
Research and Development



Ferroalloy Process Emissions Measurement

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Ferroalloy Process Emissions Measurement

by

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SUMMARY

Sampling and analysis were undertaken to characterize and quantify particulate, organic and inorganic chemical emissions in effluents from a totally sealed metallurgical furnace at a ferroalloy production facility. Effluents were sampled downstream of a Venturi scrubber during silicomanganese production (Test I) and upstream of the scrubber during ferromanganese production (Test II). Sampling and analysis methodology used was essentially that of EPA's Level 1 Environmental Assessment procedure, supplemented by a specific analysis of polynuclear aromatic hydrocarbons.

Measurements made in Test II indicated a particulate loading of 68,000 mg/m³, equivalent to 17 Kg/MW-hr. Very high levels of organics, including high molecular weight aromatic hydrocarbons, were found. Compound categories found include some polynuclear aromatic species recognized as carcinogens. High levels of arsenic were also measured in Test II. Measurement of gaseous effluent from the Venturi scrubber in Test I indicated much lower levels of all species of concern. Particulate loading was estimated to be 64 mg/m³ equivalent to 0.016 Kg/MW-hr. The major organic compound categories were simple aromatic hydrocarbons and low molecular weight polycyclics. The arsenic level was estimated to be less than 0.5 mg/m³.

In these tests, good agreement was observed between the results of Level 1 organic analysis and the specific analysis of polynuclear aromatic hydrocarbons. Good agreement was also found between the atomic absorption and spark source mass spectroscopic analyses of arsenic and antimony.

Because the two tests corresponded to different ferroalloy production processes, the results cannot provide a quantitative measure of the Venturi scrubber efficiency. However, the data imply good particulate removal efficiency. The Venturi scrubber also appears to be effective for removal of polynuclear aromatics, especially species in the higher molecular weight range that includes the recognized carcinogenic POM.

I. Introduction

Ferroalloy plants are of interest to the Environmental Protection Agency (EPA) because of their high emissions of particulates. Preliminary data from a plant in Norway showed that the closed type of metallurgical furnaces seemed to be efficient in lowering the quantities of particulate emissions. However, it was also found that these emissions contain a high percentage of polycyclic aromatic hydrocarbon materials. Further information is needed to determine the accuracy and applicability of these early findings. To supply this data, Monsanto Research Corporation was assigned by EPA to sample the emissions from the Union Carbide Ferroalloy Plant at Beauharnois, Quebec, Canada. Emissions from both the silico-manganese process and the ferromanganese process carried out in this plant were sampled. Arthur D. Little, Inc., was responsible for the analysis of these samples.

This report, which was prepared by Arthur D. Little, Inc., integrates the following information:

- sampling and on-site gas analysis data provided in rough draft form by Monsanto Research Corporation.
- Process operation data provided by Union Carbide Canada Limited.
- results of comprehensive chemical analyses by Arthur D. Little, Inc.

Chapter II presents a description of the test, including the facility, process and sampling and analysis plan. Chapter III presents the test results. Conclusions are presented in Chapter IV. Details of the analytical results are presented in the Appendices.

II. Test Description

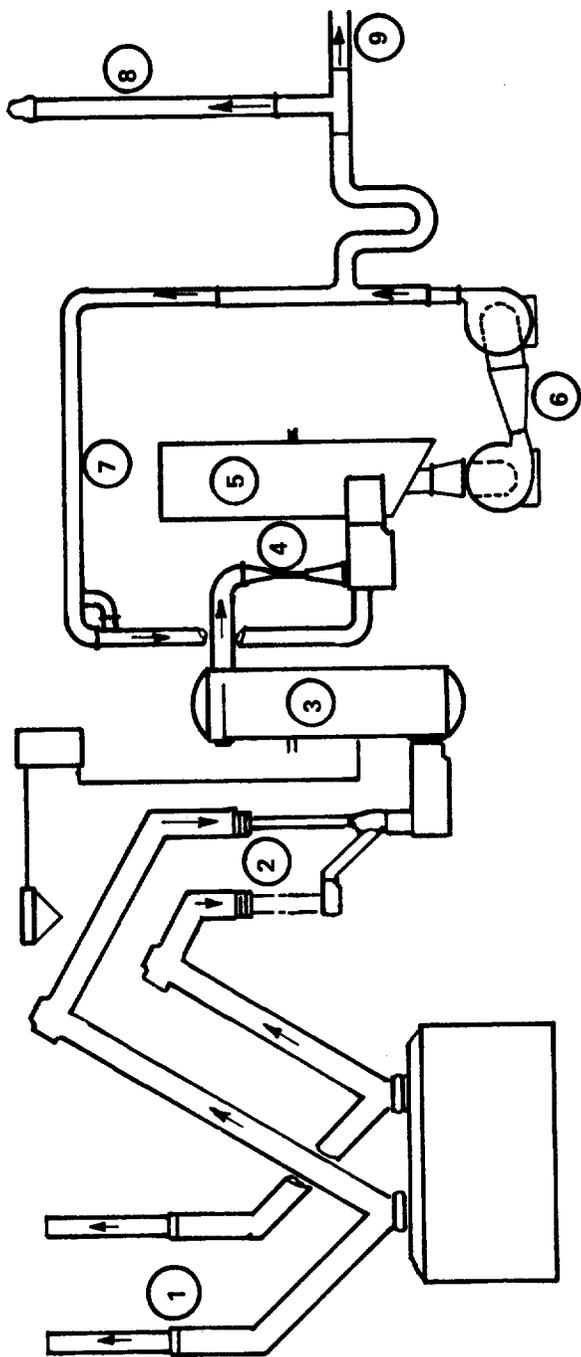
A. Description of Facility and Sampling Sites

The Union Carbide Canada Limited plant in Beauharnois, Quebec, is a modern (1974) integrated ferroalloy production facility incorporating a totally sealed electric furnace. In addition to the furnace, the plant includes facilities for: raw material preparation and storage; sintering of coke and ore fines; mix batching and delivery; and air and water pollution abatement. The closed metallurgical furnace and the associated air pollution control equipment were the focus of the tests described in this report.

The 72,000 KVA totally sealed furnace is contained in a 15 m diameter by 8.8 m deep shell, which has an air-cooled flat bottom. The inner hearth diameter is 12.1 m, and the crucible depth is 6.3 m. Three self-baking electrodes, 1.9 m diameter, are triangularly arranged at 4.75 m center-to-center distances. Additional details of furnace design are provided in Reference 1.

The air pollution abatement equipment for the closed furnace is shown schematically in Figure 1. The system includes two parallel quenchers, a coarse dust separator, a Venturi scrubber, a mist eliminator, and two fans in series. The sampling locations were upstream and downstream of the Venturi scrubber. Figure 1 also shows the bypass stacks through which furnace off-gases can be vented and then flared.

Sampling upstream of the Venturi scrubber utilized an existing 10 cm (4 in) diameter port in the 1.03 m (40.5 in) diameter bypass stack, before the flare. At this location, on the sixth floor of the furnace building, the stack temperature is normally in the range of 480 to 870°C (900 to 1600°F). The stack is under slight negative pressure at this point. The bypass stack gas typically contains about 41% carbon monoxide, 8% hydrogen, 1% oxygen and 50% carbon dioxide (dry basis), and has a moisture content of about 2%.



- 1. Bypass Stacks
- 2. Quenchers
- 3. Dust Separator
- 4. Venturi Scrubber
- 5. Mist Eliminator

- 6. Fans
- 7. Recirculation Loop
- 8. Clean Gas Stack
- 9. Incinerator Ductwork

FIGURE 1 GAS CLEANING SYSTEM

Source: Reference 1, Reproduced with permission of R. G. Ratzlaff

Sampling downstream of the Venturi scrubber was done at a point 6.1 m (20 ft) from the exit of the scrubber, using an existing port in the 0.74 m (29 in) pipe. This port is located approximately 3 m (10 ft) above the floor in a room on the third story of the furnace building. The temperature of the gas stream at this point is normally between 32 and 49°C (90 and 120°F). The stream is saturated with water and is under a positive pressure of approximately 51 cm (20 inches) of water. The major chemical components of the gas are the same as in the bypass stack: 41% carbon monoxide, 8% hydrogen, 1% oxygen and 50% carbon dioxide.

B. Description of Process

Table 1 presents the process information provided to Arthur D. Little, Inc., by Union Carbide Canada Limited, for the silicomanganese production run on August 11, 1977, and the ferromanganese production run on August 27, 1977, which were the two runs sampled.

C. Sampling Procedures

The sampling plan for these tests was prepared by Monsanto Research Corporation (MRC). A team from MRC under the direction of Mr. Darrell L. Harris performed all the sample collection and on-site gas analysis work. The methodology used was essentially that of EPA's Level 1 Environmental Assessment procedures (2), except as noted.

1. Sampling for Comprehensive Analysis

The objectives of this test program include quantitative estimation of total particulate emissions and comprehensive characterization of organic and inorganic materials emitted. To accomplish this, samples were collected using the EPA Source Assessment Sampling System (SASS) (2), shown schematically in Figure 2. This sampling train incorporates three cyclones and a filter to provide collection and size fractionation of particulates, a solid sorbent module containing XAD-2 resin for collection of organic

Table 1
Description of Process

<u>MIX ORDER (lb)</u>	<u>Aug. 11/77</u> <u>SiMn</u>	<u>Aug. 27/77*</u> <u>Std. FeMn</u>
Std. FeMn Slag	3000	-
Dried Manganese Ore (3% H ₂ O)	3000	5000
Sinter	-	1000
Dried Coke (4% H ₂ O)	1000	900
Limestone	-	500
Steel Scrap	125	-
Quartz	1000	-
Coal	250	200
 <u>OPERATING RESULTS</u>		
Average Load (while operating)(KW)	22500	17300
Operating Time (%)	98	98.5
KWH/lb. of Alloy	1.75	1.0
Production per Day (NT)	150	205
Electrode Consumption (lbs./N.T. Alloy)	60	30
 <u>SLAG COMPOSITION (%)</u>		
MnO	12.1	41.3
SiO ₂	32.4	21.1
Al ₂ O ₃	27.0	15.6
CaO	15.9	13.4
 <u>ALLOY COMPOSITION (%)</u>		
Mn	67.0	80
Si	16.0	1.0
Venturi Scrubber Water Flow Rate	90 gpm	90 gpm
Venturi Scrubber Pressure Drop	90" water	90" water

* Furnace in final stages of transition to Std. FeMn from SiMn.

