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ANALYSIS OF SAMPLES OBTAINED FROM GKT
GASIFICATION TEST OF KENTUCKY COAL—
NONPROPRIETARY VERSION

Final Report

By
S. V. Raman

Work Performed Under Contract No. AC05-78OR03054

Air Products and Chemicals, Inc.
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Technical Information Center
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United States Department of Energy

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SUBCONTRACTED R&D FINAL REPORT:

ANALYSIS OF SAMPLES OBTAINED FROM
GKT GASIFICATION TEST OF KENTUCKY COAL
NONPROPRIETARY VERSION

RADIAN CORPORATION
Austin, Texas

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Prepared for the
UNITED STATES DEPARTMENT OF ENERGY
Office of Solvent-Refined Coal Products
under Contract DE-AC05-78-OR0-3054

*Dr. Raman was affiliated with International Coal Refining Company during most of this program.

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TABLE OF CONTENTS

	<u>Page</u>
LIST OF TABLES	ii
LIST OF FIGURES	ii
ABSTRACT	iii
BACKGROUND AND EXECUTIVE SUMMARY	1
Program Objectives	1
Program Execution	1
GKT Pilot Coal Gasification Test	1
Radian Program	7
Conclusions	13
Recommendations	13
REFERENCES	14
APPENDIX: RADIANT CORPORATION REPORT	

LIST OF TABLES

<u>Table No.</u>	<u>Title</u>	<u>Page</u>
1	GKT and Radian Analytical Tasks	2
2	Ultimate Analysis of Test Coal	9
3	Pilot Gasification Results - Kentucky #9 Coal	10
4	Hydrocarbon and Trace Metal Content of the Cold Raw Gas	11
5	Analysis of Wash Water	12

LIST OF FIGURES

<u>Figure No.</u>	<u>Title</u>	<u>Page</u>
1	Flow Diagram of Coal Dust Gasification Test Unit	3
2	Raw Gas Analytical Scheme	6
3	Wash Water Analytical Scheme	8

ABSTRACT

A laboratory test program was performed to obtain detailed compositional data on the Gesellschaft für Kohle-Technologie (GKT) gasifier feed and effluent streams. GKT performed pilot gasification tests with Kentucky #9 coal and collected various samples which were analyzed by GKT and the Radian Corporation, Austin, Texas. The coal chosen had good liquefaction characteristics and a high gasification reactivity. No organic priority pollutants or PAH compounds were detected in the wash water, and solid waste leachates were within RCRA metals limits.

BACKGROUND AND EXECUTIVE SUMMARY

Program Objectives

This program was performed to obtain detailed compositional data on the Gesellschaft für Kohle-Technologie (GKT) gasifier feed and effluent streams. Adequate characterizations of the streams were to be obtained to determine the path of the major components, trace organic compounds, and trace elements.

Program Execution

GKT was subcontracted to perform pilot gasification tests with a Kentucky #9 coal in their laboratory in Essen, West Germany. These tests provided material and heat balance data, levels of carbon conversion, and syngas production rates. In addition to these analyses, GKT analyzed the raw product gas for trace organics and elements.

GKT collected samples of feed coal, effluent wash water, slag, and fly dust, preserved them according to ICRC recommendations, and shipped them to Radian Corporation, Austin, Texas for further analysis.

The division of tasks between GKT and Radian is summarized in Table 1.

GKT Pilot Coal Gasification Test

Between 24 November and 10 December 1981, GKT performed six pilot gasification test runs with a Kentucky #9 coal at various oxygen-to-carbon ratios in the feed. The tests were conducted to provide material and heat balances and data on carbon conversion and useful gas production for the SRC-I coal gasification area. Similar tests conducted in December 1979 had established design conditions for the "mixed" feed case, i.e., KMAC and coal. In addition to the usual battery of analyses, ICRC requested GKT to perform some trace organic and elemental analyses in the product raw gas and to collect effluent water, slag, and fly dust samples for later trace analyses in the U.S.

Description of the Pilot Gasifier. A schematic flow diagram of the gasification test unit is shown in Figure 1. Coal is fed to the reactor from a variable-speed screw attached to a feed hopper pressurized with nitrogen. The coal is entrained in a high-velocity oxygen stream, which

Table 1

GKT and Radian Analytical Tasks

<u>GKT</u>	<u>Radian</u>
<u>Feed coal analyses</u>	<u>Feed coal analyses</u>
1. physical properties	1. physical properties
2. ultimate analysis	2. ultimate analysis
3. ash analysis	3. ash analysis
	4. trace elements
	5. radioactivity
<u>Pilot gasification tests</u>	<u>Process feed water and wash water</u>
1. material balances	1. water quality parameters
2. heat balances	2. extractable organic priority pollutants
<u>Raw syngas analyses</u>	3. polynuclear aromatic hydrocarbons (PAHs)
1. major components	4. trace elements
2. trace hydrocarbons	5. radioactivity
3. trace elements	
<u>Wash-water analyses</u>	<u>Slag and fly dust</u>
1. major components	1. physical properties
	2. ultimate analysis
	3. ash analysis
	4. extractable organics and PAHs
	5. trace elements
	6. radioactivity
	<u>Solid leaching tests</u>
	1. leaching and analysis for elements of slag and fly dust according to EPA and ASTM methods

FIGURE 1
FLOW DIAGRAM OF COAL DUST
GASIFICATION TEST UNIT

LEGEND:

1. FEED HOPPER FOR COAL DUST	12. PACKED WASH COLUMN
2. FEED DOSING SCREW	13. WATER SEPARATOR
3. MIXING CHAMBER	14. FILTER FOR FINES
4. BURNER	15. GAS METER
5. REACTION SPACE	16. DESULFURIZER
6. REACTOR	17. COMBUSTION CHAMBER
7. ELECTRICAL HEATER	18., 19., 20. RECEIVERS FOR WATER
8. INSULATING MATERIAL	FROM 10, 11 and 12
9. QUENCH CHAMBER	21. FILTER
10. QUENCH-WATER RECEIVER	22. HEAT EXCHANGER
11. WASH COLUMN WITH SIEVE PLATES	23. PUMP

carries the material through a blow tube attached to the reactor burner block. Located in the center of the oil-cooled burner head, the oxygen/coal injection port is surrounded by a concentric steam injection pipe. The reactor is a vertical alumina tube encased in an insulated metal shell fitted with electrical heaters that provide some heat leak compensation. Temperature readings are taken from thermocouples placed at strategic locations along the reactor tube. The complete system of feed hopper, drive motor, screw, mixing chamber, and blow tube is removable as a single unit. The lower end of the reactor tube sits on a ceramic restriction orifice plate called a "drip plate," which is attached to a series of concentric alumina tubes of increasing diameter. Molten slag produced during the gasification run flows down the side of the reactor tube and drips into a quench chamber cooled by water sprays. The solidified slag, together with some entrained fly dust, is then collected in the quench water receiver. The cooled reactor exit gas, containing the bulk of the fly dust, passes from the quench chamber sequentially through a sieve plate column, a packed-bed wash column, and a water separator. The gas stream is then filtered, desulfurized in an iron oxide bed, and burned in a small flare system. The gas flow is recorded by an in-line calibrated flowmeter.

Water from the quench water receiver passes through a filter press for solids removal, and is then collected in a series of three receiving vessels. A recycle water stream is supplied to the quench chamber and the sieve plate wash column from the filter press outlet. Makeup water, approximately 30% of the total recycle flow, is supplied to the packed wash column.

Operation of the Pilot Gasifier. GKT gave the following description of unit startup:

The reactor is preheated by firing natural gas/air through the burner for approximately 20 hours before commencement of the test. The electrical heater is switched on during the preheat period. At the end of the preheat period, when the reactor temperatures are lined out, the gas burner is shut off and the coal feed assembly containing the feed material is lifted into position on top of the reactor and the flange bolted down. The burner is then fired on natural gas/air for 2-3 hours in order to restabilize the reactor temper-

ature profile. The oxygen and coal flows to the reactor tube are then established, with the coal feed initially set at approximately 1 kilogram per hour. After a 5-10 minute equilibration period, the natural gas/air rate is progressively reduced while the coal/oxygen/steam feed is increased until the coal feed levels are satisfactory... The reactor is slowly pressurized to 2 bars (30 psig) and allowed to stabilize.

Eight technicians and one supervisor operated the gasifier, collected samples, and ran the Orsat analysis for gas composition. The unit is controlled manually.

When the reactor has stabilized, a 30-min balance period commences, during which the following measurements are taken: 1) quench and cooling water flow; 2) temperature, pressure, and flow rate of coal, oxygen, steam, and nitrogen at 10-min intervals; and 3) reactor off-gas compositions by Orsat every 10 min. In addition, the gas is bubbled through various impinger solutions to obtain the trace component analyses depicted in Figure 2.

At the conclusion of the balance period, nitrogen is fed to the reactor in place of oxygen, the coal and steam flows are terminated, and a 20-min purge cycle is carried out. The run is ended by allowing the reactor to depressurize. The coal remaining in the feed hopper is then weighed, and fly dust is collected by filtering the wash water contained in the three main wash-water reservoirs. Small quantities of fly dust adhering to the walls of the quench chamber, the surface of the slag, and the wash columns, are collected in the fines filter, then dried, combined, and weighed. Slag generated in the run is recovered from the quench water receiver and from the sides of the reaction tube. The total run time, and therefore the quantity of slag and fly dust samples generated from each test, is limited by: 1) the capacity of the reactor feed hopper; 2) the capacity of the quench chamber; 3) the life of the alumina reactor tube, which is eroded by the molten slag; and 4) the tendency for progressive blockage of the drip plate orifice due to formation of a high-melting-point, alumina-contaminated slag during the run. Approximately 1.5 kg of fly dust and 50 to 100 g of slag are obtained from each run. The slag samples are contaminated by alumina

FIGURE 2
RAW GAS ANALYTICAL SCHEME

washed from the reactor tube walls, since most of the slag sticks to the sides of the reactor tube.

GKT also collected fine fly dust removed via the fines filter (unit number 14 in Figure 1), and a 1-gal wash-water sample from each gasification run.

The analyses carried out by GKT on the wash-water samples are summarized in Figure 3. Ultimate analyses of the slag and fly dust are also obtained. These results are used to complete a heat and material balance for the reactor. The elemental, heat, and mass balances are usually closed to within 1-3%.

Results. The sample tested by GKT was a Kentucky #9 coal supplied by ICRC, and was selected after three coal samples were screened. The coal chosen had good liquefaction characteristics and a high gasification reactivity.

The ultimate analysis of the test coal is shown in Table 2, and results from the six gasification tests (runs 212-216) are shown in Table 3.

Trace Compounds in the Raw Gases. The concentrations of trace heavy metals in the cold raw gas leaving the test unit are provided in Table 4. Methane was the predominant hydrocarbon found in the raw gas from the pilot gasifier, and only ethylene and acetylene were also present in measurably significant quantities. In commercial gasifier operations apart from methane, there are no higher hydrocarbons in the raw gas. It is noteworthy that the hydrocarbon concentration of the raw gas generally decreased with increasing gasifier temperature.

There is no evidence of mercaptans in the gas. The detectability limit of the analysis was 0.5 ppm (by volume). Quantities of the five trace metals, Be, Fe, Hg, Ni, and Pb, varied from 4 to 90 $\mu\text{g}/\text{Nm}^3$.

Wash-Water Analysis. The analysis of the wash water collected over each run period is shown in Table 5.

Radian Program

As mentioned earlier, samples of the feed coal, process feed water, wash water, slag, and fly dust were shipped to Radian for detailed

FIGURE 3
WASHWATER ANALYTICAL SCHEME

Table 2

Ultimate Analysis of Test Coal

	Wt % (MF ^a)
Hydrogen	4.90
Carbon	73.10
Sulfur (combustible)	2.67
Nitrogen	1.60
Ash	9.60
Oxygen	<u>8.13</u>
Total	100.00

^aMoisture-free

Table 3

Pilot Gasification Results--Kentucky #9 Coal

	Run no.					
	212	213	214	215	216	217
Coal feed rate (MF ^a ; kg/hr)	5.3	5.7	5.7	5.7	5.6	5.7
Moisture content (wt %)	2.6	2.6	2.2	2.2	2.1	2.0
Temperature (°C)						
Reactor/middle	1,770	1,880	1,963	1,884	2,030	1,912
Reactor exit	1,190	1,216	1,231	1,314	1,451	1,245
Reactor pressure (bar)	2.0	2.0	2.0	2.0	2.0	2.0
Raw gas produced (Nm ³ dry/kg MF coal)	1.735	1.771	1.777	1.715	1.743	1.791
Analysis of raw gas (vol % dry)						
CO ₂	6.1	6.8	7.8	7.6	9.6	8.3
CO	58.5	58.9	61.0	60.4	60.5	60.4
H ₂	28.9	27.6	26.4	27.0	24.3	25.2
H ₂ S ^b	0.86	0.87	0.91	0.91	0.88	0.93
COS	0.08	0.09	0.11	0.10	0.10	0.09
CH ₄	0.22	0.08	0.03	0.05	0.03	0.03
N ₂	5.2	5.5	3.8	3.7	4.6	4.9
Trace gases in raw gas (g/Nm ³ dry)						
SO ₂ ^b	The level of trace component gases produced in a small scale test unit is not representative of the levels produced in commercial gasifiers. This data is withheld from this non-proprietary report to avoid an incorrect interpretation of the levels of trace components determined in the tests.					
NH ₃ ^b						
HCN ^b						
NO _x (ppmv)						
CS ₂ (ppmv)						

^aMoisture-free.^bThese concentrations represent the concentrations in the hot, unquenched raw gas; they are based on the sum of the quantities in the cold raw gas and the wash water. In a commercial scale gasifier, concentrations of NH₃ and HCN are usually lower.

