of gas and liquid velocities for satisfactory ebullation is narrow for wide particle size distributions.

3.5.4 Correlation of Three-Phase Data

A dimensional analysis of the three-phase fluidization system following the method of Broadhurst and Becker(4) suggests a series of eight dimensionless groups, four of which are expected to be of negligible importance. The remaining four can be combined to give the same dimensionless groups as the ones used by Kim et al (5,6), namely, Froude, Reynolds, and Weber numbers. Further, the effect of the gas phase in the three-phase systems can be interpreted as a deviation from the two-phase system, so that a correlation is being sought between the difference of liquid and gas fractions and the liquid fraction in the absence of gas flow, i.e., $\epsilon_{L+G} - \epsilon_{L_{u_G=0}}$ or $\Delta\epsilon$, and gas flow, U_G .

Figures 3.2, 3.3 and 3.4 show the plots of the ΔE versus U_G for the 2mm median diameter beads of three different distribution widths, and Figures 3.5, 3.6 and 3.7 show similar plots for the 1.3mm median diameter beads. The general similarity of the shape of the curves is evident in these figures. It is also clear that the effect of the gas flow is the highest at the lowest liquid rates, suggesting that the difference between the bubble velocity and the liquid velocity is the parameter of importance. Further, the ΔE values are larger for the smaller particles than for the larger particles since the velocity of the gas bubbles relative to the liquid in the case of the smaller particles is larger than in the case of the larger particles for the same range of bed expansions.

Figures 3.2-3.7 also show bed contractions at low gas velocities in some cases. The contractions are generally very small relative to the overall bed height and in some cases appear to be a result of the more uniform mixed bed that results on introducing gas into the segregated bed obtained with liquid flow alone.

Efforts at developing correlation(s) for the three-phase data continue.

System: -8 +12 Mesh - Kerosene - N₂

Figure 3.2

Incremental Effect of Gas Flow on
Liquid + Gas Porosity, -8 +12 Mesh

System: -5 +12 Mesh-Kerosene - N₂

Figure 3.3 Incremental Effect of Gas Flow on
Liquid + Gas Porosity, -5 +12 Mesh

System: -5 +18 Mesh - Kerosene - N₂

Figure 3.4 Incremental Effect of Gas Flow on
Liquid + Gas Porosity, -5 +18 Mesh

System: -8 +25 Mesh - Kerosene - N₂

Figure 3.5 Incremental Effect of Gas Flow on Liquid + Gas Porosity, -8 +25 Mesh

System: -6 +30 Mesh - Kerosene - N_2

Figure 3.6 Incremental Effect of Gas Flow on
Liquid + Gas Porosity, -6 +30 Mesh

System: -6 +40 Mesh - Kerosene - N₂

Figure 3.7

Incremental Effect of Gas Flow on
Liquid + Gas Porosity, -6 +40 Mesh

3.6 Explosion-proof Cold Flow Test Unit

An explosion-proof cold flow test unit has been constructed for use with heptane as the liquid phase. After preliminary testing with the 1.3mm beads of runs 1032-1062, 0.5mm beads of several widths of particle size distributions will be tested with heptane.

3.7 Conclusions

1. Increasing catalyst density reduces bed expansion as expected. The effect of gas flow is also lower on the denser catalyst.
2. For 1.3mm median diameter beads even a limiting diameters ratio as high as 5.7 (-6+30 mesh), can give a satisfactory ebullated bed. A limiting diameters ratio of 8.0 (-6+40 mesh) appears to be marginal.
3. The width of the particle size distribution can be higher in a three-phase system than in a two-phase system, since the gas flow reduces the minimum fluidization velocity of the coarse fraction allowing lower liquid velocities to be used than in the absence of gas flow.
4. The two-phase (liquid-solid) fluidization of the polydisperse beads can be adequately described by applying Richardson and Zaki's correlations to the component fractions of the distributions and adding the expanded volumes.
5. Segregation and mixing of 1.3mm beads is similar to that of 2mm beads and HDS-1442A and 1442B extrudates. The beads stratify by size with liquid flow alone but are very well mixed at superficial gas velocities as low as 0.05 ft/sec.

6. Incremental bed expansion on introducing gas is greater for 1.3mm beads than for 2mm beads, and greater at lower liquid velocities than at higher liquid velocities.

3.8 References

1. V. T. Sinha, M. Butensky, T. L. Muchnick and D. Hymah, "Development of Significantly Improved Catalysts for Coal Liquefaction and Upgrading of Coal Extracts", Quarterly Progress Report No. 2, January 1-March 31, 1982.
2. D. Kunii, and O. Levenspiel, "Fluidization Engineering", John Wiley, New York (1969).
3. J. F. Davidson, and D. Harrison, "Fluidized Particles", Cambridge University Press, Cambridge (1963).
4. T. E. Broadhurst, and M. A. Becker, "Fluidization and its Applications", H. Angelino et al, eds., Cepadues Editions, Toulouse (1974).
5. S. D. Kim, C. G. J. Baker, and M. A. Bergougnou, Canadian Journal of Chemical Engineering, 50, 695 (1972).
6. S. D. Kim, C. G. J. Baker, and M. A. Bergougnou, Canadian Journal of Chemical Engineering, 53, 134 (1975).

Tasks 5 and 6: CATALYST BATCH SCREENING AND CONTINUOUS AGING TESTS

4.1 CSTR Catalyst Aging Test Unit

Catalyst aging tests will be carried out in a continuous stirred tank reactor (CSTR) installed in the same test facility as the Batch Screening Unit (1). The design incorporates elements of the Gas & Liquid Feed System developed by PETC (Pittsburgh Energy Technology Center) and the Product Collection System developed by Amoco (Amoco R&D Center). Detailed descriptions of the PETC and Amoco units are available in published reports by Polinski et al (2) and Brooks et al (3) respectively. The test unit is interfaced to a computer-based control system which permits automated operation. Reference should be made to Quarterly Progress Report No. 3 (1) for more information on the control system.

The CSTR unit has been constructed to Class 1, Division 2, Group D requirements and has been subjected to an extensive hazards and operability review. A simplified schematic of the CSTR is shown in Figure 4.1. Only major components are illustrated. A brief description is given below.

High pressure hydrogen from the Gas Compressor Control Station is filtered and passed via a solenoid valve, SV-106, to a forward pressure regulator where the pressure is reduced. The hydrogen flow rate can be either manually controlled or metered using a Brooks Supervisory Controller.

FIGURE 4.1 CSTR Coal Liquefaction Unit

The controller is installed in a nitrogen purged box which is fitted with a pressure switch connected to the computer. The temperature and pressure of the hydrogen stream entering the controller are measured and recorded by the computer. The exit stream from the Brooks System passes via a back pressure regulator through a solenoid valve, SV-101, to the flow calibration loop. The pressure of this feed stream is also monitored by the computer.

High pressure nitrogen from the Gas Compressor Control Station is filtered and passed to a forward regulator where the pressure is reduced. The pressure of the supply nitrogen is monitored by the computer. The reduced pressure stream passes via a solenoid valve, SV-102, directly to the reactor. The computer program is designed in such a way that this solenoid valve cannot be open at the same time (during normal operation) as the solenoid valve on the HP hydrogen feed (SV-101).

Coal slurry is initially prepared in a jacketted, 30-gallon vessel with the contents maintained under a nitrogen blanket. Coal is added manually to the vessel and solvent oil is pumped directly from a 55-gallon drum mounted on a portable weigh scale. The vessel contents are agitated, with the status of the agitator monitored by the computer. The contents temperature is also monitored by the computer. The prepared slurry is transferred (by the operator, as needed) using a Moyno pump to the weigh tank, V-201, which

is mounted on a load cell platform. Slurry in this tank is continuously agitated and is recirculated by a second Moyno pump. The vessel contents, under a nitrogen blanket, can be maintained at a constant temperature by an SCR. The agitator and slurry pump are monitored by the computer but are manually started/stopped. The purpose of the load cell is to provide an indication of slurry inventory and slurry flow rate. Offtakes from the slurry recirculation loop supply two high pressure (HP) slurry pumps. Provision has also been made for draining the loop and for introducing flush oil to clear any residual slurry in the lines.

One of two HP slurry pumps is selected by the operator and is started/stopped by the computer. A drain down vessel provides a means of flushing and priming each pump without contaminating the feedstock. The discharge pressure of each pump is monitored by the computer, as is the status of the pumps themselves. Each pump is provided with a relief valve. The supply line to the mix tee is protected by a pressure switch which is monitored by the computer. Provision has also been made for calibrating the pumps, prior to start up, via a back pressure regulator.

Flush oil, maintained under a nitrogen blanket, can be supplied manually to the suction side of each HP slurry pump. A high pressure pump/solenoid valve combination has been provided to inject flush oil into the reactor in the event of an interlock shutdown.

The gas from the calibration loop and the slurry from the HP slurry pump combine in a mix tee and are fed into the bottom of a one-liter stirred autoclave. The liquid level in the reactor is maintained by an overflow tube which is connected via a heat exchanger to the product collection system. The catalyst is supported in a basket secured to the agitator assembly. The reactor temperature is computer-controlled via an SCR. The temperature and pressure of the mixed feed, the reactor pressure, and the status of the autoclave agitator are all monitored by the computer. The heat exchanger on the reactor effluent line is provided with a temperature control loop. The system is protected from overpressurization by a relief valve and rupture disc assembly on the mixed feed line and by another relief valve and rupture disc assembly on the reactor itself. A "fire switch" has been provided to protect the unit in the event of a very high temperature being detected in close proximity to the reactor. A temperature "policeman" has been incorporated in the reactor temperature control system to interlock shutdown the unit in the unlikely event of a simultaneous computer fault and SCR failure. Provision has also been made for draining down the reactor into a one-liter catchpot. This can be used to check the hold-up of the reactor as determined by the position of the overflow tube.

The cooled effluent from the reactor can pass either to a product collection vessel of 3 gallons capacity, V-401, or to a sample collection vessel, V-402. In normal operation, the effluent will be directed to V-401. The gas dis-entrains from the mixture and passes to the tail gas system. Both the mixture inlet and gas exit lines are equipped with solenoid valves under computer control. These valves close if low pressures are detected in either the reactor or the tail gas system. The vessel is manually drained down, (after re-directing the slurry and gas exit to the sample collection vessel), into a waste drum which is continuously vented to the room exhaust system. Thus the flash gas which is produced in the de-pressurization is diluted with air and exhausted out of the building. The manual drain down valves are specially designed to resist highly abrasive conditions.

The sampling vessel (used daily for analytical purposes) is equipped with a flash vessel and flash gas sampling and metering systems. The flash gas flow is measured using a wet test meter equipped with a shaft encoder which is monitored by the computer. Both collection vessels are heated using SCRs and are fitted with level indicators which are monitored by the computer. They are also provided with re-pressurization loops using either H_2 hydrogen or tail gas. To prevent overpressurization, each vessel is equipped with a relief valve.