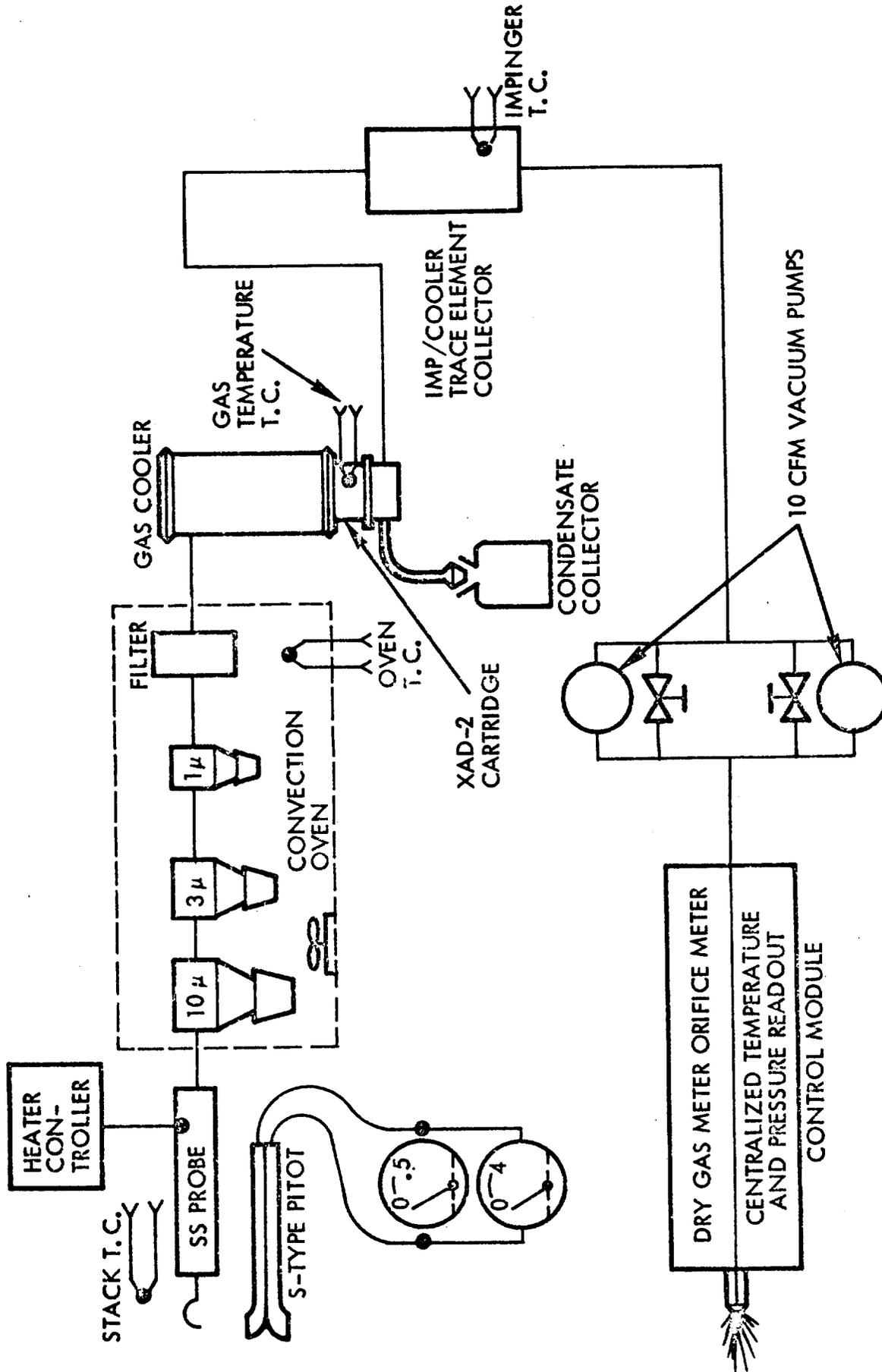


Figure 2. Source Assessment Sampling Schematic (from Reference 2)

vapors, and oxidizing impingers for collection of volatile inorganics. Several modifications to the standard SASS train were made to accommodate the special requirements of this sampling situation, especially the risk of a possible hydrogen explosion in the event of leaks from the train or the stack being sampled. Possible electrical ignition sources in the SASS train were eliminated as follows:

- The probe and oven were modified so that they could be heated by steam, rather than electricity.
- The oven and sorbent module were modified so that a nitrogen blanket could surround all spark sources on these two components.
- The console and pumps from the SASS train were located outside of the explosion hazard area, 15-25 m (50-75 ft) away.
- A 15 m (45 ft), 2.5 cm (1 in) O.D., Tygon tubing line attached to the outlet of the SASS dry test meter, to vent the gases away from the console and operators during runs.

An additional SASS train modification was to extend the Teflon-lined stainless steel braided line connecting the oven to the sorbent module, so that the probe and oven were the only train components placed on the scaffolding platforms. The sorbent module and impingers were placed on the floor below the sampling port.

The interface between the probe and the stack was accomplished by adding a packing gland to the existing port and gate valve. The probe was inserted into the packing gland which was purged with nitrogen before the gate valve was opened. During sampling, the probe nozzle was positioned in the stack at a fixed point of average velocity, determined by a preliminary traverse with a Pitot tube according to EPA Method 2. (3) The sampling system was operated as close to isokinetic conditions as was possible within the constraints of available nozzle sizes and operating parameters. The sampling

plan called for collection of 30 m³ (1060 ft³, standard) of stack gas at a rate of 1.4 to 2.4 x 10⁻³ m³/s (3 to 5 ft³/min.)

At the completion of each sampling run the train was disassembled and samples recovered according to the EPA Level 1 procedures (2).

2. Sampling for On-Site Gas Analysis

a. Carbon Monoxide, Carbon Dioxide, Oxygen and Water

It was planned to collect integrated gas samples in Tedlar bags for Orsat analysis of carbon monoxide, carbon dioxide and oxygen according to EPA Method 3 (4). It was later agreed that the readouts from the plant's instrumental analyzers, located within a few feet of the sampling port at the scrubbers outlet, could replace the Orsat analysis for that stack.

Moisture determinations in both stacks were done according to EPA Method 4 (5).

b. Organic Gases

Organic species in the -160 to +90°C boiling point range were sampled and analyzed at the plant site. Stack gases were collected in Tedlar bags. Analyses were performed using an AID portable gas chromatograph with a flame ionization detector. A 1.8 m by 6.4 mm (6' x 1/4") stainless steel Porapak Q column was operated isothermally at 50°C. The procedure was calibrated using standard gas mixtures taken to the field laboratory.

The GC system simply separates and analyzes mixtures of materials with a given boiling point range (and polarity in some cases) rather than individual pure compounds. Since the chromatogram peaks represent mixtures of materials present in a certain boiling range rather than pure,

individual compounds, the chromatographic data were reported as follows:

<u>Designation</u>	<u>B.P. Range</u>	<u>Corresponding Hydrocarbon</u>
GCI	-160 to -100	Methane, C ₁
GC2	-100 to -50	Ethane, C ₂
GC3	-50 to 0	Propane, C ₃
GC4	0 to 30	Butane, C ₄
GC5	30 to 60	Pentane, C ₅
GC6	60 to 90	Hexane, C ₆

c. Sulfur Gases

Samples were collected in gas sampling bags and the concentrations of hydrogen sulfide, carbon oxysulfide, carbon disulfide and sulfur dioxide were determined in the field. An AID Model 511 gas chromatograph with a flame photometric detector (393 nm filter) was used for the analyses. An 8 m by 3 mm (8' by 1/8') Teflon column packed with 15% UCON 50 HB 280X on 40/60 Chromosorb T was operated isothermally at 134°C. The procedure was calibrated using an AID Model 320A permeation tube system.

3. Monitoring of Carbon Monoxide Exposure

Several precautions were taken to minimize potential toxicity hazards to the sampling crew due to the high levels of carbon monoxide in the sampled streams. The plant safety procedures were explained in a lecture by Union Carbide Canada Limited personnel. The plant was equipped with continuous carbon monoxide monitors set to sound an audible alarm at the 100 ppm level. Also, plant personnel took Dräger tube readings of carbon monoxide levels in the working area every 15 minutes and cleared the area if concentrations over 100 ppm were measured. Further indication of possible carbon monoxide hazard was provided by a Monsanto Research Corporation--designed continuous monitor, set to give visible and audible alarms at the

50 ppm level. The sampling crew cleared the area when this alarm was triggered.

When sampling equipment was being inserted or removed from the stacks, sampling crew members wore trailing air masks. It was at these times that the probability of exposure to hazardous levels of carbon monoxide was greatest.

D. Analysis Procedures

The SASS train samples collected by Monsanto Research Corporation were sent to Arthur D. Little, Inc., for analysis. The samples received for analysis included eighteen components from the two SASS trains used for the two processes, two feed samples (coal and coke), and two solvent blanks corresponding to the solvents used for extraction of the sorbent condensate and for probe and cyclone rinses. For simplification, each sample has been assigned a code which is used throughout this report. Tables 2-4 identify the samples and list their codes. The analytical plan was prepared by Arthur D. Little, Inc., in consultation with the EPA project officer.

Each sample was subjected to the Level 1 analytical program, including microscopy, inorganic and organic analysis. Figures 3-5 show the actual step-by-step analysis scheme used for each sample. All samples were carried through the entire level 1 program except in those cases where the sample size was below that required for further analysis.

The samples were also analyzed for polycyclic aromatic hydrocarbons and other key related species (POM) using a GC/MS procedure.

1. Level 1 Organic Analysis

Level 1 organic analysis procedures as described in the EPA procedures manuals(2, 6) were followed. A brief summary of the various steps is given below:

Table 2

Sample Series I

Series	I
Process	Silicomanganese
Sampling Point	Outlet of Venturi Scrubber
Volume of Gas Sampled	32.12 m ³

<u>SASS Components</u>	<u>Codes</u>
cyclone catch >10 μ	IC10
cyclone catch >3 μ	IC3
cyclone catch >1 μ	IC1
filter catch	IF
probe and cyclone rinses	IPW
XAD-2 resin	IX
sorbent module condensate organic extract	ISC
Impinger soln #1 (including condensate from sorbent module)	I imp. 1
Impinger soln #2 and #3	I imp. 23

Table 3

Sample Series II

Series	II
Process	Ferromanganese
Sampling Point	Bypass ₃
Volume of Gas Sampled	1.36 m ³

<u>SASS Components</u>	<u>Codes</u>
cyclone catch >10 μ	IIC10
cyclone catch >3 μ	IIC3
cyclone catch >1 μ	IIC1
filter catch	IIF
probe and cyclone rinses	IIPW
XAD-2 resin	IIX
sorbent module condensate organic extract	IISC
Impinger soln #1 (including condensate from sorbent module	II imp. 1
Impinger soln #2 and #3	II imp. 23

Table 4

Other Samples

Coal	CL
Coke	CK
Blank (methylene chloride)	BM
Blank (methylene chloride/methanol)	BMM
Solvent blank (ADL methylene chloride)	B

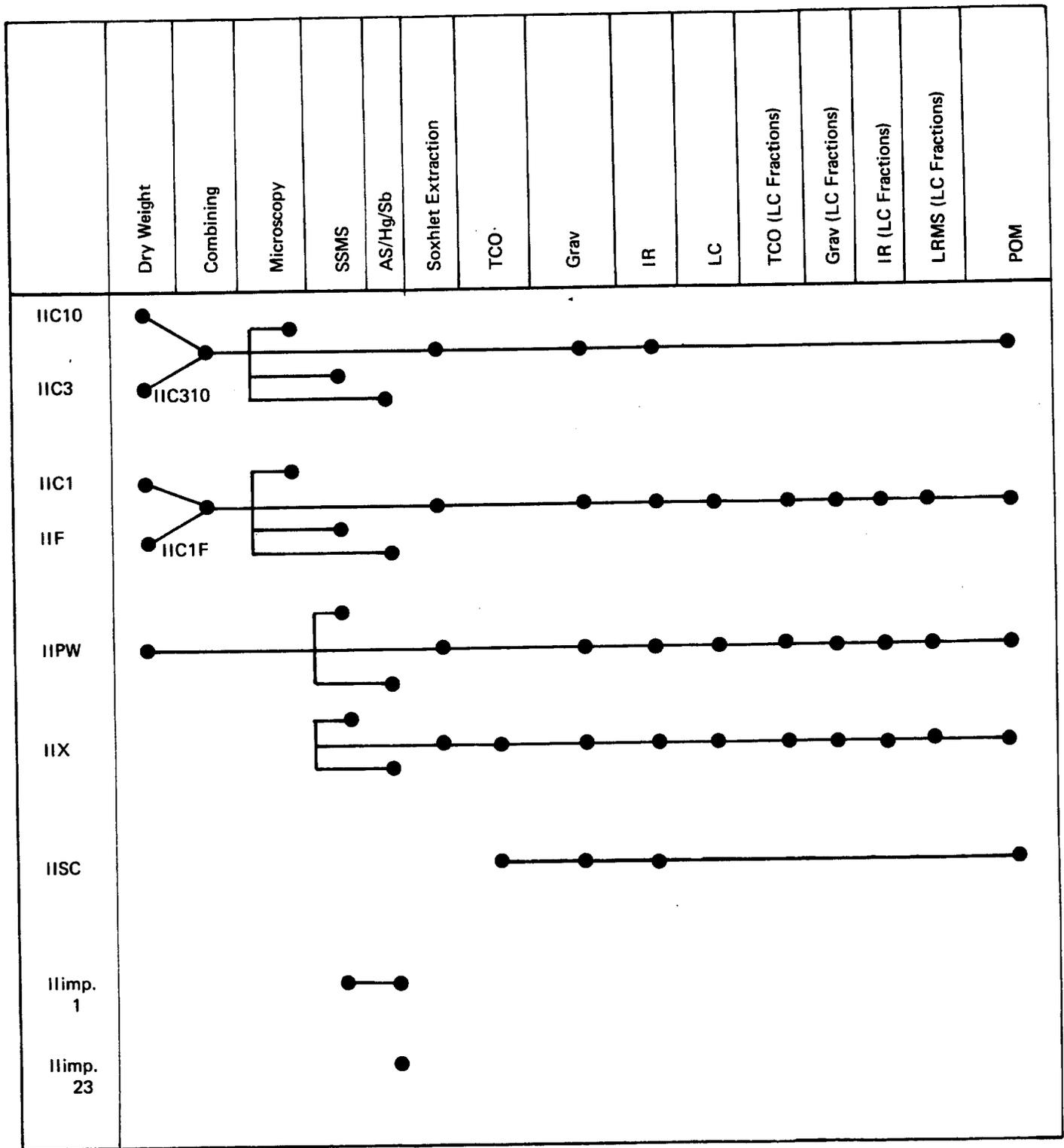


FIGURE 4 ANALYTICAL PROCEDURES FOR FERROMANGANESE SAMPLES

	Dry Weight	SSMS	AS/Hg/Sb	Soxhlet Extraction	TCO	Grav	IR	LC	TCO (LC Fractions)	Grav (LC Fractions)	IR (LC Fractions)	LRMS (LC Fractions)	POM
Coal	●	●	●	●	●	●	●	●	●	●	●	●	●
Coke	●	●	●	●	●	●	●	●	●	●	●	●	●
Blank CH ₂ Cl ₂ (Field)					●	●	●	●	●	●	●	●	●
Blank CH ₂ Cl ₂ (Lab)					●	●	●	●	●	●	●	●	●
Blank CH ₂ Cl ₂ / MeOH (Field)					●	●	●	●	●	●	●	●	●