Table 4

Hydrocarbon and Trace Metal Content of the Cold Raw Gas

	Run no.					
	212	213	214	215	216	217
Methane (ppmv)	The level of trace components in the raw gas extracted from the small scale test unit during pilot gasification tests is not comparable in practice with the level found in the raw gas produced in commercial gasifiers, particularly with respect to hydrocarbons content. In commercial gasifier operation apart from methane there are no higher hydrocarbons present in the raw gas.					
Ethane (ppmv)						
Ethylene (ppmv)						
Propylene (ppmv)						
Acetylene (ppmv)						
C ₁ -C ₃ (ppmv)	This data is withheld from this non-proprietary report to avoid an incorrect interpretation of the levels of trace components determined in the tests.					
Benzene (ppmv)						
Methylmercaptan (ppmv)						
Ethylmercaptan (ppmv)						
Isopropylmercaptan (ppmv)						
n-Propylmercaptan (ppmv)						
Be ($\mu\text{g}/\text{Nm}^3$)	3.7	5.7	-	7.1	10.4	-
Fe ($\mu\text{g}/\text{Nm}^3$)	13.1	14.5	-	18.8	22.6	18.9
Hg ($\mu\text{g}/\text{Nm}^3$)	28.6	-	-	43.5	50.6	-
Ni ($\mu\text{g}/\text{Nm}^3$)	4.5	2.96	-	6.05	6.6	4.38
Pb ($\mu\text{g}/\text{Nm}^3$)	49.7	32.2	-	12.1	90.3	58.3
Gasifier exit temperature ($^{\circ}\text{C}$)	1,190	1,216	1,231	1,314	1,451	1,245

Note: In a commercial gasifier, the hydrocarbon content of the raw gas is significantly lower than in the raw gas from the pilot gasifier; therefore, these concentrations of hydrocarbons should not be considered representative. In commercial plants, hydrocarbons heavier than methane have not been detected.

Table 5
Analysis of Wash Water

	Run no.					
	212	213	214	215	216	217
Wash water quantity (L/Nm ³ raw gas)	3.65	2.14	3.00	4.00	3.09	2.62
pH	8.5	8.6	8.5	8.3	7.9	8.0
SO ₄ ²⁻ (mg/L)	n.d.	24.6	7.5	15.4	27.0	79.4
S ₂ O ₃ ²⁻ (mg/L)	11.9	7.2	10.6	8.9	11.4	15.2
SO ₃ ²⁻ (mg/L)	0.7	5.0	11.7	7.0	4.7	2.3
NH ₄ ⁺ (mg/L)	82.4	87.8	69.4	96.2	54.1	67.0
CN ⁻ (mg/L)	122.3	95.7	50.7	70.9	32.0	33.9
SCN ⁻ (mg/L)	13.2	13.1	9.3	9.9	4.8	6.1
S ²⁻ (mg/L)	43.6	46.9	32.1	62.5	20.8	14.4
Cl ⁻ (mg/L)	50.4	53.1	-	44.1	35.2	41.0
Cl ⁻ in makeup water (mg/L)	35.8	33.3	-	28.6	27.3	32.7
ΔCl ⁻ absorbed (mg/L)	14.6	19.8	-	15.5	7.9	8.3

chemical analyses. Samples were submitted for each of the six gasification test runs.

The detailed final report from Radian is attached in its entirety. This report describes the samples analyzed, provides detailed analytical procedures, the quality assurance/quality control procedures adopted, and discusses the results in detail.

The major findings and conclusions are presented below.

Wash-Water Quality. There was little variation in the solids content of the six wash-water samples (see Table 2-4). Chloride was the predominant anionic species (25-200 mg/L). The chloride concentrations observed by Radian differ significantly from those reported by GKT (Table 5). This is (believed to be) because none of the organic compounds analyzed for were detected in any of the water samples (see Table 2-3). The level of α plus β radioactivity (~ 10 pCi/L) was below the Resource Conservation and Recovery Act (RCRA) limits (i.e., 15 pCi/L). With the exception of As and Se, all trace metal concentrations were below the limits of the National Interim Primary Drinking Water Standards (NIPDWS--Federal Register, 24 December 1975). In the Demonstration Plant, concentration of many materials would be significantly higher, because a lower water/ gas ratio would be used than in the pilot tests.

Slag and Fly Dust Samples. Small quantities of PAH compounds were detected in the slag and fly dust samples, less than 10 mg/kg in all cases. However, it was felt that the PAH values determined may be on the low side because there was evidence of irreversible sorption of the PAHs in the solid samples.

The α radioactivity level of the fly dust was about 28 pCi/g, in excess of the RCRA limit of 15 pCi/g, while that of the slag was equal to the RCRA limit.

Solid Leaching Tests. Composite samples of slag and fly dust (from all six runs) were separately subjected to water extraction by the RCRA and ASTM Procedure A methods. The filtered leachates were then analyzed for metals. The metal compositions were well within the RCRA limits.

Conclusions

This program has provided adequate analyses of gasifier feed and effluent streams for GKT coal gasification process simulation studies.

No organic priority pollutants or PAH compounds were detected in the wash water, and solid waste leachates were within RCRA metals limits.

Recommendations

Since it is anticipated that the SRC-I Demonstration Plant gasifiers will be operated on 100% coal, 100% Kerr-McGee ash concentrate (KMAC), and various blends of coal and KMAC, a similar study for KMAC gasification is recommended to determine the range of variation in effluent compositions.

References

GKT Proprietary Report No. P 1/82: Laboratory Examination and Small-Scale Test Gasification of Kentucky #9 Coal. ICRC Subcontract No. 01-11002, GKT Project No. H2-4613. (This report is retained in the ICRC Principal Project Documentation (PPD) Proprietary File ref. GKT File No. F-17-01.)

APPENDIX

RADIAN CORPORATION REPORT

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Radian Contract No. 228-004-09

ANALYSIS OF SAMPLES OBTAINED
FROM GKT GASIFICATION TEST
OF KENTUCKY COAL

Final Report

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TABLE OF CONTENTS

		<u>Page</u>
1.0	INTRODUCTION	1
2.0	RESULTS	4
2.1	Analysis of Feed Coal	6
2.2	Analysis of Wash Water and City Water	6
2.2.1	Organic Compounds	8
2.2.2	Water Quality Parameters	8
2.2.3	Metals	12
2.2.4	Radioactivity	12
2.3	Slag and Fly Dust	12
2.3.1	Organic Compounds	14
2.3.2	Ultimate, Ash and Physical Properties	14
2.3.3	Trace Metals	20
2.3.4	Radioactivity	24
2.4	Leaching Tests	24
3.0	ANALYTICAL METHODS	27
3.1	Physical Properties	27
3.2	Ash Analysis - Major Oxides	28
3.3	Ultimate Analysis	28
3.4	Organic Compounds	30
3.5	Water Quality Parameters	33
3.6	Metals	40
3.6.1	Digestive Techniques	41
3.6.2	Analytical Techniques	42
3.7	Radioactivity	44
3.8	Leaching Methods	46
4.0	QUALITY ASSURANCE/QUALITY CONTROL	47
4.1	Organic Analysis	47
4.2	Metal Analysis	51
4.3	Water Quality Analysis	57
	APPENDIX	61

LIST OF TABLES

	<u>Page</u>
2-1 Sample Inventory	5
2-2 Results of Coal Analysis	7
2-3 Radian's GC-MS Detection Limits for the Extractable Compounds for which ICRC Aqueous Samples were Analyzed	9
2-4 Wash Water and City Water Water Quality Analyses	10
2-5 Metals Data for Wash Water and City Water Samples	13
2-6 Concentration of Organic Compounds in Slag Samples	15
2-7 Concentration of Organic Compounds in Fly Dust Samples	16
2-8 Physical Properties, Ultimate and Ash Analyses of Slag Samples	17
2-9 Physical Properties, Ultimate and Ash Analyses of Fly Dust Samples	18
2-10 Trace Element and Radiochemistry Results for Slag Samples	21
2-11 Trace Element and Radiochemistry Results of Fly Dust Samples	22
2-12 Enrichment Factors for Slag and Fly Dust Samples	23
2-13 Slag RCRA and ASTM Extracts	25
2-14 Fly Dust RCRA and ASTM Extracts	26
3-1 GC-MS Conditions for ICRC Sample Extracts	32
3-2 Analytical Methods for Water Quality Analyses	34
3-3 Instrumental Conditions for ICPES and AAS Analytical Techniques	45
4-1 System Performance Requirements for Decafluorotriphenyl Phosphine (DFTPP)	49
4-2 Analysis of Quality Control Samples	50
4-3 Recovery of Labeled Compounds from Samples	52
4-4 Comparison of Results from Duplicate Analyses of Fly Dust from Run 212	53
4-5 Metals Analysis of QC Standards	54
4-6 Metals Spike Recovery	55
4-7 Metals Duplicate Analyses	56
4-8 Water Quality Duplicate Results	59
4-9 Water Quality Spike Results	60

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1.0 INTRODUCTION

International Coal Refining Company contracted with GKT to perform a pilot gasification test using Kentucky coal in December, 1981. Samples of feed coal, city water, wash water, slag and fly dust were sent to Radian for chemical analysis. These substances were analyzed for the inorganic and organic parameters specified in the ICRC request for quote 01-13121-00. The results of these analyses are presented in this report.

A flow diagram of the pilot gasifier is presented in Figure 1-1. Coal and oxygen are mixed and fed into the gasification unit. The resulting hot product gas is quenched at the bottom of the gasifier and is subjected to further gas cooling and particulate scrubbing in two wash columns: one with sieve plates and one with packing. The gas then enters a cyclone (not shown in the figure) which is located between the gas-water separator and fines filter. The gas exiting in the cyclone is filtered, metered, desulfurized, and combusted.

Slag is removed from the quench vessel at the bottom of the gasifier (identified in the figure as number 9). The makeup water to the wash column is city water. Water from the quench-water receiver and the wash columns is routed to a filter press where a solid material (fly dust) and a filtrate (wash water) are separated.

The feed coal was analyzed to determine its physical properties, ultimate and ash content, trace metals content and alpha and beta radioactivity. The results of these analyses are presented in Section 2.1

Six wash water samples (one from each pilot unit run) and one of city water were analyzed for water quality parameters, trace elements, extractable priority pollutants, polyaromatic hydrocarbons (PAH), and radioactivity. These results are summarized in Section 2.2.

LEGEND:

1. FEED HOPPER FOR COAL DUST	11. WASH COLUMN WITH SIEVE PLATES
2. FEED DOSING SCREW	12. PACKED WASH COLUMN
3. MIXING CHAMBER	13. WATER SEPARATOR
4. BURNER	14. FILTER FOR FINES
5. REACTION SPACE	15. GAS METER
6. REACTOR	16. DESULFURIZATION
7. ELECTRICAL HEATER	17. COMBUSTION CHAMBER WITH STACK
8. INSULATING MATERIAL	18., 19., 20., RECEIVER FOR WATER
9. QUENCH	FROM 10, 11 and 12
10. QUENCH-WATER RECEIVER	21. FILTER PRESS - 22. HEAT EXCHANGER - 23. PUMP

Figure 1-1. Flow Diagram of Coal Dust Gasification Test Unit

Six samples (one from each of the six pilot unit runs) each of slag and fly dust were analyzed for physical properties, ultimate and ash content, trace metals, extractable organic priority pollutants, PAH compounds and radioactivity. The results of these analyses are presented in Section 2.3.

Composites of slag were leached by the EPA procedure (Federal Register, December 28, 1978) and the ASTM procedure A. These leaching tests were also performed on a composited sample of fly dust. The limited amount of sample received precluded the inclusion of a leaching test by ASTM procedure B. The leachates obtained were analyzed for their trace element content. The results are presented in Section 2.4.

The methods used to perform these analyses are discussed in Section 3.0. EPA approved or ASTM standard methods were used when applicable throughout the program.

A rigorous quality control (QC) program was implemented for this project. The quality of the analytical results is illustrated by the QC data presented in Section 4.

Operating parameters and feed rate data were not supplied to Radian. Therefore, no attempt is made to draw conclusions about the streams based on process operating conditions.

2.0 RESULTS

Samples collected during the pilot gasification tests were received at Radian on February 3, 1982 and are listed in Table 2-1. Aqueous samples submitted for trace metal analysis had been preserved with nitric acid and shipped to Radian in polypropylene containers. Water quality parameters were determined using aliquots shipped in tightly sealed glass bottles. Samples that had been preserved by the addition of sulfuric acid were used where appropriate and unpreserved samples were analyzed for parameters adversely affected by the presence of this acid. All organic analyses were performed using the unpreserved fraction shipped in glass.

All solid samples had been packaged in polypropylene containers with the exception of small quantities of cyclone dust and filter fines which were contained in sealed plastic bags. These samples were not analyzed, and are being stored as directed by ICRC until further instructions are received.

Sections 2.1 through 2.4 present the analytical results for the following samples, respectively:

- coal - physical properties, ultimate, ash, trace elements, and radioactivity.
- wash water and city water - organics, water quality parameters, trace elements, and radioactivity;
- slag and fly dust - organics, ultimate, ash, trace metals, and radioactivity;
- slag and fly dust leaching - ASTM A and RCRA.

TABLE 2-1. SAMPLE INVENTORY

S A M P L E	TEST RUNS	PRESERVATION
Coal dust	-	none
Wash water	212-217	none
Wash water	212-217	H ₂ SO ₄
Wash water	212-217	HNO ₃
City water	-	none
City water	-	H ₂ SO ₄
City water	-	HNO ₃
Fly dust	212-217	none
Slag	212-217	none
Cyclone fly dust*	212-217	none
Filter fines*	212-217	none

*These samples were not analyzed.