The exit gas from either the product collection vessel or sample collection vessel is quenched with a small flow

of water and bubbled through a scrubbing solution in V-404. The vessel is equipped with a level indicator which is monitored by the computer. Overpressurization is prevented by a relief valve on the vessel. The scrubbed gas passes via a knock-out pot to the two-stage back pressure regulation system which permits the return of tail gas to V-401 and V-402 for repressurization. The tail gas pressure is monitored by the computer prior to back pressure regulation. The expanded, depressurized tail gas passes via another knock out pot to a wet test meter equipped with a shaft encoder monitored by the computer. Overpressurization of the system is prevented by a relief valve. The exhaust gas can be sampled, if required, before being vented.

De-ionized water in the calibrated vessel, V-601, is pumped using a HP metering pump to the scrubber vessel, V-404. The pump is controlled and monitored by the computer and the discharge stream is protected against overpressurization by a relief valve and a pressure switch.

4.2 Analytical Techniques

A wide range of techniques is to be used to analyze feeds and reaction products in the course of the catalyst development program. An evaluation of the more important methods was performed to ascertain their precision and (when possible) their accuracy when compared to independent results.

Samples from H-COAL[®] PDU runs 5, 9, and 10 as well as a sample of SRC-II heavy distillate (FSN-12) were obtained for this purpose. The PDU samples were analyzed for sulfur (two methods), nitrogen, basic nitrogen, water, ash, viscosity and density. In addition, cyclohexane, toluene, and THF insolubles were determined by pressure filtration. The SRC-II heavy distillate was tested similarly while additionally being analyzed for trace elements.

Several samples of the PDU products as well as the SRC-II heavy distillate were distilled by the ASTM D1160 method.

4.2.1. H-COAL Products

H-COAL PDU products tested include atmospheric still overheads (ASO), atmospheric still bottoms, (ASB), and vacuum still overheads (VSO) from PDU run 5 (periods 14A, 16A, and 16B), PDU run 9 (period 19B) and PDU run 10 (period 39A). A summary of the results is presented in Table 4-1.

Sulfur in samples LO-3483, LO-3487, and LO-3489 was determined by two techniques. The first was X-ray fluorescence (XRF). The application of XRF to sulfur determination yielded values with a pooled standard deviation of 0.004%. This instrumental technique, in addition to being precise, is both fast and convenient. This is in contrast to the techniques specified by ASTM Method D3177 (4) for determination of sulfur:

TABLE 4.1 - ANALYSIS OF H-COAL PDU PRODUCTS

	PDU-5 ASO LO-3483	PDU-9 ASO LO-3484	PDU-10 ASO LO-3485	PDU-5 ASB LO-3486	PDU-9 ASB LO-3487	PDU-10 ASB LO-3488	PDU-5 VSO LO-3489	PDU-9 VSO LO-3490	PDU-10 VSO LO-3491	Pooled Std. Dev. (deg. of freedom)
Sulfur-X-ray Fluorescence (%)	0.278 0.278 <u>0.281</u> s = 0.002	0.474 0.488 <u>0.492</u> s = 0.009	0.068 0.072 <u>0.072</u> s = 0.002	0.098 0.098 <u>0.100</u> s = 0.001	0.122 0.115 <u>0.131</u> s = 0.008	0.019 0.018 <u>0.018</u> s = 0.001	0.122 0.125 <u>0.123</u> s = 0.002	0.156 0.156 <u>0.155</u> s = 0.001	0.024 0.020 <u>0.020</u> s = 0.002	0.004% (18)
Sulfur- Parr Bomb (%)	0.24 0.24 <u>0.26</u> s = 0.01				0.10 0.10 <u>0.10</u> s = 0.00		0.12 0.12 <u>0.11</u> s = 0.01			0.008% (6)
Kjeldahl N%	0.50 0.49	0.45 0.45	0.31 0.32 <u>0.30</u> s = 0.01	0.45 0.53 <u>0.58</u> <u>0.33</u> s = 0.11	0.52 0.54	0.47 0.47	0.53 0.51	0.53 0.53	0.44 0.49	0.09% (5)
Basic N(%)	0.28 0.29 0.28 0.29 <u>0.29</u> s = 0.01	0.29 0.29 0.28 0.29 <u>0.30</u> s = 0.01	0.16 0.16 0.17 0.17 <u>0.17</u> s = 0.01	0.36 0.36 0.37 0.37 <u>0.37</u> s = 0.01	0.32 0.32 0.32 0.32 <u>0.32</u> s = 0.00	0.23 0.23 0.23 0.23 <u>0.23</u> s = 0.00	0.43 0.43 0.42 0.42 <u>0.42</u> s = 0.01	0.40 0.40 0.40 0.40 <u>0.40</u> s = 0.00	0.24 0.23 0.24 0.24 <u>0.24</u> s = 0.00	0.01% (36)
Water(%)	0.16 0.20 <u>0.20</u> s = 0.02	0.20 0.21	0.10 0.10	0.11 0.11	0.05 0.05	0.04 0.06 <u>0.06</u> s = 0.01	0.06 0.07 <u>0.04</u> <u>0.06</u> s = 0.01	0.08 0.07 <u>0.08</u> <u>0.08</u> s = 0.01	0.06 0.06 <u>0.07</u> <u>0.08</u> s = 0.01	0.01% (12)
Ash (%)	<0.01	<0.01	<0.01	0.04 0.03	0.04	0.06	0.02	0.01	0.02	
Viscosity @ 400C (cSt)	1.55 1.58 <u>1.58</u> s = 0.02	1.28 1.20 <u>1.28</u> s = 0.05	1.20 1.14 <u>1.27</u> s = 0.07	11.69 11.18 <u>11.68</u> s = 0.29	8.94 8.80 <u>8.88</u> s = 0.07	5.18 5.17 <u>5.20</u> s = 0.02	44.56 41.26 <u>44.65</u> s = 1.93	33.52 32.23 <u>33.52</u> s = 0.74	5.51 5.23 <u>5.52</u> s = 0.16	0.70% (18)
Viscosity @ 1350C (cSt)				1.40 1.33 <u>1.41</u> s = 0.04	1.29 1.29 <u>1.27</u> s = 0.01	1.05 1.04 <u>1.05</u> s = 0.01	2.02 2.00 <u>2.02</u> s = 0.01	1.88 1.90 <u>1.89</u> s = 0.01	1.07 1.08 <u>1.07</u> s = 0.01	0.02% (12)
Density @ 60° (g/cm³)	0.858 0.061	0.838 0.819	0.818 0.819	0.988	0.971	0.918	1.034	1.020	0.928	
Cyclohexane Insolubles (%)	0.60 0.24 0.29 <u>0.20</u> s = 0.18	0.01 0.09 0.03	0.25 0.29 <u>0.33</u> s = 0.04	2.83 2.92 <u>2.66</u> s = 0.13	1.40 1.54 <u>1.39</u> s = 0.08	0.83 0.89 <u>0.90</u> s = 0.04	0.68 0.69 <u>0.60</u> <u>0.65</u> s = 0.04	1.00 0.90 <u>0.98</u> <u>0.98</u> s = 0.05	0.23 0.27 0.27 <u>0.26</u> s = 0.02	0.9% (19)
Toluene Insolubles (%)	none found		none found	0.79 0.75 0.41 0.69 <u>0.74</u> s = 0.15	0.30 0.26 <u>0.28</u> s = 0.02	0.27 0.32 <u>0.29</u> s = 0.03	none found	0.04	0.01	0.08% (8)
THF Insolubles (%)				0.02	0.03	0.13 0.09 <u>0.13</u> s = 0.02				

1. The Eschka method - combustion with magnesium oxide/sodium carbonate,
2. Precipitation as barium sulfate after oxygen bombing, or,
3. High temperature combustion in a tube furnace.

Methods for sulfur determination which call for such wet chemical techniques are generally considered to be tedious and time consuming (5).

The second method used to determine sulfur was Parr oxygen bombing followed by Dionex ion exchange chromatography. This differs from the ASTM-accepted oxygen bombing procedure only by the method utilized to quantify sulfate (ion exchange chromatography vs. precipitation). For the three samples examined Parr bombing and XRF can be considered equivalent because the confidence interval for the mean bias between the methods (0.0183 ± 0.033 at 95% confidence) includes 0. Some care, however, must still be taken with X-ray fluorescence. It has been noted that results of XRF analysis of coal with an ash content greater than 10% are influenced by sample particle size. This influence cannot be mitigated by suitable grinding as is the case with coals having less than 10% ash (6,7). The use of XRF to analyze the coals to be used for this contract will be investigated.

The water determinations reported in Table 4.1 were made with a Photovolt Aquatest IV moisture analyzer. This instrument utilizes a coulometric Karl Fischer technique. Generation of the Karl Fischer reagent in situ along with automatic ti-

tration eliminates the traditional problems associated with the Karl Fischer technique (e.g., sensitivity and reagent odor). Furthermore this technique has an advantage in speed over azeotropic distillation methods for determining water. Even with the low values of percent water in the PDU samples, the pooled standard deviation of the results (0.01%) shows the method to be acceptably precise.

Cyclohexane, toluene, and THF insolubles were determined by pressure filtration using a modified Millipore filtration unit (8). This is a critical analysis because it is the basis for calculating percent conversion for the continuous aging reactor (1). That is: cyclohexane solubility indicates conversion to oils; toluene solubility indicates conversion to asphaltenes (plus oils); and THF solubility indicates conversion to preasphaltenes (plus oils and asphaltenes). The pressure filtration method was developed at Amoco (9) and modified by workers at PETC (5). Many solvents have been utilized in the classification of products by pressure filtration. These include hexane for oil conversions, benzene for asphaltene conversion and quinoline for preasphaltene conversion. Cyclohexane will be used because it is superior to hexane in its ability to form suspensions rather than gums. Toluene will be used instead of benzene for safety reasons. THF will be used for consistency with earlier published results. The pooled standard deviations of 0.08% for toluene insolubles and 0.09% for cyclohexane insolubles compare

well with values obtained at PETC (0.1% for THF and 0.26% for cyclohexane) (8). It should be noted, however, that much higher percent insolubles are expected for the continuous reactor product. For this reason, until the method is tested on more representative samples, the high precision of the method demonstrated by the data in Table 4.1 should be viewed with some degree of skepticism.

No C,H,N or O values are reported for these products. A Perkin-Elmer Model 240C elemental analyzer will be used to obtain C,H,N as well as direct O analyses. An evaluation of elemental analysis with the 240C (including a comparison to ASTM Ultimate analysis) will be presented in a future report.

4.2.2 SRC -II Heavy Distillate

Results of analysis of a sample of SRC-II heavy distillate are shown in Table 4.2, in comparison to those obtained by Amoco (8). In general, good agreement was obtained with the exception of cyclohexane/hexane insolubles (2.33% vs 7.66%). Repeated pressure filtrations with cyclohexane indicate that the confidence interval for this value is 2.33 ± 0.12 (at 95% confidence). It is likely that the difference arises from sample variability.