FIGURE 5 ANALYTICAL PROCEDURES FOR OTHER SAMPLES

a. Particulate Weights

The weights of the particulate samples (cyclone catches and probe and cyclone rinses) were obtained by drying the samples to constant weight in tared evaporating dishes at 50°C and cooling to room temperature in a desiccator.

b. Soxhlet Extractions

All extractions were carried out for a 24-hour period using high purity methylene chloride (Burdick and Jackson, distilled-in-glass). The following procedures were used:

- i. XAD-2 Resins - extracted with about 2500 mL of methylene chloride.
- ii. 10 μ and 3 μ cyclone catches - weighed individually and then combined. Portions of the combined particulates were removed for microscopy and inorganic analysis and the remainder extracted with 200-400 mL of methylene chloride.
- iii. 1 μ cyclone catch and filter samples - same procedure as above.

c. Total Chromatographable Organics Analysis (TCO)

The quantity of the total organic material with boiling points in the range of 100-300°C was determined by gas chromatography, using a flame ionization detector. The concentration of each sample was calculated from the ratio of the peak areas of the sample to that of the known standards. The following instrument conditions were used:

Column: 10% OV-101 on 100/120 mesh Supelcoport
Injector temperature: 270°
Detector temperature: 305°C
Temperature Program: Room temperature for 5 minutes, then programmed at 20°C/min up to 250°C
Gas flow rates: He at 30 mL/min
H₂ at 30 mL/min
Air at 300 mL/min

d. Gravimetric Analysis (Grav)

The amounts of organic material with boiling points higher than 300°C were determined by the gravimetric analysis method (Grav); one or five mL samples were pipetted into precleaned, dried, and weighed aluminum dishes, and were dried at room temperature in a desiccator to constant weight.

e. Infrared (IR)

The IR spectra of all samples as potassium bromide micro pellets were obtained on a Perkin-Elmer 521 grating spectrometer. Spectra were interpreted with the aid of references 7-10.

f. Liquid Chromatographic (LC) Separation

Samples for liquid chromatography were initially concentrated to 10 mL using Kuderna Danish apparatus followed by concentration to 1 mL under a nitrogen stream and then subjected to three consecutive solvent exchanges with cyclopentane. The resultant cyclopentane solutions were chromatographed on a silica gel column, collecting seven fractions by elution with solvent mixtures of increasing polarity.

g. Low Resolution Mass Spectroscopy (LRMS)

LRMS analysis was carried out on a Dupont 21-110B spectrometer. Both batch inlet and direct insertion probe techniques were used depending on the TCO content of the samples. Sample sizes varied from 20 µL to 50 µL. Typically, a sample was run at 15 ev and 70 ev ionization potentials over a temperature range of 70-350°C. Interpretation of the mass spectra was based on references 11-14.

2. Polycyclic Organic Matter (POM)

The polycyclic organic matter (POM) of each extract was analyzed by gas chromatography/mass spectrometry (GC/MS). A Finnigan Model 400 GC/MS with data system was used. The microprocessor controlled GC

(3% Dexsil 400 on 100/120 Supelcoport) was programmed from 170°C to 300°C after 1 minute of isothermal operation at 110°C and then held isothermally at 300°C for 30 minutes. Quantitation was based upon the selected ion chromatograms for each of the POM molecular ions. Calibration was done using a reference mixture containing selected POM compounds for specific molecular weight regions. (15-19)

3. Level 1 Inorganic Analysis

Elemental analysis was done on each sample after the appropriate sample preparation (described below) using an MS-7 Spark Source Mass Spectrometer and photographic detection system. Experiments were conducted by Commercial Testing and Engineering Co.

Particulates: Refluxed with concentrated HNO₃ and
 concentrated HCl mixture for six hours.

XAD-2 resin, coal,
 coke: Parr Bomb combustion over HNO₃

Impinger solutions: Acidified with HCl

Arsenic, mercury and antimony were determined by atomic absorption spectroscopy. A Perkin-Elmer 503 Spectrophotometer was used.

4. Microscopic Analysis

The particulates from the two SASS trains were examined under a Zeiss standard polarizing microscope. Photomicrographs were made on Ektachrome High Speed film, with samples immersed in a medium of 1.44 index to provide good contrast.

E. Problems Encountered

Despite extensive pre-test planning and preparation, several difficulties were encountered during these field tests. These are discussed briefly below.

1. Process

A problem that had a significant impact on the test program was that a major process change, from silicomanganese production to ferromanganese production, occurred between the two Monsanto Research Corporation sampling runs. This situation resulted from a combination of some plant scrubber system down time during the first Monsanto Research Corporation sampling trip, and limitations imposed on the two subsequent sampling trips by the time, schedule, and budget constraints of the Monsanto Research Corporation program.

2. Sampling System

a. Steam heating system

The steam heating system designed to control the probe and oven temperature, was found to heat the oven to about 80°C, rather than the specified 200°C. It was decided to use the electrical oven heater with a nitrogen blanket. Steam heat was used for the probe until the steam generator failed part way through the first run. It was decided that probe heating was not essential, since only about one linear foot of the probe was exposed to the ambient air.

b. Sampling for on-site analysis

Monsanto Research Corporation field crew members were unable to acquire grab gas samples for analysis of carbon monoxide, oxygen and carbon dioxide or nitrogen oxides. During one attempt to acquire a sample for nitrogen oxides analysis a sampling crew member was injured and required first aid. The plant control room data were used to estimate the concentrations of carbon monoxide, oxygen and carbon dioxide for Run 1: the same values were used for Run 2 calculations. Samples for analysis of sulfur gases and organic gases were taken as planned.

c. Sampling at the bypass stack

The port on the bypass stack had to be bored out before the probe could be inserted. During a velocity traverse on this stack, the stack was "on fire" for a time and the probe and pitot were damaged. An electrical overload, causing impinger backup, occurred when two electrical outlets were misidentified as being on independent circuits. Finally, during the sampling run the filter was found to clog in 15 minutes or less. Sample collection was stopped after the third filter had plugged; the total volume of gas sampled was 1.36 m³ instead of the intended 30 m³.

III. Test Results

A. On-Site Analyses

Data described in this section were acquired by Monsanto Research Corporation personnel.

Table 5 summarizes the sampling data acquired during the two runs with the SASS train. Sampling rates exceeded isokinetic flow by 197% in run 1 and 278% in run II, because stack gas flow rates were lower than had been expected.

Table 6 summarizes the results of the on-site analyses of stack gases. As noted in Section II.E, problems were encountered in acquiring some of the intended grab samples. There are several interesting features of the data that were acquired.

The concentrations of the major gaseous species, determined at the scrubber outlet, were somewhat different than had been expected. The abundance of the reduced species, hydrogen and carbon monoxide, was about 85% higher than anticipated, while oxygen and carbon dioxide levels were correspondingly lower. This may be due in part to preferential absorption of the oxidized components in the scrubber water.

The levels of gaseous organic species in the -160° to -50°C boiling point range (GC1 plus GC2) were three times higher downstream of the scrubber during silicomanganese production than they were upstream of the scrubber during ferromanganese production. This observation shows that the process change between runs I and II resulted in a significant change in the emissions from the facility. For this reason, the data acquired in these tests cannot be used to quantify the performance of the Venturi Scrubber.

Concentrations of sulfur gases were low and approximately the same in the two sampling runs.

Table 5

Summary of Sampling Data

Sample Series	I	II
Process	Silicomanganese	Ferromanganese
Sampling Point	Outlet of Venturi Scrubber	Bypass
Volume of Gas sampled,* m ³ (SCF)	32.1 (1130)	1.36 (48.0)
Test period, minutes	273	20
Stack temperature, °C (°F)	47 (117)	388 (730)
Stack gas velocity m/sec (ft/min)	0.0715 (14.1)	0.0852 (16.8)
Stack gas volumetric* flow rate: m ³ /sec (SCF/min)	1.51 (3200)	1.20 (2550)

* Gas volumes are corrected to standard conditions of 101 KPa (29.9" Hg) and 21.1°C (70°F).

Table 6

Results of On-Site Analyses

Sample Series	I	II
Process	Silicomanganese	Ferromanganese
Sampling Point	Outlet of Venturi Scrubber	Bypass
<u>Species</u>	<u>Concentration (v/v)</u>	
Carbon Dioxide	9.0%*	not analyzed
Carbon Monoxide	76.0%*	not analyzed
Oxygen	0.2%*	not analyzed
Hydrogen	14.8%*	not analyzed
Water	12.5%	35.6%**
Organic Gases:		
GC1 Range [†]	3000 ppm	1000 ppm
GC2 Range [†]	90 ppm	30 ppm
Sulfur Gases:		
Hydrogen Sulfide	1.5 ppm	0.95 ppm
Carbon Oxysulfide	2.47 ppm	2.11 ppm
Sulfur Dioxide	0.20 ppm	<0.05 ppm

*From readouts of plant on-line instrumentation.

**Monsanto Research Corporation believes this value to be in error; the expected value was 4-5%

†Organic gases boiling in the range of -50° to +90°C (GC3 to GC6) were not found. Those species would have had very long retention times under the GC conditions used (50°C Isothermal, Porapak Q).

During the sampling of the source, the opacity was never observed to be less than 100%. There was a heavier smoke during ferromanganese production than during silicomanganese production according to the Monsanto Research Corporation job log.

B. Results of Comprehensive Analysis

Data presented in this section are the results of analyses performed at Arthur D. Little, Inc.

1. Total Particulate Loading

The total mass of emitted particulates as well as the concentration data for the particulates in the source for both the silicomanganese and the ferromanganese processes are given in Table 7. In the effluent gas from the silicomanganese process, 88% of the particulate matter is in 3-10 μ size range. The total particulate loading in this series was found to be 64 mg/m³. Extremely high quantities of particulate matter was collected at the bypass from the ferromanganese process. A concentration of 68,000 mg/m³, relatively uniform distribution over all size ranges, was found for this stream.

Unfortunately, due to the different processes in the two series, these upstream and downstream data cannot be directly compared to reveal the efficiency of the Venturi Scrubber gas cleaning system. It is interesting to note the relatively small proportion of mass emissions in the large (10 μ) and small (filter) size ranges from the silicomanganese sample after the Venturi Scrubber.

Table 7

Total Mass of Emitted Particulates

Series No.	I	II
Process	Silicomanganese	Ferromanganese
Sampling point	Outlet of Venturi Scrubber	Bypass Stack
Volume of gas sampled	32.12 m ³	1.36 m ³
Total particulates		
10μ cyclone	0.0111 g	38.4706 g
3μ cyclone	1.8218	12.6509
1μ cyclone	0.0684	10.1065
filter	0.0319	19.3515
<u>probe and cyclone rinses</u>	<u>0.1411</u>	<u>11.9077</u>
Total	2.0743	92.4872
Total concentration		
10μ cyclone	0.34 mg/m ³	28,000 mg/m ³
3μ cyclone	56.	9,300
1μ cyclone	2.13	7,400
filter	0.99	14,000
<u>probe and cyclone rinses</u>	<u>4.4</u>	<u>8,800</u>
Total	64. mg/m ³	68,000 mg/m ³

2. Level 1 Organic Analysis

a. SASS Samples

Data on the total extractable organic material for the various SASS train components from both processes are summarized in Table 8. Very little organic matter was extracted from the particulates collected from the silicomanganese process. About 94% of the total organics was found in the XAD-2 extract, 96% of which falls into the TCO range (boiling point between 100 and 300°C). Although the concentration of organics in the sorbent condensate extract was not high, it is interesting to note that more high boiling material is present in this component.

