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SOLVENT REFINED COAL (SRC) PROCESS: TRACE ELEMENTS

Research & Development Report No. 53, Interim Report No. 31
Volume 3—Pilot Plant Development Work
Part 6—The Fate of Trace Elements in the SRC Process
For the Period August 1, 1976—July 31, 1977

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Washington State University,
Nuclear Radiation Center
Pullman, Washington

U. S. DEPARTMENT OF ENERGY

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PART 6 - THE FATE OF TRACE ELEMENTS IN THE SRC PROCESS

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MARCH, 1980

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TABLE OF CONTENTS

	Page
INTRODUCTION	2
ANALYTICAL METHODOLOGY	3
A. Sample Collection and Preparation	3
B. Neutron Activation Analysis	7
1. Sample Preparation for Irradiation	7
2. Irradiation of Samples	11
3. Post-Irradiation Treatment	11
4. Radionuclide Activity Measurement	15
5. Computation of Analytical Results	16
6. Accuracy and Precision	17
C. Atomic Absorption Analysis	23
RESULTS AND DISCUSSION	25
A. Equilibrium Set 3 Trace Element Data	25
B. Material Balance for Equilibrium Set 3 (SRC I)	30
C. Plant Effluent Water Analysis	38
ACKNOWLEDGEMENTS	42
LITERATURE CITED	43
APPENDIX I: TRACE ELEMENT DATA FOR SRC I EQUILIBRIUM SET THREE .	45
APPENDIX II: TRACE ELEMENT DATA FOR CO ADDITION STUDY AND COMPOSITES	59
APPENDIX III: TRACE ELEMENT DATA FOR PLANT EFFLUENT WATERS . . .	66

LIST OF TABLES

Table		Page
1	Pilot Plant Sample Collection Points	3
2	Run Conditions for Equilibrium Set 3	5
3	Product Yields for Equilibrium Set 3	6
4	Weight of Elements in Liquid Standards	10
5	Irradiation Procedures	12
6	Nuclear Data on Nuclides Measured	13
7	Elemental Concentrations in NBS Standard Coal (SRM 1632) and Comparison of the Average Values with Average of 4-Laboratory and NBS Values	19
8	Elemental Concentrations in NBS Standard Fly Ash (SRM 1633) and Comparison of the Average Values with Average of 4-Laboratory and NBS Values	21
9	Instrument Settings	24
10	Summary of Trace Element Data in SRC I Streams (Equilibrium Set 3)	26
11	Trace Element Reduction in SRC I Relative to Coal . . .	29
12	Material Balance for Trace Elements (Equilibrium Set 3) in SRC I Process	32
13	Materials Balances for SRC I Equilibrium Sets	33
14	SRC Pilot Plant, Vacuum Bottoms - Effects of CO Addition to Products	35
15	Comparisons of Trace Elements in Plant Effluent Waters with Process Waters, and Hamer Marsh Water	40
	APPENDIX I: Trace Element Data for SRC I Equilibrium Set Three (5 tables)	45
	APPENDIX II: Trace Element Data for CO Addition Study and Composites (2 tables)	59
	APPENDIX III: Trace Element Data for Plant Effluent Waters (9 tables).	66

ABSTRACT

Results are presented on a study of the distribution and fate of 34 trace elements in the Solvent Refined Coal Process at the pilot plant located at Fort Lewis, Washington, and operated by The Pittsburg & Midway Coal Mining Co. under contract with the U.S. Department of Energy. Neutron activation analysis was used to determine Ti, V, Ca, Mg, Al, Cl, Mn, As, Sb, Se, Hg, Br, Co, Ni, Cr, Fe, Na, Rb, Cs, K, Sc, Tb, Eu, Sm, Ce, La, Sr, Ba, Th, Hf, Ta, Ga, Zr, and Cu in feed coals, process solvent, Solvent Refined Coal (SRC), mineral residues, wet filter cake, by-product solvents, process and effluent waters and by-product sulfur. The sample points were chosen such that the major process streams were adequately described and that the major input and output materials were included. Atomic absorption spectrophotometry was used to measure the toxic elements Pb, Cd, Be in plant-derived solvents, effluent water and Hamer Marsh water. Specific methods were developed for analysis of a wide range of material compositions. The neutron activation analysis procedures were divided into short and long irradiation procedures for elements with short half lives (less than 3 hours) and intermediate to long half lives (8 hours to 5.2 years).

Data are presented for a third equilibrium set of samples from the SRC-I process and compared to two similar sets analyzed previously. A material balance (or budget) was calculated for each element from the concentration data and the yields of each process fraction.

Data are also presented on a study of carbon monoxide addition to the hydrogen stream and its effect on trace elements, and trace element data on a study of thirty-six plant effluent water samples taken during an SRC-I production run.

INTRODUCTION

In 1974 the research project on the determination of trace element behavior in the Solvent Refined Coal (SRC) processes was initiated at Washington State University. Early work involved the development and testing of methods of trace element determination using neutron activation analysis and atomic absorption spectroscopy. These methods were then used to determine the fate of trace elements in the SRC I process (solid-fuel product) and to measure trace element material balances in the SRC I process. Initial development work and the results obtained from two separate steady-state runs (denoted equilibrium sets) have been reported in a comprehensive report by Filby, Shah, Hunt, Khalil and Sautter (1). Results from the study have also been presented at national meetings and in the literature (2,3,4).

This report which includes research performed during the period August 1, 1976 through July 31, 1977 extends the initial work on the fate of trace elements in the SRC I process to a third steady-state run (herein called equilibrium set 3) in order to resolve some aberrant material balance values for certain elements. Results are also reported of a study of carbon monoxide addition to the hydrogen stream and its effect on trace elements in SRC I. In addition to the trace element data on the SRC I process, this report also includes trace element data on a study of thirty-six plant effluent samples taken during a production run of SRC I for boiler fuel test burning. The data obtained from this study should provide information on probable emissions of trace elements in aqueous effluents from a production-type facility and the effectiveness of the water treatment processes.

A. Sample Collection and Preparation

The sample collection and preparation procedure used for equilibrium set 3 was basically the same as for sets 1 and 2 reported previously (1) except for some minor modifications. The samples were collected in the pilot plant over a 24 hour period from 0800 hrs 9/13/76 through 0800 hrs 9/14/76. These samples and those prepared in the pilot plant laboratory are shown in Table 1. Sampling points are also identified in Table 1.

TABLE 1
PILOT PLANT SAMPLE COLLECTION POINTS

Sample Type	Pilot Plant Sample Point	Matrix
Raw coal	101	solid
Ground coal	105A	solid
Dust collector (coal)	-	solid
Solvent Refined Coal (SRC I)	8101	solid
Vacuum bottoms	lab prepared	solid
Mineral residue	327	solid
Wet filter cake	lab prepared	wet solid
Light oil	408	liquid
Wash solvent	410	liquid
Recycle process water - tank-oil phase	303	liquid
Process solvent	411	liquid
Precoat (filter aid)	341	solid
Recycle process water - tank-aqueous phase	304	aqueous
Bio-unit aeration	9109	aqueous
"B" Carbon filter effluent	9114	aqueous
Hamer Marsh	9117	aqueous
Bio-unit sludge	9111	solid

The pilot plant run or reaction conditions averaged over the 24-hour period are shown in Table 2. As for the previous two equilibrium sets, the sample set was only taken after the plant had achieved an approximate steady-state operation mode. Table 3 shows yields of major products obtained during the sample collection run. After collection, the samples were treated depending on their matrix type, i.e., a) solids, b) organic solvents and oils (liquids) and c) aqueous samples. Each type of sample required different procedures for the sample preparation, storage and shipment. These were:

a) Solid samples, i.e., raw coal, ground coal, wet filter cake, SRC I and vacuum bottoms. Samples were collected in cleaned glass or polyethylene containers. These containers were soaked in dilute HNO_3 for about four hours and then were washed using double distilled water followed by acetone. This step was important to remove any surface impurity or contamination before collecting the sample.

b) Liquid samples, i.e., light oil, recycle solvent, wash solvent, separator oil and total solvent accumulator oil. Samples were collected in cleaned brown glass containers and were tightly capped. Brown containers, which were cleaned as those for solid samples, were selected to prevent any photodecomposition of the organic compounds.

c) Aqueous samples, i.e., process water, effluent water and gas-liquid separator water. These samples presented some special preparation problems. The analysis of dilute aqueous solutions is difficult because many elements

- i) are present at very low concentration,
- ii) may adsorb on the walls of the containers (plastic or glass), and
- iii) may adsorb on suspended materials in the solution.

TABLE 2

RUN CONDITIONS FOR EQUILIBRIUM SET 3

Period: 0800 Hours 9/13/76 through 0800 Hours 9/14/76

Reaction Conditions	Amount	Units
Dehumidified Coal Feed	3325	lbs/hr
Moisture Free Coal Feed	3320	lbs/hr
Solvent Feed	5295	lbs/hr
Slurry Feed	8620	lbs/hr
Solvent to Coal Ratio	1.59	
Space Rate	65.5	lbs coal/(hr)(ft ³ dissolver volume)
Total Gas Feed Rate	305.0	lbs/hr
Hydrogen Feed Rate	115.3	lbs/hr
Gas Mole Weight	3.66	lbs/lb mole
Hydrogen Purity	92.4	v % (50.9 wt %)
Preheater Inlet Pressure	1609	psig
Dissolver Pressure	1534	psig

Temperatures, °F					
Preheater Outlet	Transfer Line 5' from Dissolver	Distance from Dissolver Inlet, ft			
		<u>0</u>	<u>4</u>	<u>8</u>	<u>12</u>
742	746	784	844	851	854

TABLE 3
PRODUCT YIELDS FOR EQUILIBRIUM SET 3

Component	Yield, % MFC*
Hydrogen, H_2	-2.4
Methane, CH_4	2.2
Ethane, C_2H_6	1.1
Propane, C_3H_8	1.0
Butanes, C_4H_{10}	0.4
Carbon Monoxide, CO	-0.2
Carbon Dioxide, CO_2	1.9
Hydrogen Sulfide, H_2S	1.4
Water, H_2O	5.0
Light Oil (B.P. to 380°F)	5.7
Wash Solvent (380°-480°F)	] 4.6
Process Solvent (480°F-1000°F)	
Vacuum Bottoms, SRC I	65.0
Ash	9.1
Unreacted Coal	<u>5.2</u>
	100.0

*Yield in weight percent moisture-free coal (MFC)

The rate and extent of adsorption either on the container surface or on particulates varies from one element to another and depends on the pH of the solution and the presence of complexing agents. The adsorption is often an irreversible process. Therefore, it was essential to prevent trace element precipitation or adsorption on the container walls and to remove suspended material from the aqueous solution in order to obtain reliable trace element concentration in these samples. Thus water samples were filtered and then frozen immediately after collection (5). Immediately after sampling collection, the sample was filtered through a clean 0.4 μm Nucleopore (General Electric Co.) filter in a hand-pumped Teflon filter assembly. From each sample, four filtered water samples (50 ml each), were collected in separate thin-wall high purity polyethylene bags, tied and quickly frozen. These bags were kept in other thicker wall polyethylene bags to avoid any contamination of the primary container during the preparation process or their storage. Filters and frozen-filtered samples were stored and shipped in dry ice from the pilot plant to the analytical laboratory at the Nuclear Radiation Center. All samples were kept in the freezer until prepared for neutron activation analysis.

B. Neutron Activation Analysis

1. Sample Preparation for Irradiation

For each group of samples, different preparations were employed as follows:

- a) Solid samples: After mixing, samples (about 75 to 200 mg) were accurately weighed into 2/5 dram polyethylene (Olympic Plastic Co.) vials. These vials were cleaned by soaking in dilute nitric acid for at least four hours and then cleaning with double distilled

water followed by acetone. This was required to remove any surface impurities or contaminations from the vials before use. All sample preparation was performed in a clean-air hood (Agnew-Higgins Model 43) to minimize contamination from airborne dust.

b) Liquid samples: Preparation of oils and organic solvents for irradiation was dependent on the irradiation period. Two irradiation times were used in this study. For short irradiations (1 to 10 minutes) samples were taken and weighed carefully in 2/5 dram cleaned (as for solid samples) polyethylene vials. For the longer irradiations, about 2 to 3 g of liquid samples were transferred carefully with disposable syringes (washed with distilled water and dried) to specially prepared open quartz vials. These vials were cleaned by soaking in double-distilled nitric acid for 24 hrs and subsequently washed with double-distilled water and acetone. Each quartz vial was carefully inserted into a pretreated two-dram snap-top polyethylene vial and heat sealed. Special precautions were taken to prevent any tipping of the vials' contents during handling or irradiation. Also, the 2/5 dram vials were heat sealed and double encapsulated in pre-cleaned 2-dram sealed polyethylene vials.

c) Aqueous samples: For the aqueous samples, the very low concentration of many elements required larger sample volumes (e.g., 50 ml) for analysis in order to get reliable results with good sensitivity and precision. As the irradiation positions in the reactor core are of limited size and the reactor neutron flux varies even over small distances, bulk samples could not be irradiated. Therefore, the concentration of aqueous samples by vacuum freeze-drying was adopted. Frozen aqueous samples which were received from the plant in thin

polyethylene bags were packaged into new polyethylene bags to avoid any contamination during drying. A Virtis mechanically refrigerated freeze-dryer was used, each sample was freeze-dried and the thin polyethylene bag and its residue contents were transferred into a cleaned 2/5 dram polyethylene vial. The vial was then heat sealed and transferred to a 2-dram vial as described before.

d) Standard samples: Several elemental standards were used depending on the element of interest.

- i) Four mixed aqueous standards of known concentrations, one containing Na, K and Br, the second containing Ba and Sr, the third containing Cr, Cs, Co, Fe and Zn, and the fourth containing Se, Sc, Ni and Rb were used. A 300 μ l aliquot of each standard was dried on high purity BioSil (BioRad Laboratories) and carefully sealed in 2/5 dram vials. Also, known amounts of individual standard solutions of Cu, As, Ga, La, Au, Sb, U, Ag, Eu and Tb were dried in BioSil and sealed in polyethylene vials. The concentrations of each element used are shown in Table 4.
- ii) National Bureau of Standards (NBS) Standard Reference Materials (SRM's) Coal (SRM 1632) and Fly Ash (SRM 1633) were also used as reference standard samples to check the accuracy of the analytical data in this study. Orchard Leaves (NBS SRM 1571) was used as a primary standard for Hg and Mn and as a reference standard for other elements. Bovine Liver (NBS SRM 1577), Tomato Leaves (NBS SRM 1573) and Pine Needles (NBS SRM 1575) were also reference standards for Br, Hg, Cu and other elements.
- iii) U.S. Geological Survey Standard Rocks GSP-1, PCC-1, AGV-1 and BCR-1 were also used as primary and/or reference standards. The rock GSP-1 was used as primary standard for Sm, Ce, Hf, Zr, Ta and Th.
- iv) Specpure (Matthey) Compounds S, CaCO_3 and MgO were used as standards for S, Ca and Mg.

Special care was taken to select each group of elements in the multielement aqueous standards (or solid reference standards) depending on their radionuclide half-lives and gamma-ray energies. All standards were prepared and packaged in cleaned 2/5 dram polyethylene

vials and doubly encapsulated in two-dram vials as described for solid and aqueous samples.

TABLE 4
WEIGHTS OF ELEMENTS IN LIQUID STANDARDS (300 μ l)

Element	Amount (μ g)	Element	Amount (μ g)
Mix-1			
Cr	42.12	Ag	13.28
Cs	37.41	As	24.9
Co	17.70	Au	7.10
Fe	2102	Cl	902.4
Zn	1224	Cu	21.60
Mix-2			
Sc	1.869	Ga	21.90
Se	23.37	Eu	1.883
Ni	2510	I	38.11
Rb	241.7	La	37.52
		Sb	26.14
Mix-3			
Ba	1876		
Sr	2398	Tb	9.86
		Ti	738.8
Mix-4		U	27.3
Na	51.98		
K	1208		
Br	16.78	V	30.38

2. Irradiation of Samples

Two irradiation periods were used depending on the half-lives of the induced radionuclides measured for elemental analysis. These irradiation times and the elements determined are shown in Table 5. Additional information on the nuclear reactions, radionuclides, half-life, thermal cross-section and energy levels of the emitted gamma rays are listed in Table 6 (6). All samples and corresponding standards were irradiated in the Washington State University TRIGA MARK III-fueled research reactor for a total integrated neutron flux of 3.6×10^{15} neutrons cm^{-2} on a rotator assembly for short irradiations, and 2.5×10^{17} neutrons cm^{-2} for long irradiations. In the reactor graphite-reflector irradiation position, the thermal neutron flux changes horizontally and vertically; therefore, samples were mounted on a rotator assembly. The assembly was rotated about a vertical axis during irradiation at the rate of 1 RPM to obtain a uniform horizontal neutron flux distribution. Samples and standards were arranged in tiers. Iron standard solutions dried on BioSil were used for the long irradiations as flux monitor, while Orchard Leaves (NBS) standard was used in the short irradiations.