4.2.3 D1160 Distillations

Another analytical technique which will be applied frequently is the ASTM D1160 distillation. This technique will be used to characterize solvents and products as well as for sample preparation. The actual test to be used is a modified version based on recommendations from PETC(11).

TABLE 4.2PROPERTIES OF SRC II HEAVY DISTILLATE (FSN-12)

	<u>Cyanamid</u>	<u>Amoco</u>
Specific Gravity	1.060	1.091
Distillation Residue (Modified D1160)	ca. 5	5.41
Ash, wt%	0.33	
THF insolubles, wt%	0.65	0.73
Toluene (Benzene) insolubles, wt%	0.95	(0.95)
Cyclohexane (Hexane) insolubles, wt%	2.33	(7.76)
Viscosity @ 40C, cSt	66.18	63.0
Viscosity @ 135C, cSt	2.26	2.3
Chemical Composition, wt%		
Basic N	0.55	
N (Kjeldahl)	1.08	1.06
H*	7.48	7.27
O* (by difference)	1.14	1.66
C	89.5	89.24
S	0.60	0.65
Water	0.20	0.24
H/C	1.03	0.97

*Hydrogen and oxygen percentages do not include the elemental contribution from the water.

TRACE ELEMENT COMPOSITION OF SRC II HEAVY DISTILLATE

Trace Elements, ppm	<u>Cyanamid</u>	<u>Amoco</u>
Na	50	125
Mg	23	17
Al	330	345
Si	561	450
K	66	ND
Ca	33	70
Ti	13	40
V	1	4
Cr	3	11
Mn	2	3
Fe	330	1050
Ni	1	3
Cu	1	1
Zn	1	-
Sr	1	-
Mo	-	1
Ba	1	ND
Pb	1	ND
B	-	6
Sn	-	2
TOTAL	1437	2119

ND = not detected

Among the modifications implemented are the following:

1. Use of high vacuum (approx. 0.1 torr)
2. Use of a quartz heater instead of circulating hot water
3. Use of distillation rates significantly slower than recommended in ASTM D1160

The plots in Figures 4.2 and 4.3 are the distillation curves for the H-COAL PDU vacuum still overheads and atmospheric still bottoms respectively. Figure 4.4 contains the distillation curves for replicate samples of SRC-II heavy distillate compared to values from an independent laboratory (E. W. Saybolt Co.)

Curves for the vacuum still overheads (Figure 4.2) show the similarity between the products from PDU-5 (LO-3489) and PDU-9 (LO-3490). Both have a boiling range from 525°C to 900°C with half of the material boiling below 650-700°C. The product from PDU-10 (LO-3491) has not been run yet. The curves for atmospheric still bottoms (Figure 4.3) show that they are lighter than the vacuum still overheads in general, with half of the material from PDU-9 (LO-3487) and PDU-10 (LO-3488) boiling below 575°C. Results for sample LO-3486, the atmospheric still bottoms from PDU-5, may be anomalous because of loss of a significant amount of very light material during distillation.

FIGURE 4.2
D1160 DISTILLATION
VACUUM STILL OVERHEADS

FIGURE 4.3
D1160 DISTILLATION
ATMOSPHERIC STILL BOTTOMS

FIGURE 4.4
D1160 DISTILLATION
SRC-II HEAVY DISTILLATE

Curves for SRC-II heavy distillate (Fig. 4.4) demonstrate the capability to reproduce distillations with good precision. They also show acceptable agreement with results from an independent laboratory.

4.2.4 Conclusions

In light of the objectives for the work, the data obtained demonstrate that the key analytical methods to be used in the course of the catalyst development program can be carried out with acceptable precision. To a more limited extent (for SRC-II distillate) it has been shown that these methods yield values in general agreement with those from other laboratories. However, the reliability of such comparison is questionable since coal-derived liquids are subject to chemical changes when not stored under conditions which prevent degradation: kept cold, excluding light and air (12,13). Comparison to historical data on the H-COAL PDU products was precluded by lack of pertinent values.

4.2.5 Summary of Analytical Procedures

A short description of the analytical procedures utilized for this evaluation follows. A compendium of detailed procedures will be included in a later report.

X-ray Fluorescence Sulfur (XRF)

X-rays are emitted from a chromium target at 40kv.

They impinge on the sample which absorbs the radiation and fluoresces. The sulfur fluorescence line is separated from the spectrum with a crystal monochrometer and its intensity

is measured with a proportional counter. The sulfur content is then calculated relative to a series of standards.

Parr Bomb Sulfur

The sample is fired in a Parr oxygen bomb calorimeter. The SO₂ formed is precipitated as barium sulfate and quantified with a Dionex ion exchange chromatograph.

Kjeldahl Nitrogen

Kjeldahl nitrogen is determined with a Buchi-Brinkmann Model 320/430 Nitrogen Analyzer. A sample is digested with a catalyst (mercuric oxide/potassium sulfate) and sulfuric acid. After digestion, which converts any organic nitrogen to ammonia, the sample is diluted, basified, and then steam distilled. The distillate is titrated with hydrochloric acid to quantify the ammonia.

Basic Nitrogen

Basic nitrogen compounds are determined by potentiometric titration on a Metrohm E636 Titroprocessor with perchloric acid in acetic acid/toluene. The electrode system consists of a glass electrode and a saturated calomel electrode. The titrator automatically locates the equivalence point and calculates the final results.

Water

Water is quantified on a Photovolt Aquatest IV moisture analyzer which utilizes coulometrically generated iodine to complete the Karl Fischer reagent. The sample is injected into the sample chamber and the instrument generates Karl Fischer reagent until

the water is consumed. Water detection is accomplished with a dual platinum sensing electrode. Percent water is then automatically calculated and displayed.

Ash

Ash is determined in accordance with ASTM Standard D482 "Standard Test Method for Ash from Petroleum Products". The sample is ignited and allowed to burn. The residue is reduced to ash in a muffle furnace and then weighed.

Kinematic Viscosity

Kinematic viscosity is determined according to ASTM Method D445 "Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)". The sample is introduced into a calibrated viscometer. The time it takes to flow through a capillary is measured. Kinematic viscosity is calculated from this value.

Density

Density is determined using a Mettler/Paar Model DMA 10 digital densimeter. A U-shaped hollow oscillating tube is filled with the sample and the period of the natural frequency of oscillation is measured automatically by the instrument. By comparing the period obtained with those obtained using air and water, the density of the sample can be quickly and accurately calculated.

Pressure Filtration

Pressure filtration is used to quickly determine solubility of a sample in a variety of solvents. The results are used to calculate percent conversion. Solvents typically used include cyclohexane, toluene, and tetrahydrofuran. The sample is slurried in the desired solvent and placed into a Millipore pressure

filter holder with a 100-ml solvent capacity. The inlet of the filter holder is connected to a reservoir of solvent. The filter holder is equipped with a 47-mm diameter 10- μ TEFLON® filter. The solvent reservoir is pressurized with nitrogen and the sample is filtered. The filtration residue is then dried and weighed.

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4.3 REFERENCES

1. V. T. Sinha, P. D. Kutzenco, W. J. Preston, J. S. Brinen, S. W. Graham, M. Butensky, T. L. Muchnick D. Hyman, "Development of Significantly Improved Catalysts for Coal Liquefaction and Upgrading of Coal Extracts," Quarterly Progress Report No. 3, April 1-June 30, 1982.
2. L. M. Polinski, G. J. Stiegel, R. E. Tischer, "1980 Status Review- Hydroliquefaction of Coal with Supported Catalysts," U.S.DOE Report DOE/PETC/TR-81-2, June 1981.
3. J. A. Brooks, R. J. Bertolacini, L. C. Gutberlet, D. K. Kim, "Catalyst Development for Coal Liquefaction," EPRI Report AF-190, February 1976.
4. ASTM D3177, "Standard Methods of Test for Total Sulfur in the Analysis Sample of Coal and Coke."
5. J. N. Chakrabarti, "Analytical Procedures for Sulfur in Coal Desulfurization Products" in Analytical Methods for Coal and Coal Products, Vol. I, C. Karr, Jr., ed., Academic Press, 1978, p. 306.
6. Ibid., p. 300.
7. J. Frigge, "Erdol und Kohler" Erdgas, Petrochem, 1972, pp. 447-448.
8. B. Utz, DOE (PETC), private communication to K. Matsuda, American Cyanamid Co, 3/4/82.

9. R. J. Bertolacini, L. C. Gutberlet, D. K. Kim, K. K. Robinson, "Catalyst Development for Coal Liquefaction," Report AF-574, November 1977, pp. 218-221.
10. J. A. Mahoney, "Catalyst Development for Coal Liquefaction," Proceedings of the DOE H-COAL Research TAC Meeting, September 15, 1981, pp. 8-10.
11. A. Wells, DOE (PETC), private communication to P. Kutzenko, American Cyanamid Co.
12. D. Finseth, "Maximizing Synfuel Stability", Synfuels Characterization, October 1982, pp 4-5.
13. J. R. Kershaw, D. Gray, "Ageing Studies on Coal Hydro-pyrolysis Liquids," Fuel, 59, 436 (1980).

5. Task 8: ANALYTICAL METHODS REVIEW

5.1 Introduction

Development of methods to characterize coal liquefaction catalysts focused on samples of used Amocat 1A withdrawn from the H-COAL pilot plant reactor at Catlettsburg between 6/11/82 and 7/12/82. The run was started 6/8/82 and terminated 7/12/82. The group consisted of fresh catalyst and four samples removed from the reactor intermittently during the month. Since catalyst is routinely added to and removed from the reactor, the age of the analytical sample may not be directly related to the date it was removed from the reactor. ESCA and electron microscopy work done to date has been included in this report along with a description of further work planned. Methods developed during the work with Amocat 1A will be applied to fresh and used bead catalysts generated by the current development program.

5.2 Experimental

Samples used in this study were extracted with methylene chloride for several hours at room temperature and allowed to dry. The purpose of this procedure was to remove superficial coal liquid contamination thus making the samples compatible with high vacuum techniques. Future extractions will be done using tetrahydrofuran or toluene as the solvent in order to conform with established procedures (1,2).

Bulk elemental analyses of the five samples were done using an Ortec TEFA x-ray fluorescence spectrometer. The Ortec system is energy-dispersive with a dual target x-ray tube as the source and a lithium drifted silicon detector. Quantitation is based on the LaChance-Triall equations whose constants have been determined from standards in matrices that are similar to those of the samples.

The ESCA spectra were run on an HP5950A ESCA spectrometer with an Al ($K\alpha = 1486.6$ eV) anode. The system is ion pumped and routinely runs at a pressure of 1×10^{-8} torr. The data obtained have been made semi-quantitative by correcting the peak heights for atomic cross-section and calculating relative atomic concentrations. The samples were mounted side by side on double-sided sticky tape such that none of the tape was visible.