Much larger amounts of organic matter were found in the extracts of all SASS train components, except the sorbent condensate extract, from the ferromanganese process. About 92% of the material is found in the XAD-2 extract in this case, of which about 82% was found to be high-boiling (b.p. >300°C) material.

The five extracts containing more than 0.5 mg/m³ of total organic were taken through LC separations, and the seven LC fractions collected from each extract were analyzed for TCO and Grav as well as by IR and LRMS. The LC, IR, and LRMS data are given in the Appendices. From these data, the organic species in each extract were classified into compound categories based on the results of the Level 1 analysis and the concentration of each category was estimated using the method proposed by Arthur D. Little, Inc. (20) Tables 9 to 13 show these results.

Some interesting aspects of these data are pointed out below:

1. Table 9 shows that aromatic hydrocarbons and fused aromatics having MW <216 are the major species in sample IX. Since the TCO values are much greater than the Grav values for all LC fractions,

Table 8

Total Extractable Organics, mg/m³

Process	I			II		
	Silicomanganese			Ferromanganese		
	TCO	GRAV	TOTAL	TCO	GRAV	TOTAL
Particulates extract						
10 + 3μ	—	~0.03	~0.03	—	6.6	6.6
1 + filter	—	~0.03	~0.03	—	48.	48.
probe and cyclone rinse extract	--	0.47	0.47	--	37.	37.
XAD-2 extract	45	2.18	47	205	910	1110
Sorbent condensate extract	0.57	2.02	2.59	0.41	~0.1	0.41

these species are relatively volatile materials and do not represent the major known carcinogenic POM.

- ii. The major category of compounds found in the sorbent condensate extract (ISC) is non-volatile fused aromatics having MW <216, collected mainly in LC3.
- iii. Extremely high quantities of fused aromatics over all molecular weight ranges were found in II X, especially non-volatile species. Also present in this sample were: heterocyclic nitrogen and sulfur compounds, polycyclic aromatic ketones, and a trace amount of esters. The LC separations between aromatic and polar species were very good. (Table 11)
- iv. Tables 12 and 13 show that the most abundant organic species present in the particulate extracts (II CIF and IIPW) were similar to those found in the XAD-2 extract (II X), i.e., fused aromatics in LC 3 and heterocyclic nitrogen compounds and ketones in LC 6.

The five extracts that had insufficient organic material for LC separations and subsequent analysis were examined by infrared only. By combining the IR data with the TCO and Grav results, the organic materials in each extract were very roughly categorized and approximate concentrations estimated. The data from this process, along with the data in Tables 9-13, were integrated to construct summary tables describing the concentration distribution of compound categories from each SASS train. (Tables 14 and 15)

b. Coal and Coke

Coal and coke were also extracted and analyzed. The organic species found in these samples were categorized and summarized in Tables 16 and 17. The most abundant species in coal extract were found to be elemental sulfur, aliphatic hydrocarbons, ketones, heterocyclic nitrogen compounds and fused aromatics with "low" molecular weights. Relatively small amounts of the high molecular weight fused aromatics were detected.

Table 9

ORGANIC EXTRACT SUMMARY TABLE

Sample IX, XAD-2 Extract, Silicomanganese

	LC1	LC2	LC3	LC4	LC5	LC6	LC7	Σ
Total Organics, mg/m ³	2.32	15.	19.	0.03	0.17	2.93	0.74	41.
TCO, mg	74.	480.	620.	1.1	5.6	44.	23.	1260
GRAV, mg	<0.1	0	5.0	<0.1	<0.1	50	<0.1	65

Int/mg/m³

Category	LC1	LC2	LC3	LC4	LC5	LC6	LC7	Σ
Aliphatic Hydrocarbons	100/2.3							2.3
Aromatic Hydrocarbons		100/14	100/9.4	10/<0.1*				23.
Fused Aromatics <216		10/1.4	100/9.4	10/<0.1*				11.
Heterocyclic S Compounds		1/0.1	10/0.9			10/0.7		1.7
Ketones					10/0.01*	10/0.7	1/0.1	0.8
Esters					10/0.01*	10/0.7	1/0.1	0.8
Carboxylic Acids						10/0.7		0.7
Alcohols							1/0.1	0.1
Heterocyclic N Compounds							1/0.1	0.1
Ethers							1/0.1	0.1

*Concentration estimated from LC, IR data, with reference to LRMS data of LC3 and LC6

Table 10

ORGANIC EXTRACT SUMMARY TABLE

Sample ISC., Sorbent Condensate, Silicomanganese

	LC1	LC2	LC3	LC4	LC5	LC6	LC7	Σ
Total Organics, mg/m ³	0.26	0.05	2.70	0.002	0.008	0.008	0.008	3.0
TCO, mg	0.03	0.01	18.	0.06	<0.1	<0.1	0.02	18.
GRAV, mg	8.4	1.5	68	<0.1	0.25	2.75	0.25	81.

Int/mg/m³

Category	Int/mg/m ³	LC1	LC2	LC3	LC4	LC5	LC6	LC7	Σ
Sulfur (S ₈)	100/0.2								0.2
Aliphatic Hydrocarbons	1/0.002	100/0.05*							0.05
Fused Aromatics <216			100/2.7						2.7
Fused Aromatics >216			1/0.02					**	0.02
Nitriles								100/0.004	0.004
Ketones							100/0.007	**	0.01
Esters					**	100/0.004	100/0.001	**	0.01
Ethers					**	100/0.004			0.005
Sulfides					**	100/0.004			0.005
Amides							100/0.004	**	0.004
Alcohols							100/0.004	**	0.004
Amines							100/0.004	**	0.004

* Concentration estimated from LC, IR data with reference to LRMS data of LC1

** Concentration Estimated from LC, IR data only

Table 14

Total Organics (mg/m³) for SASS Train Samples (I)
Outlet of Scrubber, Silicomanganese Process

<u>Compound Categories</u>	<u>Particulates</u>			<u>Sorbent Module</u>		<u>Total</u>
	<u>>3μ*</u>	<u>>3μ*</u>	<u>Rinses</u>	<u>Resin</u>	<u>Condens.</u>	
Aliphatic Hydrocarbons			0.2	2.3	0.05	2.5
Aromatic Hydrocarbons			0.2	23		23
Fused Aromatics <216				11	2.7	14
Fused Aromatics >216					0.02	0.02
Ether				0.1	0.005	0.1
Ketone				0.8	0.01	0.8
Alcohol	~0.01	~0.01	0.2	0.1	0.004	~0.3
Ester			0.2	0.8	0.01	1.8
Amine					0.004	~0.1
Heterocyclic N				0.1		0.1
Heterocyclic S				1.7		1.7
Carboxylic Acid				0.7		0.7
Sulfides					0.005	~0.1
Amide					0.004	~0.1
Sulfur					0.2	0.2
Nitrite					0.004	~0.1
Silicone Compounds	~0.01	~0.01	0.2			0.004

*Concentrations estimated from IR and total TCO and Grav data only.

Table 15

Total Organics (mg/m³) for SASS Train Samples II

Bypass, Ferromanganese Process

<u>Compound Categories</u>	<u>Particulates</u>		<u>Sorbent Module</u>			<u>Total</u>
	<u>>3μ*</u>	<u><3μ</u>	<u>Rinses</u>	<u>Resin</u>	<u>Condens.*</u>	
Aliphatic Hydrocarbons		~0.1	1.9	3.9		6.9
Aromatic Hydrocarbons		~0.1	~0.1	3.5		3.7
Fused Aromatics <216		0.2	14	370		380
Fused Aromatics >216	4.5	33	22	390	0.3	450
Heterocyclic S				37		37
Heterocyclic N	1.1	7.9	0.8	70	0.07	80
Ketones	0.8	6.4	7.8	53	0.05	67
Alcohols			0.02			~0.1
Esters		0.06	0.08	0.8		0.9
Carboxylic Acids		0.06		4.2		4.3

*Concentrations estimated from IR and total TCO and Grav data only.

Table 16

ORGANIC EXTRACT SUMMARY TABLE

	Sample										Σ
	LC1	LC2	LC3	LC4	LC5	LC6	LC7	Coal (CL)			
Total Organics, mg/kg	286	24.	101	35.	13.	62.	10.				530
TCO, mg	0.36	<0.01	0.80	0.014	<0.01	0.29	<0.01				1.2
GRAV, mg	24	2.0	7.7	2.9	1.1	4.9	0.86				43.

Category	Int/mg/Kg									
	100/143	100/12*	10/7.8	1/0.17	1/0.06*					
Sulfur	100/143	100/12*	10/7.8	1/0.17	1/0.06*					160
Aliphatic Hydrocarbons	100/143	100/12*								160
Fused Aromatics <216			10/7.8	1/0.17	1/0.06*					8.0
Fused Aromatics >216			100/78	100/17	100/6.4*					100
Heterocyclic Sulfur			10/7.8							7.8
Heterocyclic Nitrogen				100/17	100/6.4*	10/5.6	10/0.90*			30
Esters				1/0.17	1/0.06*	1/0.56	1/0.09*			0.88
Ketones						100/56	100/9.0*			65

*Estimated from LC and IR data, with LRMS data of adjacent LC fractions.

Table 17

ORGANIC EXTRACT SUMMARY TABLE

	Sample _____ Coke (CK)							Σ
	LC1	LC2	LC3	LC4	LC5	LC6	LC7	
Total Organics, mg/kg	158	<1.5	16	14	22	10	10	230
TCO, mg	0.36	<0.01	0.14	<0.01	<0.01	<0.01	<0.01	0.50
GRAV, mg	10.	<0.01	0.86	0.86	1.4	0.6	0.6	14.3

Category	Int/mg/kg							
Sulfur	100/156							160
Aliphatics	1/2.0		100/13**					15
Halogenated Aromatics			10/1.3**	10/1.4**				2.7
Aromatic Hydrocarbons			10/1.3**	10/1.4**				2.7
Heterocyclic N, O, S				10/1.4**	10/2.2**			3.6
Sulfides, Disulfides				10/1.4**	10/2.2**			3.6
Nitriles				10/1.4**	10/2.2**			3.6
Ethers				100/7.0**	10/2.2**			9.2
Alcohols				100/7.0**	10/2.2**		100/5.0**	19
Aldehydes, Ketones					10/2.2**		10/1.0**	8.2
Nitroaromatics					10/2.2**		10/1.0**	4.2
Amines					10/2.2**		10/1.0**	8.2
Phenols							10/1.0**	2.0
Esters, Amides							10/1.0**	2.0
Carboxylic Acids							100/5.0**	6.0
Sulfoxides							10/1.0**	2.0

** Estimated from LC and IR data, no LRMS data available.

The total amount of organics extracted from coke is low. The major portion of this seems to be elemental sulfur. The IR data indicated that aliphatic hydrocarbons, alcohols, and amines could be present as minor species.

c. Blanks

Solvent Blank (ADL Methylene Chloride): very clean, negligible amount of organic material was detected.

Methylene chloride blank (from the field); mostly aliphatic hydrocarbons, trace of silicone grease was also detected.

Blank methylene chloride/methanol: very little organic material, the non-volatile species present seem to be inorganic.

The LC data of the three blanks are given in the Appendices.

3. Polycyclic Organic Matter (POM) Analysis

The results of GC/MS POM analysis of the ferroalloy samples, expressed in terms of their concentration at the sample source, are summarized in Tables 18 and 19. The Reconstructed Gas Chromatograms are attached in Appendix A.

For the samples after the air-cleaning system (venturi scrubber) from a silicomanganese process (Series I), a total of 4.2 mg/m^3 of POM was found, 51% of which was anthracene/phenanthrene. Less than 1 mg/m^3 of fluoranthene, pyrene, chrysene and their derivatives were detected. Most of these species were found in the XAD-2 sorbent module and the sorbent condensate extracts. Even at the high sensitivity of the GC/MS method used, no POM with molecular weight over 228 was detected.