Observations are made concerning the differences in analytical results from run to run. However, since process data for the runs was not made available to Radian, conclusions are not drawn concerning these differences. In addition, the analytical results are compared to limits set by applicable regulations.

2.1 Analysis of Feed Coal

The following analyses of the feed coal were performed:

- Physical Properties including specific gravity, bulk density, particle size, and moisture content.
- Ultimate Analysis to determine the amount of carbon, hydrogen, nitrogen, sulfur, oxygen, chlorine, and ash content. Ash analysis for major oxides was also performed.
- Metals Analysis to determine the elemental concentrations of major and minor constituents in the coal.
- Radiochemical Analysis for gross alpha and beta activity.

The results of these tests are provided in Table 2-2. A result expressed as less than a value indicates the amount present is below the detection limit.

2.2 Analysis of Wash Water and City Water

Six samples of wash water and one sample of city water were analyzed for:

TABLE 2-2. RESULTS OF COAL ANALYSIS

Specific Gravity	1.32	<u>Radioactivity (pCi/g)</u>	
Bulk Density (g/mL)	0.635		
Particle Size (%)		α	< 3.4
>74 micron	5	β	12 $\pm$ 4.8
48 < x < 74	8		
31 < x < 48	18		
19 < x < 31	26		
13 < x < 19	32		
9.5 < x < 13	5		
2 < x < 9.5	6		
Moisture Content (%)	1.16		
<u>Ultimate Wt. %</u>		<u>Ash Analysis Wt. %</u>	
C (%)	72.16	K ₂ O	.167
H (%)	4.97	Na ₂ O	.149
N (%)	1.44	MgO	.076
S (%)	2.94	CaO	.462
O (%)	11.04	Fe ₂ O ₃	1.86
Cl (%)	<0.01	P ₂ O ₅	.027
		SiO ₂	5.35
Ash (%)	9.27	Al ₂ O ₃	1.67
		TiO ₂	.098
		Total	9.86%
<u>Trace Elements (mg/kg dry weight basis, unless noted)</u>			
Al	0.88%	Hg	0.33
Sb	0.76	Mo	5.6
As	5.6	Ni	12
Ba	34	P	27
Be	3.8	K	0.14%
B	43	Se	1.1
Cd	0.24	Si	2.5%
Ca	0.33%	Ag	0.11
Cr	16	Na	0.11%
Co	3.4	Tl	<0.30
Cu	9.4	Ti	590
Fe	1.3%	V	42
Pb	5.8	Zn	28
Mg	460		
Mn	44	F	50

<Indicates that the parameter was not detected. The value shown is the detection limit of the method.

- extractable organic priority pollutants,
- PAH compounds,
- water quality parameters,
- trace elements, and
- radioactivity.

The results obtained for these analyses are presented in the following subsections.

2.2.1 Organic Compounds

None of the organic compounds which were analyzed for were detected in any of the aqueous samples. Table 2-3 lists the organic compounds which were analyzed for (as requested by ICRC) by GC-MS and the detection limit for the ICRC water samples.

Section 4.0 contains results from the quality control program. One water sample was spiked with labeled compounds at 50 µg/L. The average recovery for this spike was 64 percent. The results are presented in Table 4-3.

Appendix A contains GC-MS chromatograms of all sample extracts. The internal standard, d₁₀-anthracene, is indicated by showing an extracted ion current profile (EICP) of m/e 188. This peak represents a concentration of 20 µg/L in the samples. Other organic species are present in the samples, however, these chromatograms indicate that most species are below this concentration.

2.2.2 Water Quality Parameters

The results for the water quality analyses are listed in Table 2-4. The analytical methods used are discussed in Section 3.5.

The results for the solids determinations showed only slight variation between samples. The alkalinity and pH data showed greater variability.

TABLE 2-3. RADIAN's GC-MS DETECTION LIMITS FOR THE EXTRACTABLE COMPOUNDS FOR WHICH ICRC AQUEOUS SAMPLES WERE ANALYZED*

POLLUTANT	Detection Limit, ug/L	POLLUTANT	Detection Limit, ug/L
BASE/NEUTRAL COMPOUNDS		PESTICIDES	
Acenaphthene	5	1P Aldrin	10
Acenaphthylene	2	2P α -BHC	5
Anthracene	2	3P β -BHC	10
Benzidine	10	4P γ -BHC	10
Benzo(a)anthracene	5	5P δ -BHC	10
Benzo(a)pyrene	5	6P Chlordane	20
3,4-Benzofluoranthene	5	7P 4,4'-DDT	5
Benzo(g,h,i)perylene	5	8P 4,4'-DDE	5
Benzo(k)fluoranthene	5	9P 4,4'-DDD	5
bis(2-Chloroethoxy)methane	5	10P Dieldrin	5
bis(2-Chloroethyl)ether	5	11P α -Endosulfan	10
bis(2-Chloroisopropyl)ether	10	12P β -Endosulfan	10
bis(2-Ethylhexyl)phthalate	2	13P Endosulfan	5
4-Bromophenyl phenyl ether	10	14P Endrin	10
Butyl benzyl phthalate	5	15P Endrin aldehyde	20
2-Chloronaphthalene	5	16P Heptachlor	10
4-Chlorophenyl phenyl ether	5	17P Heptachlor epoxide	10
Chrysene	5	18P PCB-1242	20
Dibenzo(a,h)anthracene	5	19P PCB-1264	20
1,2-Dichlorobenzene	5	20P PCB-1221	20
1,3-Dichlorobenzene	5	21P PCB-1232	20
1,4-Dichlorobenzene	5	22P PCB-1248	20
3,3'-Dichlorobenzidine	10	23P PCB-1260	20
Diethyl phthalate	2	24P PCB-1016	20
Dimethyl phthalate	2	25P Toxaphene	20
Di-n-butyl phthalate	2		
2,4-Dinitrotoluene	10	ADDITIONAL COMPOUNDS	
2,6-Dinitrotoluene	10	Biphenyl	2
Di-n-octyl phthalate	2	5-Methylchrysene	2
1,2-Diphenyl hydrazine	10	1-Methylnaphthalene	2
Fluoranthene	2	2-Methylnaphthalene	2
Fluorene	2	Dibenzothiophene	2
Hexachlorobenzene	10	Triphenylene	2
Hexachlorobutadiene	10	Benzo(e)pyrene	2
Hexachlorocyclopentadiene	10	Perylene	2
Hexachloroethane	10	Dibenzo(a,c)anthracene	2
Indeno(1,2,3-c,d)pyrene	5	2-Methylfluoranthene	2
Isophorone	10	Benzo(c)fluoranthene	2
Naphthalene	2	Benzo(j)fluoranthene	2
Nitrobenzene	5		
N-Nitrosodimethylamine	10		
N-Nitrosodi-n-propylamine	10		
N-Nitrosodiphenylamine	5		
Phenanthrene	2		
Pyrene	2		
1,2,4-Trichlorobenzene	5		
ACID COMPOUNDS			
2-Chlorophenol	5		
2,4-Dichlorophenol	5		
2,4-Dimethylphenol	5		
4,6-Dinitro-o-cresol	10		
2,4-Dinitrophenol	10		
3-Nitrophenol	10		
4-Nitrophenol	10		
p-Chloro-m-cresol	10		
Pentachlorophenol	10		
Phenol	5		
2,4,6-Trichlorophenol	10		

*No organic species were detected in the aqueous samples.

TABLE 2-4. WASH WATER AND CITY WATER
WATER QUALITY ANALYSES¹

P A R A M E T E R	WASH WATER						City Water
	RUN	212	213	214	215	216	217
settleable solids ²		<1	<1	<1	<1	<1	<1
floatable solids		<1	<1	<1	<1	<1	<1
total solids		675	601	538	737	559	750
total suspended solids		3	4	4	3	2	3
volatile suspended solids		2	2	2	2	1	2
total dissolved solids		592	553	505	645	532	686
conductivity ³		1170	1120	956	1200	859	1070
pH ⁴		8.27	8.33	8.31	8.22	7.92	7.99
acidity (as CaCO ₃)		<1	<1	<1	<1	<1	<1
alkalinity (as CaCO ₃)		436	464	355	421	241	206
bicarbonate (as CaCO ₃)		436	423	336	421	241	206
carbonate (as CaCO ₃)		<1	41	19	<1	<1	<1
bromide		<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
chloride		25	40	136	38	196	113
fluoride		3.3	2.4	1.6	6.2	<0.1	1.6
nitrate (as N)		8.2	6.8	6.8	31	22	44
nitrite (as N)		0.01	0.01	0.33	0.02	0.14	2.74
phosphate (as P)		13.8	14.3	15.1	14.8	5.9	4.4
total organic carbon		43	36	21	23	15	8
total inorganic carbon		ND ⁵	ND ⁵	ND ⁵	66	46	40
Kjeldahl nitrogen (as N)		87	69	69	91	48	64
formate		6	7	10	3	2	2
oil and grease		29	3	6	20	8	2
phenolics		<0.005	<0.005	<0.005	<0.005	<0.005	<0.005

¹All results in mg/L, unless otherwise indicated.

²Units are mL/L.

³Units are μ mho/cm.

⁴Units are conventional pH units.

⁵Not determined, insufficient sample.

High pH and alkalinities were found for runs 212 to 215 with lower pH and alkalinities being found for runs 216 and 217.

The amounts of chloride, fluoride, nitrate, nitrite, and phosphate showed considerable variability from run to run. Chloride was the predominant anion present in all samples. For runs 212 to 214, phosphate was present in concentrations second only to chloride. Nitrate was present in concentrations second only to chloride for runs 215 to 217. The absence of fluoride in wash water from run 216 and the significant increase in the amount of nitrite in the wash water from run 217 are also noteworthy.

The organic loading in the wash waters was relatively low. Less than detection-limit quantities of phenolics were found in all six wash waters.

The quality of the wash waters was compared with the National Interim Primary Drinking Water Standards (NIPDWS-Federal Register, 24 December 1975). The comparisons are made for information purposes only. The wash water would not necessarily have to meet these types of standards for discharge. For the water quality parameters:

- fluoride exceeds the NIPDWS limit (2 mg/L at annual average temperature of 60°F) for runs 212, 213, and 215;
- nitrate exceeds the NIPDWS limit (10 mg/L as nitrogen) for runs 215, 216, and 217, and
- total dissolved solids slightly exceed the recommended NSDWR limit (500 mg/L) for all runs.

The levels of chloride, and the pH of all wash waters were acceptable with respect to the recommended NSDWR limits. Other parameters determined are not regulated by NIPDWS or NSDWR.

2.2.3 Metals

The results obtained from analysis of wash water and city water samples are listed in Table 2-5. With the exception of arsenic and selenium content, the samples have concentrations of trace metals that are less than the acceptable limits of interim primary or proposed secondary drinking water standards, particularly in the realm of heavy metal contaminants. The arsenic values, which range from 0.029 to 0.053 mg/L, verge on the drinking water acceptable limit of 0.050 mg/L with the sample obtained from run 214 actually exceeding the acceptable limit. Selenium values from 0.020 to 0.083 mg/L exceed drinking water limits of 0.01 mg/L in all cases. The drinking water limits are cited as a point of reference, and does not imply an acceptable limit for the samples analyzed.

2.2.4 Radioactivity

The results of the radioactivity data for the composite sample of wash water are presented in Table 2-5. These results are within acceptable radiation limits of 15 PCi per liter of water or gram of solid waste prescribed by proposed limits under RCRA regulations, Federal Register, May 19, 1980.

2.3 Slag and Fly Dust

Six samples each of slag and fly dust were analyzed for the following parameters:

- extractable organic priority pollutants,
- PAH compounds,
- physical properties,
- ultimate analysis,
- ash analysis,
- trace elements, and
- radioactivity.

The results obtained for these analyses are presented in the following subsections.

TABLE 2-5. METALS DATA FOR WASH WATER AND CITY WATER SAMPLES

ELEMENT	Concentration, mg / L						
	City Water	Test 212	Test 213	Test 214	Test 215	Test 216	Test 217
Al	<0.050	0.20*	0.29	0.34	0.18*	0.33	0.57
Sb	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
As	<0.003	0.049	0.037	0.053	0.029	0.045	0.040
Ba	0.044	0.12	0.13	0.11	0.13	0.13	0.18
Be	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
B	0.14	3.9	3.7	3.7	3.8	3.1	3.7
Cd	<0.0005	0.0008*	<0.0005	<0.0005	0.0006*	<0.0005	<0.0005
Ca	34	130	120	100	130	88	120
Cr	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Co	<0.006	<0.006	<0.006	<0.006	<0.006	<0.006	<0.006
Cu	<0.001	0.009	<0.001	<0.001	<0.001	<0.001	<0.001
Fe	<0.008	5.3	4.7	1.7	3.9	0.57	0.85
Pb	<0.002	0.004*	0.005*	0.006*	<0.002	<0.002	0.003*
Mg	6.7	7.4	6.8	6.8	7.2	6.6	7.3
Mn	0.014	0.16	0.12	0.13	0.16	0.22	0.32
Hg	<0.0002	0.0003*	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002
Mo	0.005*	0.003*	<0.002	0.010*	0.004*	0.006*	0.011
Ni	<0.003	0.16	0.12	0.007*	0.075	0.091	0.073
P	<0.18	<0.18	<0.18	<0.18	<0.18	<0.18	<0.18
K	3.5	5.8	6.1	5.6	6.5	5.3	6.2
Se	0.004*	0.090	0.083	0.027	0.080	0.024	0.020
Si	<0.016	<0.016	<0.016	<0.016	<0.016	1.2	<0.016
Ag	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Na	22	29	27	27	26	21	25
Tl	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
Ti	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
V	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
Zn	<0.003	0.044	0.047	0.018	0.005*	<0.003	0.008*

Radioactivity (pCi/l for composite of six samples)

Gross: α <1.3

β 7.9 $\pm$ 2.3

*Indicates results less than five times the detection limit.