Electron microscope work was done on a JEOL-35U equipped with a wavelength-dispersive x-ray detector and a Tracor-Northern energy-dispersive x-ray detector. Samples examined in the electron microscope were mounted in epoxy and polished in order to examine cross-sectional areas of the catalyst. The Co, Mo, and Al distributions were studied with the Tracor-Northern energy-dispersive x-ray detector while the electron beam scanned the diameter of the catalyst pellet. Using the program "TASK" the electron beam strikes the sample, x-rays which are characteristic of the elements present are emitted and

detected by the energy detector. The computer stores the x-ray intensities of interest versus the distance across the pellet. Since x-ray intensity is proportional to concentration, the plots of intensity versus distance give a qualitative indication of metals distribution across the catalyst pellet.

Another program makes it possible to use the ZAF method of quantitative analysis (3). In this program intensities of the elements of interest were corrected for atomic number (Z), absorption (A), and secondary fluorescence (F). The corrected intensities were then compared with corrected intensities of known standards to give relative weight concentrations. Furthermore, the ZAF correction package provided by the manufacturer has been combined with stage movement capabilities so that several samples may be analyzed automatically. (Program 3100).

On the electron microscope it was possible to do mapping with either the energy- or wavelength-dispersive detectors. With the energy dispersive detector all of the x-rays emitted from the sample are accepted simultaneously. Therefore, very intense signals (e.g. Al in catalyst samples) saturate the detector before weak signals are intense enough to be monitored. However, the wavelength detector monitors discreet wavelengths of the emitted x-rays, so the weak signals can be counted for long periods of time without interference from strong signals. Additionally, mapping using the wavelength-dispersive detector allows the Mo and S signals to be monitored separately which is not possible with the energy-dispersive detector.

5.3 Results and Discussion

Bulk elemental information for the five Amocat 1A catalysts obtained by x-ray fluorescence has been presented in Table 5.1. There were no significant differences in the Mo or Co concentrations among the fresh and used catalysts, nor were there any significant differences between one used catalyst and another. However, there was an increase in the catalyst contaminants (Na, Ca, Mg, Ti, Fe, and K) from the fresh to the used catalysts.

ESCA survey scans of the four used catalyst samples as extrudates have been obtained. A representative scan is shown in Figure 5.1. C, O, Mo, Al, S, Ti, Ca, Fe, Mg, and a very small amount of Co were present in the ESCA spectra of the four used catalysts. Rough estimates of atomic concentration which have been calculated from the ESCA data are presented in Table 5.2.

Using the electron microscope, secondary electron images (SEI) of the five catalysts were obtained and SEI of the fresh and a representative used catalyst sample are shown in Figure 5.2. The major difference between the fresh and used catalysts was an increase in porosity with use. However, there were not any obvious differences among the various used catalysts. This may be a function of the date withdrawn not being an indication of the relative age of the catalyst since catalyst was regularly added to and removed from the reactor.

Table 5.1
Bulk Concentration of Catalyst Powders (weight %) from EDX work

Sample	Date Withdrawn	AL	MO	Co	Na	Ca	mg	Ti	Fe	K	S
164a	fresh	major	7.5	2	-	<.05	<.1	<.01	<.1	-	N.A.
164b	6/11/82	major	7	1.5	.6	.05	.1	.01	.2	.04	N.A.
164c	6/24/82	major	6.5	1.5	1	.05	.1	.02	.4	.01	N.A.
164d	7/9/82	major	6	1.5	.7	.1	.2	.03	.6	.08	N.A.
164e	7/12/82	major	6	1.5	.6	.08	.2	.02	.5	.08	N.A.

S cannot be descriminated from Mo by this technique.

Figure 5.1 ESCA Survey scan of used Amocat 1A catalyst withdrawn from Catlettsburg reactor 6/24/82. (Al K α = 1486.6eV), vacuum 1×10^{-8} torr.

Table 5.2
Atomic Concentrations (%) From ESCA data of
Catalyst Extrudates

Sample	Date Withdrawn	C	O	Ca	Mo	Al	Ti	Fe	S
164b	6/11/82	56	22	7	1	12	<1	1	>1
164c	6/24/82	60	30	2	<1	6	<1	<1	<1
164d	7/09/82	75	19	<1	<1	6	-	-	<1
164e	7/12/82	67	23	2	<1	6	<1	<1	<1

a) FRESH

b) USED

Figure 5.2 Secondary electron images of crossections of the Amocat 1A catalyst samples, beam current 1×10^{-8} amp, accelerating voltage 25kV.

a) 164a fresh catalyst

b) 164e withdrawn from the reactor 7/12/82

Using the energy-dispersive detector on the electron microscope and the "TASK" program, the average distributions of Co, Mo and Al across the catalyst pellets were obtained. Figure 5.3 shows the average distribution of Co, Mo, and Al for the fresh catalyst and for one of the used catalysts which was representative of all of the used catalysts. The distributions of all of the elements monitored across the pellets in both the fresh and used catalysts were nearly uniform. However, the total Co intensity in the used catalysts was approximately half of the total Co intensity in the fresh catalyst.

It was of interest to determine whether there was a decrease in the total Co concentration in the used catalyst or if the Co intensity in the used catalyst was decreased by the presence of a contaminant. Program 3100 results for the fresh and used catalysts indicate that the Co concentration decreases on the used catalysts relative to the fresh catalyst. However, there were some problems with this program which have not yet been resolved.

Using the energy dispersive detector on the electron microscope, representative spectra of the entire energy range for both fresh (Figure 5.4) and used catalysts (Figure 5.5) were obtained. From Figures 5.4 and 5.5 it is possible to identify Fe as the major contaminant and to note that Fe was primarily present on the edge.

Figure 5.3 Co, Mo, and Al x-ray intensities relative to distance across a catalyst pellet, beam current 2.5×10^{-10} amp, accelerating voltage 25kv.

Figure 5.4 Energy dispersive X-ray spectra of fresh Amocat
1A catalyst, beam current 2.5×10^{-10} amp, accelerating
voltage 25kv.

Figure 5.5 Energy dispersive X-ray spectra of Amocat 1A catalysts, beam current 2.5×10^{-10} amp, accelerating voltage 25kV.

- a) 164c withdrawn from the reactor 6/24/82, edge of pellet
- b) 164c withdrawn from the reactor 6/24/82, center of pellet

a) EDGE

b) CENTER

Both linescans (similar to distributions done with the energy detector) and mapping were done using the wavelength-dispersive detector on the electron microscope. One advantage of the wavelength-dispersive x-ray detector has been the ability to distinguish Mo and S peaks. Figure 5.6 showed Mo and S linescans for a used catalyst. Note that both are nearly uniformly distributed across the pellet. The Co and Mo maps of the fresh catalyst (Figure 5.7) and the S, Co, and Mo, maps of the used catalyst (Figure 5.8) were very uniform. However the Fe and Ca maps of the used catalysts (Figure 5.9) showed segregation of those elements at the edges of the catalysts. From the map of the Ti (Figure 5.9) it was not possible to identify Ti on the used catalyst pellets, possibly because the Ti concentration is below the detection limit of the instrument.

5.4 Future Work

Bulk analyses by the energy-dispersive x-ray fluorescence technique will be routinely conducted on both fresh and used catalysts in order to monitor any gross changes in the catalyst compositions. Work will be done to make the x-ray fluorescence results more quantitative by establishing better standards for used catalyst.

Further ESCA studies will include 1) examining the spent catalysts as powder (bulk) in order to establish any differences with extrudates (surface), 2) examining the

S

Mo

Figure 5.6 Wavelength dispersive linescans of used catalyst 164b which was withdrawn from the reactor 6/11/82 and which is representative of the used catalysts, beam current 2×10^{-7} amp, accelerating voltage 35kV.

Mo Map

Co Map

Figure 5.7 X-ray maps of fresh catalyst taken with the wavelength dispersive detector, beam current 4×10^{-8} amp, accelerating voltage 35kV.

- a) Co map
- b) Mo map

Figure 5.8 X-ray maps of 164d withdrawn from the reactor 7/9/82 which is representative of the used catalysts, taken with the wavelength dispersive detector, beam current 4×10^{-8} amp, accelerating voltage 35kV.

- a) Co map
- b) Mo map
- c) S map

Co

S

Mo

Figure 5.9 X-ray maps of 164d withdrawn from the reactor 7/9/82 which is representative of the used catalysts, taken with the wavelength dispersive detector, beam current 4×10^{-8} amp, accelerating 35kV.

- a) Fe map
- b) Ca map
- c) Ti map

Ca

Fe

Ti

fresh catalyst before and after extraction to see whether the extraction process is having any affect on the catalyst surface, and 3) examining the residues from the extraction process to determine whether the extraction removes any metals from the catalyst surface.

Other electron microscopy work to be done will include 1) obtaining more linescans particularly of low level contaminants like Fe and Ca, 2) improving quantitation with the energy-dispersive detector 3) developing a quantitative program for the wavelength-dispersive detector so that the Mo and S can be determined uniquely, 4) continued mapping particularly of the contaminants in the used catalysts, and 5) further studies using the spectra of the entire energy range obtained from the energy dispersive detector.

In addition to the work with the techniques described above, Auger Electron Spectroscopy (AES) and Analytical Electron Microscopy (AEM) work is planned. The AES work should include analysis of the catalyst surfaces, depth profiling into the catalyst, mapping of crossectional areas (particularly for light elements like C,O,N), and linescans across the catalyst diameter. AEM will be used to measure both morphology and elemental composition of edges and centers of the catalysts.

5.5 References

- 1) T. L. Cable, F. E. Massoth, M. G. Thomas, Fuel Processing Technology, 4, (1981) 265-275.
- 2) F. Stohl, Sandia Laboratories, personal communication, 10/82.
- 3) O. C. Wells, "Scanning Electron Microscopy", New York: McGraw-Hill, (1974), p 260-265.

APPENDIX I: TABULATION OF DATA

TASK 3

INDEX TO DATA IN APPENDIX

ITEM NO.	CATALYST	SIZE	LIQUID	RUN NOS.
29	75A BEADS 2MM HD	-6+16 MESH	KEROSENE	812-841
30	75A BEADS 2MM	-5+18 MESH	5 CP FL.	842-876
31	75A BEADS 1.3MM	-12+20	KEROSENE	877-911
32	75A BEADS 1.3MM	-10+20	KEROSENE	912-941
33	75A BEADS 1.3MM	-8+25	KEROSENE	942-971
34	75A BEADS 1.3MM	-6+25	KEROSENE	972-1001
35	75A BEADS 1.3MM	-6+30	KEROSENE	1002-1031
36	75A BEADS 1.3MM	-6+40	KEROSENE	1032-1062
37	75A BEADS 1.3MM	-6+40	3 CP FL.	1063-1097

ADDITIONAL INFORMATION RE APPENDIX

ITEM NO.	CATALYST	N/B NO.	WT. CHRGD.,GM.	COMMENT
29	75A BEADS 2MM HD	S13501-52	1414	HIGH DENSITY. WEAKER THAN NOS. 12 & 19.
30	75A BEADS 2MM	S13501-35	1213	BATCH USED IN NO 27
31	75A BEADS 1.3MM	S14178-102	564	NARROW FRACTION OF SERIES.
32	75A BEADS 1.3MM		684	120 GM OF -10+12 ADDED TO NO 31
33	75A BEADS 1.3MM		882	108 GM -8+10 & 90 GM -20+25 ADDED.
34	75A BEADS 1.3MM		1038	152 GM -6+8 ADDED
35	75A BEADS 1.3MM		1110	72 GM -25+30 ADDED
36	75A BEADS 1.3MM		1176	42 GM -30+35 & 24 GM-35+40 ADDED.
37	75A BEADS 1.3MM		1176	INTERMEDIATE VISC FLUID, SAME CHARGE.