Very high concentrations of POM were found for the Series II samples which were collected at the bypass to the air-cleaning system during a ferromanganese process. A total of 633 mg/m^3 of POM was found in these samples, 70% of which was anthracene/phenanthrene and fluoranthene, 16% of which was chrysene/benzoanthracene, benzofluoranthene, and benzo-pyrene.

Other species such as carbazole, dibenzocarbazone, perylene, indeno (1,2,3-cd) pyrene, and coronene were also found in these samples. It is interesting to note that most of the POM was in the sorbent module and very little of it was in the sorbent condensate, and also that most of the high molecular weight species were found in the particulate extracts, especially in the probe and cyclone rinses.

The substantial differences between the POM concentrations for the two series of samples shown here could be considered as an indication that the air-cleaning system used is highly effective in removing POM from effluent gases. Unfortunately, these data cannot be used as firm evidence for this, due to the different processes in the two series.

TABLE 18

GC/MS Polycyclic Organic Matter (POM) Analysis

Sample Series: I, Silicomanganese, after scrubber

Species	Sample	Concentration: mg/m ³									
		m/e	I C310	I CIF	I PW	I X	I SC	Total			
Fluorene		165+6	*		0.00012	0.86	0.62	1.5			
Anthracene/Phenanthrene		178	0.00016	0.00039	0.00064	0.83	1.30	2.1			
Carbazole		167									
Methyl-Anthracenes		192				0.42	0.028	0.070			
Isomers		192					0.018	0.018			
Fluoranthene		202	0.000058	0.00019	0.00049	0.044	0.20	0.24			
Pyrene		202	0.000042	0.00015	0.00017	0.046	0.17	0.22			
Methyl Pyrene /Methyl Fluoranthene		216					0.005	0.005			
Chrysene/Benzo(a)anthracene/etc.		228					0.016	0.016			
Methyl Chrysenes		242									
7,12-Dimethyl Benz(a)anthracene		256									
Benzo(a)pyrene		252									
Benzo(a)pyrene		252									
Perylene		252									
Methyl Benzopyrenes		266									
3-Methylcholanthene		268									
Indeno (1,2,3-cd) Pyrene		276									
Benzo(ghi)Perylene		276									
Dibenzo(a,h)anthracene		278									
Dibenzo(c,g) carbazole		267									
Dibenzo(ai & ah)pyrenes		302									
Coronene		300									
TOTAL			0.00026	0.00073	0.0014	1.82	2.40	4.2			

*All blanks are items not detected, detection limit 0.01 µg/m³

TABLE 19

GC/MS Polycyclic Organic Matter (POM) Analysis

Sample Series: II, Ferromanganese, bypass

Concentration: mg/m³

Species	Sample	m/e	II C310	II CIF	II PW	II X	II SC	Total
Fluorene		165+6	0.0014			16.3	0.0077	16.
Anthracene/Phenanthrene		178	0.054	0.014	0.62	222.	0.081	220.
Carbazole		167	*			9.6	0.014	9.6
Methyl-Anthracenes		192	0.0018		0.18	24.	0.0044	24.
Isomers		192					0.0034	0.0034
Fluoranthene		202	0.034	0.0055	2.46	220.	0.039	220.
Pyrene		202	0.019	0.0057	2.28		0.031	2.3
Methyl Pyrene/ Methyl Fluoranthene		216			0.54	14.		14.
Chrysene/Benzo(a)anthracene/etc.		228	0.048	0.026	3.40	46.	0.0063	49.
Methyl Chrysenes		242	0.00065			5.24		5.2
7,12-Dimethyl Benz(a)anthracene		256				0.58		0.58
Benzo(a)anthracene, Benzo(e)pyrene		252	0.031	0.26	3.13	47.	0.0041	51.
Benzo(a)pyrene		252						
Perylene		252	0.036	0.29	2.82		0.0041	3.1
Methyl Benzopyrenes		266		0.026	0.50	0.67		1.20
3-Methylcholanthene		268		0.053	0.34			0.39
Indeno (1,2,3-cd) Pyrene		276	0.029	0.26	0.47	5.28		6.0
Benzo(ghi)Perylene		276	0.099	0.55	0.71			1.4
Dibenzo(a,h)anthracene		278	0.0041	0.12		0.78		0.90
Dibenzo(c,g) carbazole		267	0.079					0.079
Dibenzo(ai & ah)pyrenes		302	0.15	0.23	0.16			0.54
Coronene		300	0.10	0.29	0.12			0.51
TOTAL			0.68	2.1	17.	612.	0.19	660.

*All blanks are items not detected, detection limit 0.3 µg/m³

A comparison of the data on POM concentrations obtained from Level 1 analysis and GC/MS analysis is given in Table 20. In general, the two sets of data agree with each other within an order of magnitude. In the cases of samples ISC (Series I, sorbent condensate) and IIX (Series II, sorbent module), the data are in excellent agreement with each other.

Comparison of the Level 1 and GC/MS analysis data for heterocyclic nitrogen compounds (Table 20) shows that considerably higher levels are found by the Level 1 procedure. This is an indication that the two specific compounds determined in the GC/MS analysis (carbazole and dibenzocarbazole) may constitute only a small fraction of the total heterocyclic nitrogen material. This is confirmed by the Level 1 LRMS data (e.g., Appendix A, pages A23 to A26), which show that acridines and quinolines are the most abundant heterocyclic nitrogen compounds in the ferroalloy effluent samples. The Level 1 and GC/MS results, therefore, are in satisfactory agreement for these species as well as for the polynuclear aromatic hydrocarbons.

Table 20

Total Polycyclic Organic Matter Data Comparison

<u>Series</u>	<u>I</u>		<u>II</u>	
Process	Silicomanganese		Ferromanganese	
Sampling Location	After Scrubber		Bypass	
	-----Total POM-----			
	mg/m ³		mg/m ³	
	<u>Polynuclear Aromatics</u>			
	<u>Level 1</u>	<u>GC/MS</u>	<u>Level 1</u>	<u>GC/MS</u>
SASS Sample				
C310	~0.01	0.00026	4.5	0.60
C1F	~0.01	0.00073	33	2.1
PW	~0.01	0.0014	36	17
XAD-2	11	1.8	760	602
SC	2.7	2.4	0.3	0.2
Total	14	4.2	840	650
	<u>Heterocyclic N Compounds*</u>			
SASS Sample				
C310			1.1	0.8
C1F			7.9	
PW			0.8	
XAD-2	0.1		70.	9.6
SC			0.07	~.01
Total	0.1	-	80	9.7

* Carbazole and Dibenzocarbazole were the only two heterocyclic N species determined in GC/MS analysis.

Table 21

Arsenic, Mercury, and Antimony Determinations

Sample Code	mg/m ³		
	As	Hg	Sb
Silicomanganese Series			
I C310	0.018	0.000060	0.000016
I X	0.098	0.00050	0.001
I imp 1	0.0062	0.00018	0.000025
<u>I imp 23</u>	<u>0.13</u>	<u>0.016</u>	<u>0.00020</u>
Total	0.25	0.017	0.00012
Ferromanganese Series			
II C310	24.	0.045	0.15
II ClF	15.	0.025	0.088
II PW	7.7	0.052	0.038
II X	1.03	0.014	0.019
II imp 1	0.15	0.11	0.0013
<u>II imp 23</u>	<u>0.08</u>	<u>0.26</u>	<u>0.00087</u>
Total	48.	0.51	0.30
Sample	mg/Kg		
	As	Hg	Sb
Coal	20.	0.15	0.30
Coke	20.	0.24	0.58

Table 23

Total Inorganics, Ferromanganese Series
Spark Source Mass Spectrometry Data

Sample No: Series II

<u>Concentration in mg/m³</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum		Holmium	0.03	Rhodium	
Antimony	1.9	Hydrogen	NR	Rubidium	MC
Arsenic	MC	Indium	STD	Ruthenium	0
Barium	MC	Iodine	6.0	Samarium	0.14
Beryllium	0.01	Iridium		Scandium	0.05
Bismuth	0.56	Iron	MC	Selenium	1.5
Boron	0.87	Lanthanum	0.4	Silicon	
Bromine	19	Lead	MC	Silver	1
Cadmium	6.7	Lithium	1.3	Sodium	
Calcium	MC	Lutetium	0.005	Strontium	12
Carbon	NR	Magnesium		Sulfur	0.7
Cerium	0.61	Manganese	MC	Tantalum	
Cesium	1.3	Mercury	NR	Tellurium	0.28
Chlorine	MC	Molybdenum	3.0	Terbium	0.02
Chromium	5.2	Neodymium	0.18	Thallium	3.0
Cobalt	10	Nickel	4	Thorium	0.1
Copper	34	Niobium	0.08	Thulium	0.006
Dysposium	0.05	Nitrogen	NR	Tin	0.3
Erbium	0.02	Osmium		Titanium	7.7
Europium	0.03	Oxygen	NR	Tungsten	1.2
Fluorine	MC	Palladium		Uranium	0.19
Gadolinium	0.05	Phosphorus		Vanadium	0.8
Gallium	3.6	Platinum		Ytterbium	0.03
Germanium	0.28	Potassium	MC	Yttrium	0.14
Gold		Praseodymium	0.07	Zinc	MC
Hafnium	0.002	Rhenium		Zirconium	0.56

NR - Not quantified

All blanks are elements not detected, detection limit 0.1 ppm

MC - Major component, > 68 mg/m³

Table 25

Spark Source Mass Spectrometry Data

Sample No: ICIF

<u>Concentration in $\mu\text{g}/\text{m}^3$</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum	MC	Holmium	0.019	Rhodium	
Antimony	0.034	Hydrogen	NR	Rubidium	1.1
Arsenic	2.7	Indium	STD	Ruthenium	0.16
Barium	MC	Iodine	0.002	Samarium	0.066
Beryllium	0.012	Iridium		Scandium	0.05
Bismuth	0.012	Iron	MC	Selenium	0.009
Boron	0.30	Lanthanum	0.34	Silicon	MC
Bromine	0.006	Lead	0.78	Silver	0.009
Cadmium	MC	Lithium	0.91	Sodium	MC
Calcium	MC	Lutetium	0.003	Strontium	2.1
Carbon	NR	Magnesium	MC	Sulfur	MC
Cerium	0.75	Manganese	MC	Tantalum	≤ 0.002
Cesium	0.05	Mercury	NR	Tellurium	≤ 0.001
Chlorine	0.44	Molybdenum	0.16	Terbium	0.006
Chromium	2.4	Neodymium	0.14	Thallium	0.069
Cobalt	0.16	Nickel	0.001	Thorium	0.18
Copper	0.56	Niobium	0.075	Thulium	0.002
Dysprosium	0.028	Nitrogen	NR	Tin	0.028
Erbium	0.012	Osmium		Titanium	MC
Europium	0.009	Oxygen	NR	Tungsten	0.016
Fluorine	MC	Palladium		Uranium	0.15
Gadolinium	0.016	Phosphorus	MC	Vanadium	1.0
Gallium	0.72	Platinum		Ytterbium	0.016
Germanium	0.031	Potassium	MC	Yttrium	0.26
Gold		Praseodymium	0.066	Zinc	MC
Hafnium	0.019	Rhenium	≤ 0.0006	Zirconium	0.84

Table 26

Spark Source Mass Spectrometry Data

Sample No: IPW

<u>Concentration in $\mu\text{g}/\text{m}^3$</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum	MC	Holmium	0.022	Rhodium	
Antimony	0.11	Hydrogen	NR	Rubidium	0.39
Arsenic	MC	Indium	STD	Ruthenium	
Barium	MC	Iodine	0.013	Samarium	0.061
Beryllium	0.009	Iridium		Scandium	0.04
Bismuth	0.022	Iron	MC	Selenium	0.36
Boron	0.14	Lanthanum	0.57	Silicon	MC
Bromine	2.3	Lead	1.2	Silver	0.61
Cadmium	MC	Lithium	1.9	Sodium	MC
Calcium	MC	Lutetium	0.0035	Strontium	2.7
Carbon	NR	Magnesium	MC	Sulfur	MC
Cerium	0.39	Manganese	MC	Tantalum	
Cesium	0.04	Mercury	NR	Tellurium	0.003
Chlorine	MC	Molybdenum	3.0	Terbium	0.009
Chromium	MC	Neodymium	0.184	Thallium	0.70
Cobalt	0.34	Nickel	MC	Thorium	0.23
Copper	MC	Niobium	0.14	Thulium	0.0022
Dysprosium	0.031	Nitrogen	NR	Tin	0.061
Erbium	0.018	Osmium		Titanium	MC
Europium	0.017	Oxygen	NR	Tungsten	0.066
Fluorine	MC	Palladium		Uranium	0.19
Gadolinium	0.026	Phosphorus	MC	Vanadium	1.1
Gallium	0.22	Platinum		Ytterbium	0.018
Germanium	0.061	Potassium	MC	Yttrium	0.22
Gold	0.001	Praseodymium	0.08	Zinc	MC
Hafnium	0.018	Rhenium	≤ 0.002	Zirconium	0.57