3. Post-Irradiation Treatment

Samples and standards were left after irradiation for a suitable decay period (depending on the half-lives of the radionuclides as shown in Tables 5 and 6 to allow unwanted short-lived nuclides to decay). The polyethylene vials containing standards and solid or freeze-dried aqueous samples were washed with water to remove any outer contamination and were transferred to new non-irradiated two dram vials for counting. For short irradiation samples, solid, liquid or aqueous samples and their corresponding standards were transferred immediately to new

TABLE 5
IRRADIATION PROCEDURES

Irradiation Time	Decay Time	Counting Time	Elements Determined
5-10 minutes	a) 1 minute	180 seconds	Ti, V, Mg, Ca, Al, S
	b) 30 minutes	1,000 seconds	Cl, Mn, I
8 hours	a) 14 hours	4,000 seconds	As, Br, Na, K, Sm, La, Ga, Cu, Au
	b) 21 days	80,000 seconds	Sb, Se, Hg, Ni, Co, Cr, Fe, Rb, Cs, Sc, Tb, Eu, Ce, Sr, Ba, Th, U, Hf, Ta, Zr, Zn and Ag

TABLE 6
NUCLEAR DATA ON NUCLIDES MEASURED

Element	Nuclear Reaction and Product	Half Life	$\sigma_{th} f^1$ (barns)	γ -Rays ² Measured (keV)
Al	$^{27}\text{Al}(n,\gamma)^{28}\text{Al}$	2.3 m	0.24	1,779
V	$^{51}\text{V}(n,\gamma)^{52}\text{V}$	3.8 m	4.88	1,434
S	$^{36}\text{S}(n,\gamma)^{37}\text{S}$	5.07 m	.000014	3,105
Ti	$^{50}\text{Ti}(n,\gamma)^{51}\text{Ti}$	5.79 m	0.007	320
Ca	$^{48}\text{Ca}(n,\gamma)^{49}\text{Ca}$	8.8 m	0.002	3,084
Mg	$^{26}\text{Mg}(n,\gamma)^{27}\text{Mg}$	9.5 m	0.0031	1,015
I	$^{127}\text{I}(n,\gamma)^{128}\text{I}$	25 m	6.40	443
Cl	$^{37}\text{Cl}(n,\gamma)^{38}\text{Cl}$	37 m	0.105	1,643
Mn	$^{55}\text{Mn}(n,\gamma)^{56}\text{Mn}$	2.56 h	13.3	847, 1,811
K	$^{41}\text{K}(n,\gamma)^{42}\text{K}$	12.4 h	0.08	1,525
Cu	$^{63}\text{Cu}(n,\gamma)^{64}\text{Cu}$	12.8 h	3.10	511
Ga	$^{71}\text{Ga}(n,\gamma)^{72}\text{Ga}$	14.1 h	2.10	834
Na	$^{23}\text{Na}(n,\gamma)^{24}\text{Na}$	15.0 h	0.53	1,368
As	$^{75}\text{As}(n,\gamma)^{76}\text{As}$	26.3 h	5.0	559
Br	$^{81}\text{Br}(n,\gamma)^{82}\text{Br}$	35.3 h	1.58	777
La	$^{139}\text{La}(n,\gamma)^{140}\text{La}$	40.2 h	8.90	1,597
Sm	$^{152}\text{Sm}(n,\gamma)^{153}\text{Sm}$	46.8 h	56.0	103
Au	$^{197}\text{Au}(n,\gamma)^{198}\text{Au}$	64.7 h	98.8	412
Ba	$^{130}\text{Ba}(n,\gamma)^{131}\text{Ba}$	12.0 d	0.009	496
U	$^{235}\text{U}(n,f)^{140}\text{La}$	12.8 d	4.16	1,596
Rb	$^{85}\text{Rb}(n,\gamma)^{86}\text{Rb}$	18.7 d	0.65	1,079
Th	$^{232}\text{Th}(n,\gamma)^{233}\text{Th} \rightarrow ^{233}\text{Pa}$	27.0 d	7.40	312

Table 6 (continued)

Element	Nuclear Reaction and Product	Half Life	$\sigma_{th} f^1$ (barns)	γ -Rays ² Measured (keV)
Cr	$^{50}\text{Cr}(n,\gamma)^{51}\text{Cr}$	27.8 d	0.73	320
Ce	$^{140}\text{Ce}(n,\gamma)^{141}\text{Ce}$	32.5 d	0.53	145
Hf	$^{180}\text{Hf}(n,\gamma)^{181}\text{Hf}$	42.5 d	3.52	482
Fe	$^{58}\text{Fe}(n,\gamma)^{59}\text{Fe}$	45.6 d	0.003	1,099
Hg	$^{202}\text{Hg}(n,\gamma)^{203}\text{Hg}$	46.9 d	1.19	279
Sb	$^{123}\text{Sb}(n,\gamma)^{124}\text{Sb}$	60.3 d	1.41	1,691
Sr	$^{84}\text{Sr}(n,\gamma)^{85}\text{Sr}$	64.0 d	0.004	514
Zr	$^{94}\text{Zr}(n,\gamma)^{95}\text{Zr}$	64.0 d	0.014	757
Ni	$^{58}\text{Ni}(n,p)^{58}\text{Co}$	71.3 d	-	811
Tb	$^{159}\text{Tb}(n,\gamma)^{160}\text{Tb}$	72.1 d	46.0	879
Sc	$^{45}\text{Sc}(n,\gamma)^{46}\text{Sc}$	83.9 d	13.0	889
Ta	$^{181}\text{Ta}(n,\gamma)^{182}\text{Ta}$	115.0 d	21.0	1,121
Se	$^{74}\text{Se}(n,\gamma)^{75}\text{Se}$	120 d	0.26	265
Zn	$^{64}\text{Zn}(n,\gamma)^{65}\text{Zn}$	243 d	0.23	1,116
Ag	$^{109}\text{Ag}(n,\gamma)^{110m}\text{Ag}$	255 d	1.46	658
Cs	$^{133}\text{Cs}(n,\gamma)^{134}\text{Cs}$	2.05 y	28.0	797
Co	$^{59}\text{Co}(n,\gamma)^{60}\text{Co}$	5.26 y	37.0	1,333
Eu	$^{151}\text{Eu}(n,\gamma)^{152}\text{Eu}$	12.7 y	2,820	1,408

¹ $\sigma_{th} f$ --product of thermal neutron (n, γ) capture cross section (barns) and isotopic fraction of target nuclide. All reactions involved are (n, γ) reactions except the fast-neutron reaction, $^{58}\text{Ni}(n,p)^{58}\text{Co}$ used to measure Ni.

² Energies in keV taken from Filby et al. (6).

two dram vials after the entrapped air was flushed out with a syringe to remove the major part of the ^{41}Ar activity. For long irradiations of liquid samples, samples were carefully transferred from the quartz vials to clean glass petri dishes (5 cm diameter). The quartz vials were rinsed with the minimum amount of benzene to transfer any remaining sample to the petri dish. Powdered silica was slowly added to the petri dish containing the irradiated oil or organic solvent and mixed carefully to form a homogeneous gel. The dish was then sealed and stored at 4°C. Standards were also transferred to glass petri dishes to ensure the same counting geometry in a similar manner except water was used in making the silica gel.

4. Radionuclide Activity Measurement

Ge(Li) gamma-ray spectroscopy was used for all gamma-ray emitting radionuclides activity determinations. The gamma-ray spectrometer consisted of a Ge(Li) detector (Princeton Gamma-Tech; volume, 60 cm³), Princeton Gamma-Tech Preamplifier (Model RG-11), Tennelec 203 BLR amplifier, Nuclear Data Model 2200 4096 channel analyzer and a Kennedy 3112 7-track magnetic tape unit. The system resolution was 2.3 keV measured at the ^{60}Co 1332.5 keV line and the peak/Compton ratio was 30:1.

For the determination of Ti, V, Al, Mg, Ca and S, the analyzer was set to record a 0 to 4096 keV gamma-ray spectrum in 2048 channels. Samples and standards were allowed to decay for 2 min and then were counted for 300 seconds on the gamma-ray spectrometer for ^{51}Ti , ^{52}V , ^{28}Al , ^{27}Mg , ^{49}Ca and ^{37}S . The start and end times of each count relative to the time at the end of the irradiation were noted. For the determination of Cl, Mn, and I, the gamma-ray spectrometer was calibrated to record 0-2048 keV in 1 keV/channel. After 30 min decay time, to allow the short-lived

nuclides ^{28}Al , ^{27}Mg , ^{49}Ca and ^{37}S to decay, samples and standards were counted for at least 1000 seconds for ^{38}Cl , ^{56}Mn and ^{128}I . All spectra were recorded on 7-track IBM compatible magnetic tape.

For long irradiations all samples and standards were counted for approximately 4000 seconds to measure ^{24}Na , ^{42}K , ^{82}Br , ^{153}Sm , ^{64}Cu , ^{76}As , ^{72}Ga , ^{198}Au and ^{140}La radionuclides. After a decay period of not less than 17 days, samples and corresponding standards were counted again for 10,000 to 80,000 seconds to determine ^{141}Ce , ^{75}Se , ^{203}Hg , ^{233}Pa (for Th), ^{51}Cr , ^{181}Hf , ^{131}Ba , ^{85}Sr , ^{134}Cs , ^{58}Co (for Ni), $^{110\text{m}}\text{Ag}$, ^{160}Ib , ^{95}Zr , ^{46}Sc , ^{86}Rb , ^{59}Fe , ^{65}Zn , ^{182}Ta , ^{60}Co , ^{152}Eu , ^{140}La (for U) and ^{124}Sb . The gamma-ray spectra were recorded on magnetic tape.

5. Computation of Analytical Results

In all INAA procedures described above, gamma-ray spectra were transferred from 7-track tape to 9-track IBM magnetic tape and gamma-ray peak areas of all nuclides were computed by the FOURIER spectrum analysis program on the IBM 360-67 computer. The computer programs were previously developed at W.S.U. to identify the radionuclides present from their characteristic gamma-ray energies (7,8,9), calculate net gamma-ray peak areas in sample and standard spectra and convert net peak areas to the appropriate elemental weights by direct comparison to known standards, thus calculating the concentration of elements in the original samples. The concentrations were calculated from

$$C = \frac{A_{sa} W_{st}}{A_{st} W_{sa}}$$

where C = concentration of element in sample ($\mu\text{g/g}$)
 A_{sa} = peak area/sec of nuclide of interest of sample
 A_{st} = peak area/sec of nuclide in standard
 W_{st} = weight (μg) of element in standard
 W_{sa} = weight (g) of sample.

All peak areas (A_{sa} and A_{st}) were corrected for decay to a common point in time. The statistical error associated with radioactivity counting was also calculated by the FOURIER program and the standard deviation of each concentration value calculated. For the very short half-lived radionuclides, such as ^{51}Ti , ^{52}V , ^{28}Al , ^{27}Mg , ^{49}Ca and ^{37}S , decay during counting corrections were applied using Hoffman and Van Camerik method (10).

6. Accuracy and Precision

National Bureau of Standards Reference Materials (SRM's) Coal (SRM 1632) and Coal Fly Ash (SRM 1633) were analyzed with the samples and other standards to evaluate the accuracy and precision of every irradiation run. The analytical concentration values for 28 elements in SRC 1632 and SRM 1633 standards are shown in Table 7 and 8 respectively. The values shown in Tables 7 and 8 were obtained during the period August 1, 1976 through December 31, 1978 both in equilibrium set 3 analysis and SRC II work. The data shown represent three separate irradiations and the averages of two determinations for each irradiation. The comparison of these average values, the average results from a four laboratory comparison which indicates analyses by Washington State University (11,12,13) and the certified NBS values are also shown in Tables 7 and 8. It is clear that the results of this study agree very well with the reported and

certified values as was previously conducted in equilibrium sets 1 and 2 studies (1).

Blank corrections were made for all irradiations. This correction was essential for aqueous samples due to their low elemental concentrations. Blank corrections were applied for vials and polyethylene bags for aqueous samples. A study of the effect of blank values on final analyses was made by using three methods for preparation and counting of coal and fly ash (NBS) standards. These are:

- i) samples in 2/5 dram irradiated vials were transferred to non-irradiated 2 dram vials and counted,
- ii) samples were transferred after irradiation to a petri dish using water to form a homogeneous gel and counted, and
- iii) samples were weighed into polyethylene 3x 4 cm thin-wall bags which were transferred after irradiation to non-irradiated 2/5 dram vials and inserted into 2 dram vials for counting.

Tables 7 and 8 show results obtained from the three methods which indicate no significant difference from different blank values effect. The neutron flux differences between sample and standard due to their different irradiation position levels were corrected by using factors calculated from flux monitors counting. Errors may result if overlapping peak energies are very close and unresolvable, as for 279.1 keV of ^{203}Hg and 279.3 keV of ^{75}Se in mercury determination and for 1115.5 keV of ^{65}Zn and 1120.5 keV of ^{46}Sc for zinc determination. Because there are no other peaks for these mercury and zinc nuclides, appropriate corrections were made for the overlapping peaks by obtaining the interfering peak area from that of another peak of the interfering nuclide (e.g., 264.7 keV of ^{75}Se and 889.3 keV of ^{46}Sc) (7,8). As presented in the nuclear data table, the 1015 keV ^{27}Mg peak energy was used for magnesium determination because ^{56}Mn 846.7 keV peak overlaps with the highest intensity peak 843.8 keV for magnesium nuclide. Also, the 889.3

TABLE 7

ELEMENTAL CONCENTRATIONS IN NBS STANDARD COAL (SRM 1632) AND COMPARISON OF THE AVERAGE
VALUES WITH AVERAGE OF 4-LABORATORY AND NBS VALUES

Element (ppm)	Coal ^{1*}	Coal ^{2*}	Coal ^{3*}	Average ⁺	4-Laboratory Average**	NBS Value**
As	6.35 ± 0.14	6.25 ± 0.12	6.55 ± 0.15	6.41 ± 0.17	5.5 ± 0.4	6.5 ± 1.4
Sb	3.91 ± 0.10	2.82 ± 0.10	4.00 ± 0.15	3.72 ± 0.66	3.8 ± 1.3	3.9 ± 1.3
Se	3.10 ± 0.07	2.90 ± 0.08	2.85 ± 0.12	2.91 ± 0.21	3.4 ± 0.2	3.4 ± 0.2
Br	19.1 ± 0.4	19.5 ± 0.3	19.6 ± 0.4	19.4 ± 0.23	19.3 ± 1.6	19.3 ± 1.9
Ni	17.8 ± 2.8	12.2 ± 2.3	17.2 ± 4.3	16.4 ± 3.8	18.0 ± 3.0	18.0 ± 4.0
Co	5.75 ± 0.28	5.64 ± 0.38	5.32 ± 0.33	5.56 ± 0.25	5.7 ± 0.3	5.7 ± 0.4
Cr	20.4 ± 1.6	17.8 ± 1.9	19.2 ± 2.0	19.4 ± 1.1	19.8 ± 0.8	19.7 ± 0.9
Fe (%)	0.88 ± 0.01	0.86 ± 0.01	0.82 ± 0.01	0.85 ± 0.04	0.85 ± 0.03	0.84 ± 0.04
Na	393 ± 3	383 ± 3	392 ± 3	390 ± 7	414 ± 19	414 ± 20
Rb	20.9 ± 0.9	19.7 ± 1.0	21.1 ± 1.8	20.8 ± 0.9	21.0 ± 2.0	21.0 ± 2.0
Cs	1.31 ± 0.05	1.50 ± 0.03	1.34 ± 0.09	1.36 ± 0.11	1.4 ± 0.1	1.4 ± 0.1
K (%)	0.286 ± 0.007	0.286 ± 0.005	0.279 ± 0.007	0.283 ± 0.004	0.28 ± 0.03	0.28 ± 0.03
Sc	3.73 ± 0.01	3.51 ± 0.01	3.49 ± 0.01	3.59 ± 0.17	3.8 ± 0.2	3.7 ± 0.3
Tb	0.22 ± 0.01	0.24 ± 0.01	0.21 ± 0.02	0.22 ± 0.02	0.23 ± 0.05	0.23 ± 0.05
Eu	0.33 ± 0.06	0.33 ± 0.05	0.30 ± 0.07	0.32 ± 0.02	0.31 ± 0.02	0.33 ± 0.04
Sm	1.68 ± 0.01	1.48 ± 0.01	1.63 ± 0.01	1.60 ± 0.10	1.7 ± 0.1	1.7 ± 0.2
Ce	20.1 ± 0.1	17.9 ± 0.1	18.0 ± 0.1	18.8 ± 1.3	19.5 ± 1.0	19.5 ± 1.0
La	10.7 ± 0.1	10.2 ± 0.2	10.5 ± 0.2	10.5 ± 0.2	10.7 ± 0.5	10.7 ± 1.2
Sr	162 ± 8	166 ± 8	150 ± 13	159 ± 10	156 ± 14	161 ± 16
Ba	358 ± 11	322 ± 14	349 ± 27	347 ± 27	353 ± 25	353 ± 30
Th	3.10 ± 0.01	3.19 ± 0.02	2.90 ± 0.04	3.04 ± 0.14	3.2 ± 0.2	3.2 ± 0.2
U	1.32 ± 0.19	0.90 ± 0.18	1.49 ± 0.41	1.30 ± 0.24	1.41 ± 0.07	1.41 ± 0.2
Hf	0.98 ± 0.02	0.95 ± 0.02	0.94 ± 0.03	0.96 ± 0.04	0.85 ± 0.12	0.96 ± 0.05
Ta	0.25 ± 0.02	0.25 ± 0.02	0.23 ± 0.02	0.24 ± 0.03	0.22 ± 0.02	0.24 ± 0.04

Table 7 (continued)

Element (ppm)	Coal ^{1*}	Coal ^{2*}	Coal ^{3*}	Average ⁺	4-Laboratory Average**	NBS Values**
Ga	5.47 ± 0.26	5.24 ± 0.41	5.28 ± 0.26	5.35 ± 0.20	-	5.4 ± 0.3
Zr	33.2 ± 5.9	29.1 ± 4.9	34.5 ± 9.0	32.9 ± 6.2	-	-
Cu	16.9 ± 0.5	22.9 ± 4.0	15.0 ± 0.5	18.3 ± 4.1	-	18.0 ± 2.0
Zn	38.2 ± 2.7	26.0 ± 2.7	34.5 ± 2.8	33.0 ± 6.3	30.0 ± 10.0	30.0 ± 10.0

¹Counted in 2/5 dram irradiated vials.

²Counted in petri dish.

³Counted in thin wall polyethylene bags.

*Average of two determinations plus counting statistical error.

**Reference

⁺ $\bar{X} \pm S_{\bar{X}}$ where $\bar{X}$ is average of six values.