<Indicates that the parameter was not detected. The value shown is the detection limit of the method.

2.3.1 Organic Compounds

Table 2-3 lists the organic species for which analyses were performed. The concentrations of organic species in the slag and fly dust samples are summarized in Tables 2-6 and 2-7, respectively. These tables list the organic species found in the samples. The other organic species listed in Table 2-3 were not detected in the samples; the detection limit ($\mu\text{g/kg}$) is virtually identical to the water detection limit listed in Table 2-3.

As summarized in Tables 2-6 and 2-7, these samples contained low levels of polynuclear aromatic hydrocarbons (PAHs), dibenzothiophene and phenol. A sample spiked with labeled PAHs had extremely poor recovery as summarized in Table 4-3. This result indicates that the sample matrix irreversibly sorbs PAHs and that the concentrations listed in Tables 2-6 and 2-7 may be low. Appendix A contains GC-MS chromatograms of the sample extracts. Naphthalene is the predominant compound in the sample extracts.

The organic compound eluting at 24-25 minutes on the chromatograms^{*} is di-n-octyl phthalate, a common plasticizer. This compound probably originates from the plastic sample container and was, therefore, not quantified, although it is listed as an extractable priority pollutant.

2.3.2 Ultimate, Ash and Physical Properties

The physical properties, ultimate and ash data for the slag samples is presented in Table 2-8. The information for fly dust samples is provided in Table 2-9. The specific gravity of the dust remained constant throughout the study, but the specific gravity of the slag varied significantly. The same trend is observed for the bulk density of the samples. The variation in the slag is apparently influenced by the small amount of sample available and the large variations in particle size in the samples submitted for analysis. The specific gravity of each slag sample (obtained using ground

^{*}Chromatograms are presented in the Appendix.

TABLE 2-6. CONCENTRATION OF ORGANIC COMPOUNDS IN SLAG SAMPLES*

C O M P O U N D	Molecular Weight	C O N C E N T R A T I O N, mg/kg					
		T E S T R U N					
		212	213	214	215	216	217**
Naphthalene	128	0.34	0.29	0.20	0.35	3.5	1.6
1-methylnaphthalene	142	0.22	0.19	0.14	0.18	1.9	0.89
2-methylnaphthalene	142	0.067	0.046	0.044	0.068	0.83	0.31
Biphenyl	154	0.097	0.089	0.081	0.12	0.69	0.27
Acenaphthene	154	0.022	0.016	0.028	0.037	0.32	0.17
Acenaphthylene	152	0.003	0.002	0.007	ND	0.041	ND
Fluorene	166	0.030	0.025	0.030	0.025	0.011	ND
Dibenzothiophene	184	0.002	ND	0.003	ND	0.002	ND
Phenanthrene	178	0.099	0.14	0.079	0.064	0.099	0.18
Fluoranthene	202	0.026	0.050	0.019	0.005	0.020	ND
Pyrene	202	0.053	0.061	0.046	0.009	0.037	ND
RADIAN SAMPLE NUMBER		4559	4564	4569	4575	4579	4585
GC-MS FILE NUMBER		21313	21312	21311	21314	21315	21316
DATE INJECTED		3-30-82	3-30-82	3-30-82	3-31-82	3-31-82	3-31-82

*Only those compounds detected in one or more samples are listed. The complete list of compounds determined in this study is presented in Table 2-3.

**Sample size only 0.63 g.

ND=Not Detected

TABLE 2-7. CONCENTRATION OF ORGANIC COMPOUNDS IN FLY DUST SAMPLES*

C O M P O U N D	Molecular Weight	C O N C E N T R A T I O N , mg/kg					
		T E S T R U N					
		212	213	214	215	216	217
Naphthalene	128	8.1	4.1	4.1	3.2	4.4	4.8
1-methylnaphthalene	142	0.36	0.91	0.82	0.25	1.1	0.82
2-methylnaphthalene	142	0.26	0.59	0.64	0.16	0.77	0.45
Biphenyl	154	0.15	0.35	0.26	0.082	0.35	0.19
Acenaphthene	154	0.13	0.45	0.29	0.086	0.27	0.23
Acenaphthylene	152	1.9	3.4	2.2	1.0	2.3	2.2
Fluorene	166	0.095	0.29	0.27	0.11	0.30	0.22
Dibenzothiophene	184	0.064	0.72	0.059	0.024	0.077	0.050
Phenanthrene	178	0.23	0.18	0.12	0.059	0.16	0.010
Anthracene	178	ND	0.015	0.010	ND	0.013	0.004
Fluoranthene	202	0.12	0.073	0.030	0.020	0.045	0.038
Pyrene	202	0.82	0.27	0.77	0.055	0.11	0.075
Chrysene	228	ND	ND	**	**	**	**
Benz(a)anthracene	228	ND	ND	0.008	0.007	0.009	0.007
Triphenylene	228	ND	ND	**	**	**	**
Phenol	94	0.14	0.36	0.59	0.17	0.73	0.61
RADIAN SAMPLE NUMBER		4560	4565	4570	4574	4580	4584
GC-MS FILE NUMBER		21306	21301	21302	21308	21304	21305
DATE INJECTED		3-30-82	3-29-82	3-29-82	3-30-82	3-30-82	3-30-82

*Only those compounds detected in one or more samples are listed. The complete list of compounds determined in this study is presented in Table 2-3.

**Trace level of 228 isomer reported as benz(a)anthracene

ND=Not Detected

TABLE 2-8. PHYSICAL PROPERTIES, ULTIMATE
AND ASH ANALYSES OF SLAG SAMPLES

Physical Properties	Test Run					
	212	213	214	215	216	217
Specific Gravity	1.77	1.78	1.68	2.18	2.39	2.15
Bulk Density (g/mL)	1.50	1.29	1.62	1.08	1.27	1.43
Particle Size > 3/4"	0	23.1%	0	66.3%	63.1%	0
1/4" < x < 3/4"	36.3%	64.0%	29.9%	32.3%	20%	66.8%
200 mesh < x < 1/4"	63.7%	12.9%	70.1%	1.4%	16.8%	33.2%
< 200 mesh	0	0	0	0	0.1%	0
Moisture Content (%)	0.020	0.100	0.090	0.010	0.005	0.010
<u>Ultimate</u>						
<u>Analysis, Wt %</u>						
C	0.64	0.48	0.44	0.08	1.00	0.65
H	0.11	0.07	0.11	0.05	0.12	0.14
N	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
O	0.13	0.10	0.05	0.18	0.20	0.08
S	0.44	0.87	0.23	0.51	0.08	0.19
Cl	0.04	< 0.02	< 0.02	< 0.02	< 0.02	0.81
Ash	100.1	100.3	99.4	99.5	99.3	100.1
<u>Ash</u>						
<u>Analysis, Wt %</u>						
K ₂ O	0.828	0.948	1.03	0.864	0.264	0.720
Na ₂ O	0.770	0.945	1.08	1.27	0.716	1.05
MgO	0.481	0.548	0.598	0.482	0.199	0.398
CaO	3.92	4.20	4.90	2.94	2.24	3.22
Fe ₂ O ₃	13.7	17.2	20.0	10.0	6.40	12.9
P ₂ O ₅	0.053	0.053	0.057	0.032	0.021	0.039
SiO ₂	30.0	38.0	30.0	26.0	13.0	26.0
Al ₂ O ₃	23.0	34.0	34.0	53.0	70.0	51.0
TiO ₂	0.520	0.585	0.618	0.468	0.234	0.451
Total	73.3	96.5	92.3	95.1	93.1	95.8

TABLE 2-9. PHYSICAL PROPERTIES, ULTIMATE AND
ASH ANALYSES OF FLY DUST SAMPLES

Physical Properties	Test Run					
	212	213	214	215	216	217
Specific Gravity	1.85	1.90	1.95	1.94	1.95	1.94
Bulk Density (g/mL)	0.34	0.35	0.33	0.37	0.37	0.34
Particle Size (%) > 74 micron	29	28	26	23	24	26
45 < x < 74 micron	19	18	12	20	21	13
38 < x < 45 micron	1	2	4	0	0	2
< 38 micron	51	52	58	57	55	59
Moisture Content (%)	0.44	0.47	0.42	0.46	0.48	0.40
Ultimate						
Analysis, Wt %						
C	65.1	63.0	55.8	59.7	53.6	54.0
H	0.32	0.36	0.12	0.24	0.28	0.14
N	0.72	0.54	0.47	0.60	0.42	0.52
O	2.05	2.03	1.98	2.40	2.17	2.23
S	2.33	2.39	2.37	2.78	2.51	2.76
Cl	< 0.02	0.61	< 0.02	0.13	< 0.02	0.48
Ash	31.97	32.4	39.2	35.9	42.3	41.8
Ash						
Analysis, Wt %						
K ₂ O	0.588	0.588	0.804	0.648	0.816	0.804
Na ₂ O	0.365	0.392	0.567	0.486	0.635	0.678
MgO	0.267	0.267	0.351	0.292	0.351	0.351
CaO	0.896	0.910	1.29	1.06	1.25	1.18
Fe ₂ O ₃	5.72	5.58	7.15	6.15	7.29	7.15
P ₂ O ₅	0.034	0.030	0.037	0.032	0.044	0.050
SiO ₂	18.4	17.3	21.4	19.0	23.5	23.5
Al ₂ O ₃	6.43	6.05	7.94	6.62	8.12	7.75
TiO ₂	0.384	0.367	0.484	0.418	0.501	0.484
Total	33.1	31.5	40.0	34.7	42.5	41.9

material) provides a much better indication of this property of the slag than does the bulk density. The ash analysis of slag obtained from run 212 is significantly low. However, this value is an average of values obtained by duplicate digestion and quadruplicate analysis.

The ultimate analyses of the slag and fly dust indicate the following:

- The ash content of the fly dust ranges from 31.97 - 42.27 percent in the six runs. The ash contents of the slag are greater than 99 percent in all runs.
- The carbon content of the fly dust ranges from 53.6 - 65.1 percent in the six runs. The carbon content of the slag is ≤ 1 percent in all runs.
- The sulfur content of the fly dust ranges from 2.33 - 2.78 percent in the six runs. The slag sulfur content ranges from 0.08 - 0.8 percent.
- The chlorine content of the samples varied considerably, from 0-0.81 percent for slag and from 0-0.61 percent for the fly dust.
- The nitrogen contents of the samples were fairly consistent, ranging from 0.42 - 0.72 percent for the fly dust and <0.02 percent for all of the slag samples.

The fly dust ash analysis is reasonably consistent from run to run. The ratio of major compounds (Fe_2O_3 , SiO_2 , and Al_2O_3) is consistent from run to run and with the coal ash analysis (Table 2-2).

The slag ash analyses show high variation from run to run, particularly for Fe_2O_3 , SiO_2 , and Al_2O_3 . Many of the results indicate that there may have been difficulty in obtaining representative samples.

The sum of the major oxide analyses for the slag and fly dust agree reasonably well with the ash content obtained from the ultimate analysis.

2.3.3 Trace Metals

The results for the metals analysis of slag are presented in Table 2-10 and the values obtained for the analysis of fly dust are presented in Table 2-11.

In order to evaluate the enrichment or depletion of trace elements in the fly dust and slag compared to the coal, Table 2-12 was prepared. If no enrichment or depletion occurs, the trace element content is related to the ash content by the following:

$$\frac{\text{Trace element concentration (slag or fly dust)}}{\text{Trace element concentration (coal)}} = \frac{\text{ash content (slag or fly dust)}}{\text{ash content (coal)}}$$

If the ratio of a trace element in slag or fly dust to coal is less than the ratio of the ash contents, then the trace element has been depleted with respect to the coal. Should this ratio be greater than that of the ash content of slag or fly dust to the ash content of coal, enrichment of the slag or fly dust is indicated. Based upon the ratios calculated for each element in Table 2-12 the following several trends are observed.

	<u>Slag</u>	<u>Fly Dust</u>
Elements depleted relative to coal	As, Sb, B, Cd, Cu, F, Pb, Hg, Mg, Mo, Ni, K, Se, Si, Ag, Tl, Ti, and Zn	F, Hg, and Se
Elements enriched relative to coal	Al	B, Cd, Cu, and Ag

TABLE 2-10. TRACE ELEMENT AND RADIOCHEMISTRY RESULTS FOR SLAG SAMPLES

ELEMENT	Concentration, mg/kg [†]					
	Test 212	Test 213	Test 214	Test 215	Test 216	Test 217
Al	12%	18%	18%	28%	37%	27%
Sb	<0.75	<0.75	<0.75	<0.75	<0.75	<0.75
As	33	23	34	11	11	27
Ba	270	250	240	250	88	160
Be	35	32	33	17	12	23
B	180	28	60	30	8.0	33
Cd	<0.13	0.30*	<0.13	<0.13	<0.13	<0.13
Ca	2.8%	3.0%	3.5%	2.1%	1.6%	2.3%
Cr	220	210	120	14	37	72
Co	35	39	39	27	7.5	30
Cu	26	19	12	33	8.0	16
F	42	44	26	38	42	28
Fe	9.6%	12%	14%	7.0%	4.5%	9.0%
Pb	1.5*	<0.75	<0.75	<0.75	1.2*	<0.75
Hg	2900	3300	3600	2900	1200	2400
Mn	470	420	400	270	170	280
Hg	0.24	0.082	0.039	0.051	0.066	0.031
Mo	13	8.1	10	8.7	1.3	7.4
Ni	32	67	52	54	10	69
P	230	230	250	140	93	170
K	6900	7900	8600	7200	2200	6000
Se	<0.75	<0.75	<0.75	<0.75	<0.75	<0.75
Si	14%	13%	14%	12%	5.9%	12%
Ag	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25
Na	5700	7000	8000	9400	5300	7800
Tl	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
Ti	3100	3500	3700	2800	1400	2700
V	310	270	290	150	75	180
Zn	30	30	18	4.8	3.8	90

Radiochemistry (composite sample pCi/g)

Gross: 2 13 ± 3.3
3 40 ± 5.7

[†]Results in mg/kg dryweight, unless noted.