Table 27

Spark Source Mass Spectrometry Data

Sample No: Ix

		<u>Concentration in $\mu\text{g}/\text{m}^3$</u>			
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum		Holmium		Rhodium	
Antimony		Hydrogen		Rubidium	
Arsenic		Indium	STD	Ruthenium	
Barium		Iodine		Samarium	
Beryllium		Iridium		Scandium	
Bismuth		Iron		Selenium	
Boron		Lanthanum		Silicon	
Bromine		Lead		Silver	
Cadmium		Lithium		Sodium	MC
Calcium		Lutetium		Strontium	
Carbon	NR	Magnesium		Sulfur	
Cerium		Manganese		Tantalum	
Cesium		Mercury	NR	Tellurium	
Chlorine		Molybdenum		Terbium	
Chromium		Neodymium		Thallium	
Cobalt		Nickel		Thorium	
Copper		Niobium		Thulium	
Dysprosium		Nitrogen	NR	Tin	
Erbium		Osmium		Titanium	
Europium		Oxygen	NR	Tungsten	
Fluorine		Palladium		Uranium	
Gadolinium		Phosphorus		Vanadium	
Gallium		Platinum		Ytterbium	
Germanium		Potassium		Yttrium	
Gold		Praseodymium		Zinc	MC
Hafnium		Rhenium		Zirconium	

Table 28

Spark Source Mass Spectrometry Data

Sample No: I imp 1

Concentration in $\mu\text{g}/\text{m}^3$					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum		Holmium		Rhodium	
Antimony		Hydrogen		Rubidium	
Arsenic	6	Indium	STD	Ruthenium	
Barium	200	Iodine	2	Samarium	
Beryllium		Iridium		Scandium	
Bismuth		Iron	100	Selenium	10
Boron		Lanthanum		Silicon	
Bromine		Lead		Silver	
Cadmium	2	Lithium		Sodium	
Calcium		Lutetium		Strontium	
Carbon	NR	Magnesium		Sulfur	500
Cerium		Manganese	20	Tantalum	
Cesium		Mercury	NR	Tellurium	
Chlorine		Molybdenum	100	Terbium	
Chromium	70	Neodymium		Thallium	
Cobalt	3	Nickel		Thorium	
Copper		Niobium	0.4	Thulium	
Dysprosium		Nitrogen	NR	Tin	1
Erbium		Osmium		Titanium	20
Europium		Oxygen	NR	Tungsten	
Fluorine		Palladium		Uranium	
Gadolinium		Phosphorus		Vanadium	
Gallium		Platinum		Ytterbium	
Germanium		Potassium		Yttrium	
Gold		Praseodymium		Zinc	
Hafnium		Rhenium		Zirconium	

Table 29

Spark Source Mass Spectrometry Data

Sample No: II C 310

<u>Concentration in $\mu\text{g}/\text{m}^3$</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum		Holmium	0.019	Rhodium	
Antimony	0.67	Hydrogen	NR	Rubidium	MC
Arsenic	MC	Indium	STD	Ruthenium	
Barium	MC	Iodine	2.0	Samarium	0.075
Beryllium	0.0037	Iridium		Scandium	0.026
Bismuth	0.30	Iron	MC	Selenium	0.53
Boron	0.37	Lanthanum	0.19	Silicon	
Bromine	11.	Lead	140.	Silver	0.34
Cadmium	2.8	Lithium	1.1	Sodium	
Calcium	MC	Lutetium	0.0038	Strontium	4.1
Carbon	NR	Magnesium		Sulfur	MC
Cerium	0.30	Manganese	MC	Tantalum	
Cesium	0.64	Mercury	NR	Tellurium	0.15
Chlorine	MC	Molybdenum	1.0	Terbium	0.0075
Chromium	4.9	Neodymium	0.11	Thallium	2.1
Cobalt	7.5	Nickel	3.2	Thorium	0.075
Copper	17.	Niobium	0.037	Thulium	0.0037
Dysprosium	0.030	Nitrogen	NR	Tin	0.19
Erbium	0.015	Osmium		Titanium	4.9
Europium	0.011	Oxygen	NR	Tungsten	0.56
Fluorine	MC	Palladium		Uranium	0.075
Gadolinium	0.022	Phosphorus		Vanadium	0.60
Gallium	1.6	Platinum		Ytterbium	0.019
Germanium	0.19	Potassium	MC	Yttrium	0.075
Gold		Praseodymium	0.037	Zinc	MC
Hafnium		Rhenium		Zirconium	0.30

Table 30

Spark Source Mass Spectrometry Data

Sample No: II C 1 F

<u>Concentration in mg/m³</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum		Holmium	0.0087	Rhodium	
Antimony	0.87	Hydrogen	NR	Rubidium	9.7
Arsenic	MC	Indium	STD	Ruthenium	
Barium	MC	Iodine	3.5	Samarium	0.043
Beryllium	0.0043	Iridium		Scandium	0.022
Bismuth	0.11	Iron	MC	Selenium	0.35
Boron	0.41	Lanthanum	0.11	Silicon	
Bromine	6.5	Lead	20.	Silver	0.15
Cadmium	2.8	Lithium	0.065	Sodium	
Calcium	MC	Lutetium	<0.002	Strontium	7.1
Carbon	NR	Magnesium		Sulfur	
Cerium	0.17	Manganese	MC	Tantalum	
Cesium	0.54	Mercury	NR	Tellurium	0.086
Chlorine		Molybdenum	1.3	Terbium	0.011
Chromium	0.13	Neodymium	0.043	Thallium	0.26
Cobalt	1.7	Nickel	0.065	Thorium	
Copper	10.	Niobium	0.0065	Thulium	<0.002
Dysprosium	0.021	Nitrogen	NR	Tin	0.086
Erbium	0.0065	Osmium		Titanium	2.8
Europium	0.015	Oxygen	NR	Tungsten	0.50
Fluorine	MC	Palladium		Uranium	0.043
Gadolinium	0.022	Phosphorus		Vanadium	0.17
Gallium	1.5	Platinum		Ytterbium	0.0065
Germanium	0.043	Potassium	MC	Yttrium	0.043
Gold		Praseodymium	0.022	Zinc	MC
Hafnium		Rhenium		Zirconium	0.15

Table 31

Spark Source Mass Spectrometry Data

Sample No: II pw

<u>Concentration in mg/m³</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum		Holmium	0.0026	Rhodium	
Antimony	0.41	Hydrogen	NR	Rubidium	5.2
Arsenic	MC	Indium	STD	Ruthenium	
Barium	MC	Iodine	0.44	Samarium	0.026
Beryllium	0.0008	Iridium		Scandium	0.0026
Bismuth	0.15	Iron	MC	Selenium	0.23
Boron	0.087	Lanthanum	0.096	Silicon	
Bromine	2.0	Lead	MC	Silver	0.096
Cadmium	1.1	Lithium	0.22	Sodium	
Calcium	MC	Lutetium	<0.0008	Strontium	0.53
Carbon	NR	Magnesium		Sulfur	
Cerium	0.14	Manganese	MC	Tantalum	
Cesium	0.14	Mercury	NR	Tellurium	0.044
Chlorine		Molybdenum	0.59	Terbium	0.0017
Chromium	0.17	Neodymium	0.026	Thallium	0.57
Cobalt	0.70	Nickel	0.44	Thorium	0.017
Copper	7.1	Niobium	0.0018	Thulium	<0.0008
Dysprosium	0.0044	Nitrogen	NR	Tin	0.017
Erbium	0.0026	Osmium		Titanium	0.070
Europium	0.0053	Oxygen	NR	Tungsten	0.13
Fluorine	5.5	Palladium		Uranium	0.070
Gadolinium	0.0053	Phosphorus		Vanadium	0.070
Gallium	0.53	Platinum		Ytterbium	0.0026
Germanium	0.053	Potassium	MC	Yttrium	0.017
Gold		Praseodymium	0.0088	Zinc	MC
Hafnium	0.0017	Rhenium		Zirconium	0.11

Table 32

Spark Source Mass Spectrometry Data

Sample No: II x

<u>Concentration in mg/m³</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum		Holmium		Rhodium	
Antimony		Hydrogen	NR	Rubidium	
Arsenic		Indium	STD	Ruthenium	
Barium		Iodine		Samarium	
Beryllium		Iridium		Scandium	
Bismuth		Iron		Selenium	
Boron		Lanthanum		Silicon	
Bromine		Lead		Silver	
Cadmium		Lithium		Sodium	MC
Calcium		Lutetium		Strontium	
Carbon	NR	Magnesium		Sulfur	
Cerium		Manganese		Tantalum	
Cesium		Mercury	NR	Tellurium	
Chlorine		Molybdenum		Terbium	
Chromium		Neodymium		Thallium	
Cobalt		Nickel		Thorium	
Copper		Niobium		Thulium	
Dysprosium		Nitrogen	NR	Tin	
Erbium		Osmium		Titanium	
Europium		Oxygen	NR	Tungsten	
Fluorine		Palladium		Uranium	
Gadolinium		Phosphorus		Vanadium	
Gallium		Platinum		Ytterbium	
Germanium		Potassium		Yttrium	
Gold		Praseodymium		Zinc	
Hafnium		Rhenium		Zirconium	

Table 33

Spark Source Mass Spectrometry Data

Sample No: II imp 1

<u>Concentration in mg/m³</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum		Holmium		Rhodium	
Antimony		Hydrogen	NR	Rubidium	
Arsenic	.1	Indium	STD	Ruthenium	
Barium		Iodine		Samarium	
Beryllium		Iridium		Scandium	
Bismuth		Iron		Selenium	0.4
Boron		Lanthanum		Silicon	
Bromine		Lead		Silver	0.4
Cadmium		Lithium		Sodium	
Calcium		Lutetium		Strontium	
Carbon	NR	Magnesium		Sulfur	0.7
Cerium		Manganese	0.7	Tantalum	
Cesium		Mercury	NR	Tellurium	
Chlorine		Molybdenum		Terbium	
Chromium		Neodymium		Thallium	
Cobalt		Nickel	0.5	Thorium	
Copper		Niobium	0.04	Thulium	
Dysprosium		Nitrogen	NR	Tin	
Erbium		Osmium		Titanium	
Europium		Oxygen	NR	Tungsten	
Fluorine	MC	Palladium		Uranium	
Gadolinium		Phosphorus		Vanadium	
Gallium	0.05	Platinum		Ytterbium	
Germanium		Potassium	MC	Yttrium	
Gold		Praseodymium		Zinc	
Hafnium		Rhenium		Zirconium	

Table 34

Spark Source Mass Spectrometry Data

Sample No: coal

<u>Concentration in mg/Kg</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum	>110	Holmium		Rhodium	
Antimony	0.9	Hydrogen	NR	Rubidium	1
Arsenic	11	Indium	STD	Ruthenium	
Barium	810	Iodine	0.2	Samarium	0.8
Beryllium	0.1	Iridium		Scandium	1
Bismuth	220.	Iron	MC	Selenium	3
Boron		Lanthanum	5	Silicon	39
Bromine	2	Lead	9.	Silver	1
Cadmium	2	Lithium	40	Sodium	MC
Calcium	860	Lutetium		Strontium	37
Carbon	NR	Magnesium	350	Sulfur	MC
Cerium	7.	Manganese	MC	Tantalum	
Cesium	0.1	Mercury	NR	Tellurium	
Chlorine		Molybdenum	6	Terbium	0.1
Chromium	26	Neodymium	1	Thallium	
Cobalt	2	Nickel	12	Thorium	≤1
Copper	12	Niobium	1	Thulium	
Dysprosium		Nitrogen	NR	Tin	3
Erbium		Osmium		Titanium	300
Europium	0.2	Oxygen	NR	Tungsten	
Fluorine		Palladium		Uranium	≤0.8
Gadolinium	0.3	Phosphorus	780	Vanadium	9
Gallium	2	Platinum	120.	Ytterbium	
Germanium	≤2.	Potassium	MC	Yttrium	4
Gold		Praseodymium	1	Zinc	33
Hafnium		Rhenium		Zirconium	74.