TABLE 8

ELEMENTAL CONCENTRATIONS IN NBS STANDARD FLY ASH (SRM 1633) AND COMPARISON OF THE AVERAGE
VALUES WITH AVERAGE OF 4-LABORATORY AND NBS VALUES

Element (ppm)	Fly Ash 1*	Fly Ash 2*	Average ⁺	4-Laboratory Average**	NBS value**
As	56.2 ± 0.3	58.7 ± 0.3	57.5 ± 1.6	60.7 ± 2.4	58.0 ± 4.0
Sb	6.27 ± 0.23	7.05 ± 0.21	6.66 ± 0.46	6.9 ± 0.5	6.9 ± 0.6
Se	9.57 ± 0.44	10.2 ± 0.3	9.89 ± 0.56	10.2 ± 1.3	10.2 ± 1.4
Br	7.62 ± 0.39	8.04 ± 0.38	7.83 ± 0.31	12.0 ± 4.0	12.0 ± 4.0
Ni	120 ± 16	119 ± 13	120 ± 4	92.0 ± 6.0	98.0 ± 9.0
Co	36.0 ± 0.3	38.4 ± 0.2	37.2 ± 1.4	41.8 ± 1.3	41.5 ± 1.2
Cr	124 ± 2	127 ± 1	126 ± 2	128 ± 5	127 ± 6
Fe (%)	6.10 ± 0.04	6.26 ± 0.06	6.18 ± 0.10	6.25 ± 0.30	6.20 ± 0.30
Na	2990 ± 8	2940 ± 10	2965 ± 31	3200 ± 300	3200 ± 400
Rb	102 ± 5	134 ± 6	118 ± 18	125 ± 10	125 ± 10
Cs	7.69 ± 0.12	7.77 ± 0.09	7.73 ± 0.10	8.9 ± 1.0	8.6 ± 1.1
K (%)	1.59 ± 0.01	1.63 ± 0.01	1.61 ± 0.02	1.72 ± 0.03	1.61 ± 0.15
Sc	26.3 ± 0.1	26.1 ± 0.1	26.2 ± 0.3	27.3 ± 1.0	27.0 ± 1.0
Tb	2.01 ± 0.07	1.50 ± 0.06	1.75 ± 0.3	1.9 ± 0.3	1.9 ± 0.3
Eu	2.44 ± 0.05	2.37 ± 0.04	2.41 ± 0.08	2.3 ± 0.1	2.5 ± 0.4
Sm	12.6 ± 0.1	12.9 ± 0.1	12.8 ± 0.2	12.4 ± 0.9	12.4 ± 0.9
Ce	146 ± 1	142 ± 1	144 ± 3	146 ± 10	146 ± 15
La	75.5 ± 0.4	79.5 ± 0.5	77.5 ± 2.4	82.0 ± 2.0	82.0 ± 2.0
Sr	1360 ± 42	1420 ± 37	1390 ± 53	1700 ± 300	1700 ± 300
Ba	2640 ± 73	2540 ± 70	2590 ± 70	2700 ± 200	2700 ± 200
Th	23.3 ± 0.1	24.3 ± 0.1	23.8 ± 0.6	25.4 ± 1.5	24.8 ± 2.2
U	11.7 ± 1.4	10.5 ± 1.4	11.1 ± 0.9	12.0 ± 0.5	12.0 ± 0.5
Hf	8.27 ± 0.11	7.58 ± 0.09	7.93 ± 0.44	7.0 ± 1.1	7.9 ± 0.4

Table 8 (continued)

Element (ppm)	Fly Ash 1*	Fly Ash 2*	Average +	4-Laboratory Average**	NBS value**
Ta	1.81 ± 0.09	1.56 ± 0.08	1.69 ± 0.15	1.7 ± 0.1	1.8 ± 0.3
Ga	25.6 ± 0.3	29.6 ± 0.6	27.5 ± 2.4	--	--
Zr	301 ± 30	273 ± 32	285 ± 29	301 ± 20	301 ± 20
Cu	126 ± 15	112 ± 13	119 ± 11	--	--
Zn	226 ± 3	--	226 ± 3	216 ± 25	216 ± 25

¹ Counted in 2/5 gram irradiated vials.

² Counted in petri dish

*Average of two determination plus counting statistical error.

**Reference (1)

$\bar{x} \pm \frac{s}{\sqrt{x}}$ where $\bar{x}$ is average of four values.

keV gamma-ray energy peak was better than 1120.5 keV peak for ^{46}Sc determination due to ^{65}Zn overlapping peak. For ^{64}Cu analysis, 511.0 keV/1368.4 keV ^{24}Na peak area correction had been made.

C. Atomic Absorption Analysis

Water samples from equilibrium set 3 were analyzed by atomic absorption for Pb, Cd and Be. The technique was also used to measure Pb, Cd, and Be in a series of carbon filter effluent samples taken during SRC I production runs. The frozen water samples were acidified with 16 M HNO_3 after partial thawing (1% HNO_3 v/v final conc.). Atomic absorption measurements were made with the carbon rod atomizer (CRA) Varian AA-5 spectrophotometer directly using 3 μl aliquots of the acidified solutions. The procedure of standard additions was used for Pb and Cd, with one standard addition only. Full details of procedures used have been given previously (1).

The standard additions procedure was initially used for Be. This was abandoned in favor of an external standard when it became apparent that Be, if present, was below detection limits and the sample matrix effect on Be was an enhancement of atomic absorption. Thus the use of the external standard results in a slightly high value for the limit. This was acceptable in view of the time saved by avoiding standard additions. A value of 0.0015 absorbance units was chosen as the detection limit for Be measurement.

For Pb the samples showed a severe matrix effect, suppressing the signal by as much as a factor of 6. By changing from step atomization to ramp atomization this suppression was reduced to a factor of about 2. Even though it was evident that these samples contained very little Pb, the standard additions procedure was used to assure that Pb, if present, would be detected. The value of the limit of Pb concentration depends on the sample matrix, optical alignment, and the laboratory procedure. Variation in the limit numbers in the

table should not be taken as indicative of actual Pb levels.

Cadmium was detected in all samples, ranging in concentration from 0.6 to 10 ppb. Incomplete removal of matrix material at the relatively low temperature of the ash cycle has sometimes been a problem in past work with Cd. By going to ramp atomization it has been possible to separate the absorption peaks of background and Cd. Details of the Cd procedure will appear in a separate report.

Instrument settings are summarized in Table 9. Argon at a flow rate setting of 6.5 was used as the inert gas to surround the CRS assembly. Background measurements were made with the H₂ Continuum Lamp. Cooling water was on full force.

TABLE 9
Instrument Settings

	Pb	Cd	Be
Lamp Current, mA	7	4	7
Wave Length, nm	217.0	228.8	234.9
Slit Width, microns	300	300	300
Slit Height, mm	4.3	4.3	4.3
Spectral Band Width, nm	.99	.99	.99
Dry Cycle	7v - 30 sec to 8v - 25 sec		
Ash Cycle	8v - 20 sec	7v - 20 sec	8v - 20 sec
Rate	2	2-1/2	2-1/2
Cut-off	7v	7, 7-1/2v	7-1/2v

RESULTS AND DISCUSSION

A. Equilibrium Set 3 Trace Element Data

Complete trace element data for trace elements determined by neutron activation analysis and atomic absorption spectrophotometry in materials from equilibrium set 3 are shown in Appendix 1. In Appendix 1, the precision values quoted are for single standard deviations derived from counting statistics in the case of the neutron activation analyses and replicate carbon rod firings for the atomic absorption analyses of Pb, Be, and Cd. The data obtained by neutron activation analysis were obtained for all samples whereas the data for Pb, Be, and Cd obtained by atomic absorption are only for the aqueous phases. The data for the process fractions important in obtaining a material balance for each trace element are summarized in Table 10 for 34 elements. No precision values are given in Table 10 since these have been included in Appendix 1. The materials from process streams analyzed were ground coal (GC), Solvent Refined Coal I (SRC), wet filter cake (WFC), light oil (LO), process recycle solvent (PRS), process water (PW), and effluent water (EW). In contrast to the two previous equilibrium sets analyzed (1), equilibrium set 3 did not contain a pyridine insolubles (PI) sample as the representative residue material. Instead, the wet filter cake (WFC) sample was analyzed. Since the pyridine insolubles are obtained by treatment of the wet filter cake with pyridine, the wet filter cake represents the composition of the insoluble SRC residue plus some remaining SRC and process solvent. Inspection of the data in Table 10 shows that most elements, with the exception of Ti, Br, Cl, As, Mn, V and Ga are depleted in SRC I by at least an order of magnitude compared to the input coal. Such depletion is consistent with the data from the first two equilibrium sets (1). As was

Table 10

SUMMARY OF TRACE ELEMENT DATA IN SRC I STREAMS^a
(EQUILIBRIUM SET 3)

Element (ppm)	GC	SRC	WFC	LO	PRS	WS	PW	EW
Ti	600	311	1,450	<1.3	5.01	<0.9	<0.6	<0.3
V	37.4	7.94	127	<0.01	0.19	<0.01	<0.003	0.038
Ca	1,320	82.0	4,660	<10	<9	<6	<6	11.7
Al	0.90**	80.0	3.62**	0.92	1.30	<0.2	0.70	0.23
Cl	293	113	1,110	6.19	66.8	89.3	<1.02	7.97
Mn	23.6	8.9	84.0	<0.001	1.23	0.11	<0.002	0.042
As	5.70	2.17	11.0	0.010	0.38	0.015	0.66	<4*
Sb	2.00	0.17	5.00	<1	6.80*	<2.0	<1.0*	0.49*
Se	3.00	0.070	7.80	0.023	0.019	0.017	0.021	0.85*
Hg	30.0*	<0.14	107*	7.43*	<0.1*	5.66*	12.1*	0.31*
Br	5.20	6.60	18.1	0.018	0.52	0.011	0.007	0.038
Ni	12.6	1.23	31.3	<0.14	<0.4	<0.2	<0.1	0.039
Co	2.80	0.24	7.80	0.010	0.037	0.015	0.007	0.67*
Cr	12.8	3.68	47.3	0.012	4.51	0.020	0.016	<0.003*
Fe	1.65**	214	4.91**	0.86	107	7.59	0.35	0.17
Na	238	5.63	545	0.78	0.38	0.42	0.11	86.9
Rb	15.0	<0.13	59.8	<0.02	<0.07	--	<0.02	<0.002
Cs	0.87	<0.005	2.40	2.89*	3.71*	5.43*	2.25*	<0.001

Table 10 (continued)

Element (ppm)	GC	SRC	WFC	LO	PRS	WS	PW	EW
K	1,830	9.20	6,810	<0.1	<0.1	0.08	0.047	<3.4
Sc	1.90	0.13	4.30	0.063*	5.49*	<0.1*	0.039*	<0.03*
Tb	0.14	0.010	0.32	<0.6*	<2*	<1*	<0.5*	<0.02*
Eu	0.13	0.010	0.56	1.40*	2.00*	2.31*	1.11*	<0.02*
Sm	1.00	0.050	2.80	<0.046*	2.59*	<0.05*	<0.04*	<0.11*
Ce	9.60	0.16	34.2	<7*	<0.01	<9*	<7*	0.34*
La	4.82	0.10	18.0	<0.7	2.87*	<0.7*	<0.3*	<1.5*
Sr	37.0	<1.5	60.0	<0.3	<0.6	<0.5	<0.3	<0.02
Ba	74.0	2.12	102	<0.3	<0.7	<0.6	<0.2	<0.03
Th	1.40	0.060	3.40	<0.7*	5.25*	<1*	<0.7*	<0.04*
Hf	0.48	0.020	1.14	<0.7*	<2*	<1*	<0.6*	<0.04*
Ta	0.15	0.022	0.26	<0.9*	3.36*	<2*	<0.7	0.057*
Ga	3.08	1.26	6.40	6.15*	0.017	<2*	0.49*	<0.02
Zr	64.0	5.10	173	<0.2	--	0.24	<0.14	--
Cu	6.15	--	80.5	0.037	0.28	9.61*	8.93*	<0.01*
Zn	60.3	<1	<33	<0.02	0.50	0.054	<0.02	0.26

NOTE: GC = Ground Coal; WFC = Wet Filter Coal; PRS = Process Recycle Solvent; PW = Process Water; LO = Light Oil; WS = Wash Solvent; EW = Effluent Water.

*Values in ppb.

**Values in percent.

^aError terms are not given in order to reduce space.

observed for equilibrium sets 1 and 2, Ti shows only moderate depletion in SRC (factor of 2) and the Ti content of SRC I (311 ppm) is higher than that of any other trace metal. In order to compare the behavior of the trace elements in equilibrium set 3 to equilibrium sets 1 and 2, the concentration ratios, (SRC I concentration)/(coal concentration) and the percentage depletion in SRC I relative to coal are shown in Table 11. Illinois coals were used in all runs and similar behavior in all sets is shown by most elements. It is clear that most elements in all runs are efficiently removed from coal with 75% or more reduction. Except for Cl, Br and Ti, all elements are significantly depleted (50% or more) in the SRC I product and many elements show 90% or greater reduction. Some depletion differences for certain elements were observed from one set to another. Chromium shows greater reduction (88%) in equilibrium set 1 while the percentage reduction was lower in the equilibrium sets 2 (63%) and 3 (71%). Vanadium in set 2 and As in set 3 showed lower reduction than in the other two sets. Also, the Ta depletion was higher in set 3 than for sets 1 and 2. Gallium decreased by 81% in set 2 but only 50% and 59% reductions were observed in sets 1 and 3 respectively. The reduction of Cl, Mn and Hg were within the 40-71% range. These differences in behavior are presumably related to the forms in which these elements exist in coal and/or possible mineral transformations during the solvent refining process. There is a wide range of SRC/Coal values as shown in Table 11. Values between 0.001 and 0.03 are found for K, Na, Cs and Fe, while Ti, Cl and Hg have values higher than 0.3.

Titanium was the only element that was consistently depleted less than 50% relative to feed coal for all sets. The high content of Ti in SRC I has been noted by other authors (14). The anomalously high Ti content of SRC I and its distinct behavior from other elements is discussed later and a

Table 11
TRACE ELEMENT REDUCTION IN SRC-I RELATIVE TO COAL

Element	Equilibrium Set #1		Equilibrium Set #2		Equilibrium Set #3	
	SRC/Coal	% Reduction	SRC/Coal	% Reduction	SRC/Coal	% Reduction
Ti	0.88	12	0.79	26	0.52	48
V	0.15	85	0.47	53	0.21	79
Ca	0.12	88	0.19	81	0.06	94
Mg	0.08	92	--	--	--	--
Al	0.02	98	0.03	97	0.01	99
Cl	0.62	38	0.34	66	0.39	61
Mn	0.60	40	0.40	60	0.38	62
As	0.16	84	0.11	89	0.38	62
Sb	0.08	92	0.04	96	0.09	91
Se	0.06	94	0.03	97	0.02	98
Hg	0.35	65	0.41	59	--	--
Br	1.70	+70	1.33	+33	1.27	+27
Ni	--	--	--	--	0.10	90
Co	0.04	96	0.05	95	0.09	91
Cr	0.12	88	0.37	63	0.29	71
Fe	0.01	99	0.01	99	0.01	99
Na	0.03	97	0.04	96	0.02	98
Rb	--	--	0.02	98	--	--
Cs	0.03	97	--	--	--	--
K	0.003	100	0.001	100	0.005	100
Sc	0.22	78	0.15	85	0.07	93
Tb	0.12	88	0.09	91	0.07	93
Eu	0.21	79	0.14	86	0.08	92
Sm	0.11	89	0.07	93	0.05	95
Ce	0.02	98	0.02	98	0.02	98
La	0.02	98	0.01	99	0.02	98
Th	0.11	89	0.10	90	0.04	96
Hf	0.16	84	0.12	88	0.04	96
Ta	0.33	67	0.29	71	0.15	85
Ga	0.50	50	0.19	81	0.41	59
Zr	0.25	75	0.08	92	0.08	92
Ba	0.11	89	--	--	0.03	97
Cu	0.10	90	0.08	92	--	--

possible explanation proposed. In all equilibrium sets, there are increases in the Br concentration in SRC I relative to feed coal. The reason for the Br increase may be an undetermined source of contamination. The relatively large increases (70, 33, and 27% for sets 1, 2 and 3 respectively) rule out a simple concentration effect due to removal of mineral matter, assuming that all the Br in the original coal is organically bound and remains so in the SRC product. The relatively low concentrations of Br in the process solvents (1.0, 0.75 and 0.52 ppm for sets 1, 2 and 3 respectively) also indicate that make-up solvent is not a source of Br contamination in the process. A possible explanation is that the filtration step in SRC I process may be the source of contamination. However, the concentration of Br in the filteraid materials used on the rotary filters could not be determined in the equilibrium set 1 and 2 materials because of the very high Na content in these samples. However, the Br content of the precoat material was measured for equilibrium set 3 (Appendix 1) and the value of 0.842 ppm indicates that this is not the source of the Br.

B. Material Balance for Equilibrium Set 3 (SRC I)

The trace element material balances for equilibrium sets 1 and 2 (1) were calculated from measured process yields of SRC I, oils, etc., as a fraction of moisture free coal (MFC). In those sets, the residual material chosen to represent the residue in the process was the pyridine insolubles, derived from wet filter cake (WFC). The WFC was laboratory prepared from the reacted coal solution and was uncontaminated by filteraid, but because the WFC contained process solvent and some SRC I material, it was not used as a basis for the material balance calculations. The equilibrium set 3, however, did not contain pyridine insolubles but only wet filter cake and no plant process yields were available for this material. Hence it was assumed that the K content of

the wet filter cake represented 100% of the K in the coal (MFC). This assumption is reasonable since the K content of the input coal was 1830 ppm, the SRC I K content was 9.20 ppm and that of WFC, 6810 ppm. Using these data a process yield of 26.8% for the wet filter cake was obtained and used in the material balance calculations. Using the measured process yields 65% for SRC I, 5.7% for light oil, 4.6% for wash solvent, 5.0% for process water, and arbitrarily assigned 5% for process solvent (see Ref. 1), and the trace element data from Table 10, the trace element material balances were calculated. These are shown in Table 12. The values for each trace element for each material are the product of the concentrations of the element in the material times the fractional yield.