Run No.	Liq. Vel (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Bed Height		MANOMETRIC READINGS, CM. OF WATER									
			Settld In.	Measurd In.										
RN	UL	UG	H0	HM	M1	M0	M2	M3	M4	M5	M6	M7	M8	M9
812	.086	.000	18.00	20.0	-38.0	.0	37.7	61.8	61.0	61.5	61.3	60.3	60.4	60.3
813	.086	.012	18.00	20.0	-35.3	.0	33.5	57.3	54.2	52.0	49.1	46.0	42.5	39.4
814	.086	.025	18.00	21.0	-34.0	.0	31.8	55.5	51.4	47.0	42.6	37.4	32.4	27.3
815	.086	.050	18.00	21.0	-31.3	.0	29.8	52.5	47.3	40.8	34.3	26.8	19.0	11.5
816	.086	.100	18.00	21.0	-29.8	.0	29.3	48.7	42.4	32.4	22.5	11.7	.3	-11.0
817	.086	.150	18.00	22.0	-29.0	.0	27.5	43.6	36.0	23.3	10.8	-2.7	-17.8	-32.6
818	.127	.000	18.00	23.0	-34.9	.0	31.2	60.0	63.5	63.5	63.5	62.9	62.5	62.3
819	.127	.012	18.00	23.0	-31.5	.0	29.5	57.5	61.0	58.6	56.0	53.0	50.0	47.0
820	.127	.025	18.00	23.0	-30.3	.0	28.5	55.0	58.9	55.1	51.1	46.5	41.8	37.0
821	.127	.050	18.00	23.0	-27.8	.0	26.5	51.7	54.2	48.8	43.0	36.3	29.0	21.7
822	.127	.100	18.00	23.0	-26.0	.0	25.5	47.7	48.8	40.3	31.4	21.3	10.3	-1.3
823	.127	.150	18.00	23.0	-25.0	.0	23.0	44.3	43.5	32.2	19.8	7.5	-6.6	-21.4
824	.168	.000	16.50	27.0	-30.6	.0	27.0	52.3	69.5	69.3	68.8	68.6	68.3	68.3
825	.168	.012	16.50	27.0	-26.8	.0	24.5	48.5	67.3	64.9	62.2	59.1	56.6	53.6
826	.168	.025	16.50	27.0	-26.3	.0	23.8	46.8	63.8	61.0	56.8	52.5	48.3	43.7
827	.168	.050	16.50	26.0	-23.8	.0	22.2	43.8	58.6	54.3	48.3	41.8	34.8	27.8
828	.168	.100	16.50	26.0	-22.0	.0	21.3	41.3	52.9	46.4	38.1	27.9	17.9	7.7
829	.168	.150	16.50	26.0	-20.5	.0	19.8	39.0	48.2	39.0	28.0	15.0	1.8	-11.0
830	.209	.000	16.00	31.0	-27.0	.0	23.2	45.5	65.0	73.0	72.9	72.5	72.3	72.0
831	.209	.012	16.00	30.0	-23.7	.0	21.5	42.0	61.4	67.5	65.5	62.4	59.8	57.0
832	.209	.025	16.00	30.0	-23.7	.0	20.3	40.0	57.5	62.9	59.7	55.0	50.5	46.0
833	.209	.050	16.00	30.0	-20.4	.0	19.3	37.2	53.4	57.5	52.0	45.4	39.0	32.0
834	.209	.100	15.50	30.0	-19.0	.0	17.6	34.6	50.0	52.0	44.2	35.2	25.5	15.5
835	.209	.150	15.50	30.0	-17.8	.0	16.8	33.0	46.0	45.0	35.0	22.8	10.7	-1.7
836	.250	.000	15.50	38.0	-23.7	.0	19.8	39.3	56.8	71.0	77.3	77.3	77.0	77.0
837	.250	.012	15.50	37.0	-20.8	.0	18.3	36.0	52.3	66.0	69.2	66.8	64.2	62.0
838	.250	.025	15.50	37.0	-19.7	.0	17.5	33.5	48.0	59.8	62.6	58.4	53.7	49.3
839	.250	.050	15.50	37.0	-17.8	.0	15.6	30.0	42.8	53.0	54.2	48.3	41.3	34.0
840	.250	.100	15.50	36.0	-16.0	.0	14.0	27.5	41.0	49.4	47.0	38.5	28.8	18.8
841	.250	.150	15.50	35.0	-14.5	.0	13.2	26.2	37.7	44.3	39.2	28.3	16.2	3.3

Run No.	Liq. Vel (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Bed Height		MANOMETRIC READINGS, CM. OF WATER									
			Settled In.	Measured In.										
RN	UL	UG	H0	HM	M1	M0	M2	M3	M4	M5	M6	M7	M8	M9
842	.129	.000	19.00	48.0	-24.6	.0	11.8	28.0	44.0	57.2	68.6	77.1	80.0	80.0
843	.103	.000	19.00	39.0	-25.0	.0	17.7	36.7	54.5	68.5	77.3	77.2	77.2	77.0
844	.076	.000	19.00	31.5	-27.0	.0	22.0	44.4	64.3	73.0	74.0	73.5	73.3	73.0
845	.049	.000	19.00	26.0	-29.0	.0	27.5	52.8	68.5	69.0	69.3	69.0	68.3	68.0
846	.022	.000	19.00	21.5	-32.0	.0	34.2	61.8	62.4	62.0	61.7	61.3	60.8	60.3
847	.129	.200	19.00	46.0	-3.5	.0	13.2	19.5	24.4	26.9	26.4	20.2	7.0	-8.0
848	.103	.200	19.00	36.0	-10.0	.0	10.8	19.0	25.5	28.0	24.0	12.8	-2.7	-19.8
849	.076	.200	19.00	31.0	-13.3	.0	12.8	22.2	28.5	27.4	17.5	2.8	-14.5	-33.3
850	.049	.200	19.00	30.0	-17.0	.0	16.0	25.3	29.5	22.7	8.0	-8.7	-28.0	-47.0
851	.022	.200	19.00	24.0	-19.3	.0	18.0	25.7	25.2	13.8	-1.8	-19.8	-39.0	-58.8
852	.129	.150	19.00	40.0	-12.3	.0	6.4	14.0	20.5	25.0	27.0	23.7	14.9	3.0
853	.103	.150	19.00	36.0	-12.8	.0	10.8	20.8	28.0	33.0	30.1	20.5	8.1	-6.0
854	.076	.150	19.00	33.0	-15.0	.0	14.0	25.0	33.8	34.2	25.2	12.5	-1.9	-17.0
855	.049	.150	18.50	29.0	-18.5	.0	16.3	28.0	34.9	29.8	18.4	5.0	-9.7	-25.7
856	.022	.150	18.50	26.0	-20.6	.0	20.0	30.7	30.0	19.0	5.0	-9.6	-26.3	-43.0
857	.129	.100	18.50	46.0	-13.0	.0	8.5	18.0	26.5	33.6	37.2	34.8	27.0	17.3
858	.103	.100	18.50	40.0	-15.5	.0	11.0	22.8	34.0	40.3	39.1	32.0	22.2	11.2
859	.076	.100	18.50	33.0	-17.4	.0	15.3	29.0	40.8	42.6	36.0	26.3	15.3	3.8
860	.049	.100	18.50	28.0	-20.0	.0	20.1	35.5	43.0	37.8	28.0	17.0	5.2	-7.0
861	.022	.100	18.50	23.0	-21.0	.0	24.0	38.0	36.0	26.2	15.4	3.7	-8.7	-22.3
862	.129	.050	18.50	43.0	-18.8	.0	7.0	18.8	30.0	39.5	46.0	46.0	40.5	34.5
863	.103	.050	18.50	36.0	-19.0	.0	12.0	25.8	39.0	49.5	50.9	46.0	39.3	32.2
864	.076	.050	18.50	31.0	-20.5	.0	16.0	33.0	48.0	52.0	48.2	42.0	35.0	27.6
865	.049	.050	18.50	28.0	-22.8	.0	21.5	40.5	51.3	49.0	43.0	36.0	29.0	21.0
866	.022	.050	18.50	21.0	-24.0	.0	28.0	45.0	42.6	36.1	29.3	21.5	13.5	5.0
867	.129	.025	18.50	48.0	-18.0	.0	9.0	22.5	34.8	45.7	54.3	57.5	55.0	51.5
868	.103	.025	18.50	35.0	-19.8	.0	13.7	28.9	43.7	56.4	59.5	57.3	53.1	48.8
869	.076	.025	18.50	31.0	-21.8	.0	17.3	36.0	53.2	58.7	56.7	53.1	49.2	44.8
870	.049	.025	18.50	26.0	-23.4	.0	22.5	43.6	57.0	56.0	52.8	48.8	44.5	39.2
871	.022	.025	18.50	20.0	-22.5	.0	34.3	52.7	51.5	47.5	43.3	39.0	34.3	28.5
872	.129	.012	18.00	44.0	-17.3	.0	11.6	25.0	38.0	49.6	59.7	65.5	63.6	61.3
873	.103	.012	18.00	36.0	-20.3	.0	14.4	31.0	46.8	61.2	64.0	63.0	60.2	58.2
874	.076	.012	18.00	32.0	-23.0	.0	16.0	35.2	53.8	62.0	60.4	58.0	55.7	53.3
875	.049	.012	18.00	26.0	-24.5	.0	21.3	43.5	59.3	59.0	56.8	54.5	51.8	48.5
876	.022	.012	18.00	20.0	-27.0	.0	32.0	51.5	50.8	48.6	46.0	43.2	40.2	36.5