*Indicates that the parameter was not detected. The value shown is the detection limit of the method.

TABLE 2-11. TRACE ELEMENT AND RADIOCHEMISTRY RESULTS
OF FLY DUST SAMPLES

	Concentration, - mg / kg†					
	Test 212	Test 213	Test 214	Test 215	Test 216	Test 217
Al	3.4%	3.2%	4.2%	3.5%	4.3%	4.1%
Sb	2.8	2.0	3.8	3.4	4.9	3.7
As	19	20	29	26	36	29
Ba	97	100	130	120	140	50
Be	12	12	15	14	16	16
B	330	380	510	430	500	490
Cd	1.8	1.8	3.6	3.1	4.4	1.3
Ca	6400	6500	9200	7600	8900	8400
Cr	91	72	91	78	83	100
Co	13	15	19	16	19	20
Cu	87	49	66	63	93	120
F	92	72	60	70	60	90
Fe	4.0%	3.9%	5.0%	4.3%	5.1%	5.0%
Pb	20	25	34	26	46	46
Mg	1600	1600	2100	1750	2100	2100
Mn	120	120	160	130	150	150
Hg	0.016	0.021	0.010	0.019	0.018	0.006
Mo	20	23	31	27	34	32
Ni	50	54	74	60	78	82
P	150	130	160	140	190	220
K	4900	4900	6700	5400	6800	6700
Se	1.7	1.6	2.4	2.3	2.4	3.7
Si	8.6%	8.1%	10%	8.9%	11%	11%
Ag	0.82	0.62	0.66	0.88	1.7	1.0
Na	2700	2900	4200	3600	4700	5020
Tl	<0.50	1.8	1.9	2.6	4.1	3.8
Ti	2300	2200	2900	2500	3000	2900
V	140	140	180	160	180	190
Zn	120	140	160	160	220	290

Radiochemistry (composite sample pCi/g)

Gross: α 28 $\pm$ 5.5
 β 43 $\pm$ 6.0

†Results in mg/kg dryweight unless noted.

<Indicates that the parameter was not detected. The value shown is the detection limit of the method.

TABLE 2-12. ENRICHMENT FACTORS FOR SLAG AND FLY DUST SAMPLES

ELEMENT	TEST RUN											
	212		213		214		215		216		217	
	SLAG*	FLYDUST**	SLAG	FLYDUST	SLAG	FLYDUST	SLAG	FLYDUST	SLAG	FLYDUST	SLAG	FLYDUST
Al	13.6	3.9	20.4	3.6	20.4	4.8	31.8	4.0	42.0	4.9	30.7	4.7
Sb	1	3.7	1	2.6	1	5.0	1	4.5	1	6.4	1	4.9
As	5.9	3.4	4.1	3.6	6.1	5.2	1.96	4.5	1.96	6.4	4.8	5.1
Ba	7.9	2.9	7.4	2.9	7.1	3.8	7.4	3.5	2.6	4.1	4.7	1.5
Be	9.2	3.2	8.4	3.2	8.7	3.9	4.5	3.7	3.16	4.2	6.1	4.2
B	4.2	7.7	0.65	8.8	1.4	11	0.70	10	0.19	12	0.77	11
Cd	< 0.54	7.5	<1.2	7.5	<0.54	15	<0.54	13	<0.54	18	<0.54	5.4
Ca	8.5	1.9	9.1	1.97	10.6	2.8	6.4	2.3	4.8	2.7	7.0	2.5
Cr	13.8	5.7	13.1	4.5	7.5	5.7	0.88	4.9	2.3	5.2	4.5	6.3
Co	10.3	3.8	11.5	4.4	11.5	5.6	7.9	4.7	2.2	5.6	8.8	5.9
Cu	2.8	9.3	2.02	5.2	1.28	7.02	3.5	6.7	0.85	9.9	1.70	12
F	0.84	1.8	0.88	1.4	0.52	1.2	0.76	1.4	0.84	1.2	0.56	1.8
Fe	7.4	3.1	9.2	3.0	10.8	3.8	5.4	3.3	3.5	3.9	6.9	3.8
Pb	0.26	3.4	<1.3	4.3	<1.3	5.9	<1.3	4.5	.21	7.9	<1.3	7.9
Mg	6.3	3.5	7.17	3.5	7.83	4.6	6.30	3.8	2.60	4.6	5.21	4.6
Mn	10.7	2.7	9.5	2.7	9.09	3.6	6.14	3.0	3.86	3.4	6.37	3.4
Hg	0.72	.048	0.25	.063	0.12	.030	0.15	.057	0.20	.054	0.093	.018
Mo	2.32	3.6	1.45	4.1	1.79	5.5	1.56	4.8	0.23	6.1	1.32	5.7
Ni	2.67	4.2	5.58	4.5	4.33	6.2	4.5	5.0	0.83	6.5	5.75	6.8
P	8.5	5.6	8.5	4.8	9.3	5.9	5.18	5.2	3.44	7.0	6.30	8.1
K	4.93	3.5	5.64	3.5	6.14	4.8	5.14	3.9	1.57	4.9	4.29	4.8
Se	<.68	1.5	<.68	1.4	<.68	2.2	<.68	2.1	<.68	2.2	<.68	3.4
Si	5.6	3.4	5.2	3.2	5.6	4.0	4.8	3.6	5.2	4.4	4.8	4.4
Ag	<2.3	7.5	<2.3	5.6	<2.3	6.0	<2.3	8.0	<2.3	15	<2.3	9.1
Na	5.2	2.5	6.4	2.6	7.27	3.8	8.54	3.3	4.81	4.3	7.1	4.6
Tl	<1.6	<1.7	<1.6	<6	<1.6	<6	<1.6	<9	<1.6	<14	<1.6	<13
Ti	5.25	3.9	5.93	3.7	6.27	4.9	4.74	4.2	2.37	5.1	4.58	4.9
V	7.4	3.3	6.4	3.3	6.9	4.3	3.6	3.8	3.8	4.3	4.30	4.5
Zn	1.07	4.3	1.07	5.0	0.64	5.7	0.17	5.7	0.14	7.9	3.2	10
Ash***	10.8	3.4	10.8	3.5	10.7	4.2	10.7	3.9	10.7	4.6	10.8	4.5

*Ratio of slag elemental concentration to coal elemental concentration

**Ratio of fly dust elemental concentration to coal elemental concentration

***Ratio of slag or fly dust ash content (by ultimate) to the coal ash content (by ultimate)

<Indicates that the parameter was not detected. The value shown is the detection limit of the method.

The above observations are not absolute, but do give an indication of the elements that may be found in the product gas or wash water stream.

2.3.4 Radioactivity

The radiochemistry (gross alpha and beta) data for the slag and fly dust composites is provided in Tables 2-10 and 2-11, respectively. The gross alpha value for the fly dust (28 ± 5.5 pCi/g) exceeds the proposed RCRA limit of 15 pCi/g and the slag value is approximately equal to the proposed limit. There is currently no regulation for gross Beta.

2.4 Leaching Tests

The results of the EPA and ASTM leachates of the slag and fly dust are presented in Tables 2-13 and 2-14, respectively. In all cases, the levels of elements found are within acceptable limits for the regulation.

The RCRA limit for each of the eight regulated metals is given below, along with the leachate concentrations for each metal:

	<u>RCRA Limit</u> <u>mg/L</u>	<u>Slag</u> <u>mg/L</u>	<u>Fly Dust</u> <u>mg/L</u>
As	5	<0.003	<0.003
Ba	100	0.058	0.15
Cd	2	0.006	0.077
Cr	5	0.009	0.007
Pb	5	<0.002	<0.002
Hg	0.2	<0.0002	<0.0002
Se	1	<0.003	0.004
Ag	5	<0.001	<0.001

The concentrations of all eight metals are less than 4 percent of the RCRA limits.

TABLE 2-13. SLAG RCRA AND ASTM EXTRACTS

ELEMENT	CONCENTRATION, mg/L	
	RCRA	ASTM A
Al	<0.050	0.10
Sb	<0.003	<0.003
As	<0.003	<0.003
Ba	0.058	0.041
Be	0.001*	<0.0005
B	0.054	0.030*
Cd	0.006	0.0008*
Ca	15	17
Cr	0.009	<0.001
Co	0.008*	<0.006
Cu	0.018	<0.001
Fe	3.4	0.058
Pb	<0.002	<0.002
Mg	0.59	0.71
Mn	0.080	0.021
Hg	<0.0002	<0.0002
Mo	0.008*	0.009*
Ni	0.019	<0.003
P	0.18	<0.18
K	1.1	0.56
Se	<0.003	<0.003
Si	0.40	1.1
Ag	<0.001	<0.001
Na	0.38	1.2
Tl	<0.003	<0.003
Ti	0.029	<0.005
V	0.024	<0.003
Zn	0.044	<0.003

*Indicates values less than five times the detection limit.

<Indicates that the parameter was not detected. The value shown is the detection limit of the method.

TABLE 2-14. FLY DUST RCRA AND ASTM EXTRACTS

ELEMENT	CONCENTRATION, mg/L	
	RCRA	ASTM A
Al	<0.050	<0.050
Sb	<0.003	0.005*
As	<0.003	<0.003
Ba	0.15	0.099
Be	0.001*	<0.0005
B	0.61	0.10
Cd	0.077	0.004
Ca	120	340
Cr	0.007	<0.001
Co	0.050	<0.006
Cu	0.029	<0.001
Fe	<0.008	0.016*
Pb	<0.002	<0.002
Mg	5.0	14
Mn	0.71	0.48
Hg	<0.0002	<0.0002
Mo	0.006*	0.006*
Ni	0.30	0.063
P	<0.18	<0.18
K	9.4	16
Se	0.004*	0.015
Si	5.5	4.5
Ag	<0.001	<0.001
Na	7.9	19
Tl	<0.003	<0.003
Ti	0.019*	<0.005
V	0.019	<0.003
Zn	2.0	0.013

*Indicates values less than five times the detection limit.

<Indicates that the parameter was not detected. The value shown is the detection limit of the method.

3.0 ANALYTICAL METHODS

The methods used for analysis of samples submitted to Radian are described in the following subsections. All procedures are in accordance with the methods proposed to ICRC in section 3 of Radian's proposal number 228-484-1-456.

3.1 Physical Properties

The physical properties of the feed coal, fly dust, and slag were determined by the following methods.

Specific Gravity

Specific gravity of the coal, fly dust, and slag is determined by adding a known weight of solid into a tared water-filled pycnometer. After weighing the pycnometer, the specific gravity is calculated using the sample dry weight, the pycnometer weight with water only, and the pycnometer weight with water and solids.

Bulk Density

The bulk density is determined by the measurement of the weight of a known volume of sample. Dry sample is placed in a tared container of known volume and the weight of solid per volume calculated.

Particle Size

Slag and particle size analysis is performed using a series of sieves ranging from 75 mm to 4.75 mm. The particle sizing of the coal and flydust is performed by placing the sample in a sedimentation cylinder in a constant temperature bath. A hydrometer is used to determine settling rates, which by use of a nomograph denotes particle size.

Moisture Content

Moisture content is obtained by drying solid samples to constant weight in an oven set at 103-105°C. This procedure is in accordance with ASTM D2216.

3.2 Ash Analysis - Major Oxides

Determination of major species in the ash analysis was performed on the lithium borate fusion described in section 3.6.1. The sample is analyzed by Inductively Coupled Plasma Emission Spectroscopy (ICPES) for each of the major metal constituents present. The oxide content is obtained by a calculation to convert the concentration of the metal present to the concentration of the oxide (i.e. Ca + CaO).

3.3 Ultimate Analysis

Carbon and Hydrogen are determined by combusting a sample in pure oxygen at 1050°C. The carbon is converted to CO₂ and the hydrogen to H₂O. The gases pass through a water trap and nitrogen oxide scrubber into a carbon dioxide coulometer where CO₂ in the gas stream is quantitatively determined. Trapped water is quantitatively converted to CO₂ which is determined by a second coulometer.

Oxygen

A weighed sample is pyrolyzed in nitrogen. The pyrolysis products are passed over carbon at 1100°C so that all oxygen is converted to carbon monoxide. After removal of all potential interferents, the CO is oxidized to CO₂ and passed into a CO₂ coulometer for quantification.

Sulfur

A Fisher Model 470 sulfur analyzer is used for sulfur analysis. The sample is combusted in oxygen at 1300°C and the SO₂ produced is titrated with iodine.

Nitrogen

The sample is combusted in pure oxygen. Carbon dioxide is used as the carrier gas to sweep the combustion products from the combustion zone over oxidation catalysts to remove potential interferents.

The gases are passed over copper to remove excess oxygen and reduce nitrogen oxides to nitrogen gas. The gas is passed into a nitrometer where the volume of nitrogen produced is measured.

Ash

A weighed sample is placed into a tared platinum crucible. The sample is then ashed at 750°C. The procedure is repeated until a residue of constant weight is observed.