A comparison of the material balances for each element calculated from all process fractions of equilibrium set 3 with the two previous equilibrium sets is shown in Table 13. The material balances are based on assumption of 100% balance for K in coal. For equilibrium set 1, the values range from 53% (Mg) to 156% (Br). Except for Br (156%) and Mg (71%), all values lie within the range of 81% to 150% with an average of $111 \pm 6\%$. For set 2, the values range from 82% (Cl) to 357% (As). Except for As (357%), Ca (293%), Cr (272%), Ni (248%), Ti (176%) and V (177%), all balances lie within the range 83% to 145% with an average of $114 \pm 6\%$. For equilibrium set 3, the values range from 56% (Ta) to 351% (Cu). Except for Cu (351%) and Br (173%), all balances lie within the range 65% to 129% with an average of $89 \pm 5\%$. Except for the high values noted, the ranges for the 3 sets may be regarded as excellent given the assumptions made and the errors associated with obtaining representative samples from the process streams in a large operating pilot plant.

There are a number of explanations for the high values observed for some elements. The high value for As in set 2 is probably due to an anomalous

TABLE 12

MATERIAL BALANCE FOR TRACE ELEMENTS (EQUILIBRIUM SET 3) IN SRC I PROCESS

Material	SRC	WFC	LO	RPS	WS	RPW	Total	Coal	Mat. Bal.
Fraction (ppm)	0.65	0.268	0.057	0.05	0.046	0.05	1.12	1.00	(percent)
Ti	202	389	--	0.251	--	--	591	600	98.5 ± 14.5
V	5.16	33.9	--	0.010	--	--	39.1	37.4	104 ± 6
Ca	53.3	1,248	--	--	--	--	1,301	1,320	98.6 ± 25.0
Al	52.0	9,700	0.053	0.065	--	0.035	9,752	9,040	108 ± 6
Cl	73.5	296	0.353	3.34	4.11	--	377	293	129 ± 6
Mn	5.79	22.5	--	0.062	0.005	--	28.4	23.6	120 ± 5
As	1.41	2.95	0.001	0.019	0.001	0.003	4.38	5.70	76.9 ± 3.2
Sb	0.111	1.34	--	--	--	--	1.45	2.00	72.5 ± 3.8
Se	0.046	2.09	0.001	0.001	0.001	0.001	2.14	3.00	71.3 ± 4.2
Hg (ppb)	--	28.7	0.424	--	0.260	0.610	29.9	30.0	99.9 ± 12.6
Br	4.29	4.85	0.001	0.026	0.001	--	9.17	5.20	173 ± 9
Ni	0.80	8.39	--	--	--	--	9.19	12.6	72.9 ± 15.8
Co	0.156	2.09	0.001	0.002	0.001	--	2.25	2.80	81.2 ± 3.7
Cr	2.39	12.7	0.001	0.226	0.001	0.001	15.3	12.8	120 ± 5
Fe (%)	0.014	1.32	--	0.001	--	--	1.34	1.65	80.7 ± 4.1
Na	3.66	173	0.045	0.019	0.019	0.005	177	238	74.2 ± 3.6
Rb	--	16.0	--	--	--	--	16.0	15.0	107 ± 10
Cs	--	0.643	--	--	--	--	0.64	0.87	74.0 ± 4.0
K	5.98	1,825	--	--	0.004	0.003	1,831	1,830	100 ± 5
Sc	0.085	1.15	--	--	--	--	1.24	1.90	65.1 ± 3.0
Tb	0.007	0.086	--	--	--	--	0.092	0.14	65.9 ± 4.6
Eu	0.007	0.150	--	--	--	--	0.157	0.13	121 ± 7
Sm	0.03	0.75	--	--	--	--	0.78	1.00	78.3 ± 3.7
Ce	0.104	9.17	--	--	--	--	9.27	9.60	96.6 ± 4.7
La	0.065	4.82	--	--	--	--	4.89	4.82	101 ± 6
Th	0.039	0.911	--	--	--	--	0.95	1.40	67.9 ± 3.2
Hf	0.013	0.306	--	--	--	--	0.319	0.48	66.4 ± 3.6
Ta	0.014	0.070	--	--	--	--	0.084	0.15	56.1 ± 9.7
Ga	0.82	1.72	--	0.001	--	--	2.54	3.08	82.3 ± 4.5
Zr	3.32	46.4	--	--	0.011	--	49.7	64.0	77.6 ± 8.5

TABLE 13

MATERIAL BALANCES FOR SRC I EQUILIBRIUM SETS

Element	Set #1 (%)	Set #2 (%)	Set #3 (%)	Average (%)
Ti	149	176	99	141
V	101	177	104	127
Ca	146	293*	99	123
Mg	53	88	-	71
Al	92	102	108	101
Cl	88	82	129	100
Mn	118	143	120	127
As	106	357*	77	92
Sb	137	118	73	109
Se	119	88	71	93
Hg	93	109	100	100
Br	156	145	173	158
Ni	133	248*	73	103
Co	129	115	81	108
Cr	117	272*	120	119
Fe	112	105	81	99
Na	142	112	74	109
Rb	78	119	107	101
Cs	96	98	74	89
K	100	100	100	100
Sc	95	145	65	102
Tb	81	143	66	97
Eu	94	105	121	107
Sm	98	119	78	98
Ce	105	115	97	106
La	112	104	101	106
Ba	99	118	-	109
Th	97	127	68	97
Hf	101	141	66	103
Ta	94	135	56	95
Ga	110	73	82	88
Zr	128	102	78	103
Cu	140	123	351*	132

NOTE: Material balances as % MFC (moisture free coal).

*Values not considered for the average calculations.

analysis since the other equilibrium sets give good balances for As. If the material balance for As in Set 2 is based on the mineral residue value (81.2 ppm) rather than the apparently anomalous value in the pyridine insolubles, a material balance of 83.6% is obtained. Evidently, the wet filter cake (and derived pyridine insolubles) was not representative for As when the sample was taken. The Sb and Se balances closed well in all sets. The high balances for Ni, Cr, and V in set 2 may be due to corrosion in the construction materials during sampling since problems were encountered in the pilot plant at that time. However, the balances are good in the two other equilibrium sets. Bromine in all sets is consistently high which may be due to a source of contamination and has been discussed previously (1). For Hg, which may be appreciably volatile as Hg^0 under the process conditions, the material balances are 93%, 109% and 100% for sets 1, 2 and 3, respectively, indicating that the Hg balance is excellent for all sets within experimental error. The anomalous value for Cu in set 3 could arise from the erosion of bronze valves in the plant. It can be seen that the alkali metals (Na, K, Cs and Rb), the rare earth elements (La, Ce, Sm, Eu and Tb) and Fe show acceptable material balances for all equilibrium sets in SRC I process.

During the period September 28th to October 6th, 1976, the hydrogen gas stream was adjusted to contain 10% CO in an attempt to produce more process solvent. As a number of elements can form carbonyls, i.e., Co, Cr, Ni, Mn, Fe, etc., (e.g., $\text{Ni}(\text{CO})_4$, $\text{Fe}(\text{CO})_5$, etc.) which could recur with aromatic and aliphatic species produced from coal and concentrate in the solid SRC I, it was decided to compare trace element concentrations of SRC I prior to, and after CO addition. Pilot plant analyses of SRC I indicated that during CO addition the ash content rose from 0.08% to 0.20% and dropped to 0.08% after CO addition. Table 14 shows the elemental concentrations of SRC I produced

TABLE 14

EFFECT OF CO ADDITION ON SRC I PRODUCT

Addition of CO	609 Before	SRC I PRODUCT 610 During	611 After
Ash Content (%)	0.08	0.20	0.08
Ti (ppm)	209 $\pm$ 6	462 $\pm$ 10	386 $\pm$ 8.8
V (ppm)	7.5 $\pm$ 0.08	12.6 $\pm$ 0.14	9.0 $\pm$ 0.09
Ca (ppm)	<55	<65	<45
Mg (ppm)	<111	<134	<134
Al (ppm)	60 $\pm$ 2	290 $\pm$ 4	156 $\pm$ 1.7
Cl (ppm)	117.6 $\pm$ 2.4	123.4 $\pm$ 2.5	16.10 $\pm$ 2.4
Mn (ppm)	14.7 $\pm$ 0.16	10.0 $\pm$ 0.12	9.27 $\pm$ 0.12
I (ppm)	2.23 $\pm$ 0.12	2.0 $\pm$ 0.14	1.7 $\pm$ 0.07

TABLE 14 (Cont.)

EFFECT OF CO ADDITION ON SRC I PRODUCT

Addition of CO	SRC I PRODUCT		
	609 Before	610 During	611 After
As (ppm)	1.22 $\pm$ 0.01	2.0 $\pm$ 0.01	1.6 $\pm$ 0.02
Sb (ppm)	0.086 $\pm$ 0.003	0.25 $\pm$ 0.014	0.07 $\pm$ 0.003
Se (ppm)	0.06 $\pm$ 0.006	0.18 $\pm$ 0.02	0.07 $\pm$ 0.008
Hg (ppb)	-	-	-
Br (ppm)	6.56 $\pm$ 0.03	5.11 $\pm$ 0.04	5.80 $\pm$ 0.06
Ni (ppm)	1.86 $\pm$ 0.20	140 $\pm$ 0.4	1.35 $\pm$ 0.22
Co (ppm)	0.22 $\pm$ 0.002	0.38 $\pm$ 0.006	0.18 $\pm$ 0.002
Cr (ppm)	8.43 $\pm$ 0.05	8.0 $\pm$ 0.1	4.2 $\pm$ 0.04
Fe (ppm)	288 $\pm$ 4	383 $\pm$ 9	162 $\pm$ 3
Na (ppm)	10.61 $\pm$ 0.05	12.3 $\pm$ 0.10	9.12 $\pm$ 0.10
Rb (ppm)	<0.2	<0.4	<0.4
Cs (ppm)	<0.005	0.06 $\pm$ 0.005	<0.006
K (ppm)	4.77 $\pm$ 0.43	5.6 $\pm$ 0.6	<2.1

TABLE 14 (Cont.)

EFFECT OF CO ADDITION ON SRC I PRODUCT

Addition of	SRC I PRODUCT		
	609 Before	610 During	611 After
Sc (ppm)	0.28 ± 0.001	0.98 ± 0.004	0.43 ± 0.001
Tb (ppm)	0.032 ± 0.001	0.014 ± 0.002	0.004 ± 0.006
Eu (ppm)	0.017 ± 0.0004	0.023 ± 0.001	0.016 ± 0.0004
Sm (ppm)	0.14 ± 0.001	0.30 ± 0.002	<0.1
Ce (ppm)	0.25 ± 0.006	0.86 ± 0.03	0.31 ± 0.008
La (ppm)	0.08 ± 0.002	0.16 ± 0.006	0.11 ± 0.008
Sr (ppm)	<2.2	<10	<3.4
Ba (ppm)	1.9 ± 0.5	51.2 ± 9.9	17.4 ± 2.6
Th (ppm)	0.11 ± 0.001	0.45 ± 0.007	0.23 ± 0.002
Hf (ppm)	0.03 ± 0.001	0.14 ± 0.005	0.06 ± 0.002
Ta (ppm)	0.028 ± 0.001	0.092 ± 0.005	0.041 ± 0.002
Ga (ppm)	0.70 ± 0.02	2.0 ± 0.02	1.30 ± 0.02
Zr (ppm)	4.7 ± 0.5	12.1 ± 2.0	5.2 ± 0.6
Cu (ppm)	2.29 ± 0.02	2.8 ± 0.03	1.85 ± 0.03
Zn (ppm)	<0.1	<0.1	<0.1

during the CO addition test period. Firstly, the increase in the ash content is paralleled by an increase in most trace elements in the SRC I obtained during CO addition compared to SRC I obtained prior to and after CO addition. This is indicated for trace elements which would not be expected to form carbonyl-type compounds, e.g. Ti, Al, V, As, Sb, Se, K, rare earths, Ta, and Zr. There is a corresponding increase in Fe and Co, but not in Mn, Cr, Ni levels. The effects of CO addition are thus not clearly related to carbonyl formation but are related in some way to increased ash content. These effects should be investigated further. Data on laboratory prepared Vacuum Bottoms show trends similar to the SRC I data and are included in Appendix II.

Also included in Appendix II are data on four 2-month composite samples: SRC I, mineral residue, ground coal, and sulfur. These were obtained in July-August, 1976.

C. Plant Effluent Water Analysis

In order to operate in an environmentally acceptable manner, the pilot plant water wastes, i.e., process waters, coolant water, run-off water, etc., are treated by a combination of $Al(OH)_3$ flocculation, biosludge treatment, and activated carbon filtration prior to discharge into Hamer Marsh. Appendix I shows trace element data for process water, bio-unit aeration water, plant effluent water, and Hamer Marsh water. As was observed for equilibrium sets 1 and 2 the high Hg, Se, and As levels seen in the process water have been reduced by the treatment process to essentially natural levels (Hamer Marsh).

In an attempt to evaluate the emission of trace elements in plant effluent waters during production runs of SRC I, the plant effluent from the carbon filtration system was sampled during production runs of SRC I (period July - September 1976) and the neutron activation analysis data for each

sample (one or two collection runs) are shown in Appendix III. Also shown in Appendix III are the values for atomic absorption determination of Pb, Cd, and Be for each sample.

To illustrate the relationship of trace element contents of process waters, plant effluent waters, and Hamer Marsh water the mean trace element contents of three SRC I process waters (equilibrium sets 1 - 3), three Hamer Marsh waters, 3 plant effluent waters, and line supply water are shown in Table 15.

Inspection of the data in Table 15 show that, except for Ni, there are no statistically significant differences between the trace element composition of the plant effluent water and Hamer Marsh water. The process water, on the other hand, shows significantly higher values of Hg, Se and Cr than does plant effluent water, thus indicating that the treatment and dilution process is satisfactory for trace element removal from process waters.

TABLE 15

COMPARISONS OF TRACE ELEMENTS IN PLANT EFFLUENT
WATERS WITH PROCESS WATERS, AND HAMER MARSH WATER

Element [*]	Process Waters ^{**}	Plant Effluent Water ^{**} (Pt 9114)	Hamer Marsh Water [*]	Line Supply
Ti (ppm)	<0.3	<1.0	<0.3	<0.1
V	<10	26 ± 19 (34)	<50	1.0
Ca (ppm)	7.11 (1)	9.0 ± 3.2 (33)	7.9 ± 5.3	7.0
Al	470 ± 99	630 ± 640 (34)	390 ± 280	160
Cl (ppm)	36.2 ± 21.4	3.9 ± 1.2	5.7 ± 4.9	2.5
Mn	30 ± 14	32 ± 15	21 ± 9	2.0
As	7.6 ± 2.7	<10	<5	<5
Sb	0.66 ± 0.34	0.63 ± 0.38	1.3 ± 1.1	0.12
Se	453 ± 400	0.64 ± 0.32	0.53 ± 0.33	<0.1
Hg	75 ± 47	0.54 ± 0.39	0.60 ± 0.35	0.30
Br	24 ± 12	42.2 ± 7.5	29 ± 6.6	26.8
Ba	<100	21 ± 23 (13)	<100	<20
Sc	0.13 ± .07 (2)	0.022 ± 0.011 (26)	0.065 ± 0.06	7.4
Zn	440 ± 400	310 ± 250 (26)	73 ± 15	30
Th		0.06 ± 0.14 (13)		0.01
Ni	9.0 ± 7.1 (2)	40 ± 26	8.3 ± 3.2	10
Cu (ppm)	0.24 (1)	1.2 ± 0.8 (20)	<0.03	0.20
Na (ppm)	2.9 ± 3.1	61 ± 35	31 ± 21	5.3
Rb	1.3 ± 0.9	1.2 ± 0.8 (19)	0.61 ± 0.42	<1
Cs	0.04 ± .02	0.062 ± 0.024 (28)	0.04 ± .04	0.02
K (ppm)	0.46 ± 0.37 (2)	7.3 ± 3.9 (5)	0.93 (1)	0.9
Co	0.26 ± 0.14	0.70 ± 0.47	0.35 ± .10	0.22
Cr	9.4 ± 2.8	3.5 ± 1.8 (33)	6.2 (1)	3.3
Fe (ppm)	0.62 ± 0.62	0.18 ± 0.10	0.47 ± 0.31	0.07
Pb		1.4 ± 0.6 (9)		1.3
Cd		2.8 ± 2.1	0.15 (1)	1.5
Be		<0.7	<10	<0.2

* and ** (See Notes next page)

Notes to Table 15:

* All values in ppb except where noted

** Replicates: Process waters 3] except where noted
Plant effluent 35] in parentheses
Hamer Marsh 3]

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APPENDIX I

TRACE ELEMENT DATA FOR SRC I EQUILIBRIUM SET THREE

TABLE 1
SRC PILOT PLANT, EQUILIBRIUM SET 3

	Raw Coal (572-05)	Ground Coal (572-06)	Solvent Refined Coal (572-11)	Mineral Residue (572-09)	Vacuum Bottom (580)
Ti (ppm)	410 $\pm$ 75	600 $\pm$ 65	311 $\pm$ 17	2,956 $\pm$ 540	218 $\pm$ 23
V (ppm)	97.5 $\pm$ 2.0	37.4 $\pm$ 1.1	7.94 $\pm$ 0.10	216.0 $\pm$ 5.5	7.5 $\pm$ 0.2
Ca (ppm)	1,195 $\pm$ 200	1,320 $\pm$ 285	82 $\pm$ 23	17,280 $\pm$ 1,175	<200
Mg (ppm)	<1500	<1500	<165	<4000	<200
Al (ppm)	9,170 $\pm$ 117	9,040 $\pm$ 134	80 $\pm$ 3	5.9 $\pm$ 1.2(%)	110 $\pm$ 5
Cl (ppm)	283 $\pm$ 5	293 $\pm$ 4	113 $\pm$ 4	1,113 $\pm$ 51	29 $\pm$ 1.5
Mn (ppm)	22.5 $\pm$ 0.22	23.6 $\pm$ 0.24	8.9 $\pm$ 0.2	138 $\pm$ 1.2	2.9 $\pm$ 0.14
I (ppm)	1.4 $\pm$ 0.21	1.1 $\pm$ 0.14	1.21 $\pm$ 0.1	<0.8	2.6 $\pm$ 0.11

TABLE 1 (Cont.)
SRC PILOT PLANT, EQUILIBRIUM SET 3

	Raw Coal (572-05)	Ground Coal (572-06)	Solvent Refined Coal (572-11)	Mineral Residue (572-09)	Vacuum Bottom (580)
As (ppm)	7.2 $\pm$ 0.08	5.7 $\pm$ 0.08	2.17 $\pm$ 0.05	75.2 $\pm$ 0.8	<5
Sb (ppm)	3.80 $\pm$ 0.04	2.0 $\pm$ 0.04	0.17 $\pm$ 0.003	9.8 $\pm$ 0.20	0.04 $\pm$ 0.003
Se (ppm)	4.47 $\pm$ 0.08	3.0 $\pm$ 0.08	0.07 $\pm$ 0.005	16.33 $\pm$ 0.46	0.06 $\pm$ 0.001
Hg (ppb)	53.5 $\pm$ 4.0	30.0 $\pm$ 2.5	<14	<10	<14
Br (ppm)	4.9 $\pm$ 0.17	5.2 $\pm$ 0.18	6.6 $\pm$ 0.14	--	3.50 $\pm$ 0.13
Ni (ppm)	16.0 $\pm$ 1.1	12.6 $\pm$ 1.6	1.23 $\pm$ 0.12	121.4 $\pm$ 12.2	1.40 $\pm$ 0.17
Co (ppm)	3.78 $\pm$ 0.024	2.80 $\pm$ 0.023	0.24 $\pm$ 0.002	25.0 $\pm$ 0.18	0.28 $\pm$ 0.004
Cr (ppm)	13.7 $\pm$ 0.14	12.8 $\pm$ 0.17	3.68 $\pm$ 0.03	144.7 $\pm$ 1.42	1.83 $\pm$ 0.02
Fe (ppm)	16,580 $\pm$ 245	16,490 $\pm$ 247	214.2 $\pm$ 2.8	123,000 $\pm$ 1,830	81.3 $\pm$ 1.7
Na (ppm)	154.8 $\pm$ 1.14	238 $\pm$ 1.7	5.63 $\pm$ 0.13	2988 $\pm$ 61	<10
Rb (ppm)	14.7 $\pm$ 0.55	15.0 $\pm$ 1.0	<0.13	71.3 $\pm$ 4.5	<0.2
Cs (ppm)	0.85 $\pm$ 0.008	0.87 $\pm$ 0.014	<0.005	4.81 $\pm$ 0.09	4.5 $\pm$ 1.1
K (ppm)	1642 $\pm$ 25	1826 $\pm$ 28	<10	8881 $\pm$ 390	<10

TABLE 1 (Cont.)