Run No.	Liq. Vel (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Bed Height		MANOMETRIC READINGS, CM. OF WATER										
			Settled In.	Measured In.											
RN	UL	UG	HO	HM	M1	HO	M2	M3	M4	M5	M6	M7	M8	M9	
877	.023	.000	10.00	11.0	-24.0	.0	8.5	10.4	10.0	9.6	7.4	7.4	7.0	6.0	
878	.023	.012	10.00	10.0	-22.4	.0	6.4	6.5	4.8	2.5	-2.3	-3.7	-6.7	-10.0	
879	.023	.025	10.00	10.0	-21.0	.0	6.0	5.5	2.0	-1.7	-8.4	-11.4	-16.8	-21.8	
880	.023	.050	10.00	12.0	-18.3	.0	6.0	4.3	-1.3	-7.2	-16.4	-22.8	-31.3	-39.5	
881	.023	.100	10.00	13.0	-14.7	.0	5.3	1.7	-7.3	-17.8	-30.7	-41.5	-53.5	-67.0	
882	.023	.150	10.00	16.0	-13.5	.0	1.4	-5.9	-17.6	-30.7	-45.6	-60.3	-75.8	-92.5	
883	.023	.200	10.00	16.0	-11.3	.0	.0	-9.8	-23.8	-40.0	-57.2	-75.2	-95.0	.0	
884	.043	.000	10.00	13.5	-20.0	.0	14.8	16.0	15.9	15.7	13.0	13.0	12.8	11.8	
885	.043	.012	10.00	13.0	18.3	.0	14.3	15.0	12.8	10.7	5.8	4.0	1.3	-2.3	
886	.043	.025	10.00	14.0	-17.0	.0	13.0	13.0	9.7	5.8	-5	-3.5	-8.4	-13.9	
887	.043	.050	10.00	16.0	-15.5	.0	10.8	10.0	5.0	-1.1	-9.7	-15.2	-23.3	-31.5	
888	.043	.100	10.00	18.0	-13.0	.0	8.3	6.0	-2.3	-12.3	-24.8	-35.3	-47.3	-59.8	
889	.043	.150	10.00	19.0	-12.5	.0	3.8	-2.2	-12.3	-24.5	-38.0	-52.5	-67.2	-82.1	
890	.043	.200	10.00	19.0	-10.8	.0	1.6	-6.0	-18.1	-33.3	-49.8	-67.2	-85.3	.0	
891	.064	.000	10.00	14.5	-20.8	.0	15.8	15.3	14.5	14.6	15.3	14.0	14.0	13.8	
892	.064	.012	10.00	15.0	-19.0	.0	13.7	13.4	11.6	9.7	7.5	5.3	2.4	-.3	
893	.064	.025	10.00	15.0	-18.3	.0	12.0	11.0	8.3	4.5	1.0	-2.8	-7.0	-11.8	
894	.064	.050	10.00	16.0	-16.7	.0	10.2	8.2	4.0	-1.8	-7.8	-14.0	-21.4	-28.8	
895	.064	.100	10.00	18.0	-14.0	.0	6.7	4.0	-4.0	-13.0	-23.3	-33.2	-45.0	-56.2	
896	.064	.150	10.00	19.0	-11.4	.0	4.3	-.7	-10.7	-23.2	-36.6	-50.3	-65.3	-81.0	
897	.064	.200	10.00	19.0	-9.9	.0	10.8	-3.8	-16.7	-31.0	-47.0	-64.0	-82.0	-100.0	
898	.084	.000	10.00	17.5	-18.0	.0	14.2	18.4	17.8	17.7	18.3	17.2	17.0	16.0	
899	.084	.012	10.00	16.0	-17.3	.0	13.6	16.3	14.5	13.0	11.3	9.3	6.7	4.4	
900	.084	.025	10.00	17.0	-16.2	.0	12.7	14.2	11.8	8.5	5.4	2.2	-2.3	-6.5	
901	.084	.050	10.00	18.0	-15.0	.0	11.0	11.8	8.0	3.0	-2.3	-7.9	-14.2	-20.5	
902	.084	.100	10.00	19.0	-12.2	.0	7.4	6.0	-1.2	-9.4	-19.1	-29.4	-40.8	-52.0	
903	.084	.150	10.00	19.0	-10.0	.0	4.8	2.0	-6.0	-17.1	-29.2	-43.3	-57.3	-72.2	
904	.084	.200	10.00	19.0	-6.0	.0	2.8	-1.8	-12.0	-25.7	-41.0	-57.0	-74.4	-92.8	
905	.104	.000	10.00	22.5	-15.0	.0	11.3	21.8	24.0	23.8	24.5	23.8	23.3	23.0	
906	.104	.012	10.00	21.0	-14.0	.0	11.7	19.8	19.3	17.4	16.0	13.7	11.0	8.5	
907	.104	.025	10.00	22.0	-13.2	.0	11.2	17.0	15.6	12.5	9.7	5.8	1.3	-3.0	
908	.104	.050	10.00	23.0	-12.0	.0	9.8	14.0	11.2	6.0	1.1	-5.8	-12.3	-19.2	
909	.104	.100	10.00	23.0	-9.8	.0	6.5	8.0	2.8	-5.5	-14.0	-24.5	-35.5	-46.0	
910	.104	.150	10.00	23.0	-7.7	.0	4.4	3.0	-3.7	-14.3	-26.0	-40.0	-54.0	-69.0	
911	.104	.200	10.00	23.0	-6.0	.0	1.8	-1.0	-9.7	-22.0	-36.3	-52.5	-69.7	-87.5	

Run No.	Liq. Vel (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Bed Height		MANOMETRIC READINGS, CM. OF WATER									
			Settled In.	Measured In.										
RN	UL	UG	HO	HM	M1	M0	M2	M3	M4	M5	M6	M7	M8	M9
912	.024	.000	12.00	13.0	-27.0	.0	16.0	15.8	15.5	15.4	16.8	15.5	15.5	15.3
913	.024	.025	12.00	13.0	-24.9	.0	11.8	10.5	7.8	4.5	1.5	-3.2	-7.4	-12.3
914	.024	.050	12.00	14.0	-22.0	.0	10.8	9.0	4.2	-1.4	-7.0	-14.2	-21.0	-29.0
915	.024	.100	12.00	16.0	-19.3	.0	8.3	5.2	-3.4	-12.5	-23.0	-34.0	-46.3	-58.5
916	.024	.150	12.00	17.0	-16.3	.0	5.8	1.0	-9.6	-22.2	-36.5	-51.8	-67.0	-84.0
917	.024	.200	12.00	17.0	-14.8	.0	3.5	-2.7	-15.4	-30.8	-47.5	-66.0	-85.5	.0
918	.051	.000	12.00	17.5	-22.0	.0	17.5	22.8	22.7	22.7	23.7	22.4	22.2	22.2
919	.051	.025	12.00	17.0	19.5	.0	15.7	18.0	15.5	11.5	8.0	3.2	-1.7	-6.5
920	.051	.050	12.00	18.0	-18.3	.0	14.0	15.5	12.0	6.5	1.0	-6.0	-13.0	-20.3
921	.051	.100	12.00	19.0	-15.5	.0	10.5	11.4	4.9	-3.8	-13.0	-23.7	-35.3	-47.0
922	.051	.150	12.00	20.0	-13.5	.0	7.8	6.8	-2.5	-13.4	-26.8	-41.2	-56.0	-71.7
923	.051	.200	12.00	20.0	-11.5	.0	5.3	2.5	-7.8	-22.0	-37.5	-55.3	-72.0	-91.0
924	.077	.000	12.00	21.0	-19.8	.0	15.5	26.2	26.0	26.0	26.8	25.7	25.5	25.4
925	.077	.025	12.00	20.0	-17.8	.0	14.8	21.2	19.3	16.2	13.3	9.0	5.0	1.0
926	.077	.050	12.00	21.0	-16.0	.0	13.3	18.5	15.0	10.0	4.7	-2.0	-8.5	-16.0
927	.077	.100	12.00	22.0	-14.2	.0	10.2	12.5	7.5	-.8	-10.0	-20.5	-31.3	-43.0
928	.077	.150	12.00	22.0	-11.5	.0	8.0	8.3	1.7	-9.4	-22.0	-36.0	-50.0	-64.7
929	.077	.200	12.00	22.0	-9.3	.0	5.8	4.2	-4.2	-17.4	-32.8	-49.5	-67.0	-84.8
930	.104	.000	12.00	27.0	-16.8	.0	12.4	22.8	31.3	31.3	32.2	31.5	31.3	31.0
931	.104	.025	12.00	24.0	-14.3	.0	12.3	22.3	23.8	20.8	18.0	14.0	10.3	6.4
932	.104	.050	12.00	25.0	-13.3	.0	11.0	19.0	18.7	14.8	9.7	3.2	-3.0	-10.5
933	.104	.100	12.00	26.0	-11.0	.0	8.8	13.0	11.3	4.0	-5.0	-15.0	-25.3	-36.5
934	.104	.150	12.00	27.0	-9.4	.0	6.4	8.5	4.3	-5.5	-17.4	-30.8	-45.2	-59.5
935	.104	.200	12.00	27.0	-7.5	.0	5.0	5.0	-1.5	-13.0	-27.0	-43.0	-59.2	-77.2
936	.132	.000	12.00	35.0	-14.8	.0	10.2	19.5	26.8	33.2	35.0	34.3	34.3	34.3
937	.132	.025	12.00	30.0	-12.0	.0	10.3	19.4	25.0	24.0	21.8	18.0	13.8	10.5
938	.132	.050	12.00	30.0	-11.0	.0	9.3	16.8	20.3	18.0	13.7	8.0	2.0	-4.8
939	.132	.100	12.00	31.0	-8.8	.0	7.0	11.8	11.8	6.4	-1.2	-10.8	-21.3	-31.6
940	.132	.150	12.00	32.0	-7.0	.0	4.8	7.3	5.3	-2.0	-12.2	-25.2	-38.3	-52.4
941	.132	.200	12.00	32.0	-5.5	.0	3.3	3.2	-1.0	-10.3	-21.8	-37.0	-53.0	-69.5