3.4 Organic Compounds

The identification and quantitation of organic species was accomplished by gas chromatography-mass spectrometry (GC-MS). Analyses were conducted according to EPA Method 625, published in the December 3, 1979, Federal Register, with a slight modification. The method modification consisted of using a 25-meter Hewlett-Packard wide bore fused silica capillary column as the analytical column for the sample extracts. This column improves the chromatography resolution and the method sensitivity and has been approved by the EPA Las Vegas laboratory for use on hazardous waste samples. Radian also modified the GC conditions in order to achieve improved resolution of the polynuclear aromatic hydrocarbons (PAHs). Figure 3-1 illustrates the chromatography of the PAHs. Analytical conditions are summarized in Table 3-1.

Samples were analyzed by GC-MS after a solvent extraction and concentration step. Water samples were extracted at both basic and acid pH with methylene chloride in continuous extractors. The extracts were combined prior to analysis. Slag and ash samples were extracted in Soxhlet extractors with methylene chloride. All sample extracts were concentrated by Kuderna-Danish evaporation.

Identification of organic species was accomplished by examination of the stored mass spectral data. As specified in the EPA method, identification was based on the occurrence of key fragment ions at known retention times.

Quantitation of organic species was achieved by the internal standard technique, relative to d_{10} -anthracene.

TABLE 3-1. GC-MS CONDITIONS FOR ICRC SAMPLE EXTRACTS

Instrument	Hewlett-Packard 5985
Ionization voltage	70eV
Ionization mode	Electron impact
Scan rate	1 scan/second
Scan range	45 + 450 amu
Column	SE-54 fused silica capillary (25m) [J&W DB-5]
H ₂ flow rate	30 cm/sec
Initial temperature	35°C
Initial hold	3 min
Program rate	30°/min for 5.5 min, then 4°/min
Final temperature	300°C
Final hold	20 min
Injector temperature	35°C
Injection	on-column
Sample size	1 µL

3.5 Water Quality Parameters

This section describes the analytical methods used to determine the various water quality parameters. The parameters determined and the methods used are summarized in Table 3-2. The analytical method for each parameter is discussed below in the order listed in Table 3-2.

Classical wet and instrumental methods were employed to determine most of the water quality parameters. The classical methods used were drawn from the EPA Methods for the Analysis of Water and Wastes (1979) and the APHA Standard Methods for the Examination of Water and Wastewater (Fifteenth Ed., 1981). Total inorganic carbon was determined according to the method outlined by the Beckman Instrument Company. Formates were determined by an ion chromatographic method refined at Radian for the analysis of gasification wastewater.

Settleable and Floatable Solids

Settleable and floatable solids were determined by EPA Method 160.5 and Standard Method 206A, respectively. Both parameters were determined using unpreserved samples.

Settleable solids are determined by filling an Imhoff cone, allowing settling, and recording the volume of settled matter. The results are reported in mL/L.

Floatable solids are determined using a one-liter Imhoff cone fitted with a stopcock. The sample is mixed with a paddle mixer and allowed to settle. Most of the sample is then drawn off. The floatable solids remaining in the cone are drawn through a tared, glass fiber filter. The filter is dried at 105°C for two hours and the floatable solids are determined by weighing. The floatable solids are calculated and reported in mg/L.

TABLE 3-2. ANALYTICAL METHODS FOR WATER QUALITY ANALYSES

PARAMETER	METHOD EMPLOYED	SPLIT ANALYZED
Settleable solids	EPA 160.5 ¹ (volumetric)	Unpreserved
Floatable solids	SM 206A ² (gravimetric)	Unpreserved
Total solids	EPA 160.3 (gravimetric)	Unpreserved
Total suspended solids	EPA 160.2 (gravimetric)	Unpreserved
Volatile suspended solids	EPA 160.4 (gravimetric)	Unpreserved
Total dissolved solids	EPA 160.1 (gravimetric)	Unpreserved
Conductivity	EPA 120.1 (conductivity)	Unpreserved
pH	EPA 150.1 (potentiometric)	Unpreserved
Alkalinity	SM 403 (titrimetric)	Unpreserved
Bicarbonate	SM 403 (titrimetric)	Unpreserved
Carbonate	SM 403 (titrimetric)	Unpreserved
Acidity	EPA 305.1 (titrimetric)	Unpreserved
Bromide	SM 405 (spectrophotometric)	Unpreserved
Chloride	EPA 325.3 (titrimetric)	Unpreserved
Fluoride	EPA 340.2 (potentiometric)	Unpreserved
Nitrate	SM 418D (spectrophotometric)	Unpreserved
Nitrite	EPA 354.1 (spectrophotometric)	Unpreserved
Phosphate	EPA 365.2 (spectrophotometric)	H ₂ SO ₄ preserved
Total organic carbon	EPA 415.1 (combustion/IR)	H ₂ SO ₄ pres.
Total inorganic carbon	Beckman Instrument manual	Unpreserved
Kjeldahl nitrogen	EPA 351.3 (digestion, distillation, titrimetric)	H ₂ SO ₄ preserved
Formate	Ion chromatographic	Unpreserved
Oil and Grease	EPA 413.1 (extraction, gravimetric)	H ₂ SO ₄ preserved
Phenolics	EPA 420.1 (distillation, spectrophotometric)	H ₂ SO ₄ preserved

¹EPA Methods for the Chemical Analysis of Water and Wastes (1979)²APHA Standard Methods for the Examination of Water and Wastewater (1981)

Total, suspended, volatile suspended, and dissolved solids

Total solids (TS), total suspended solids (TSS), total volatile suspended solids (TVSS) and total dissolved solids (TDS) were determined gravimetrically using:

- total solids - EPA Method 160.3,
- total suspended solids - EPA Method 160.2,
- total volatile suspended solids - EPA Method 160.4, and
- total dissolved solids - EPA Method 160.1.

All four determinations were performed on unpreserved samples.

Total solids are determined by transferring a homogeneous aliquot to a tared beaker, evaporating to dryness at 105°C, and weighing the residue and beaker. Total suspended solids are determined by filtering an aliquot of well-mixed sample through a tared, glass fiber filter and weighing the residue retained following drying at 105°C. To determine total volatile solids, the glass fiber filter is ignited at 550°C in a muffle furnace and weighed. Total dissolved solids are determined by transferring filtrate from the TSS determination to a tared beaker, evaporating to dryness at 180°C, and weighing the beaker and residue. Results for all four solids determinations are calculated and reported in mg/L.

Conductivity

Conductivity was measured using EPA Method 120.1, an instrumental method. A Lab-Line® Lectro Mho-meter, Model Mark V, was used to perform the determinations on unpreserved samples. The temperature of each sample was measured prior to analysis and the temperature compensating dial set to this temperature. A 0.0100 N KCl solution was used to calibrate the instrument. All results are reported in $\mu\text{mho}/\text{cm}$ at 25°C.

pH, alkalinity, bicarbonate, carbonate

The determination of pH and alkalinity was performed by EPA Method 150.1 and Standard Method 403, respectively. Bicarbonate and carbonate content was calculated from the alkalinity data. Unpreserved samples were used for these determinations.

The pH of the samples is determined potentiometrically with a Corning Model 130 pH meter equipped with a commercially available combination electrode. The meter is standardized before use with a commercially available buffer solution (pH 7). The standardization is checked with a second buffer (pH 4). The pH of each sample is read and recorded to 0.01 pH units.

Following determination of pH, the alkalinity titration is performed. Aliquots are titrated with standardized sulfuric acid to pH 8.3 and then to pH 4.5. The course of the titration is followed potentiometrically and the volume of acid to reach each end-point is read and recorded. Total, bicarbonate, and carbonate alkalinites are calculated from these data. All alkalinity results are reported in mg/L as CaCO_3 .

Acidity

Acidity was determined using EPA Method 305.1. The pH of unpreserved sample is determined and a measured amount of standard sulfuric acid is added to lower the pH to four or less. The resulting solution is treated with H_2O_2 , boiled for several minutes, and cooled. A potentiometric titration is then performed with standard NaOH to pH 8.2. Acidity results are calculated and reported in mg/L as CaCO_3 .

Bromide

Bromide was determined spectrophotometrically by Standard Method 405. Unpreserved samples were analyzed for bromide. In the presence of

chloramine-T, bromide is oxidized to bromine and bromination of phenol red takes place. The intensity of red-violet compound formed is measured spectrophotometrically using a Bausch and Lomb Spectronic 20. The bromide concentration in the sample is calculated and reported in mg/L bromide.

Chloride

Chloride was determined titrimetrically by EPA Method 325.3. An acidified aliquot is titrated with standardized mercuric nitrate in the presence of a mixed diphenylcarbazone-bromophenol blue indicator. The end point is signaled by the formation of the blue-violet mercury diphenylcarbazone complex. The mercuric nitrate is standardized against a standard sodium chloride solution. The chloride concentration is calculated and reported in mg/L chloride.

Fluoride

Fluoride was determined potentiometrically using EPA Method 340.2 and standard additions. All measurements are performed with a Corning Model 130 pH meter equipped with an Orion fluoride selective electrode and reference electrode. Unpreserved sample is analyzed. A 5.0 mL aliquot is transferred to a polyethylene beaker, the pH is adjusted to 5.25 with glacial acetic acid, and 5 mL of citrate buffer is added. The citrate buffer is added to buffer the sample as well as release complexed fluoride and provide constant ionic strength. The fluoride selective and reference electrodes are immersed in the sample and allowed to equilibrate for five minutes in the magnetically stirred sample. The potential is then read and recorded. At this point, 0.10 mL of 1000 ppm fluoride solution is added. The electrodes are allowed to equilibrate for five minutes before the final potential is measured. The change in potential is used to calculate the fluoride content in the aliquot. The fluoride concentration is reported in mg/L fluoride.

Nitrate

Nitrate was determined spectrophotometrically in unpreserved sample using Standard Method 418D. Sulfite-urea solution and antimony solution are added to each sample to remove interferences by nitrite and chloride, respectively. The solution is then acidified and treated with chromotropic acid. The intensity of the resulting yellow color is measured spectrophotometrically. The nitrate concentration is calculated and reported as mg/L nitrogen.

Nitrite

Nitrite was determined spectrophotometrically in unpreserved sample using EPA Method 354.1. Nitrite reacts with sulfanilamide in acid medium to form an intermediate diazonium salt. The diazonium salt is coupled with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a reddish purple azo dye. The intensity of this dye is determined spectrophotometrically. Nitrite content is calculated and reported in mg/L nitrogen.

Phosphate

Phosphate was determined spectrophotometrically on acid (H_2SO_4) preserved samples using EPA Method 365.2. A preliminary, persulfate digestion is performed to release organically bound phosphate. Following digestion, ammonium molybdate and potassium antimonyl tartrate are reacted with the phosphate in acidic medium. The heteropoly acid-phosphomolybdic acid formed is reduced by ascorbic acid to molybdenum blue. The intensity of the intensely colored molybdenum blue is measured spectrophotometrically. Phosphate content is calculated and reported as mg/L phosphorus.

Total organic carbon, total inorganic carbon

Total organic carbon (TOC) and total inorganic carbon (TIC) were determined instrumentally with a Beckman Model 915B Total Organic Carbon

Analyzer. TOC was determined according to EPA Method 415.1. Acid preserved sampled ($\text{pH} < 2$ by H_2SO_4) is sparged with nitrogen to remove inorganic carbon. The sparged solution is injected into the instrument where organic carbon is oxidized to carbon dioxide in a 850°C furnace. The carbon dioxide produced is measured with an infrared detector. TOC is calculated and reported in mg/L carbon.

Total inorganic carbon was determined according to the method outlined in the manual for the Model 915B TOC analyzer. Unpreserved sample was injected into the instrument and onto phosphoric acid on silica. Inorganic carbon is released as carbon dioxide and the carbon dioxide is measured by an infrared detector. TIC is calculated and reported as mg/L carbon.

Total Kjeldahl Nitrogen

Total Kjeldahl nitrogen was determined titrimetrically following digestion and distillation according to EPA Method 351.3. Acid (H_2SO_4) preserved sample is digested in the presence of concentrated sulfuric acid and selenate. The digest is treated with concentrated base and the ammonia liberated is distilled into indicating boric acid solution. A Labconco microstill is used for the distillation. The amount of ammonia distilled is determined by titration with standard sulfuric acid. Total Kjeldahl nitrogen is calculated and reported as mg/L nitrogen.

Formate

Formate was determined by ion chromatography using the technique of standard additions. A Dionex Model 12 ion chromatograph was used for all determinations. Formate standards were prepared from formic acid which had been previously standardized. The eluent used for 0.0020 M sodium tetraborate. A conventional conductivity detector was used to quantitate the formate eluted.

Oil and Grease

Oil and grease was determined gravimetrically following extraction with Freon. A modification of EPA Method 413.1 was used. Aliquots of acid (H_2SO_4) preserved samples were taken from the large containers. These aliquots were extracted successively with Freon. The Freon extracts were combined in a tared boiling flask. The Freon was evaporated to dryness with a rotary evaporator. The flask was then weighed to determine Freon soluble oil and grease. Oil and grease are calculated and reported in mg/L.

Phenolics

Phenolics were determined spectrophotometrically following a preliminary distillation and chloroform extraction. EPA Method 420.1 was used. Acid (H_2SO_4) preserved sample is distilled and the distillate collected for analysis. Phenolics in the distillate is reacted with 4-amino-antipyrine in the presence of potassium ferricyanide at pH 10. The reddish-brown antipyrine dye formed is extracted into chloroform and determined spectrophotometrically. Phenolic content is calculated and reported in mg/L phenol.

3.6 Metals

It is vitally important to the analysis of solid and aqueous samples to utilize the proper digestion technique along with the appropriate analytical technique in order to achieve the most accurate result possible.