NR - Not quantified

All blanks are elements not detected, detection limit 0.1 ppm

MC - Major component, >1 g

Table 35

Spark Source Mass Spectrometry Data

Sample No: coke

<u>Concentration in mg/Kg</u>					
<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>	<u>Element</u>	<u>Conc.</u>
Aluminum	MC	Holmium		Rhodium	
Antimony	1	Hydrogen	NR	Rubidium	14
Arsenic	14	Indium	STD	Ruthenium	
Barium	240	Iodine	0.3	Samarium	2
Beryllium	0.5	Iridium		Scandium	4
Bismuth	3	Iron	MC	Selenium	1
Boron		Lanthanum	14	Silicon	MC
Bromine	6	Lead	7	Silver	3
Cadmium	3	Lithium	46	Sodium	MC
Calcium	MC	Lutetium		Strontium	110
Carbon	NR	Magnesium	MC	Sulfur	MC
Cerium	10	Manganese	560	Tantalum	
Cesium	1	Mercury	NR	Tellurium	<0.8
Chlorine		Molybdenum	12	Terbium	0.1
Chromium	38	Neodymium	4	Thallium	
Cobalt	10	Nickel	17	Thorium	3
Copper	30	Niobium	7	Thulium	
Dysprosium		Nitrogen	NR	Tin	5
Erbium		Osmium		Titanium	MC
Europium	0.3	Oxygen	NR	Tungsten	
Fluorine		Palladium		Uranium	4
Gadolinium	0.5	Phosphorus	710	Vanadium	41
Gallium	5	Platinum	0.8	Ytterbium	
Germanium	2	Potassium	MC	Yttrium	5
Gold		Praseodymium	2	Zinc	110
Hafnium		Rhenium		Zirconium	210

NR - Not quantified

All blanks are elements not detected, detection limit 0.1 ppm

MC - Major component, >1 g

5. Microscopic Analysis

The photomicrographs of IC310 and IC1F were made at 1/10 second and those of IIC310 and IIC1F were made at 1/5 second.

The observations on the four particulate samples examined are as follows:

- IC310 - Consisting mainly of isotropic spheres ranging in sizes from 3 to 10 μm , with a few larger, up to 30 μm . While predominantly colorless, the spheres did include some that were opaque, red, green, and yellow. A few non-spherical birefringent particles were also present. All particles had refractive indices greater than 1.515.
- IC1F - Appearing to be identical to IC310, except that the spheres were less agglomerated.
- IIC310 - Containing mostly opaque particles mostly less than 1 μm diameter, but some up to 6 μm in diameter. The larger particles might have been agglomerates of smaller particles. A few birefringent needle like particles were also seen. The refractive indices were greater than 1.515.
- IIC1F - Appearing to be the same as IIC310 except very few particles larger than 1 μm .

IV. Conclusions

These tests at a closed metallurgical furnace ferroalloy production facility were directed towards determination of emissions of particulate and polycyclic organic material.

The particulate emission data acquired during these tests are presented in Table 36. An appropriate reference point for evaluating the particulate loading in the effluent is provided by the new source performance standards for ferroalloy production facilities. These specify that emissions of particulate matter from a control device shall not exceed 0.23 kg/mw-hr while standard ferromanganese or silicomanganese is being produced, and that opacity shall not exceed 15% (21). Observations made by the sampling team indicate that opacity exceeded the U.S. new source performance standard. The measured loadings of 17 kg/mw-hr upstream of the scrubber during ferromanganese production indicate that an efficient particulate control device (>98.6% removal) is required in order to meet the standard. Measurements made downstream of the Venturi scrubber during silicomanganese production show a particulate loading of 0.016 kg/mw-hr. This is well within the new source performance standards. Because of the process change, these data cannot be used to obtain a quantitative estimate of scrubber efficiency for particulate control. At least part of the observed thousand-fold difference in particulate loading between the two tests may be due to the process change. However, it is also quite probable that the Venturi scrubber did have sufficient capacity to control the ferromanganese production particulate emissions at or below the 0.23 kg/mw-hr performance standard at the scrubber exit.

The results of the organic analysis are summarized in Table 37, which lists all categories of compounds found to be present at concentrations of 0.5 mg/m³ or higher. Extremely high quantities of organic materials were found in the ferromanganese effluent gas at the bypass to the gas cleaning system, upstream of the Venturi scrubber. Fused aromatic

Table 36

Summary of Particulate Emission Data

Sampling Site	Upstream of Venturi	Downstream of Venturi
Process	Ferromanganese	Silicomanganese
Effluent Flow Rate		
m ³ /sec	1.20	1.51
m ³ /hr	4300	5400
Particulated Concentration		
mg/m ³	68000	64
Particulate Emissions		
kg/hr	290	0.35
Average Furnace Power		
MW (megawatt)	17.3	22.5
Particulate Emissions		
kg/MW-hr	17	0.016

Table 37

Summary of Organic Analysis Results: Major Components

Concentration, mg/m³ *

Process	Ferromanganese	Silicomanganese
Sampling Site	Upstream of Venturi	Downstream of Venturi
<u>Compound Categories</u>		
Aliphatic Hydrocarbons	6.9	2.5
Aromatic Hydrocarbons	3.7	23
Fused Aromatics < 216 MW	380	14
Fused Aromatics > 216 MW	450	0.02
Heterocyclic N	80	0.1
Heterocyclic S	37	1.7
Ketones	67	0.8
Esters	0.9	1.8
Carboxylic Acids	4.3	0.7
Organic Gases (GC1 & GC2)	1,030ppm	3,090ppm

* Gas volumes are corrected to standard conditions of 101 KPa (29.9" Hg) and 21.1°C (70°F).

hydrocarbons having a wide range of molecular weights were identified. The presence of fused aromatics of molecular weight greater than 216 at 450 mg/m^3 is of particular concern, since this compound category includes some polycyclic aromatic hydrocarbons recognized as carcinogens. Moderate amounts of heterocyclic nitrogen and sulfur compounds as well as polycyclic aromatic ketones were also found in these samples. The concentration of carcinogenic material could be very high in this unscrubbed gas stream.

On the other hand, the major organic compound categories found in the silicomanganese effluent gas after it had passed through a Venturi Scrubber were simple aromatic hydrocarbons and "low" molecular weight fused aromatics, both in the TCO range. The concentration of carcinogenic species appears to be low.

It is significant to note that the Level 2 GC/MS analysis gave results that were in very good agreement with the qualitative and quantitative data generated in the Level 1 organic analysis. (See Tables 18-20).

Because of the different processes sampled, one cannot use these data to quantify the effectiveness of the gas cleaning system for removal of potentially harmful organic species from the effluent. However, examination of the process data allows some inferences to be drawn. The major sources of polycyclic organic material in the ferroalloy process effluents are the self baking carbon electrodes and the coal and coke added to the feed. Table 38 summarizes process data which show that these two potential sources of polycyclic organic material were of comparable magnitude in the two tests. It is reasonable to hypothesize, therefore, that comparable quantities and types of POM compounds were produced in the two ferroalloy processes. The emissions data, also summarized in Table 38, show that total organics collected by the SASS train and aromatic hydrocarbon levels are lower by more than an order of magnitude for the samples collected downstream of the

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scrubber. Furthermore, the emissions of high molecular weight POM are lower by more than four orders of magnitude for samples collected at the Venturi exit. These reductions in POM emissions are almost certainly too large to be accounted for by the process change alone. The Venturi scrubber appears to be effective for POM removal and especially efficient for species in the molecular weight range ($MW > 216$) that includes the recognized carcinogenic POM. This is consistent with the fact that the higher molecular weight POM have lower volatility, are more condensable, and are probably scrubbed from the quenched gas stream as particulate material (condensed, or adsorbed on solid particulate).

The on-site gas analysis data indicated that emissions of gaseous hydrocarbons (GC1 and GC2, b.p. $< 50^{\circ}\text{C}$) were higher in the silicomanganese test than in the ferromanganese test, by a factor of three. Levels of these gaseous species would of course be essentially unaffected by the wet scrubber. It could be possible that these results indicate a significant shift in the chemical composition of the organic emissions, with the silicomanganese process yielding a much higher gas-to-POM ratio than the ferromanganese process. This seems unlikely in view of the general similarity of the two ferroalloy process chemistries. The most plausible conclusion from these results is that total organic emissions (gases plus SASS) may have been somewhat higher in the silicomanganese test than in the ferromanganese, and that the scrubber was even more effective for POM removal than the data in Table 38 imply.

Inorganic chemical emissions from ferroalloy plants were not the major focus of these tests. However, two features of the inorganic analysis data are worthy of comment. First it should be noted that, while trace metal levels in the effluent from the Venturi scrubber during silicomanganese production were low (generally $< 1 \text{ mg/m}^3$), the estimated arsenic emission level is $250 \text{ } \mu\text{g/m}^3$ (Table 21).

Comparison of this estimate with the EPA Multimedia Environmental Goals - Minimum Acute Toxicity Effluent (MEG-MATE) criterion of $2 \mu\text{g}/\text{m}^3$ for arsenic and its compounds (22), suggests that more extensive, Level 2 analyses of arsenic in ferroalloy plant emissions may be warranted.

Second, it is interesting to compare the results of the atomic absorption spectroscopic (AAS) and spark source mass spectroscopic (SSMS) analyses for the two elements that were determined by both techniques. Table 39 presents the results of the arsenic and antimony determinations for a number of the SASS train sample components. (Samples for which the SSMS result was "major component" are generally omitted from the table). The agreement between the AAS and SSMS data for arsenic is surprisingly good. In fact, the agreement is generally much better than could be expected, considering that individual SSMS determinations are uncertain within a factor of two or three. The agreement between AAS and SSMS for antimony is not quite so good. Note, however, that the antimony concentrations are low about 10^3 times lower than arsenic levels. The two sets of antimony data do agree, within a factor of ten, for the two samples corresponding to concentrations of about $100 \mu\text{g}/\text{m}^3$. Since the MATE value for antimony is $500 \mu\text{g}/\text{m}^3$ (22), these results suggest that SSMS analysis may be as adequate for both antimony and arsenic, as it is for analysis of other trace inorganics at Level 1.

Table 38

Summary of Process and Effluent Parameters

Sampling Site	Upstream of Venturi	Downstream of Venturi
Process	Ferromanganese	Silicomanganese
Electrode Consumption		
lb/day*	6,150	9,000
lb/m ³ of stack gas **	0.059	0.069
Coal/coke content of Feed, %*	14.5	14.9
<u>Emissions, mg/m³</u>		
Total Organics (SASS train)	1,200	50
Total Aromatics	830	26
Aromatics of MW > 216	450	0.02
Volatile Organics (GC1 & GC2)	1,030	3,090

* Calculated from data in Table 1

** Calculated from data in Tables 1 and 6

Table 39

Comparison of AAS and SSMA Data for
Arsenic and Antimony in Selected Samples

<u>Sample</u>	<u>Arsenic</u> mg/m ³		<u>Antimony</u> μg/m ³	
	<u>AAS</u>	<u>SSMS</u>	<u>AAS</u>	<u>SSMS</u>
<u>Particulates</u>				
I C 310	0.018	0.022	0.02	0.39
II C 310	24	MC* (>27)	150	660
II C IF	25	MC* (>21)	88	840
<u>Impingers</u>				
I Imp I	0.0062	0.0068	0.025	n.d.**
II Imp I	0.15	0.15	1.3	n.d.**
<u>Solids Parr Bombed for SSMS</u>				
I x	0.098	n.d.**	1.	n.d.**
II x	1.03	n.d.**	19.	n.d.**
Coal	20 mg/kg	11 mg/kg	0.3 mg/kg	0.9 mg/kg
Coke	20 mg/kg	14 mg/kg	0.6 mg/kg	1 mg/kg

* Major Component

** Not detectable, or < 0.1 ppm weight in sample analyzed.

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APPENDIX A

LEVEL 1 ORGANIC ANALYSIS DATA

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LC REPORT

SAMPLE: IX, XAD-2 extract, Silicomanganese

	TCO mg	GRAV mg	Total mg	Concentration ⁵ mg/M ³
Total Sample ¹	1440	70	1510	47
Taken for LC ²	57.4	2.8	60.	
Recovered ³	50.3	2.6	53	