SRC PILOT PLANT, EQUILIBRIUM SET 3

	Raw Coal (572-05)	Ground Coal (572-C6)	Solvent Refined Coal (572-11)	Mineral Residue (572-09)	Vacuum Bottom (580)
Sc (ppm)	2.0 $\pm$ 0.003	1.9 $\pm$ 0.008	0.13 $\pm$ 0.0004	13.7 $\pm$ 0.06	0.13 $\pm$ 0.0005
Tb (ppm)	0.13 $\pm$ 0.004	0.14 $\pm$ 0.006	0.010 $\pm$ 0.001	670 $\pm$ 36	<0.002
Eu (ppm)	0.15 $\pm$ 0.002	0.13 $\pm$ 0.003	0.010 $\pm$ 0.0003	1.29 $\pm$ 0.02	0.011 $\pm$ 0.0004
Sm (ppm)	1.16 $\pm$ 0.011	1.0 $\pm$ 0.010	0.05 $\pm$ 0.003	10.5 $\pm$ 0.13	<0.1
Ce (ppm)	9.53 $\pm$ 0.04	9.60 $\pm$ 0.07	0.16 $\pm$ 0.04	106.0 $\pm$ 0.5	0.13 $\pm$ 0.006
La (ppm)	4.50 $\pm$ 0.10	4.82 $\pm$ 0.10	0.10 $\pm$ 0.02	48.4 $\pm$ 1.2	<0.1
Sr (ppm)	<20	37 $\pm$ 6.5	<20	558 $\pm$ 40	<20
Ba (ppm)	76.0 $\pm$ 5.9	74.0 $\pm$ 12.0	2.12 $\pm$ 0.54	623 $\pm$ 65	1.9 $\pm$ 0.45
Th (ppm)	1.30 $\pm$ 0.007	1.40 $\pm$ 0.002	0.06 $\pm$ 0.001	10.3 $\pm$ 0.07	0.05 $\pm$ 0.001
Hf (ppm)	0.45 $\pm$ 0.007	0.48 $\pm$ 0.01	0.02 $\pm$ 0.001	3.26 $\pm$ 0.07	0.03 $\pm$ 0.001
Ta (ppm)	0.14 $\pm$ 0.006	0.15 $\pm$ 0.008	0.022 $\pm$ 0.001	0.97 $\pm$ 0.06	0.026 $\pm$ 0.002
Ga (ppm)	2.92 $\pm$ 0.08	3.08 $\pm$ 0.09	1.26 $\pm$ 0.04	17.90 $\pm$ 0.80	<5
Zr (ppm)	112 $\pm$ 4	64 $\pm$ 4.5	5.1 $\pm$ 0.32	558 $\pm$ 34	2.7 $\pm$ 0.41
Cu (ppm)	8.32 $\pm$ 0.10	6.15 $\pm$ 0.07	<10	107.0 $\pm$ 7.5	<10
Zn (ppm)	27.2 $\pm$ 0.3	60.3 $\pm$ 0.6	<1	252.2 $\pm$ 3.0	<0.2

TABLE 2

SRC PILOT PLANT, EQUILIBRIUM SET 3

	Coal Dust (572-07)	Wet Filter Cake (572-10)	Bio-unit Sludge (572-12)
Ti (ppm)	410 $\pm$ 140	1,450 $\pm$ 200	<370
V (ppm)	38.5 $\pm$ 1.7	126.5 $\pm$ 2.7	382 $\pm$ 5
Ca (ppm)	9,210 $\pm$ 900	4,655 $\pm$ 600	3,064 $\pm$ 500
Mg (ppm)	3,071 $\pm$ 1,200	<1400	3,973 $\pm$ 1,000
Al (ppm)	18,630 $\pm$ 1,440	36,240 $\pm$ 299	25,000 $\pm$ 800
Cl (ppm)	475 $\pm$ 12	1,105 $\pm$ 20	663.5 $\pm$ 30.0
Mn (ppm)	85.7 $\pm$ 1.0	84.0 $\pm$ 0.7	64.3 $\pm$ 0.65
I (ppm)	1.9 $\pm$ 0.6	4.4 $\pm$ 0.68	18.1 $\pm$ 2.5

TABLE 2 (Cont.)

SRC PILOT PLANT, EQUILIBRIUM SET 3

	Coal Dust (572-07)	Wet Filter Cake (572-10)	Bio-unit Sludge (572-12)
As (ppm)	28.8 $\pm$ 0.27	11.0 $\pm$ 0.2	3.8 $\pm$ 0.8
Sb (ppm)	3.3 $\pm$ 0.14	5.0 $\pm$ 0.10	<1
Se (ppm)	3.4 $\pm$ 0.09	7.8 $\pm$ 0.17	7.2 $\pm$ 0.13
Hg (ppb)	63 $\pm$ 5	107 $\pm$ 9.2	4,106 $\pm$ 293
Br (ppm)	5.45 $\pm$ 0.32	18.1 $\pm$ 0.48	13.1 $\pm$ 2.1
Ni (ppm)	22 $\pm$ 2	31.3 $\pm$ 5.8	21.2 $\pm$ 1.5
Co (ppm)	7.2 $\pm$ 0.04	7.8 $\pm$ 0.06	3.8 $\pm$ 0.03
Cr (ppm)	19.5 $\pm$ 0.2	47.3 $\pm$ 0.40	71.8 $\pm$ 0.6
Fe (ppm)	37,960 $\pm$ 559	49,100 $\pm$ 391	26,300 $\pm$ 390
Na (ppm)	580 $\pm$ 3	645 $\pm$ 5	6,282 $\pm$ 128
Rb (ppm)	22.0 $\pm$ 0.7	59.8 $\pm$ 2.0	10.2 $\pm$ 1.8
Cs (ppm)	1.34 $\pm$ 0.02	2.4 $\pm$ 0.04	0.20 $\pm$ 0.008
K (ppm)	11.9 $\pm$ 1.9	6,813 $\pm$ 98	887 $\pm$ 86

TABLE 2 (Cont.)

SRC PILOT PLANT, EQUILIBRIUM SET 3

	Coal Dust (572-07)	Wet Filter Cake (572-10)	Bio-unit Sludge (572-12)
Sc (ppm)	3.07 ± 0.01	4.3 ± 0.01	0.80 ± 0.004
Tb (ppm)	0.40 ± 0.008	0.32 ± 0.01	0.11 ± 0.006
Eu (ppm)	0.07 ± 0.001	0.56 ± 0.01	0.07 ± 0.001
Sm (ppm)	3.15 ± 0.03	2.8 ± 0.03	<1
Ce (ppm)	18.6 ± 0.07	34.2 ± 0.2	3.9 ± 0.08
La (ppm)	13.6 ± 0.25	18.0 ± 0.3	1.77 ± 0.5
Sr (ppm)	<100	60 ± 10	<50
Ba (ppm)	125 ± 6	102 ± 12	196 ± 55
Th (ppm)	2.4 ± 0.01	3.4 ± 0.02	1.8 ± 0.02
Hf (ppm)	0.70 ± 0.01	1.14 ± 0.02	0.32 ± 0.01
Ta (ppm)	0.19 ± 0.009	0.26 ± 0.05	0.02 ± 0.004
Ga (ppm)	3.51 ± 0.23	6.4 ± 0.25	7.2 ± 1.4
Zr (ppm)	94.0 ± 5.1	173 ± 13	48.1 ± 3.6
Cu (ppm)	<20	80.5 ± 0.6	<10
Zn (ppm)	766 ± 3	<33	394 ± 1.7

TABLE 3

SRC PILOT PLANT, EQUILIBRIUM SET 3

	Recycle Process Water S.P. 303 (9/13/76) (572-1) Oil Phase	Light Oil S.P. 408 (9/13/76) (572-2)	Wash Solvent S.P. 410 (9/13/76) (572-3)	Process Solvent S.P. 411 (9/13/76) (572-4)
As (ppb)	67.5 ± 0.7	9.54 ± 0.51	15.3 ± 0.6	378 ± 2
Sb (ppb)	<1.04	<1.25	<2.14	6.80 ± 1.01
Se (ppb)	20.9 ± 1.9	22.6 ± 1.9	17.2 ± 3.5	19.0 ± 2.6
Hg (ppb)	12.1 ± 1.3	7.43 ± 1.08	5.66 ± 1.32	<0.101
Br (ppb)	6.67 ± 0.38	17.5 ± 0.9	10.5 ± 0.9	524 ± 6
Ni (ppm)	<0.116	<0.139	<0.241	<0.362
Co (ppb)	7.27 ± 0.35	10.0 ± 0.5	14.6 ± 0.7	37.2 ± 1.0
Cr (ppb)	15.6 ± 2.3	11.9 ± 2.0	19.5 ± 3.0	4513 ± 66
Fe (ppm)	0.347 ± 0.086	0.861 ± 0.179	7.59 ± 0.42	107 ± 3
Na (ppm)	0.105 ± 0.001	0.780 ± 0.005	0.420 ± 0.004	0.375 ± 0.003
Rb (ppb)	<18.9	<23.9	--	<69.4
Cs (ppb)	2.25 ± 0.22	2.89 ± 0.25	5.43 ± 0.47	3.71 ± 0.56
K (ppm)	0.047 ± 0.012	<0.114	0.081 ± 0.020	<0.100

TABLE 3 (Cont.)

SRC PILOT PLANT, EQUILIBRIUM SET 3

	Recycle Process Water S.P. 303 (9/13/76) (572-1) Oil Phase	Light Oil S.P. 408 (9/13/76) (572-2)	Wash Solvent S.P. 410 (9/13/76) (572-3)	Process Solvent S.P. 411 (9/13/76) (572-4)
Sc (ppb)	0.039 ± 0.009	0.063 ± 0.011	<0.095	5.49 ± 0.08
Tb (ppb)	<0.479	<0.575	<1.06	<1.71
Eu (ppb)	1.11 ± 0.11	1.40 ± 0.13	2.31 ± 0.21	1.99 ± 0.18
Sm (ppb)	<0.036	<0.046	<0.053	2.59 ± 0.045
Ce (ppb)	<6.68	<6.64	<9.34	<11.2
La (ppb)	<0.253	<0.742	<0.680	2.87 ± 0.31
Sr (ppm)	<0.256	<0.289	<0.455	<0.556
Ba (ppm)	<0.217	<0.258	<0.642	<0.651
Th (ppb)	<0.665	<0.718	<1.20	5.25 ± 0.36
Hf (ppb)	<0.609	<0.711	<1.15	<1.37
Ta (ppb)	<0.743	<0.877	<1.55	3.36 ± 1.00
Ga (ppb)	0.494 ± 0.147	6.15 ± 0.47	<1.47	16.9 ± 0.5
Zr (ppm)	<0.139	<0.159	0.238 ± 0.060	--
Cu (ppb)	8.93 ± 0.45	37.2 ± 1.8	9.61 ± 1.23	279 ± 2
Zn (ppb)	<21.8	<26.4	53.8 ± 12.3	503 ± 21
Ag (ppb)	<1.93	<2.24	<3.42	<4.98
Au (ppb)	<0.162	<0.088	<0.109	<0.068

TABLE 4
SRC PILOT PLANT, EQUILIBRIUM SET 3

	Precoat at Atmospheric Tank S.P. 341 (9/13/76) (572-8)	Water from Separator Holding Tank #54452 (564)	Oil from Separator Holding Tank #54452 (565)
As (ppb)	473 ± 68	<4.33	273 ± 2
Sb (ppb)	183 ± 5	<2.08	22.4 ± 1.4
Se (ppb)	143 ± 12	282 ± 5	323 ± 6
Hg (ppb)	12.6 ± 1.0	27.2 ± 5.1	0.977 ± 0.080
Br (ppb)	842 ± 167	<7.05	1531 ± 17
Ni (ppm)	7.63 ± 0.37	<0.200	0.780 ± 0.129
Co (ppb)	233 ± 4	13.0 ± 0.7	110 ± 2
Cr (ppb)	9514 ± 137	<10.9	963 ± 16
Fe (ppm)	713 ± 17	<0.786	41.4 ± 1.2
Na (ppm)	1968 ± 22	3.65 ± 0.02	0.461 ± 0.004
Rb (ppb)	1828 ± 85	<43.2	<76.1
Cs (ppb)	94.4 ± 2.3	3.65 ± 0.31	3.96 ± 0.64
K (ppm)	272 ± 34	<0.238	<0.120

SRC PILOT PLANT, EQUILIBRIUM SET 3

	Precoat at Atmospheric Tank S.P. 341 (9/13/76) (572-8)	Water from Separator Holding Tank #54452 (564)	Oil from Separator Holding Tank #54452 (565)
Sc (ppb)	272 ± 1	<0.084	3.89 ± 0.08
Tb (ppb)	44.8 ± 2.1	<0.910	<2.16
Eu (ppb)	29.0 ± 1.0	2.02 ± 0.20	1.92 ± 0.17
Sm (ppb)	1300 ± 14	<0.096	0.757 ± 0.034
Ce (ppb)	1396 ± 8	<6.81	<10.9
La (ppb)	1048 ± 59	<2.15	1.25 ± 0.24
Sr (ppm)	12.2 ± 0.8	<0.423	<0.625
Ba (ppm)	25.8 ± 0.9	<0.462	<0.772
Th (ppb)	231 ± 2	<0.936	3.77 ± 0.38
Hf (ppb)	65.9 ± 1.5	<0.991	1.90 ± 0.39
Ta (ppb)	19.3 ± 1.3	<1.44	1.14 ± 0.33
Ga (ppb)	<1.31	<3.29	80 ± 1
Zr (ppm)	5.26 ± 0.53	--	--
Cu (ppb)	7104 ± 53	0.663 ± 0.001	185 ± 2
Zn (ppb)	<151	62 ± 12	11811 ± 104
Ag (ppb)	132 ± 16	63.6 ± 3.9	<5.52
Au (ppb)	<7.0	<0.029	<0.113

TABLE 5

SRC PILOT PLANT, EQUILIBRIUM SET 3

	"B" Carbon Filter Effluent Point 9114 571-01 (9/76)	Hamer Marsh Point 9117 571-02 (9/76)	Bio-unit Aeration Point (9109) (571-03)	Recycle Process Water Tank Water (571-04)
Tl (ppm)	<0.248	<0.319	<0.307	<0.361
V (ppm)	0.038 ± 0.002	0.089 ± 0.004	<0.039	<0.003
Ca (ppm)	11.7 ± 1.2	14.0 ± 1.4	12.6 ± 0.9	7.11 ± 1.05
Hg (ppm)	<1.82	2.51 ± 0.76	<3.09	<2.74
Al (ppm)	0.232 ± 0.035	0.256 ± 0.075	<0.415	<0.365
Cl (ppm)	7.97 ± 0.30	11.4 ± 0.3	6.35 ± 0.34	59.3 ± 2.2
Mn (ppm)	0.042 ± 0.002	0.030 ± 0.002	<0.034	<0.015
I (ppm)	<3.9	<3.6	0.021 ± 0.007	<0.016
Pb (ppb)	<2.3	<2.3	<1.9	-
Cd (ppb)	1.3 ± 0.2	2.5 ± 0.4	<1.2	-
Be (ppb)	<0.8	<0.9	<0.2	-

TABLE 5 (Cont.)