3.6.1 Digestive Techniques

Digestive techniques used on solid samples include a perchloric acid digestion, a lithium borate fusion, and a sodium carbonate fusion. Aqueous samples are prepared for analysis using the digestion for total metals in "Methods of Chemical Analysis of Water and Wastes," EPA600/4-79-020, section 4.1.3. Leachate samples are analyzed without digestion.

Perchloric Acid Digestion

The perchloric acid digestion (PAD) has been found by Radian to have the widest application for analysis of metal constituents. The digestion is a modification of the method described by McQuaker, et.al. ("Digestion of Environmental Material for Analysis by ICPEs." Analytical Chemistry, Vol, 51, No. 7, June, 1978) differing primarily in the amount of acid used. The procedure entails the addition of nitric (HNO_3) and hydrofluoric (HF) acids to a dried sample. The sample is covered, heated to reflux, and then allowed to cool. Perchloric acid (HClO_4) is added and the sample taken to dryness. The final step is repeated until the baked sample takes on a grey color. Deionized water and dilute hydrochloric acid (HCl) are added and the sample is warmed to effect complete dissolution. The sample is then cooled and taken to volume. ULTREX acids and teflon digestive beakers are used to prevent contamination.

A low temperature plasma asher (IPC Model PM-148) is used to reduce the carbon content of the coal and fly dust samples. This action eliminates the potential for an explosive reaction between the carbon and perchloric acid, while minimizing volatile trace element loss.

Lithium Borate Fusion

A lithium borate fusion as outlined by Perkin-Elmer Corporation's "Methods Manual" is used for the determination of major ion species in solid samples. The procedure involves the mixing of a portion of sample (.1-.2 g) with lithium meta-borate in a disposable, ultra pure graphite crucible. The mixture is fused at 1050°C and the melt poured into dilute HCl for dissolution. The sample is then taken to volume with deionized water.

Sodium Carbonate Fusion

The sodium carbonate fusion is utilized for the analysis of boron and fluoride in solid samples. A portion of sample (0.3-0.5 g) is mixed with powdered sodium carbonate in a porcelain crucible, which is then fused at 850°C, dissolved in HCl, and taken to volume.

EPA Water Digestion

Aqueous samples are digested using the digestion described in "Chemical Analysis of Water and Wastes" U.S. EPA. The digestion entails the treatment of an aliquot of the wash water with nitric acid followed by evaporation to near dryness. This step is repeated twice with the final addition being dilute HCl after which the sample is diluted to volume. ULTREX acids are used to prevent contamination.

3.6.2 Analytical Techniques

Inductively Coupled Argon Plasma Emission Spectroscopy (ICPES)

ICPES is a multielement methodology which provides for simultaneous analysis of up to 40 elements with detection limits rivaling atomic absorption spectroscopy (AAS). The instrument used in this study is an Applied Research Laboratories model 34000B vacuum spectrometer with off-peak background

correction capabilities. It is equipped with a PDP 11/04 mini-computer with dual floppy disks for data manipulation and storage. For this study, the ICPEES is utilized for the analysis of

Aluminum	Calcium	Iron	Molybdenum
Boron	Cobalt	Potassium	Sodium
Barium	Chromium	Magnesium	Nickel
Beryllium	Copper	Manganese	Phosphorous
Silicon	Titanium	Vanadium	Zinc

in solid, aqueous, and leachate samples. Major species are analyzed on the lithium borate fusion, minor and trace species on the perchloric acid digestion, and boron on the sodium carbonate fusion.

The analysis is conducted in accordance with "Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," U.S. EPA Method 200.7, Nov. 1980 and McQuaker, et. al. referenced in section 3.6.1.

Instrument calibration is performed for each element of interest prior to each analysis set. Standards are prepared in the same acid matrix as samples to prevent error due to acid content.

Atomic Absorption Spectroscopy

In this study, Atomic Absorption Spectroscopy (AAS) is used for the determination of:

Silver	Cadmium	Lead	Selenium
Arsenic	Mercury	Antimony	Thallium

Graphite furnace AAS is utilized for all elements except mercury. Graphite furnace AAS allows for the determination of pico-gram quantities of metal constituents. The technique involves the introduction of micro-liter aliquots onto a graphite tube mounted in the optical pathway of the

spectrometer. The aliquot is dried and charred to remove interferent species and atomized to produce atomic vapor which produces an absorption peak which is compared to standards for quantification. Analyses are performed in accordance with "Chemical Analysis of Water and Wastes," U.S. EPA, March 1979. The dry, char and atomization temperature programs used are as specified by instrument manufacturers' methods manuals.

Mercury in coal, dust, and slag samples is determined by ignition in an oxygen bomb with absorption in KMnO_4 . The resultant solution is analyzed according to methodology described in "Chemical Analysis of Water and Wastes" referenced above. Aqueous and leachate samples are analyzed directly by the methodology described in this reference.

Instrumental conditions for ICPES and AAS techniques are presented in Table 3-3.

3.7 Radioactivity

Liquid samples are evaporated to a small volume, transferred to a tared stainless steel planchet, evaporated to dryness under infrared lamps, reweighed, and counted for gross alpha or gross beta activity. The ASTM methods D1943-66 (1977) and D1890-66 (1977) are used for alpha and beta determinations respectively. Samples of coal, flydust, or slag are mounted by filtering a slurry of the pulverized solid in a minimum of deionized water through a tared membrane filter. The solids are then dried under infrared lamps, reweighed, mounted on stainless steel planchets, and counted for gross alpha or gross beta activity. Sample areal densities (for either liquid or solid samples) are kept below 5 mg/cm^2 . Reported activities are corrected for self-absorption and instrument counting efficiency.

TABLE 3-3. INSTRUMENTAL CONDITIONS FOR ICPES AND
AAS ANALYTICAL TECHNIQUES

I C P E S			
ELEMENT	Wavelength	Detection Limit (mg/L)	EPA Method
Al	237.31	0.050	200.7
Ba	455.40	0.001	
Be	234.86	0.0005	
B	182.64	0.010	
Cd	226.50	0.002	
Ca	315.89	0.040	
Cr	267.72	0.001	
Co	228.62	0.006	
Cu	324.74	0.001	
Fe	259.94	0.008	
Mg	279.08	0.030	
Mn	257.61	0.001	
Mo	202.03	0.002	
Ni	221.65	0.003	
P	213.63	0.18	
K	766.49	0.050	
Si	251.61	0.020	
Na	589.59	0.010	
Ti	368.50	0.005	
V	289.33	0.003	
Zn	202.55	0.003	

- 1 meter spectrometer
 - scanning entrance slit
 - programmable nebulizer tip desalter
 - PDP 11/04 mini-computer
- Forward Power-1250 watts
Reflected Power- <5
Spectrometer Vacuum-63 microns

A A S						
ELEMENT	Wave-length	Analysis Temperatures (°C)			Detection Limit	EPA Method
		Dry	Char	Atomization		
Sb	217.0	100	450	2400	.003	204.2
As	193.7	100	450	2100	.003	206.2
Cd	228.8	100	450	2200	.0005	213.2
Pb	283.3	100	450	2100	.002	239.2
Se	196	100	450	2500	.003	270.2
Ag	328.6	100	1300	2700	.001	272.2
Tl	277.6	100	450	2100	.003	279.2

3.8 Leaching Methods

RCRA Leaching

RCRA leachings are performed by weighing 100 g of the solid to be leached into an amber glass jar and 1600 milliliters of DI water is added. The initial pH is recorded and the initial pH adjusted to a pH of 5 with 0.5 N acetic acid. A maximum of 400 milliliters of acid may be used during the leaching process. The solution is continuously stirred for 24 hours. During leaching, the pH is routinely monitored and readjusted to pH 5 with either the acetic acid or 0.25 N sodium hydroxide. After 24 hours, the leachate is filtered through a 0.45 μ m filter and diluted to 2000 milliliters deionized water. The leachates were analyzed according to the method described in Section 3.6.2.

ASTM Procedure A

This extraction procedure is a water shake extraction. A weighed portion of sample is mixed with a volume of water equal to four times the mass of the solid. The closed container is agitated for 48 hours and filtered through a 0.45 μ m filter and analyzed for metal content. The leachates were analyzed according to the method described in Section 3.6.2.

4.0 QUALITY ASSURANCE/QUALITY CONTROL

All analyses are subjected to a strict quality assurance/quality control program. Periodic QA audits are submitted by the corporate quality assurance officer to verify the reliability of the analytical data. Each laboratory functions under a quality control program designed to monitor all results. Any problems that may arise are quickly traced and then solved. Quality control measures may vary in each laboratory, depending on the function of the laboratory. However, the program common to all the laboratories consists of:

- daily system evaluation,
- completion of instrument logs,
- daily calibration,
- analysis of blanks,
- analysis of quality control standards, and
- analysis of spiked samples.

4.1 Organic Analysis

Specific QC activities were employed by the organic laboratories during the analyses. These activities encompass the requirements of EPA Method 625 as well as additional measures developed at Radian. The primary QC measures were:

- daily GC-MS system performance evaluation,
- analysis of standards,
- analysis of quality control samples,
- analysis of blanks, and
- analysis of spiked (labeled compounds) samples.

System Performance Evaluation

A daily system performance evaluation was conducted by determining the mass spectral intensities of several key ions of decafluorotriphenyl phosphine (DFTPP). A list of the criteria required for a satisfactory performance is found in Table 4-1. The system met these criteria on the days samples were analyzed.

Analysis of Standards (Calibration)

Solutions containing the priority pollutants and PAHs at known concentrations (standards) prepared by Supelco, Inc. (Bellefonte, PA) were analyzed daily to determine the instrument response and sensitivity. These analyses were used to establish calibration curves for quantitation.

Analysis of Quality Control Samples

Radian periodically analyzes a set of quality control samples. These samples contain selected priority pollutants at known levels in solvent. For this program the results of the quality control analyses are summarized in Table 4-2.

Analysis of Blanks

A Radian laboratory water blank, treated in the same manner as the samples, was analyzed and no compounds were detected.

TABLE 4-1. SYSTEM PERFORMANCE REQUIREMENTS FOR
DECAFLUOROTRIPHENYL PHOSPHINE (DFTPP)

<u>MASS</u>	<u>DFTPP CRITERION</u>
51	30-60% Mass 198
68	<2% Mass 69
70	<2% Mass 69
127	40-60% Mass 198
197	<1% Mass 198
198	Base Peak
199	5-9% Mass 198
275	10-30% Mass 198
365	>1% Mass 198
441	< Mass 443
442	>40% Mass 198
443	17-23% Mass 442

TABLE 4-2. ANALYSIS OF QUALITY CONTROL SAMPLES

C O M P O U N D S	Spike Level ($\mu\text{g/mL}$)	Average* Concentration Measured ($\mu\text{g/mL}$)	Average* Percent Recovery
Hexachloroethane	10	12	120
Hexachlorobutadiene	20	30	150
Isophorone	50	75	150
Acenaphthene	15	10	67
Fluorene	14	11	79
Hexachlorobenzene	11	13	118
1,2-Diphenylhydrazine	20	13	65
Pyrene	25	33	132
di-n-Octyl phthalate	20	29	145

*Average of six determinations

Analysis of Spiked Samples

Separate aliquots of one aqueous sample and one ash sample were spiked with deuterium labeled polynuclear aromatic hydrocarbons before extraction. The compounds were spiked at 50 µg/L for the water sample and 1.14 µg/g for the ash sample. The recovery of these compounds is shown in Table 4-3.

The recoveries for the ash sample indicate that the organic compounds are sorbed by the sample. Since the sample spikes did not contain the naturally abundant species, these analyses also served for a duplicate sample determination. No organics were found in the spiked water sample, agreeing with the initial analysis. A comparison of the results obtained from the duplicate analyses is shown in Table 4-4.

4.2 Metal Analysis

Analyses in the Atomic Spectroscopy laboratory were verified by specific QC activities. These measures included the analysis of blanks, spikes, QC standards, and replicates at the frequency of ten percent. The primary QC measures were:

- daily system performance evaluation of both AA and ICPES,
- daily analysis of calibration standards,
- daily analysis of QC samples,
- daily analysis of spiked samples,
- daily analysis of replicate samples, and
- daily analysis of blanks.

TABLE 4-3. RECOVERY OF LABELED COMPOUNDS FROM SAMPLES *

C O M P O U N D	Percent Recovery	
	Wash Water Run 216	Fly Dust Run 212
Naphthalene-d ₈	42	5.7
Acenaphthene-d ₁₀	56	3.4
Fluorene-d ₁₀	58	1.3
Anthracene-d ₁₀	66	<1.0
Phenanthrene-d ₁₀	67	<1.0
Pyrene-d ₁₀	68	<1.0
Chrysene-d ₁₂	66	<1.0
Benz(a)anthracene-d ₁₂	72	<1.0
Benzo(a)pyrene-d ₁₂	74	<1.0
Dibenzothiophene-d ₈	70	<1.0

*50 µg/L of each labeled compound was added to the aqueous sample.

1.1 µg/g of each labeled compound was added to the fly dust samples.

TABLE 4-4. COMPARISON OF RESULTS FROM DUPLICATE ANALYSES OF FLY DUST FROM RUN 212

C O M P O U N D	C O N C E N T R A T I O N , $\mu\text{g/g}$	
	ORIGINAL ANALYSIS*	REPEAT ANALYSIS**
Naphthalene	8.1	7.5
1-Methylnaphthalene	0.36	0.45
2-Methylnaphthalene	0.26	0.31
Biphenyl	0.15	0.17
Acenaphthene	0.13	0.17
Acenaphthylene	1.9	2.8
Fluorene	0.095	0.14
Dibenzothiophene	0.064	0.095
Phenanthrene	0.23	0.34
Fluoranthene	0.12	0.25
Pyrene	0.82	1.3
Phenol	0.14	0.20

*Results from run 212 reported in Table 2-7

**Sample was also spiked with labeled compounds

System Performance Evaluation

All operating conditions with the ICPES were monitored and controlled by the dedicated computer. Daily evaluation of millivolt intensities insure the required sensitivity and reliability. Instrument operating logs were maintained for each AA unit. Instrumental response to various standards were compared to historic results. Any deviation in operating parameters was immediately noted and evaluated.