Run No.	Liq. Vel (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Bed Height		MANOMETRIC READINGS, CM. OF WATER									
			Settled In.	Measured In.										
RN	UL	UG	H0	HM	M1	M0	M2	M3	M4	M5	M6	M7	M8	M9
942	.016	.000	16.00	17.0	-22.4	.0	26.1	30.9	30.9	30.7	30.9	30.4	30.0	30.0
943	.016	.025	16.00	15.5	-23.5	.0	22.0	21.8	19.0	15.3	11.8	7.5	2.7	-2.5
944	.016	.050	16.00	16.0	-21.5	.0	19.0	19.8	15.4	9.5	3.4	-3.7	-11.4	-19.3
945	.016	.100	16.00	19.0	-19.3	.0	14.4	16.4	9.4	.3	-9.5	-21.2	-33.0	-46.3
946	.016	.150	16.00	21.0	-17.4	.0	10.7	10.7	2.0	-10.5	-24.8	-40.8	-56.0	-75.0
947	.016	.200	16.00	22.0	-17.0	.0	7.0	6.8	-3.4	-18.5	-35.6	-54.0	-74.8	-98.0
948	.039	.000	16.00	20.0	-24.8	.0	20.5	34.8	34.8	34.8	33.7	34.7	34.5	34.0
949	.039	.025	16.00	21.0	-21.8	.0	19.0	30.7	29.7	26.3	23.4	19.0	15.0	10.5
950	.039	.050	16.00	21.0	-20.3	.0	17.3	26.4	24.4	19.0	13.8	7.0	.0	-7.3
951	.039	.100	16.00	24.0	-18.0	.0	13.4	20.3	17.0	8.7	-.7	-11.8	-22.8	-35.0
952	.039	.150	16.00	24.0	-15.0	.0	10.8	15.2	10.0	-.5	-13.7	-27.5	-43.0	-59.0
953	.039	.200	16.00	25.0	-13.4	.0	8.8	10.6	3.5	-9.0	-24.4	-42.0	-60.4	-79.8
954	.063	.000	16.00	24.0	-22.0	.0	18.0	33.0	39.0	38.5	39.3	38.4	38.5	38.4
955	.063	.025	15.50	24.5	-19.0	.0	16.8	30.2	31.4	31.4	28.3	24.2	20.2	16.0
956	.063	.050	15.50	25.0	-18.0	.0	15.8	27.8	28.5	24.0	18.8	12.5	5.6	-1.3
957	.063	.100	15.50	26.0	-15.6	.0	12.7	21.8	20.4	13.2	4.2	-6.0	-17.4	-28.8
958	.063	.150	15.50	27.0	-13.4	.0	10.1	15.3	12.9	3.2	-8.5	-22.3	-37.7	-53.5
959	.063	.200	15.50	28.5	12.3	.0	7.9	10.6	6.7	-4.2	-19.1	-36.6	-54.0	-73.0
960	.086	.000	15.50	31.5	-19.5	.0	15.0	27.9	38.6	43.8	44.1	43.5	43.5	43.5
961	.086	.025	15.50	26.5	-16.6	.0	14.1	28.0	34.9	33.7	31.0	27.3	22.7	18.9
962	.086	.050	15.50	28.5	-15.4	.0	13.5	25.1	30.1	27.5	22.5	16.3	9.5	2.9
963	.086	.100	15.50	29.5	-13.4	.0	11.0	19.8	22.0	16.5	8.3	-1.7	-12.3	-23.0
964	.086	.150	15.50	31.0	-11.8	.0	8.8	15.0	14.1	6.8	-4.3	-17.5	-31.3	-46.2
965	.086	.200	15.50	33.0	-9.8	.0	7.3	10.0	8.4	-1.0	-14.2	-29.6	-46.8	-64.0
966	.109	.000	15.50	35.5	-17.8	.0	13.7	25.0	35.1	42.7	45.0	44.5	44.5	44.2
967	.109	.025	15.50	29.0	-14.5	.0	12.7	24.6	34.1	35.0	32.5	28.5	24.5	19.9
968	.109	.050	15.50	30.0	-13.1	.0	11.3	21.5	29.0	28.8	25.2	19.5	13.1	6.3
969	.109	.100	15.30	30.5	-11.5	9.5	17.5	21.5	18.0	10.8	1.6	0.7	19.7	.0
970	.109	.150	15.50	31.5	-9.5	.0	7.4	12.8	14.1	8.4	-1.6	-13.9	-27.6	-42.5
971	.109	.200	15.50	32.5	-8.0	.0	6.1	8.8	7.5	-.5	-12.4	-27.4	-43.5	-61.4

Run No.	Liq. Vel (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Bed Height		MANOMETRIC READINGS, CM. OF WATER									
			Settld In.	Measurd In.										
RN	UL	UG	HO	HM	M1	H0	H2	H3	H4	H5	H6	H7	H8	H9
972	.030	.000	18.00	21.0	-23.9	.0	24.1	44.0	44.1	44.4	44.4	43.3	43.2	43.3
973	.030	.025	18.00	21.5	-25.3	.0	22.9	36.8	35.6	31.8	28.3	24.1	19.4	14.3
974	.030	.050	18.00	23.0	-23.5	.0	20.6	33.2	31.5	26.2	20.1	13.5	6.8	-.7
975	.030	.100	18.00	24.5	-21.5	.0	16.0	26.1	24.0	15.3	5.7	-4.9	-16.7	-29.3
976	.030	.150	18.00	26.5	-18.6	.0	13.4	20.4	17.0	6.2	-7.2	-21.0	-36.5	-53.0
977	.030	.200	18.00	26.5	-17.3	.0	10.1	14.3	10.0	-2.5	-18.3	-35.7	-54.5	-75.2
978	.050	.000	18.00	25.5	-24.8	.0	21.5	37.3	47.7	47.7	47.9	47.5	47.3	47.3
979	.050	.025	18.00	24.0	21.7	.0	19.5	37.1	41.8	39.6	36.7	32.3	28.1	24.0
980	.050	.050	18.00	25.0	-20.5	.0	18.0	33.1	37.1	32.9	27.5	21.3	15.0	7.6
981	.050	.100	18.00	26.0	-18.4	.0	14.8	25.8	28.5	22.0	13.3	2.5	-8.3	-19.8
982	.050	.150	18.00	27.0	-16.1	.0	12.2	20.3	20.5	11.8	-.5	-13.9	-29.1	-45.0
984	.068	.000	18.00	28.0	-22.7	.0	18.5	34.4	37.3	49.3	49.3	49.0	48.7	48.5
985	.068	.025	18.00	26.5	-19.2	.0	17.5	34.2	43.6	41.6	38.4	34.6	30.6	26.0
986	.068	.050	18.00	28.0	-17.8	.0	16.8	31.0	38.7	35.5	30.5	24.1	17.5	11.0
987	.068	.100	18.00	29.0	-15.6	.0	14.3	25.4	29.8	24.5	15.6	5.5	-5.1	-16.3
988	.068	.150	18.00	30.5	-13.9	.0	11.1	19.5	21.6	14.6	2.8	-9.9	-24.4	-39.6
989	.068	.200	18.00	31.5	-12.7	.0	9.3	15.0	15.0	6.7	-7.0	-23.5	-40.0	-58.1
990	.090	.000	18.50	34.5	-20.8	.0	16.6	29.9	42.1	51.1	52.8	52.4	52.3	52.0
991	.090	.025	18.50	31.5	-16.7	.0	15.4	29.0	41.5	43.5	41.0	37.1	32.8	28.3
992	.090	.050	18.50	31.0	-15.7	.0	14.3	27.0	36.9	37.2	32.7	28.4	20.1	13.0
993	.090	.100	18.50	32.0	-13.3	.0	12.5	22.0	28.7	27.2	20.2	10.6	.0	-10.8
994	.090	.150	18.50	33.0	-12.0	.0	10.0	17.0	21.1	17.0	7.7	-5.5	-18.6	-23.5
995	.090	.200	18.50	34.0	-10.8	.0	8.6	13.4	14.8	9.5	2.0	-16.6	-33.0	-50.3
996	.109	.000	18.50	45.0	-26.5	.0	6.7	19.2	30.0	38.6	45.3	49.0	49.5	49.6
997	.109	.025	18.50	35.0	-15.7	.0	13.4	26.0	38.0	44.0	43.8	40.8	37.5	33.2
998	.109	.050	18.50	33.0	-14.2	.0	13.3	24.6	34.8	38.9	36.1	31.2	26.0	18.9
999	.109	.100	18.50	34.0	-11.7	.0	11.8	21.2	28.0	28.1	23.0	14.2	5.3	-5.5
1000	.109	.150	18.50	35.5	-10.4	.0	9.1	15.7	20.0	18.9	12.4	1.9	-10.3	-24.6
1001	.109	.200	18.50	36.5	-9.1	.0	7.7	12.7	14.1	10.8	2.8	-9.7	-24.6	-41.6

Run No.	Liq. Vel (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Bed Height		MANOMETRIC READINGS, CM. OF WATER									
			Settled In.	Measured In.										
RN	UL	UG	H0	HM	M1	M0	M2	M3	M4	M5	M6	M7	M8	M9
1002	.026	.000	18.50	21.5	-29.3	.0	25.7	46.6	46.6	46.1	46.1	45.8	45.6	45.4
1003	.026	.025	18.50	20.5	-26.7	.0	24.2	39.8	39.3	36.2	32.0	28.0	23.9	18.9
1004	.026	.050	18.50	22.0	-24.4	.0	22.4	36.4	35.9	31.0	25.0	18.8	12.0	4.3
1005	.026	.100	18.50	24.5	-22.1	.0	17.7	28.7	27.8	20.1	10.7	.4	-9.9	-21.6
1006	.026	.150	18.50	25.5	-20.8	.0	14.0	21.5	19.0	9.4	-3.1	-16.4	-30.8	-46.2
1007	.026	.200	18.50	26.5	-19.2	.0	10.8	16.1	12.0	.5	-14.0	-31.0	-49.5	-68.7
1008	.045	.000	18.50	24.5	-26.2	.0	22.0	40.5	47.7	47.7	47.5	47.2	47.0	46.7
1009	.045	.025	18.50	24.5	-23.4	.0	21.2	39.5	42.9	40.3	36.7	33.1	29.0	24.9
1010	.045	.050	18.50	25.5	-22.2	.0	19.1	35.0	37.7	33.5	27.5	21.3	14.9	7.6
1011	.045	.100	18.50	26.0	-19.8	.0	16.5	27.5	29.3	22.9	13.7	4.0	7.0	-17.8
1012	.045	.150	18.50	27.5	-17.3	.0	13.4	22.3	21.5	13.2	1.0	-12.5	-27.1	-41.8
1013	.045	.200	18.50	28.5	-16.2	.0	10.4	17.8	15.4	5.5	-8.4	-23.2	-40.8	-59.0
1014	.065	.000	18.50	28.5	-23.8	.0	18.7	35.4	48.3	50.2	50.3	50.0	50.0	49.8
1015	.065	.025	18.50	27.5	-21.0	.0	18.0	25.5	43.8	42.0	39.2	35.3	31.5	27.0
1016	.065	.050	18.50	28.0	-19.7	.0	16.8	32.0	39.0	36.1	31.0	24.8	18.5	11.6
1017	.065	.100	18.50	29.0	-18.8	.0	15.0	27.0	30.8	26.0	17.1	7.9	-2.0	-13.0
1018	.065	.150	18.50	30.5	-15.0	.0	11.7	20.6	23.0	16.8	6.0	-6.7	-20.1	-34.7
1019	.065	.200	18.00	31.5	-14.7	.0	9.8	16.0	16.2	9.4	-3.2	-18.1	-34.8	-52.0
1020	.084	.000	18.50	33.5	-22.3	.0	16.6	32.0	44.0	52.2	52.5	52.3	52.0	51.7
1021	.084	.025	18.50	29.0	-18.5	.0	16.5	31.8	43.7	44.3	41.7	38.3	34.4	30.3
1022	.084	.050	18.50	30.3	-17.9	.0	15.3	29.2	38.8	38.1	33.3	27.3	21.3	14.8
1023	.084	.100	18.50	31.5	-15.3	.0	12.6	23.5	29.9	27.8	20.8	11.9	1.8	-8.4
1024	.084	.150	18.50	32.5	-12.8	.0	11.4	19.3	22.8	18.3	9.0	-2.7	-16.3	-29.5
1025	.084	.200	18.50	33.8	-11.8	.0	8.8	15.0	15.5	9.5	-2.0	-15.6	-32.1	-47.4
1026	.104	.000	18.50	41.0	-20.4	.0	14.5	27.8	38.8	46.8	52.4	53.5	53.5	53.3
1027	.104	.025	18.50	32.5	-15.1	.0	14.0	27.0	38.3	43.5	42.5	39.6	36.2	32.3
1028	.104	.050	18.50	33.5	-13.3	.0	13.3	25.3	34.8	37.5	34.5	29.7	23.5	17.1
1029	.104	.100	18.50	34.3	-11.5	.0	11.5	20.8	27.0	27.5	22.3	14.2	4.9	-5.0
1030	.104	.150	18.50	35.5	-11.3	.0	8.4	15.4	19.1	16.9	10.4	-.8	-12.8	-26.6
1031	.104	.200	18.50	36.5	-9.1	.0	7.4	12.2	12.7	9.0	.5	-13.1	-27.8	-43.4