SRC PILOT PLANT, EQUILIBRIUM SET 3

	Effluent Water Point 9114 (571-01)	Hamer Marsh Water Point 9117 (571-02)	Bio-unit Aeration Water Point 9109 (571-03)	Recycle Process Water Tank Water (571-04)
As (ppb)	<3.84	<6.06	<6.16	5.80 ± 0.18
Sb (ppb)	0.486 ± 0.036	0.911 ± 0.046	1.43 ± 0.05	0.312 ± 0.022
Se (ppb)	0.850 ± 0.050	0.250 ± 0.053	0.415 ± 0.061	287 ± 2
Hg (ppb)	0.308 ± 0.011	0.414 ± 0.013	0.699 ± 0.020	98.4 ± 1.7
Br (ppb)	37.5 ± 2.2	35.9 ± 2.3	59.8 ± 2.5	38.2 ± 0.5
Ni (ppb)	39 ± 2	6 ± 1	10 ± 2	<11
Co (ppb)	0.673 ± 0.017	0.312 ± 0.013	0.458 ± 0.016	0.163 ± 0.008
Cr (ppb)	<2.93	<3.62	<4.15	<4.16
Fe (ppm)	0.174 ± 0.009	0.239 ± 0.011	0.183 ± 0.011	0.227 ± 0.009
Na (ppm)	86.9 ± 8.5	45.0 ± 4.4	55.7 ± 5.4	<0.335
Rb (ppb)	<1.88	<1.99	<3.06	2.30 ± 0.57
Cs (ppb)	<0.104	<0.044	<0.110	<0.103
K (ppm)	<3.44	<3.01	3.76 ± 1.12	<0.129

TABLE 5 (Cont.)

SRC PILOT PLANT, EQUILIBRIUM SET 3

	Effluent Water Point 9114 (571-01)	Hammer Marsh Water Point 9117 (571-02)	Bio-unit Aeration Water Point 9109 (571-03)	Recycle Process Water Tank Water (571-04)
Sc (ppb)	< 0.026	< 0.014	< 0.033	< 0.057
Tb (ppb)	< 0.018	< 0.014	< 0.025	< 0.027
Eu (ppb)	< 0.024	< 0.029	< 0.028	< 0.022
Sm (ppb)	< 0.107	< 0.227	< 0.181	< 0.042
Ce (ppb)	0.343 $\pm$ 0.055	0.265 $\pm$ 0.074	0.430 $\pm$ 0.086	< 1.52
La (ppb)	< 1.47	< 2.81	< 2.43	0.343 $\pm$ 0.063
Sr (ppb)	< 14.9	65.8 $\pm$ 4.5	< 28.1	< 28.0
Ba (ppb)	< 30.9	< 36.8	< 67.8	< 82.6
Th (ppb)	< 0.043	< 0.043	< 0.067	< 0.135
U (ppb)	< 0.362	< 0.422	< 0.511	< 0.393
Hf (ppb)	< 0.035	< 0.030	< 0.071	< 0.078
Ta (ppb)	0.057 $\pm$ 0.011	< 0.038	< 0.051	0.055 $\pm$ 0.007
Ga (ppb)	< 22.2	< 22.2	< 32.2	< 1.18
Zr (ppb)	-	-	-	-
Cu (ppb)	< 8.23	< 3.56	< 10.5	< 51.6
Zn (ppm)	0.263 $\pm$ 0.002	0.069 $\pm$ 0.001	0.365 $\pm$ 0.002	1.005 $\pm$ 0.003
Ag (ppb)	< 0.232	< 1.94	< 8.28	< 0.032
Au (ppb)	< 0.040	< 0.085	0.044 $\pm$ 0.010	0.009 $\pm$ 0.002

APPENDIX II

TRACE ELEMENT DATA FOR CO ADDITION STUDY AND COMPOSITES

TABLE 1
SRC PILOT PLANT, EFFECTS OF
CO ADDITION ON VACUUM BOTTOMS

	606 Before CO	Vacuum Bottoms 607 During CO	608 After CO
Ash Content (%)	0.05	0.24	0.15
Ti (ppm)	192 $\pm$ 28	433 $\pm$ 8.4	425 $\pm$ 17
V (ppm)	9.9 $\pm$ 0.30	11.0 $\pm$ 0.12	11.5 $\pm$ 0.32
Ca (ppm)	160 $\pm$ 49	<55	<90
Mg (ppm)	<200	<167	<180
Al (ppm)	830 $\pm$ 10	260 $\pm$ 3	706 $\pm$ 53
Cl (ppm)	53.5 $\pm$ 13.5	38.3 $\pm$ 1.53	32.3 $\pm$ 2.5
Mn (ppm)	3.67 $\pm$ 0.20	9.37 $\pm$ 0.13	13.56 $\pm$ 0.18
I (ppm)	<1.0	0.83 $\pm$ 0.07	0.67 $\pm$ 0.16

TABLE 1 (Cont.)

SRC PILOT PLANT, EFFECTS OF
CO ADDITION ON VACUUM BOTTOMS

Addition of CO	Vacuum Bottoms		
	606 Before	607 During	608 After
As (ppm)	1.12 ± 0.006	1.55 ± 0.02	2.13 ± 0.03
Sb (ppm)	0.013 ± 0.002	0.044 ± 0.006	0.064 ± 0.007
Se (ppm)	0.07 ± 0.005	0.20 ± 0.02	0.16 ± 0.02
Hg (ppb)	1.56 ± 0.24	<10	<10
Br (ppm)	1.3 ± 0.01	2.34 ± 0.04	2.5 ± 0.06
Ni (ppm)	1.52 ± 0.12	1.4 ± 0.4	2.0 ± 0.5
Co (ppm)	0.28 ± 0.002	0.31 ± 0.004	0.41 ± 0.004
Cr (ppm)	1.72 ± 0.02	3.0 ± 0.04	4.2 ± 0.07
Fe (ppm)	70.4 ± 1.3	92.8 ± 2.6	820 ± 11.0
Na (ppm)	12.3 ± 0.06	10.0 ± 0.11	44.0 ± 0.41
Rb (ppm)	<0.2	<0.4	<1.2
Cs (ppm)	0.006 ± 0.001	<0.02	<0.01
K (ppm)	7.1 ± 0.6	6.88 ± 0.73	11.8 ± 2.3

TABLE 1 (Cont.)

SRC PILOT PLANT, EFFECTS OF
CO ADDITION ON VACUUM BOTTOMS

Addition of CO	606 Before	Vacuum Bottoms 607 During	608 After
Sc (ppm)	0.170 $\pm$ 0.005	1.03 $\pm$ 0.003	1.16 $\pm$ 0.003
Tb (ppm)	0.010 $\pm$ 0.0005	0.009 $\pm$ 0.001	0.07 $\pm$ 0.002
Eu (ppm)	0.008 $\pm$ 0.002	0.031 $\pm$ 0.001	0.033 $\pm$ 0.001
Sm (ppm)	0.070 $\pm$ 0.0004	<0.02	<0.02
Ce (ppm)	0.15 $\pm$ 0.004	0.62 $\pm$ 0.01	0.87 $\pm$ 0.02
La (ppm)	0.06 $\pm$ 0.002	0.20 $\pm$ 0.01	0.25 $\pm$ 0.01
Sr (ppm)	<1.8	<7.3	7.4 $\pm$ 1.8
Ba (ppm)	3.30 $\pm$ 0.60	11.2 $\pm$ 1.4	65.2 $\pm$ 8.8
Th (ppm)	0.08 $\pm$ 0.001	0.52 $\pm$ 0.003	0.55 $\pm$ 0.005
Hf (ppm)	0.03 $\pm$ 0.001	0.15 $\pm$ 0.003	0.18 $\pm$ 0.004
Ta (ppm)	0.02 $\pm$ 0.001	0.09 $\pm$ 0.004	0.10 $\pm$ 0.005
Ga (ppm)	0.58 $\pm$ 0.02	1.72 $\pm$ 0.03	3.0 $\pm$ 0.06
Zr (ppm)	2.02 $\pm$ 0.30	10.6 $\pm$ 1.2	20.0 $\pm$ 2.1
Cu (ppm)	6.6 $\pm$ 0.04	12.1 $\pm$ 0.09	14.2 $\pm$ 0.15
Zn (ppm)	<0.08	<0.3	<0.3

TABLE 2

SRC PILOT PLANT, JULY-AUGUST COMPOSITES

	Solvent Refined Coal 575-01	Mineral Residue 575-02	Ground Coal 575-03	Sulfur 575-04
Ti (ppm)	327.0 $\pm$ 13.3	2642 $\pm$ 544	589 $\pm$ 100	<50
V (ppm)	10.0 $\pm$ 0.2	182.0 $\pm$ 3.6	35.9 $\pm$ 1.0	8.1 $\pm$ 0.25
Ca (ppm)	182 $\pm$ 38	10,260 $\pm$ 890	2492 $\pm$ 370	<580
Mg (ppm)	<358	<4000	<1400	<162
Al (ppm)	150 $\pm$ 3	56,900 $\pm$ 648	12,000 $\pm$ 140	30.0 $\pm$ 3
Cl (ppm)	101.2 $\pm$ 2.5	1081 $\pm$ 55	248.0 $\pm$ 4.5	20 $\pm$ 2
Mn (ppm)	15.3 $\pm$ 0.2	140.0 $\pm$ 1.0	35.0 $\pm$ 0.3	1.85 $\pm$ 0.07
I (ppm)	1.38 $\pm$ 0.14	<21	1.53 $\pm$ 0.28	<0.3

TABLE 2 (Cont.)

SRC PILOT PLANT, JULY-AUGUST COMPOSITES

	Solvent Refined Coal (575-01)	Mineral Residue (575-02)	Ground Coal (575-03)	Sulfur (575-04)
Sc (ppm)	0.74 $\pm$ 0.002	11.0 $\pm$ 0.04	2.87 $\pm$ 0.01	0.12 $\pm$ 0.01
Tb (ppm)	0.011 $\pm$ 0.001	1.26 $\pm$ 0.02	0.37 $\pm$ 0.01	<0.004
Eu (ppm)	0.04 $\pm$ 0.001	1.31 $\pm$ 0.01	0.30 $\pm$ 0.004	<0.01
Sm (ppm)	0.34 $\pm$ 0.007	10.6 $\pm$ 0.14	2.0 $\pm$ 0.02	<0.04
Ce (ppm)	0.63 $\pm$ 0.01	80.2 $\pm$ 0.24	19.1 $\pm$ 0.10	<0.4
La (ppm)	0.24 $\pm$ 0.02	53.0 $\pm$ 1.3	10.8 $\pm$ 0.17	<0.2
Sr (ppm)	<6.4	492.0 $\pm$ 23.9		<16
Ba (ppm)	8.03 $\pm$ 1.45	325.4 $\pm$ 26.2	96.4 $\pm$ 16.02	<12
Th (ppm)	0.28 $\pm$ 0.003	8.5 $\pm$ 0.04	2.1 $\pm$ 0.02	<0.03
Hf (ppm)	0.12 $\pm$ 0.003	2.3 $\pm$ 0.04	0.5 $\pm$ 0.01	0.20 $\pm$ 0.01
Ta (ppm)	0.07 $\pm$ 0.004	0.54 $\pm$ 0.03	0.16 $\pm$ 0.01	<0.005
Ga (ppm)	1.41 $\pm$ 0.06	16.2 $\pm$ 0.83	4.08 $\pm$ 0.10	<0.3
Zr (ppm)	13.2 $\pm$ 1.2	401.6 $\pm$ 21.8	80.4 $\pm$ 6.0	<8
Cu (ppm)	<10	141.9 $\pm$ 1.08	17.03 $\pm$ 0.15	<10
Zn (ppm)	<0.2		48.03 $\pm$ 0.62	14.4 $\pm$ 0.51

TABLE 2 (Cont.)

SRC PILOT PLANT, JULY-AUGUST COMPOSITES

	Solvent Refined Coal (575-01)	Mineral Residue (575-02)	Ground Coal (575-03)	Sulfur (575-04)
As (ppm)	2.73 ± 0.06	68.3 ± 0.8	18.7 ± 0.13	<0.2
Sb (ppm)	0.46 ± 0.04	15.6 ± 1.03	3.16 ± 0.14	<0.01
Se (ppm)	0.11 ± 0.01	14.5 ± 0.32	2.93 ± 0.09	<0.24
Hg (ppb)	<100	<100	73.1 ± 6.2	<100
Br (ppm)	6.30 ± 0.14	10.2 ± 2.0	4.40 ± 0.18	<0.3
Ni (ppm)	1.25 ± 0.35	99.4 ± 6.3	23.9 ± 2.2	<1.5
Co (ppm)	0.38 ± 0.004	29.43 ± 0.18	6.53 ± 0.05	0.09 0.006
Cr (ppm)	5.26 ± 0.05	120.0 ± 1.03	17.32 ± 0.23	<0.35
Fe (ppm)	370 ± 6	$116,110 \pm 713$	$25,070 \pm 374$	56 ± 4
Na (ppm)	13.7 ± 0.22	$3,242 \pm 66$	203.5 ± 1.45	158.3 ± 0.94
Rb (ppm)	<0.5	105.0 ± 3.5	17.1 ± 1.3	<0.3
Cs (ppm)	<0.01	4.50 ± 0.05	0.97 ± 0.02	0.07 0.007
K (ppm)	9.2 ± 2.7	$8,205 \pm 363$	$1,870 \pm 28$	<15

APPENDIX III

TRACE ELEMENT DATA FOR PLANT EFFLUENT WATERS

TABLE 1

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	9/20/76 (54468)	9/21/76 (54475)	9/22/76 (54478)	9/23/76 (55720)	9/24/76 (55725)
Ti (ppm)	<0.3	<0.3	<0.2	<0.2	<0.4
V (ppm)	0.065 ± 0.003	0.062 ± 0.003	0.042 ± 0.002	0.037 ± 0.002	0.040 ± 0.002
Ca (ppm)	6.20 ± 1.1	4.80 ± 1.3	8.50 ± 0.7	9.9 ± 0.8	9.50 ± 0.9
Al (ppm)	0.23 ± 0.02	0.28 ± 0.02	0.24 ± 0.02	0.42 ± 0.02	0.85 ± 0.03
Cl (ppm)	3.8 ± 0.2	4.2 ± 0.2	2.4 ± 0.1	4.1 ± 0.2	3.8 ± 0.2
Mn (ppm)	0.035 ± 0.002	0.037 ± 0.002	0.024 ± 0.002	0.031 ± 0.002	0.029 ± 0.003
I (ppm)	<0.02	<0.02	0.029 ± 0.010	0.012 ± 0.005	0.10 ± 0.015

TABLE 1 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	9/20/76 (54468)	9/21/76 (54475)	9/22/76 (54478)	9/23/76 (55720)	9/24/76 (55725)
As (ppb)	<5.0	<2.5	<3.6	<3.5	2.0 $\pm$ 0.5
Sb (ppb)	1.84 $\pm$ 0.03	0.52 $\pm$ 0.018	2.11 $\pm$ 0.04	0.77 $\pm$ 0.02	0.82 $\pm$ 0.02
Se (ppb)	0.9 $\pm$ 0.04	0.83 $\pm$ 0.034	0.9 $\pm$ 0.05	0.70 $\pm$ 0.04	0.64 $\pm$ 0.03
Hg (ppb)	0.4 $\pm$ 0.01	0.34 $\pm$ 0.01	1.0 $\pm$ 0.03	0.41 $\pm$ 0.01	0.44 $\pm$ 0.01
Br (ppb)	49.0 $\pm$ 1.7	47.4 $\pm$ 1.0	56.4 $\pm$ 1.6	40.4 $\pm$ 1.6	47.0 $\pm$ 1.0
Ni (ppm)	0.033 $\pm$ 0.001	0.08 $\pm$ 0.001	0.043 $\pm$ 0.001	0.02 $\pm$ 0.001	0.10 $\pm$ 0.001
Co (ppb)	0.35 $\pm$ 0.006	0.5 $\pm$ 0.008	0.55 $\pm$ 0.009	1.2 $\pm$ 0.01	0.8 $\pm$ 0.009
Cr (ppb)	2.62 $\pm$ 0.05	2.3 $\pm$ 0.04	1.74 $\pm$ 0.05	19.2 $\pm$ 0.14	3.10 $\pm$ 0.05
Fe (ppm)	0.2 $\pm$ 0.005	0.22 $\pm$ 0.005	0.15 $\pm$ 0.005	0.15 $\pm$ 0.004	0.10 $\pm$ 0.003
Na (ppm)	40.0 $\pm$ 0.2	34.6 $\pm$ 0.1	37.7 $\pm$ 0.2	33.6 $\pm$ 0.12	27.0 $\pm$ 0.1
Rb (ppb)	0.82 $\pm$ 0.15	0.51 $\pm$ 0.13	0.66 $\pm$ 0.20	0.92 $\pm$ 0.13	3.66 $\pm$ 0.21
Cs (ppb)	0.04 $\pm$ 0.004	0.06 $\pm$ 0.004	0.05 $\pm$ 0.006	0.07 $\pm$ 0.004	0.12 $\pm$ 0.005
K (ppm)	3.8 $\pm$ 0.9	<4	<5	<3	<4

TABLE 1 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	9/20/76 (54468)	9/21/76 (54475)	9/22/76 (54478)	9/23/76 (55720)	9/24/76 (55725)
Sc (ppb)	0.02 ± 0.001	0.01 ± 0.0004	0.05 ± 0.001	0.02 ± 0.001	0.04 ± 0.001
Tb (ppb)	<0.01	<0.01	<0.01	<0.01	<0.01
Eu (ppb)	0.008 ± 0.001	0.005 ± 0.001	0.010 ± 0.002	<0.004	0.002 ± 0.0007
Sm (ppb)	<0.16	<0.09	<0.1	<0.1	<0.06
Ce (ppb)	0.03 ± 0.008	<0.1	<0.13	<0.1	<0.1
La (ppb)	<1.4	<0.7	<1	<1	<0.7
Sr (ppb)	<13	<7	<8	<7	<9
Ba (ppb)	<9	<10	<10	4 ± 1	10 ± 3
Th (ppb)	0.01 ± 0.003	0.012 ± 0.003	0.02 ± 0.003	<0.01	<0.01
Hf (ppb)	<0.02	0.01 ± 0.003	<0.02	0.014 ± 0.004	<0.017
Ta (ppb)	<0.02	0.005 ± 0.001	0.02 ± 0.005	0.02 ± 0.003	0.03 ± 0.003
Ga (ppb)	<37	<34	<46	<28	<33
Zr (ppm)	0.03 ± 0.002	0.01 ± 0.001	0.042 ± 0.002	0.013 ± 0.001	0.011 ± 0.001
Cu (ppm)	<0.1	<0.1	<0.1	<0.1	<0.1
Zn (ppm)	0.19 ± 0.001	<1	0.43 ± 0.002	<1	<1