Analysis of Calibration Standards

Daily calibration standards were prepared from commercially available atomic absorption solutions. Standards were prepared in solutions which matched the acid matrix of the sample.

Analysis of QC Samples

The accuracy of the calibration standards was checked by analyzing EPA QC standards. NBS standards were digested and analyzed with the samples. Digestion of NBS samples provides a check on digestion efficiency. Results of the analyses are presented in Table 4-5.

Analysis of Spiked Samples

Random samples were spiked after the digestion and before the analysis. This verifies the analytical method. These results are presented in Table 4-6.

Analysis of Replicate Samples

Samples were chosen at random for duplicate digestion and analysis. Results of duplicate analyses are presented in Table 4-7.

TABLE 4-5. METALS ANALYSIS OF QC STANDARDS

ELEMENT	Coal NBS 1632*		Ash NBS 1633*		Water EPA QC**	
	Certified	Analyzed	Certified	Analyzed	Certified	Analyzed
Al	1.7%	1.5%	12.7%	12%	0.70	0.72
Sb	3.6	3.4	6.9	6.4	0.0082	0.0074
As	5.8	6.1	61	65	0.040	0.034
Ba	344	350	2700	2400	-	-
Be	1.4	1.6	12	18	0.75	0.74
B	41	70	480	430	-	-
Cd	0.24	0.23	1.2	1.1	0.0015	0.0012
Ca	4100	4000	4.6%	4.3%	-	-
Cr	20	23	130	120	0.15	0.15
Co	5.7	5.9	40	55	0.50	0.50
Cu	17	15	127	120	0.25	0.25
Fe	8500	8100	6.2%	6.2%	0.60	0.66
Pb	28	26	72	68	0.018	0.020
Mg	1800	1900	1.6%	1.2%	-	-
Mn	42	45	490	470	0.35	0.32
Hg	(0.50)	0.68	0.14	0.10	-	-
Mo	3.1	3.0	29	26	-	-
Ni	15	17	98	90	0.25	0.21
P	71	79	(880-3300)	550	-	-
K	2800	3000	1.7%	1.2%	-	-
Se	3.0	2.6	9.6	8.8	0.040	0.040
Si	2.8%	2.5%	22%	19%	-	-
Ag	0.060	0.070	(.25-1.0)	0.34	0.049	0.043
Na	380	390	3200	3300	-	-
Tl	(0.60)	0.40	4.0	3.0	-	-
Ti	950	1050	7200	6100	-	-
V	35	33	216	250	0.75	0.73
Zn	36	34	213	200	0.20	0.21

*Results in µg/g, unless noted.

**Results in mg/L, unless noted.

() Reported, but not certified.

TABLE 4-6. METALS SPIKE RECOVERY

ELEMENT	SLAG TEST #215* (%)	WASTEWATER #213* (%)
Al	88	107
Sb	78	110 (WW #214)
As	88 (Slag #216)	96 (WW #216)
Ba	93	110
Be	98	104
B	104	108
Cd	88	97
Ca	93	97
Cr	97	95
Co	94	97
Cu	108	93
Fe	110	104
Pb	97 (Flydust #216)	92 (City Water)
Mg	93	98
Mn	98	95
Hg	109 (Slag #217)	104 (WW #212)
Mo	89	93
Ni	91	110
P	110	106
K	97	104
Se	87 (Slag #212)	100
Si	93	103
Ag	98 (Slag #212), 88 (Flydust #212)	94
Na	104	110
Tl	91 (Flydust #217)	104 (WW #215)
Ti	110	93
V	107	95
Zn	93	98

*other samples noted by ()

TABLE 4-7. METALS DUPLICATE ANALYSES

ELEMENT	Flydust #216 (µg/g)		City Water (mg/L)	
	A	B	A	B
Al	4.3%	4.4%	<.05	<.05
Sb	5.1	4.9	<.003	<.003
As	36	37	<.003	<.003
Ba	140	140	0.044	0.043
Be	16	16	<.0005	<.0005
B	490	480	0.14	0.14
Cd	4.3	4.5	<.0005	<.0005
Ca	8400	8300	34	36
Cr	83	84	<.001	<.001
Co	19	18	<.006	<.006
Cu	93	97	<.001	<.001
Fe	5.1%	5.1%	<.008	<.008
Pb	46	47	<.002	<.002
Mg	2100	2080	6.7	6.6
Mn	150	150	0.014	0.014
Hg	.0053	.0056	<.0002	<.0002
Mo	34	35	0.005	0.006
Ni	77	78	<.003	<.003
P	190	195	<.18	<.18
K	6800	6750	3.4	3.6
Se	2.6	2.8	0.004	<.003
Si	11%	11%	<.016	<.016
Ag	1.7	1.6	<.001	<.001
Na	4700	4600	22	23
Tl	4.0	4.1	<.003	<.003
Ti	3000	3100	<.005	<.005
V	170	190	<.003	<.003
Zn	220	210	<.003	<.003

<Indicates that the parameter was not detected. The value shown is the detection limit of the method.

Analysis of Blanks

A digestion blank consisting of all reagents used in the dissolution of the sample was analyzed with the samples. The blanks were analyzed and no contaminants were found.

4.3 Water Quality Analysis

Specific QC measures in the Water Quality laboratory were employed. The primary QC measures were:

- daily system evaluation,
- daily analysis of calibration standards,
- analysis of replicate samples,
- analysis of spikes and distillation standards,
- use of standard additions for certain parameters.

System Evaluation

Instrumentation required for the determination of the various parameters was checked and calibrated on the day of the analysis. Instrument logs are maintained to monitor use and operation.

Analysis of Calibration Standards

Calibration standards are prepared from commercially available standards or from reagent-grade chemicals. All standards are within the limits stated in "Methods for Chemical Analysis of Water and Wastes," EPA, March 1979.

Analysis of Duplicates

Samples were analyzed in replicate, when sample volume permitted. The results are presented in Table 4-8.

Analysis of Spikes

Certain parameters with known interferences were spiked during the analysis. Parameters that require a distillation were verified by the distillation of a standard. Formate was analyzed using the method of additions. Spikes and other QC results are presented in Table 4-9.

TABLE 4-8. WATER QUALITY DUPLICATE RESULTS

P A R A M E T E R	S A M P L E	R E S U L T S
pH	Wastewater Test #215	8.24, 8.21
Alkalinity	Wastewater Test #216	243, 244 mg/L
Bicarbonate	Wastewater Test #216	243, 244 mg/L
Acidity	Wastewater Test #215	< 1, < 1 mg/L
Bromide	Wastewater Test #216	< .01, < .01 mg/L
Chloride	City Water	27, 27 mg/L
Total Solids	Wastewater Test #215	754, 737 mg/L
Total Suspended Solids	Wastewater Test #216	< 1.0, 2.0 mg/L
Total Volatile Suspended Solids	Wastewater Test #216	0.5, 0.9 mg/L
Total Dissolved Solids	Wastewater Test #216	501, 532 mg/L
Specific Conductance	City Water	348, 350 mhos
TKN	Wastewater Test #217	64, 64 mg/L
Nitrate	Wastewater Test #217	44, 41 mg/L
Nitrite	City Water	< .01, < .01 mg/L
Phosphate	Wastewater Test #216	5.7, 5.9 mg/L

TABLE 4-9. WATER QUALITY SPIKE RESULTS

P A R A M E T E R	S A M P L E	SPIKE RECOVERY
Bromide	City Water	84%
Nitrite	Wastewater Test #214	88%
Phosphate	City Water	89%
Phenolics	Distillation Standard	99.8%
TKN	Standard	105%

APPENDIX

GC-MS CHROMATOGRAMS OF
ICRC SAMPLE EXTRACTS

The GC-MS chromatograms obtained for the samples analyzed are included in the appendix for reference purposes. The lower trace is the chromatogram obtained for a 20 µg/mL d₁₀-anthracene internal standard.

FRN21168CRN7
45< M <450

[CNC 4558-2-1625(R-W), 2-25-82, 0-2200 DN-CDL

08FSCC126M], PROTOCOL 6033, 1UL, 910-20

STARTING MASS

Figure A-1. GC/MS Chromatogram of Radian Sample 4558 (Wash Water, Run 212)

FRN 21166 CRN 18
45 < M < 450

[CRC 4563-]-[625(I/N)], 2-26-82, 0-2200

ON-COL: DBEFSCC(26), PROTOCOL 5033.1UL

STARTING MASS 45

Figure A-2. GC/MS Chromatogram of Radian Sample 4563 (Wash Water, Run 213)

FRN21163CRN8

45< M <450

[CNC 4608-1-1625(R/N).2-16-82.0-2200 0W-COL.

DB5FBC(25M).PROTOCOL 5033.1UL. 010-20PP

STARTING MASS

Figure A-3. GC/MS Chromatogram of Eadian Sample 4568 (Wash Water, Run 214)

FRN 21167 CRN 18

45< M < 450

CRC 4573-1-1625(A/N). 2-20-82.0-2200 ON-COL

DBSFSCC(25K). PROTOCOL 5C33.1UL. #10-20

STARTING MASS 45

Figure A-4. GC/MS Chromatogram of Radian Sample 4573 (Wash Water, Run 215)

FRN 21160 CRN 17

45 < M < 450

ICRC 4578-1-11661A/N1, 2-25-82, G-2200 ON-COL.

DBSFSCC125M1, PROTOCOL 5033, IUL, #10-20

STARTING MASS 45

Figure A-5. GC/MS Chromatogram of Radian Sample 4578 (Wash Water, Run 216)

FRN21161 CRN 7

45< M <450

[CRC 4583-1]-1626(A/N): 2-26-82, P-2200, CN-COL

08679CC(26K), PROTOCOL 5033, IUL, 810-20

STARTING MASS

Figure A-6. GC/MS Chromatogram of Radian Sample 4583 (Wash Water, Run 217)

FRN 21159 CRN 17

45 < M < 450

ICR4588-1-16251A/N/1.2-25-82.0-220C. 0N-CO1

DBSFCCI(2SM). PROTOCO1 5033.1UL. #10-20

STARTING MASS 45

Figure A-7. GC/MS Chromatogram of Radian Sample 4588 (City Water)

FRN 21306 CRN 36

45< M < 400

[CRC 4580(217), 3-30-82.0=2100

DBFSCC. PROTOCOL 5033. .SUL. #10-20

STARTING MASS 45

Figure A-8. GC/MS Chromatogram of Radian Sample 4560 (Fly Dust, Run 212)

FRN 21301 CRN 35
45 < M < 400

!CRC 4565(=213).3-29-82.0=2400
DBSFSEC.PROTOCOL 5033.IUL. \$10-2C
STARTING MASS 45

Figure A-9. GC/MS Chromatogram of Radian Sample 4565 (Flydust, Run 213)

Figure A-10. GC/MS Chromatogram of Radian Sample 4570 (Fly Dust, Run 214)

FRN 21308 CRN 36

45 < M < 400

ICRC 4574 (215), 3-30-82, G=2400

DBSFSCC, PROTOCOL 5033, IUL, 910-20

STARTING MASS 45

Figure A-11. GC/MS Chromatogram of Radian Sample 4574 (Flydust, Run 215)

CRC 4580(216).3 30-82.0-2400

DB5FSCC.PLOT0001 5033.1UL #10-20

STARTING MASS 45

FRN 21304 CRN 35
45 M 400

FRN 21305 CRN 36

45 < M < 400

ICRC 45841(217), 3-30-82, 0-2400

DBSFCC, PROTOCOL 5033, IUL, #10-20

STARTING MASS 45

Figure A-13. GC/MS Chromatogram of Radian Sample 4584, Fly Dust Run 217

FRN 21313 CRN 36

45 < M < 400

ICRC-45591 (212):3/30/82.DA[N=2400V. ON-COLUMN.

5033:25M-DB5 FSCC.910A=2000-.15L.

STARTING MASS 45

Figure A-14. GC/MS Chromatogram of Radian Sample 4559 (Slag, Run 212)

FRN 213.2 CRN 36

45 < M < 400

[CRC-4564] 213.2:3/30/82.06 [N=2400V] DN-COLUMN

S033:25H-DBS FSCC. S10R=2000-.15L

STARTING MASS 45

Figure A-15. GC/MS Chromatogram of Radian Sample 4564 (Slag, Run 213)

ICRC-45691-214):3/30/82.GAIN=2400V. ON-COL JMW.

5033:25M-085 FSC. *10A=2000-.15L.

STARTING MASS 45

Figure A-16. GC/MS Chromatogram of Radian Sample 4569 (Slag, Run 214)

FRN 21314 CRN 38

45 < M < 400

LCRC 4575 (215), 3-31-82, 0-2400

OBSFSCC, PROTOCOL 5023, IUL, \$1C-20

STARTING MASS 45

Figure A-17. GC/MS Chromatogram of Radian Sample 4575 (Slag, Run 215)

FRN 21315 CRN 38

45 < M < 400

ICRC 4579(216).3-31-82.G=2400

DBSFSCC.PROTOCOL 5033.IUL.510-20.

STARTING MASS -5

Figure A-18. GC/MS Chromatogram of Radian Sample 4579 (Slag, Run 216)

[CRC456X] = 217, 3-31-82, 0=2400
DBSFSC.PROTOCOL 5033. JUL. 910-20
STARTING MASS 45

Figure A-19. GC/MS Chromatogram of Radian Sample 4585 (Slag, Run 217)