Run No.	Liq. Vel (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Bed Height		MANOMETRIC READINGS, CM. OF WATER									
			Settled In.	Measured In.										
RN	UL	UG	HO	HM	M1	M0	M2	M3	M4	M5	M6	M7	M8	M9
1032	.027	.000	18.00	21.0	-23.2	.0	26.5	46.9	46.8	46.8	46.6	46.2	46.0	45.1
1033	.027	.025	18.00	20.5	-26.0	.0	24.8	38.8	37.4	34.1	30.7	27.2	23.0	19.0
1034	.027	.050	18.00	21.5	-24.6	.0	21.0	33.3	32.1	27.0	21.4	14.8	7.9	.8
1035	.027	.100	18.00	22.3	-22.1	.0	17.5	26.9	23.8	15.5	6.3	-3.7	-15.3	-26.7
1036	.027	.150	18.00	24.5	-20.2	.0	13.4	20.0	15.7	5.6	-7.7	-21.3	-36.8	-52.5
1037	.027	.200	18.00	26.5	-16.5	.0	13.5	16.2	11.3	-1.1	-17.6	-34.6	-52.6	-72.7
1038	.027	.012	18.00	19.3	-27.3	.0	27.4	40.5	40.0	37.7	35.4	32.7	30.2	27.1
1039	.039	.000	18.00	22.0	-26.1	.0	23.9	43.3	44.0	43.8	43.8	43.2	42.8	42.6
1040	.039	.025	18.00	20.8	-23.3	.0	23.2	38.6	38.5	35.4	31.4	27.3	23.0	18.8
1041	.039	.050	18.00	22.5	-23.0	.0	20.5	33.8	33.7	28.7	22.5	16.3	9.3	1.8
1042	.039	.100	18.00	24.5	-21.1	.0	16.1	26.5	25.0	17.1	7.3	-3.0	-13.8	-25.0
1043	.039	.150	18.00	25.8	-18.8	.0	12.9	19.8	17.3	7.8	-4.9	-18.8	-33.8	-50.3
1044	.039	.200	18.00	26.5	-17.1	.0	9.9	15.7	12.5	.6	-14.0	-30.0	-48.5	-66.3
1045	.050	.000	17.50	24.0	-25.6	.0	21.5	39.0	44.5	43.9	43.8	43.5	43.3	43.0
1046	.050	.025	17.50	23.0	-23.3	.0	20.3	36.9	38.8	35.7	32.8	28.7	24.4	20.4
1047	.050	.050	17.50	25.0	-22.6	.0	18.7	34.2	35.3	31.2	26.2	20.5	14.9	8.7
1048	.050	.100	17.50	26.0	-19.5	.0	15.7	26.2	27.0	19.9	11.4	1.8	-8.6	-20.2
1049	.050	.150	17.50	27.3	-17.3	.0	12.7	20.1	19.8	10.7	.8	-13.6	-28.4	-42.2
1050	.050	.200	17.50	28.3	-14.4	.0	10.0	15.1	12.8	2.5	-11.0	-27.1	-45.0	-63.2
1051	.060	.000	17.50	27.5	-23.4	.0	19.1	35.3	46.5	46.7	46.5	46.3	46.2	46.0
1052	.060	.025	17.50	23.3	-20.5	.0	18.9	35.3	41.2	39.0	35.8	32.2	28.3	24.4
1053	.060	.050	17.50	24.5	-19.6	.0	17.2	31.8	35.7	31.6	26.2	20.5	13.9	7.0
1054	.060	.100	17.50	26.0	-17.5	.0	14.3	24.8	26.7	20.1	11.7	1.7	-9.4	-21.3
1055	.060	.150	17.50	28.5	-16.1	.0	11.9	20.0	20.3	12.3	1.0	-11.8	-24.2	-38.8
1056	.060	.200	17.50	29.3	-12.8	.0	10.1	15.3	13.0	3.5	-10.4	-25.6	-43.1	-60.6
1057	.073	.000	17.50	29.0	-22.3	.0	18.3	33.4	45.7	47.0	46.8	46.6	46.3	46.0
1058	.073	.025	17.50	25.5	-19.1	.0	16.8	32.9	40.1	39.1	36.0	33.0	29.0	24.8
1059	.073	.050	17.50	27.0	-17.5	.0	16.0	29.9	35.9	32.7	27.8	21.9	15.5	8.8
1060	.073	.100	17.50	28.0	-16.0	.0	13.5	23.9	27.4	22.1	14.3	4.6	-6.3	-16.3
1061	.073	.150	17.50	29.5	-14.1	.0	11.2	19.3	20.1	12.7	2.3	-10.2	-24.5	-39.0
1062	.073	.200	17.50	30.8	-12.2	.0	8.8	14.0	13.3	5.5	-7.0	-22.1	-39.5	-57.5

Run No.	Liq. Vel (Sup.) Ft/Sec	Gas Vel (Sup.) Ft/Sec	Bed Height		MANOMETRIC READINGS, CM. OF WATER									
			Settld In.	Measured In.										
RN	UL	UG	H0	HM	M1	M0	M2	M3	M4	M5	M6	M7	M8	M9
1063	.013	.000	17.50	19.0	-26.5	.0	26.5	41.0	40.8	40.3	40.2	40.6	40.1	39.8
1064	.013	.025	17.50	20.0	-26.7	.0	22.3	30.2	29.0	26.3	22.1	18.0	13.5	8.5
1065	.013	.050	17.50	22.0	-23.5	.0	19.5	28.8	27.0	21.9	15.4	8.8	2.0	-6.0
1066	.013	.100	17.50	23.5	-20.5	.0	15.8	22.9	19.7	11.4	1.3	-10.2	-22.4	-36.3
1067	.013	.150	17.50	24.5	-19.0	.0	12.5	16.7	12.0	2.0	-12.3	-28.2	-41.4	-63.1
1068	.013	.200	17.50	26.0	-16.6	.0	9.6	12.0	6.1	-6.1	-23.8	-42.5	-63.0	-84.8
1069	.013	.000	17.50	17.9	-29.8	.0	25.9	36.7	36.1	36.3	36.0	36.3	35.8	35.8
1070	.018	.000	17.50	20.0	-29.0	.0	24.5	40.0	39.4	39.3	39.3	39.3	39.0	38.8
1071	.018	.025	17.50	21.5	-24.5	.0	22.9	33.3	32.7	29.8	25.4	21.0	16.3	11.1
1072	.018	.050	17.50	22.5	-23.1	.0	19.5	29.5	28.5	23.5	17.0	9.8	2.5	-6.0
1073	.018	.100	17.50	24.0	-20.3	.0	15.0	22.7	20.1	12.0	1.3	-9.9	-22.8	-36.8
1074	.018	.150	17.50	25.3	-16.9	.0	13.3	18.2	14.4	3.5	-10.6	-25.8	-42.2	-60.4
1075	.018	.200	17.50	26.5	-15.2	.0	9.8	12.7	6.8	-4.5	-22.1	-40.3	-60.3	-82.3
1076	.018	.000	17.50	19.5	-32.0	.0	22.0	36.0	35.7	36.0	35.8	35.8	35.6	35.6
1077	.036	.000	17.50	26.5	-25.3	.0	19.8	36.0	35.1	45.2	45.4	45.6	45.6	45.6
1078	.036	.025	17.50	24.0	-22.7	.0	19.3	35.2	38.1	36.0	32.8	28.7	24.0	20.1
1079	.036	.050	17.50	25.0	-19.8	.0	18.0	31.1	33.2	29.2	23.5	17.2	10.2	2.5
1080	.036	.100	17.50	26.3	-16.8	.0	15.1	24.8	24.8	18.0	8.6	2.3	-13.8	-26.7
1081	.036	.150	.00	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0
1002	.036	.200	17.50	29.0	-13.8	.0	9.5	13.5	9.0	-1.0	-16.4	-34.3	-53.6	-74.3
1083	.036	.000	17.50	24.8	-26.6	.0	19.6	36.6	42.3	42.3	42.3	42.2	42.2	42.0
1084	.054	.000	17.50	29.0	-24.2	.0	18.3	33.5	44.1	45.6	45.8	45.7	45.7	45.7
1085	.054	.025	17.50	26.0	-19.6	.0	17.1	32.4	37.7	36.5	34.0	30.4	26.0	21.7
1086	.054	.050	17.50	26.5	-18.5	.0	16.3	29.5	34.5	32.0	27.2	21.3	15.3	8.5
1087	.054	.100	17.50	29.0	-16.3	.0	13.3	23.3	26.8	23.0	14.5	5.6	-4.6	-15.8
1088	.054	.150	17.50	30.0	-13.0	.0	11.5	18.0	17.1	9.8	-2.0	-15.7	-31.3	-47.4
1089	.054	.200	17.50	31.0	-11.1	.0	9.5	12.5	10.2	1.5	-13.5	-30.2	-48.8	-69.0
1090	.054	.000	17.50	29.5	-24.9	.0	15.0	29.2	40.4	43.6	43.9	43.9	43.7	43.4
1091	.073	.000	17.50	35.0	-20.1	.0	14.6	27.0	36.8	44.3	49.2	50.0	50.4	50.4
1092	.073	.025	17.50	28.0	-16.9	.0	15.8	29.8	38.5	38.6	36.0	32.6	28.7	24.6
1093	.073	.050	17.50	29.0	-14.8	.0	13.3	24.5	31.8	32.0	28.0	22.5	16.1	9.5
1094	.073	.100	17.50	30.5	-12.8	.0	11.6	19.8	24.0	21.4	14.0	4.3	-6.5	-18.4
1095	.073	.150	17.50	32.0	-10.7	.0	8.9	14.3	15.9	11.1	1.0	-11.5	-25.7	-41.2
1096	.073	.200	17.50	39.0	-9.0	.0	7.8	11.1	9.8	4.5	-8.6	-23.8	-40.5	-59.4
1097	.073	.000	17.50	36.0	-21.8	.0	13.4	25.8	36.3	43.3	46.5	46.5	46.3	46.3