TABLE 2
TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	9/27/76 (55742)	9/28/76 (55755)	9/29/76 (58708)	10/1/76 (58721)	10/7/76 (58759)
Ti (ppm)	<0.3	<0.40	<0.3	<0.3	<0.2
V (ppm)	0.023 ± 0.001	0.031 ± 0.001	0.053 ± 0.001	0.056 ± 0.001	0.031 ± 0.001
Ca (ppm)	8.55 ± 0.73	15.8 ± 1.1	8.57 ± 0.70	6.12 ± 0.65	6.15 ± 0.64
Al (ppm)	0.289 ± 0.020	3.31 ± 0.06	0.254 ± 0.019	0.393 ± 0.011	0.190 ± 0.008
Cl (ppm)	4.74 ± 0.12	4.95 ± 0.18	5.93 ± 0.15	4.22 ± 0.12	3.074 ± 0.115
Mn (ppm)	0.032 ± 0.003	0.036 ± .004	0.073 ± 0.003	0.018 ± 0.003	0.020 ± 0.002
I (ppm)	0.017 ± 0.003	<0.02	<0.008	<0.013	<0.045

TABLE 2 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	9/27/76 (55742)	9/28/76 (55755)	9/29/76 (58708)	10/1/76 (58721)	10/7/76 (58759)
As (ppb)	<6	<4	<6.0	<7	<10
Sb (ppb)	0.60 $\pm$ 0.02	0.92 $\pm$ 0.02	0.54 $\pm$ 0.02	0.71 $\pm$ 0.03	1.0 $\pm$ 0.03
Se (ppb)	0.87 $\pm$ 0.04	0.54 $\pm$ 0.05	0.66 $\pm$ 0.06	0.9 $\pm$ 0.07	0.85 $\pm$ 0.04
Hg (ppb)	0.75 $\pm$ 0.02	0.57 $\pm$ 0.02	1.2 $\pm$ 0.03	1.8 $\pm$ 0.07	0.27 $\pm$ 0.01
Br (ppb)	49.0 $\pm$ 2.4	44.5 $\pm$ 1.2	45.5 $\pm$ 2.3	29 $\pm$ 2	
Ni (ppm)	0.07 $\pm$ 0.001	0.07 $\pm$ 0.001	0.03 $\pm$ 0.001	0.04 $\pm$ 0.02	0.11 $\pm$ 0.001
Co (ppb)	0.76 $\pm$ 0.01	0.64 $\pm$ 0.009	0.45 $\pm$ 0.008	1.14 $\pm$ 0.02	0.84 $\pm$ 0.01
Cr (ppb)	24.0 $\pm$ 0.20	8.2 $\pm$ 0.09	16.2 $\pm$ 0.16	9.3 $\pm$ 0.13	2.5 $\pm$ 0.06
Fe (ppm)	0.27 $\pm$ 0.01	0.18 $\pm$ 0.004	0.27 $\pm$ 0.006	0.22 $\pm$ 0.008	0.16 $\pm$ 0.005
Na (ppm)	47.4 $\pm$ 0.27	42.5 $\pm$ 0.21	46.5 $\pm$ 0.3	44.3 $\pm$ 0.3	--
Rb (ppb)	1.0 $\pm$ 0.15	1.0 $\pm$ 0.24	1.1 $\pm$ 0.2	0.82 $\pm$ 0.2	1.9 $\pm$ 0.2
Cs (ppb)	0.06 $\pm$ 0.003	0.04 $\pm$ 0.007	0.10 $\pm$ 0.006	0.08 $\pm$ 0.01	0.05 $\pm$ 0.005
K (ppm)	<9.7	<5.0	<9	<6	<10

TABLE 2 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	9/27/76 (55742)	9/28/76 (55755)	9/29/76 (58708)	10/1/76 (58721)	10/7/76 (58759)
Sc (ppb)	0.02 ± 0.001	0.03 ± 0.001	<0.002	0.02 ± 0.001	0.03 ± 0.001
Tb (ppb)	<0.01	0.01 ± 0.003	<0.01	<0.01	<0.01
Eu (ppb)	<0.003	0.01 ± 0.002	0.008 ± 0.002	0.05 ± 0.003	0.01 ± 0.001
Sm (ppb)	<0.20	<0.07	<0.15	<0.2	<0.1
Ce (ppb)	<0.13	<0.1	<0.1	<0.3	3.2 ± 0.03
La (ppb)	<1.5	<1.0	<1.5	1.6 ± 0.4	<1
Sr (ppb)	<7	<10	<10	<11	<8
Ba (ppb)	5 ± 1	20 ± 2	40 ± 2	<21	$1,438 \pm 9$
Th (ppb)	<0.01	0.03 ± 0.003	0.02 ± 0.004	<0.04	0.06 ± 0.004
Hf (ppb)	<10	0.01 ± 0.003	0.02 ± 0.004	<0.03	0.21 ± 0.005
Ta (ppb)	0.01 ± 0.003	0.13 ± 0.01	0.025 ± 0.005	<0.03	0.06 ± 0.006
Ga (ppb)	<10	<10	<10	<25	<10
Zr (ppm)	0.01 ± 0.001	0.02 ± 0.002	0.01 ± 0.002	0.02 ± 0.003	0.02 ± 0.002
Cu (ppm)	<0.1	<0.1	<0.1	53 ± 0.03	<0.1
Zn (ppm)	<0.1	0.91 ± 0.004	0.25 ± 0.001	0.31 ± 0.01	0.4 ± 0.002

TABLE 3

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	10/8/76 (58768)	10/8/76 L.S. (52215)	10/11/76 (58783)	10/12/76 (58788)	10/13/76 (58789)
Ti (ppm)	<0.2	<0.1	<0.2	<0.2	<0.2
V (ppm)	0.042 ± 0.001	0.001 ± 0.000	0.043 ± 0.001	0.040 ± 0.001	0.037 ± 0.001
Ca (ppm)	8.594 ± 0.667	7.042 ± 0.448	8.669 ± 0.556	6.524 ± 0.808	8.31 ± 0.96
Al (ppm)	0.844 ± 0.014	0.163 ± 0.007	0.121 ± 0.020	<0.137	0.219 ± 0.016
Cl (ppm)	5.41 ± 0.16	2.47 ± 0.08	5.27 ± 0.16	6.48 ± 0.21	5.23 ± 0.16
Mn (ppm)	0.023 ± 0.003	0.002 ± 0.000	0.016 ± 0.003	0.010 ± 0.002	0.012 ± 0.002
I (ppm)	0.012 ± 0.005	<0.006	--	--	<0.03

TABLE 3 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	10/8/76 (58768)	Line Supply (52215)	10/11/76 (58783)	10/12/76 (58788)	10/13/76 (58789)
As (ppb)	<5	<1.5	<3	<5	<4
Sb (ppb)	0.53 $\pm$ 0.02	0.12 $\pm$ 0.01	0.44 $\pm$ 0.02	0.43 $\pm$ 0.02	0.63 $\pm$ 0.03
Se (ppb)	1.25 $\pm$ 0.04	<0.05	0.63 $\pm$ 0.03	0.9 $\pm$ 0.05	0.9 $\pm$ 0.07
Hg (ppb)	0.42 $\pm$ 0.014	0.3 $\pm$ 0.03	0.54 $\pm$ 0.02	0.5 $\pm$ 0.02	1.7 $\pm$ 0.07
Br (ppb)	55 $\pm$ 2	26.8 $\pm$ 0.6	1.7 $\pm$ 1	43.6 $\pm$ 2	35 $\pm$ 1.6
Ni (ppm)	0.03 $\pm$ 0.001	0.01 $\pm$ 0.001	0.05 $\pm$ 0.001	0.02 $\pm$ 0.001	0.06 $\pm$ 0.002
Co (ppb)	0.42 $\pm$ 0.006	0.22 $\pm$ 0.007	2.9 $\pm$ 0.03	0.34 $\pm$ 0.01	0.7 $\pm$ 0.01
Cr (ppb)	2.1 $\pm$ 0.05	3.3 $\pm$ 0.08	7.0 $\pm$ 0.08	2.8 $\pm$ 0.08	6.6 $\pm$ 0.10
Fe (ppm)	0.14 $\pm$ 0.004	0.07 $\pm$ 0.004	0.13 $\pm$ 0.004	0.14 $\pm$ 0.001	0.2 $\pm$ 0.007
Na (ppm)	42.7 $\pm$ 0.2	5.3 $\pm$ 0.04	21.8 $\pm$ 0.1	31.1 $\pm$ 0.2	30.0 $\pm$ 0.2
Rb (ppb)	0.7 $\pm$ 0.14	<1	<1	<1	2.1 $\pm$ 0.2
Cs (ppb)	0.05 $\pm$ 0.004	0.02 $\pm$ 0.005	0.04 $\pm$ 0.004	0.07 $\pm$ 0.008	0.1 $\pm$ 0.01
K (ppm)	10 $\pm$ 2	0.9 $\pm$ 0.12	<3	<4	7.6 $\pm$ 1.0

TABLE 3 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	10/8/76 (58768)	Line Supply (52215)	10/11/76 (58783)	10/12/76 (58788)	10/13/76 (58789)
Sc (ppb)	0.02 ± 0.001	7.4 ± 0.3	0.02 ± 0.001	0.02 ± 0.001	0.03 ± 0.001
Tb (ppb)	<0.01	<0.01	<0.01	<0.01	<0.01
Eu (ppb)	0.005 ± 0.009	0.01 ± 0.002	0.004 ± 0.001	0.06 ± 0.003	0.05 ± 0.003
Sm (ppb)	<0.14	<0.1	<0.1	<0.3	<0.11
Ce (ppb)	<0.1	0.07 ± 0.02	<0.1	0.3 ± 0.05	0.17 ± 0.03
La (ppb)	<1.2	<0.6	<1	<2	<2
Sr (ppb)	<6	<6	<7	<7	<10
Ba (ppb)	<10	<15	<10	<15	<11
Th (ppb)	0.01 ± 0.003	0.01 ± 0.002	<0.01	<0.02	0.02 ± 0.005
Hf (ppb)	<0.01	<0.02	0.01 ± 0.003	<0.02	<0.03
Ta (ppb)	0.02 ± 0.004	<0.02	0.04 ± 0.006	<0.02	0.02 ± 0.005
Ga (ppb)	<22	<5	<25	<15	<15
Zr (ppm)	0.011 ± 0.001	<0.005	0.007 ± 0.001	0.010 ± 0.002	0.016 ± 0.003
Cu (ppm)	<0.1	0.2 ± 0.01	<0.1	0.4 ± 0.03	0.5 ± 0.05
Zn (ppm)	0.14 ± 0.001	0.03 ± 0.001	0.08 ± 0.001	0.20 ± 0.001	1.16 ± 0.004

TABLE 4
TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	10/14/76 (50201)	10/15/76 (50206)	10/18/76 (50222)	10/19/76 (50228)	10/25/76 (50282)
Ti (ppm)	<0.4	<0.2	<0.3	<0.2	0.131 ± 0.051
V (ppm)	0.037 ± 0.001	0.033 ± 0.001	0.013 ± 0.001	0.012 ± 0.001	0.006 ± 0.001
Ca (ppm)	9.480 ± 0.83	5.11 ± 1.0	8.95 ± 1.257	8.870 ± 0.869	14.4 ± 0.902
Al (ppm)	0.425 ± 0.010	0.179 ± 0.009	0.709 ± 0.029	0.513 ± 0.019	1.398 ± 0.020
Cl (ppm)	5.22 ± 0.15	4.79 ± 0.11	5.04 ± 0.171	2.80 ± 0.078	2.60 ± 0.065
Mn (ppm)	0.017 ± 0.003	0.016 ± 0.002	0.017 ± 0.003	0.015 ± 0.002	0.038 ± 0.002
I (ppm)	<0.011	<0.012	<0.018	<0.013	<0.008

TABLE 4 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	10/14/76 (50201)	10/15/76 (50206)	10/18/76 (50222)	10/19/76 (50228)	10/25/76 (50282)
As (ppb)	<7	<3	<7	<6	<4
Sb (ppb)	0.55 ± 0.02	0.34 ± 0.01	0.50 ± 0.01	0.38 ± 0.02	0.65 ± 0.02
Se (ppb)	1.4 ± 0.08	0.9 ± 0.04	0.6 ± 0.02	0.20 ± 0.03	0.23 ± 0.03
Hg (ppb)	0.77 ± 0.03	0.52 ± 0.02	0.43 ± 0.01	0.17 ± 0.02	0.14 ± 0.01
Br (ppb)	49 ± 2	38 ± 1	38 ± 2	38 ± 2	35 ± 1
Ni (ppm)	0.05 ± 0.001	0.02 ± 0.01	0.022 ± 0.0005	<0.005	0.03 ± 0.001
Co (ppb)	0.56 ± 0.01	0.2 ± 0.005	0.34 ± 0.005	<0.2	0.52 ± 0.007
Cr (ppb)	4.23 ± 0.08	3.4 ± 0.04	3.4 ± 0.04	2.7 ± 0.13	3.3 ± 0.05
Fe (ppm)	0.22 ± 0.003	0.12 ± 0.004	0.08 ± 0.002	0.14 ± 0.008	0.30 ± 0.006
Na (ppm)	37.3 ± 0.2	35.8 ± 0.1	48.5 ± 0.3	38.6 ± 0.3	30.0 ± 0.2
Rb (ppb)	<2.4	0.6 ± 0.1	0.78 ± 0.11	<1.5	2.2 ± 0.23
Cs (ppb)	0.06 ± 0.007	0.03 ± 0.003	0.05 ± 0.003	0.05 ± 0.01	0.08 ± 0.005
K (ppm)	<6	3.0 ± 0.8	<9	<4	<6

TABLE 4 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	10/14/76 (50201)	10/15/76 (50206)	10/18/76 (50222)	10/19/76 (50228)	10/25/76 (50282)
Sc (ppb)	0.03 ± 0.001	0.01 ± 0.0003	0.01 ± 0.0003	<0.002	0.04 ± 0.001
Tb (ppb)	<0.01	<0.01	<0.01	<0.01	<0.01
Eu (ppb)	<0.01	0.006 ± 0.001	0.003 ± 0.0007	0.01 ± 0.002	0.004 ± 0.001
Sm (ppb)	<0.2	<0.1	<0.2	<0.3	<0.1
Ce (ppb)	0.52 ± 0.03	<0.04	0.17 ± 0.013	0.22 ± 0.04	0.07 ± 0.01
La (ppb)	<2	<1	<2	<2	<1
Sr (ppb)	<24	<6	<6	<13	<7
Ba (ppb)	<30	<9	<8	<28	14 ± 4
Th (ppb)	<0.04	0.52 ± 0.02	<0.01	0.04 ± 0.008	<0.01
Hf (ppb)	0.05 ± 0.01	<0.01	<0.01	<0.04	<0.02
Ta (ppb)	<0.05	0.01 ± 0.002	<0.01	<0.03	0.03 ± 0.004
Ga (ppb)	<20	<20	<20	<25	<20
Zr (ppm)	0.05 ± 0.004	0.01 ± 0.001	0.01 ± 0.001	<0.01	0.02 ± 0.002
Cu (ppm)	<0.1	<0.1	<0.1	0.45 ± 0.005	<0.1
Zn (ppm)	<0.1	0.07 ± 0.001	<0.1	0.34 ± 0.002	0.33 ± 0.002

TABLE 5
TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	10/26/76 (50298)	10/27/76 (51994)	10/28/76 (51977)	10/29/76 (51967)	11/1/76 (51929)
Ti (ppm)	<0.20	<0.30	<0.25	<0.26	<0.5
V (ppm)	0.004 ± 0.001	0.002 ± 0.001	0.004 ± 0.001	0.006 ± 0.001	0.008 ± 0.001
Ca (ppm)	7.92 ± 1.29	13.2 ± 0.9	10.8 ± 1.1	9.71 ± 0.77	8.42 ± 1.02
Al (ppm)	1.16 ± 0.022	1.75 ± 0.04	1.55 ± 0.029	1.05 ± .03	0.66 ± 0.024
Cl (ppm)	4.14 ± 0.14	3.79 ± 0.12	3.88 ± 0.13	3.58 ± 0.13	3.71 ± 0.15
Mn (ppm)	0.043 ± 0.003	0.054 ± 0.004	0.065 ± 0.004	0.046 ± 0.004	0.030 ± 0.004
I (ppm)	<0.024	<0.009	<0.011	<0.015	<0.025

TABLE 5 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	10/26/76 (5C298)	10/27/76 (51994)	10/28/76 (51977)	10/29/76 (51967)	11/1/76 (51929)
As (ppb)	<10	<4	<6	<8	<8
Sb (ppb)	0.43 $\pm$ 0.03	0.43 $\pm$ 0.02	0.59 $\pm$ 0.03	0.47 $\pm$ 0.02	0.96 $\pm$ 0.03
Se (ppb)	0.38 $\pm$ 0.03	0.18 $\pm$ 0.03	0.21 $\pm$ 0.05	0.24 $\pm$ 0.02	1.0 $\pm$ 0.05
Hg (ppb)	0.42 $\pm$ 0.03	1.01 $\pm$ 0.05	0.26 $\pm$ 0.04	0.22 $\pm$ 0.01	0.22 $\pm$ 0.01
Br (ppb)	38.5 $\pm$ 3.0	37.3 $\pm$ 2	38.4 $\pm$ 1.8	44.7 $\pm$ 2.3	46.4 $\pm$ 2.5
Ni (ppm)	0.05 $\pm$ 0.002	0.05 $\pm$ 0.002	0.02 $\pm$ 0.002	0.02 $\pm$ 0.001	0.02 $\pm$ 0.001
Co (ppb)	0.7 $\pm$ 0.02	0.31 $\pm$ 0.01	0.56 $\pm$ 0.02	0.46 $\pm$ 0.01	0.46 $\pm$ 0.01
Cr (ppb)	2.1 $\pm$ 0.09	3.7 $\pm$ 0.1	3.9 $\pm$ 0.22	2.5 $\pm$ 0.08	2.4 $\pm$ 0.09
Fe (ppm)	0.11 $\pm$ 0.006	0.52 $\pm$ 0.01	0.15 $\pm$ 0.011	0.10 $\pm$ 0.005	0.20 $\pm$ 0.01
Na (ppm)	118.0 $\pm$ 0.7	30.8 $\pm$ 0.2	44.7 $\pm$ 0.3	56.1 $\pm$ 0.4	81.7 $\pm$ 0.5
Rb (ppb)	<1	<1	<2.5	<1	<2
Cs (ppb)	<0.03	<0.04	<0.06	0.03 $\pm$ 0.006	0.07 $\pm$ 0.007
K (ppm)	12.0 $\pm$ 3.4	<2	<4	<6	<16

TABLE 5 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	10/26/76 (50298)	10/27/76 (51994)	10/28/76 (51977)	10/29/77 (51967)	11/1/76 (51929)
Sc (ppb)	0.009 ± 0.001	<0.002	<0.003	0.01 ± 0.0005	<0.002
Tb (ppb)	<0.01	<0.01	<0.01	<0.01	<0.01
Eu (ppb)	0.007 ± 0.002	0.005 ± 0.001	<0.013	0.008 ± 0.001	0.045 ± 0.003
Sm (ppb)	<0.2	<0.1	<0.3	<0.4	<0.14
Ce (ppb)	0.27 ± 0.04	0.34 ± 0.05	<0.3	0.5 ± 0.04	0.61 ± 0.04
La (ppb)	<3	<2.0	<2	<3	<2
Sr (ppb)	<7	<10	<23	<5	<9
Ba (ppb)	<12	<16	<47	<13	<20
Th (ppb)	<0.022	<0.024	<0.05	<0.02	0.02 ± 0.006
Hf (ppb)	<0.02	<0.03	<0.07	<0.02	<0.03
Ta (ppb)	<0.02	0.02 ± 0.005	<0.06	<0.01	<0.02
Ga (ppb)	<25	<20	<25	<25	<35
Zr (ppm)	0.01 ± 0.002	0.01 ± 0.002	0.02 ± 0.005	0.007 ± 0.002	0.02 ± 0.002
Cu (ppm)	2.5 ± 0.2	0.28 ± 0.01	0.60 ± 0.02	0.95 ± 0.03	1.2 ± 0.1
Zn (ppm)	0.14 ± 0.002	0.02 ± 0.005	<0.06	<0.01	<0.02

TABLE 6
TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	11/2/76 (51920)	11/3/76 (59675)	11/4/76 (52023)	11/5/76 (52052)	11/8/76 (59401)
Tl (ppm)	<0.3	<0.4	<0.5	<0.7	<0.5
V (ppm)	0.004 ± 0.006	0.010 ± 0.001	0.015 ± .001	0.005 ± 0.002	0.006 ± 0.001
Ca (ppm)	6.68 ± 0.97	10.2 ± 1.3	9.02 ± 1.62	7.20 ± 1.56	8.10 ± 1.2
Al (ppm)	0.829 ± 0.019	0.353 ± 0.053	0.378 ± 0.029	0.765 ± 0.153	0.588 ± 0.054
Cl (ppm)	4.52 ± 0.22	3.17 ± 0.22	3.81 ± 0.20	2.45 ± 0.30	2.95 ± 0.19
Mn (ppm)	0.043 ± 0.005	0.042 ± .006	0.056 ± 0.007	0.052 ± 0.006	0.024 ± 0.006
I (ppm)	<0.02	<0.04	<0.03	<0.07	<0.03

TABLE 6 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	11/2/76 (51920)	11/3/76 (59675)	11/4/76 (52023)	11/5/76 (52052)	11/8/76 (59401)
As (ppb)	<9	<9	<6	<9	<11
Sb (ppb)	0.61 $\pm$ 0.03	0.41 $\pm$ 0.02	0.36 $\pm$ 0.02	0.40 $\pm$ 0.02	0.51 $\pm$ 0.03
Se (ppb)	0.58 $\pm$ 0.06	0.73 $\pm$ 0.06	0.51 $\pm$ 0.05	0.20 $\pm$ 0.02	0.50 $\pm$ 0.07
Hg (ppb)	0.25 $\pm$ 0.03	0.30 $\pm$ 0.02	0.17 $\pm$ 0.01	0.30 $\pm$ 0.03	0.40 $\pm$ 0.03
Br (ppb)	36.8 $\pm$ 3.6	47.3 $\pm$ 3.2	23.0 $\pm$ 2.0	42.3 $\pm$ 2.3	42.4 $\pm$ 3.1
Ni (ppm)	0.03 $\pm$ 0.002	0.04 $\pm$ 0.001	0.008 $\pm$ 0.001	0.01 $\pm$ 0.001	0.04 $\pm$ 0.001
Co (ppb)	1.3 $\pm$ 0.02	0.60 $\pm$ 0.01	0.66 $\pm$ 0.01	1.0 $\pm$ 0.01	0.84 $\pm$ 0.01
Cr (ppb)	3.3 $\pm$ 0.10	3.2 $\pm$ 0.07	3.6 $\pm$ 0.08	2.6 $\pm$ 0.10	3.5 $\pm$ 0.10
Fe (ppm)	0.14 $\pm$ 0.007	0.16 $\pm$ 0.005	0.10 $\pm$ 0.005	0.15 $\pm$ 0.006	0.25 $\pm$ 0.007
Na (ppm)	70.9 $\pm$ 0.4	100.0 $\pm$ 0.6	49.8 $\pm$ 0.3	101.2 $\pm$ 0.7	127.1 $\pm$ 0.8
Rb (ppb)	<1.3	0.9 $\pm$ 0.2	1.4 $\pm$ 0.35	<1	<1
Cs (ppb)	0.07 $\pm$ 0.01	0.09 $\pm$ 0.007	0.05 $\pm$ 0.007	<0.02	0.07 $\pm$ 0.01
K (ppm)	<8	<14	<6	<18	<20

TABLE 6 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	11/2/76 (51920)	11/3/76 (59675)	11/4/76 (52023)	11/5/76 (52052)	11/8/76 (59401)
Sc (ppb)	0.011 ± 0.001	0.01 ± 0.001	0.02 ± 0.001	<0.005	0.02 ± 0.001
Tb (ppb)	<0.01	<0.01	<0.01	<0.01	<0.01
Eu (ppb)	0.06 ± 0.003	0.05 ± 0.002	0.04 ± 0.002	0.01 ± 0.001	0.07 ± 0.003
Sm (ppb)	<0.2	<0.2	<0.15	<0.3	<0.2
Ce (ppb)	0.44 ± 0.06	<0.15	0.57 ± 0.03	<0.2	0.11 ± 0.02
La (ppb)	<4	<3	<2	<2	<3
Sr (ppb)	<10	<6	<8	<6	<9
Ba (ppb)	13 ± 3	<10	91 ± 5	<16	13 ± 4
Th (ppb)	<0.03	<0.02	0.02 ± 0.004	<0.02	0.02 ± 0.005
Hf (ppb)	<0.03	<0.02	0.02 ± 0.004	<0.02	<0.03
Ta (ppb)	0.02 ± 0.005	<0.02	<0.02	<0.01	<0.03
Ga (ppb)	<40	<45	<40	<40	<45
Zr (ppm)	0.02 ± 0.002	0.010 ± 0.001	0.01 ± 0.002	<0.007	0.01 ± 0.003
Cu (ppm)	1.18 ± 0.10	2.0 ± 0.2	1.17 ± 0.10	2.0 ± 0.1	2.8 ± 0.31
Zn (ppm)	0.32 ± 0.002	0.16 ± 0.005	0.27 ± 0.001	0.25 ± 0.002	0.33 ± 0.003

TABLE 7
TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	11/9/76 (52070)	11/22/76 (52113)	11/23/76 (52120)	11/24/76 (52127)	11/25/76 (52128)
Ti (ppm)	<0.8	<0.6	<0.7	0.655 ± 0.2	<0.4
V (ppm)	<0.004	0.032 ± 0.002	0.020 ± 0.002	0.023 ± 0.002	0.015 ± 0.001
Ca (ppm)	3.82 ± 1.4	<4.0	<4.4	6.70 ± 1.33	7.85 ± 1.16
Al (ppm)	0.410 ± 0.10	0.297 ± 0.022	0.222 ± 0.060	0.158 ± 0.057	0.310 ± 0.024
Cl (ppm)	3.52 ± 0.19	3.80 ± 0.31	3.09 ± 0.17	2.941 ± 0.209	1.54 ± 0.16
Mn (ppm)	0.038 ± 0.008	0.022 ± 0.006	0.015 ± 0.005	0.026 ± 0.007	0.028 ± 0.006
I (ppm)	<0.03	<0.1	<0.03	<0.04	<0.04

TABLE 7 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	11/9/76 (52079)	11/22/76 (52113)	11/23/76 (52120)	11/24/76 (52127)	11/25/76 (52128)
As (ppb)	<10	<10	<9	<10	<10
Sb (ppb)	0.36 $\pm$ 0.02	0.46 $\pm$ 0.02	0.41 $\pm$ 0.03	0.50 $\pm$ 0.02	0.5 $\pm$ 0.02
Se (ppb)	0.39 $\pm$ 0.06	0.42 $\pm$ 0.02	1.15 $\pm$ 0.05	0.43 $\pm$ 0.03	0.48 $\pm$ 0.03
Hg (ppb)	0.37 $\pm$ 0.03	0.70 $\pm$ 0.04	0.34 $\pm$ 0.02	0.47 $\pm$ 0.03	0.64 $\pm$ 0.03
Br (ppb)	46 $\pm$ 3	44 $\pm$ 2	49 $\pm$ 3	52 $\pm$ 3	44 $\pm$ 3
Ni (ppm)	0.03 $\pm$ 0.001	0.09 $\pm$ 0.002	0.013 $\pm$ 0.001	0.02 $\pm$ 0.001	0.01 $\pm$ 0.001
Co (ppb)	0.60 $\pm$ 0.01	1.0 $\pm$ 0.02	0.40 $\pm$ 0.01	0.54 $\pm$ 0.01	0.41 $\pm$ 0.01
Cr (ppb)	3.1 $\pm$ 0.10	1.7 $\pm$ 0.10	2.20 $\pm$ 0.08	3.0 $\pm$ 0.08	2.4 $\pm$ 0.09
Fe (ppm)	0.18 $\pm$ 0.006	0.13 $\pm$ 0.006	0.14 $\pm$ 0.006	0.14 $\pm$ 0.005	0.15 $\pm$ 0.006
Na (ppm)	126.0 $\pm$ 0.9	117.0 $\pm$ 1.0	117.0 $\pm$ 1.0	107.0 $\pm$ 0.8	120.0 $\pm$ 1.0
Rb (ppb)	<1.2	<1	<1	1.0 $\pm$ 0.2	0.7 $\pm$ 0.2
Cs (ppb)	0.09 $\pm$ 0.01	<0.03	0.06 $\pm$ 0.006	<0.03	0.01 $\pm$ 0.004
K (ppm)	<16	<23	<22	<14	<14

TABLE 7 (Cont.)

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

	11/9/76 (52070)	11/22/76 (52113)	11/23/76 (52120)	11/24/76 (52127)	11/25/76 (52128)
Sc (ppb)	0.02 ± 0.001	<0.002	0.02 ± 0.001	<0.002	<0.001
Tb (ppb)	<0.01	<0.01	<0.01	<0.01	<0.01
Eu (ppb)	0.05 ± 0.003	0.01 ± 0.002	0.06 ± 0.003	0.02 ± 0.002	0.01 ± 0.001
Sm (ppb)	<0.4	<0.2	<0.3	<0.4	<0.4
Ce (ppb)	0.12 ± 0.03	0.21 ± 0.04	0.2 ± 0.04	0.13 ± 0.03	0.13 ± 0.04
La (ppb)	<3	<3	<3	<3	<3
Sr (ppb)	<9	<8	<6	<8	<6
Ba (ppb)	17 ± 4	<16	12 ± 3	21 ± 3	11 ± 3
Th (ppb)	<0.03	<0.02	<0.02	<0.02	<0.02
Hf (ppb)	<0.03	<0.02	<0.02	<0.02	<0.02
Ta (ppb)	<0.02	<0.02	<0.02	<0.02	<0.02
Ga (ppb)	<40	<40	<40	<35	<30
Zr (ppm)	0.013 ± 0.003	<0.008	0.01 ± 0.002	<0.01	<0.007
Cu (ppm)	0.66 ± 0.06	2.6 ± 0.2	1.6 ± 0.1	1.9 ± 0.10	1.4 ± 0.10
Zn (ppm)	0.35 ± 0.002	0.37 ± 0.002	0.13 ± 0.001	0.17 ± 0.002	0.18 ± 0.002

TABLE 8

TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

11/26/76 (52139)	
<hr/>	
Tl (ppm)	<0.5
V (ppm)	0.010 ± 0.001
Ca (ppm)	20.8 ± 1.7
Al (ppm)	0.433 ± 0.047
Cl (ppm)	0.98 ± 0.11
Mn (ppm)	0.045 ± 0.006
I (ppm)	<0.023

TABLE 8 (Cont.)
TRACE ELEMENTS IN CARBON FILTER EFFLUENTS

11/26/76 (52139)			
As (ppb)	<6	Sc (ppb)	0.02 ± 0.001
Sb (ppb)	0.74 ± 0.03	Tb (ppb)	<0.01
Se (ppb)	0.23 ± 0.04	Eu (ppb)	0.05 ± 0.003
Hg (ppb)	0.57 ± 0.03	Sm (ppb)	<0.2
Br (ppb)	36 ± 2	Ce (ppb)	1.12 ± 0.05
		La (ppb)	<3
Ni (ppm)	0.05 ± 0.002		
Co (ppb)	0.79 ± 0.01	Sr (ppb)	<9
Cr (ppb)	3.5 ± 0.09	Ba (ppb)	<20
Fe (ppm)	0.50 ± 0.01	Th (ppb)	<0.03
Na (ppm)	30.2 ± 0.2	Hf (ppb)	<0.03
Rb (ppb)	<1.3	Ta (ppb)	<0.03
Cs (ppb)	<0.09	Ga (ppb)	<24
K (ppm)	<4	Zr (ppm)	0.02 ± 0.002
		Cu (ppm)	0.47 ± 0.03
		Zn (ppm)	0.47 ± 0.003

TABLE 9

LEAD, CADMIUM, AND BERYLLIUM CONTENT OF SRC
PLANT EFFLUENT WATERS (Pt. 9114)^a

Sample Date and Number	Elemental Concentration		
	Pb (ppb)	Cd (ppb)	Be (ppb)
9/20/76 (54468)	<35.0	2.5 ± 0.1	<0.7
9/21/76 (54475)	<3.7	3.5 ± 0.3	<0.5
9/22/76 (54478)	2.0 ± 0.5	3.3 ± 0.2	<0.2
9/23/76 (55720)	<14.3	5.5 ± 0.5	<0.9
9/24/76 (55725)	2.2 ± 0.6	10 ± 1	<0.2
9/27/76 (55742)	1.5 ± 0.4	3.0 ± 0.3	<0.16
9/28/76 (55755)	1.0 ± 0.3	2.0 ± 0.2	<0.12
9/29/76 (58708)	0.8 ± 0.2	1.5 ± 0.2	<0.14
10/1/76 (58721)	<2.4	4.1 ± 0.2	<0.12
10/7/76 (58759)	<1.2	1.6 ± 0.2	<0.12
10/8/76 (58768)	0.4 ± 0.4	1.8 ± 0.2	<0.12
10/8/76 (52215) (Line Supply)	1.3 ± 0.2	1.5 ± 0.1	<0.12

TABLE 9 (Cont.)

LEAD, CADMIUM, AND BERYLLIUM CONTENT OF SRC
PLANT EFFLUENT WATERS (Pt. 9114)^a

Sample Date and Number	Elemental Concentration		
	Pb (ppb)	Cd (ppb)	Be (ppb)
10/11/76 (58783)	<2.0	1.7 ± 0.1	<0.2
10/12/76 (58788)	1.5 ± 0.3	1.3 ± 0.1	<0.2
10/13/76 (58789)	<2.8	1.3 ± 0.1	<0.2
10/14/76 (50201)	<1.8	5.4 ± 0.7	<0.13
10/15/76 (50206)	<1.0	3.2 ± 0.2	<0.2
10/18/76 (50222)	<3.7	1.2 ± 0.1	<0.2
10/19/76 (50228)	<3.5	4.7 ± 0.5	<0.2
10/25/76 (50282)	<1.8	7.7 ± 0.4	<0.2
10/26/76 (50298)	<1.2	3.3 ± 0.3	<0.2
10/27/76 (51994)	<1.6	1.7 ± 0.1	<0.2
10/28/76 (51977)	<2.0	2.0 ± 0.1	<0.2
10/29/76 (51967)	<2.2	2.0 ± 0.1	<0.2

TABLE 9 (Cont.)

LEAD, CADMIUM AND BERYLLIUM CONTENT OF SRC
PLANT EFFLUENT WATERS (Pt. 9114)^a

Sample Date and Number	Elemental Concentration		
	Pb (ppb)	Cd (ppb)	Be (ppb)
11/1/76 (51929)	<3.3	1.4 ± 0.1	<0.2
11/2/76 (51920)	1.6 ± 0.5	4.8 ± 0.2	<0.2
11/3/76 (59675)	1.9 ± 0.4	2.0 ± 0.2	<0.2
11/4/76 (52023)	<4.3	2.8 ± 0.1	<0.2
11/5/76 (52052)	<3.9	1.1 ± 0.1	<0.2
11/8/76 (59401)	<3.9	3.6 ± 0.3	<0.2
11/9/76 (52070)	<6.6	3.2 ± 0.2	<0.2
11/22/76 (52113)	<3.1	0.9 ± 0.2	<0.2
11/23/76 (52120)	<4.8	0.8 ± 0.1	<0.2
11/24/76 (52127)	<2.6	0.8 ± 0.1	<0.2
11/25/76 (52128)	<7.4	0.6 ± 0.1	<0.2
11/26/76 (52139)	<3.6	0.6 ± 0.1	<0.2

Footnotes to Table of Values:

- a) The ± errors reported were derived from the precision (standard deviation from the mean) of atomic absorption measurements from successive firings of the carbon rod atomizer. Sample 10/8/76 is included in this group for comparison with the Line Supply sample.