Fraction	TCO ⁴ mg	GRAV ⁴ mg	Total ⁴ mg	Concentration ⁵ mg/M ³
1	74	ND ⁶	74.	2.32
2	484	10.	494.	15.4
3	628	5.0	633	19.7
4	1.10	ND	1.10	0.03
5	5.61	ND	5.61	0.17
6	44.	50.	94.	2.93
7	23.	ND	23.	0.74

1. Quantity in entire sample, determined before LC
2. Portion of whole sample used for LC, actual mg
3. Quantity recovered from LC column, actual mg
4. Total mg computed back to total sample
5. Total mg divided by total volume
6. NOT detectable

LRMS REPORT

SAMPLE: IX-2, XAD-2 extract, silicomanganese

Major Categories

Intensity	Category	MW Range
100	Aromatic Hydrocarbons	92-196
10	Fused Aromatics <216	178-202
1	Heterocyclic Sulfur Compounds	184

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	Naphthalene	128	C ₁₀ H ₈
10	Toluene	92	C ₇ H ₈
10	Xylene	106	C ₈ H ₁₀
10	Indene	116	C ₉ H ₈
10	Methyl Naphthalene	142	C ₁₁ H ₁₀
10	Biphenylene / Acenaphthylene	152	C ₁₂ H ₈
10	Anthracene / phenanthrene	178	C ₁₄ H ₁₀
1	Alkyl Naphthalenes	156-170	C ₁₂ H ₁₂ - C ₁₃ H ₁₄
1	Alkyl Biphenyls / Acenaphthalenes	168-196	C ₁₃ H ₁₂ - C ₁₅ H ₁₆
1	Dibenzothiophene	184	C ₁₂ H ₈ S
1	methyl Anthracene / phenanthrene	192	C ₁₅ H ₁₂
1	Pyrene, etc	202	C ₁₆ H ₁₀

Other

LRMS REPORT

SAMPLE: IX-3 XAD-2 extract, silico manganese

Major Categories

Intensity	Category	MW Range
100	Aromatic Hydrocarbons	116-202
100	Fused Aromatics < 216	166-202
10	Heterocyclic S compds.	184

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	Naphthalene	128	C ₁₀ H ₈
100	Biphenylene / Acenaphthylene	152	C ₁₂ H ₈
100	Fluorene	166	C ₁₃ H ₁₀
100	Anthracene	178	C ₁₄ H ₁₀
10	Indene	116	C ₉ H ₈
10	Alkyl naphthalenes	142, 156	C ₁₁ H ₁₀ , C ₁₂ H ₁₂
10	Biphenyl / Acenaphthalene	154	C ₁₂ H ₁₀
10	Alkyl Biphenyls / Acenaphthalene	168-196	C ₁₃ H ₁₂ - C ₁₅ H ₁₆
10	Bibenzothiophene	184	C ₁₂ H ₈ S
10	methyl Anthracene / phenanthrene	192	C ₁₅ H ₁₂
10	Pyrene: etc.	202	C ₁₆ H ₁₀

Other

IR REPORT

SAMPLE: IX-5, LCS, XAO extract, Venturi scrubber

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
No significant IR absorptions			

IR REPORT

SAMPLE: IX-7, LC7, XAO extract, Venturi scrubber

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
No significant IR absorptions			

LC REPORT

SAMPLE: ISC, Sorbent condensate, Silicomanganese

	TCO mg	GRAV mg	Total mg	Concentration ⁵ mg/M ³
Total Sample ¹	18.3	65	83.3	2.59
Taken for LC ²	14.7	52.	67.	
Recovered ³	14.7	65.3	80.	

Fraction	TCO ⁴ mg	GRAV ⁴ mg	Total ⁴ mg	Concentration ⁵ mg/M ³
1	0.03	8.44	8.47	0.26
2	0.01	1.50	1.51	0.05
3	18.3	68.5	87.	2.70
4	0.06	ND.	0.06	0.002
5	ND	0.25	0.25	0.008
6	ND	2.75	2.75	0.008
7	0.02	0.25	0.27	0.008

1. Quantity in entire sample, determined before LC
2. Portion of whole sample used for LC, actual mg
3. Quantity recovered from LC column, actual mg
4. Total mg computed back to total sample
5. Total mg divided by total volume
6. Not detectable

IR REPORT

SAMPLE: ISC-3, LC3, sorbent cond. Venturi scrubber

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3100-3000	m	CH, aromatic	
2950-2850	W	CH, aliphatic	
1600-900	m	numerous sharp peaks, aromatic ring	
810, 730, 710	S	aromatic ring	

IR REPORT

SAMPLE: ISC-4, LC4, sorbent cond. Venturi scrubber

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3400	W	OH or NH	
3100-3000	W	CH, aromatic	
3000-2850	S	CH, aliphatic	
1740	W	C=O ester, or overture	
1600, 1500	W	aromatic ring	
1250, 1080	S	Si-CH ₃ , Si-O	
1020, 800	S	ether	
1200	S	Sulfite, ether	
1050	S	sulfoxide	
800-700	m	aromatic	

LC REPORT

SAMPLE: IX, XAD-2 extract Ferromanganese

	TCO mg	GRAV mg	Total mg	Concentration ⁵ mg/M ³
Total Sample ¹	279	1230	1510	1110
Taken for LC ²	18.6	74.2	92.8	
Recovered ³	17.0	68.2	85.2	

Fraction	TCO ⁴ mg	GRAV ⁴ mg	Total ⁴ mg	Concentration ⁵ mg/M ³
1	2.25	3.0	5.25	3.86
2	9.45	ND ⁶	9.45	6.95
3	195.	867	1062.	781
4	1.21	48	49	36
5	6.15	3.0	9.15	6.73
6	29.7	90	120.	88.
7	11.	12.	23.	17.

1. Quantity in entire sample, determined before LC
2. Portion of whole sample used for LC, actual mg
3. Quantity recovered from LC column, actual mg
4. Total mg computed back to total sample
5. Total mg divided by total volume
6. Not detectable

LRMS REPORT

SAMPLE: ILX-3, XAD-2 extract, Ferromanganese

Major Categories

Intensity	Category	MW Range
100	fused Aromatics <216	152-210
100	fused Aromatics >216	216-350
10	Heterocyclic S compds	184

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	Anthracene / phenanthrene	178	C ₁₄ H ₁₀
100	Pyrene / Fluoranthene	202	C ₁₆ H ₁₀
100	Chrysene, Benzanthracenes	228	C ₁₈ H ₁₂
100	Benzo[a]pyrene, perylene	252	C ₂₀ H ₁₂
10	Acenaphthalene	152	C ₁₂ H ₁₀
10	Fluorene	166	C ₁₃ H ₁₀
10	methyl Acenaphthalene	168	C ₁₃ H ₁₂
10	Dibenzothiophene	184	C ₁₂ H ₈ S
10	methyl anthracene	192	C ₁₅ H ₁₂
10	Benzo[a]fluorenes	216	C ₁₇ H ₁₂
10	Benzo[a]naphthalene	218	C ₁₇ H ₁₄

Other

10	PAH at m/e 230-302
1	PAH at m/e 190-350

LRMS REPORT

SAMPLE: IX-4, XAD2 extract, Ferromanganese

Major Categories

Intensity	Category	MW Range
100	Heterocyclic N compds	167-267
100	Fused Aromatics >216	228-302
10	Fused Aromatics <216	178-202
10		

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	carbazole	167	C ₁₃ H ₉ N
100	Benzocarbazole	217	C ₁₆ H ₁₁ N
100	Benzo pyrenes	252	C ₂₀ H ₁₂
10	Anthracene/phenanthrene	178	C ₁₄ H ₁₀
10	methyl carbazole	181	C ₁₄ H ₁₁ N
10	Dimethyl carbazole	195	C ₁₅ H ₁₃ N
10	pyrene	202	C ₁₆ H ₁₀
10	Benzo anthracenes, chrysene	228	C ₁₈ H ₁₂
10	methyl Benzo Carbazole	231	C ₁₇ H ₁₃ N
10	Dibenzocarbazole	267	C ₂₀ H ₁₃ N
10	Benzo perylene	276	C ₂₂ H ₁₂
10	methyl cholanthrene	268	C ₂₁ H ₁₆
10	Dibenzo chrysenes	302	C ₂₂ H ₁₄

Other

10	PAH at m/e 191, 241, 243, 245, 257, 258, 326
1	PAH at m/e 200 to over 400

LRMS REPORT

SAMPLE: IX-5, XAD-2 extract, Ferromanganese

Major Categories

Intensity	Category	MW Range
100	Heterocyclic N compds	167-253
100	Ketones	230-280

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	Carbazole	167	C ₁₂ H ₉ N
100	Benzoanthrone	230	C ₁₇ H ₁₀ O
10	Naphthoisoxyamid	153	C ₁₁ H ₇ N
10	methyl carbazole	181	C ₁₃ H ₁₁ N
10	4 ring Heterocyclic N	203	C ₁₅ H ₉ N
10	Benzo carbazole	217	C ₁₆ H ₁₁ N
10	4 ring N	227	C ₁₇ H ₉ N
10	5 ring N	253	C ₁₉ H ₁₁ N
10	Dibenzo fluorenone	280	C ₂₁ H ₁₂ O

Other

10	PAH at m/e 254
1	Heterocyclic N, m/e 129-379
1	Oxygen-compds m/e 180-380

LRMS REPORT

SAMPLE: IX-6, XAD-2 extract, Ferromanganese

Major Categories

Intensity	Category	MW Range
100	Heterocyclic N compds	179-303
100	Ketones	180-304
10	Carboxylic Acids	122

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	Acridine	179	C ₁₃ H ₉ N
100	Fluorenone	180	C ₁₃ H ₈ O
100	4 ring Heterocyclic N	203	C ₁₅ H ₉ N
100	" " O	204	C ₁₅ H ₈ O
100	Anthraquinoline	229	C ₁₇ H ₁₁ N
100	Benzanthrone	230	C ₁₇ H ₁₀ O
10	Benzoic Acid	122	C ₇ H ₆ O ₂
10	methyl Acridine	193	C ₁₄ H ₁₁ N
10	methyl fluorenone	194	C ₁₄ H ₁₀ O
10	Dimethyl Acridine	207	C ₁₅ H ₁₃ N
10	Anthraquinone	208	C ₁₄ H ₈ O ₂
10	Benzocubazole	217	C ₁₆ H ₁₁ N
10	methyl Anthraquinoline	243	C ₁₈ H ₁₃ N
10	5 ring N	255	C ₁₉ H ₁₁ N
10	6 ring N	303	
10	Dibenz acridine	279	C ₂₁ H ₁₃ N

Other

10	PAH at m/e 219, 244, 254, 268, 280, 304
1	PAH at m/e 265-380

LRMS REPORT

SAMPLE: IX-7. XAD-2 Extract. Ferromanganese

Major Categories

Intensity	Category	MW Range
10	Heterocyclic N Compds	129-303
10	Ketones	230-280
1	Esters	136

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
10	Acridine	179	C ₁₃ H ₉ N
10	4 ring N	203	C ₁₅ H ₉ N
10	Benzo carbazole	217	C ₁₆ H ₁₁ N
10	Anthraquinoline	229	C ₁₇ H ₁₁ N
10	Benzanthrone	230	C ₁₇ H ₁₀ O
10	5. ring Heterocyclic N	253	C ₁₉ H ₁₁ N
10	6 " " " N	279	C ₂₁ H ₁₃ N
10	Dibenzofluorenone	280	C ₂₁ H ₁₂ O
1	Quinoline	129	C ₉ H ₇ N
1	Alkyl quinolines	143-171	C ₁₀ H ₉ N - C ₁₂ H ₁₃ N
1	methyl Benzoate	136	C ₈ H ₈ O ₂
1	methyl Acridine	193	C ₁₄ H ₁₁ N

Other

1	PAH at m/e 200-329

IR REPORT

SAMPLE: IX-5, LC5, XAD extract, Ferromanganese

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3400	W	NH or OH	
3050	W	CH, aromatic	
2220	W	C≡N, N≡C=O	
1700	W	C=O	
1600, 1450	W	sharp bands, aromatic ring	
750, 700	M	aromatic subst.	

IR REPORT

SAMPLE: IX-6, LC6, XAD extract, Ferromanganese

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3500-3100	W	OH or NH	
3100-3000	W	CH, aromatic, olefinic	
3000-2800	W	CH, aliphatic	
1710	S	C=O	
1670	W	C=C	
1600, 1500	W	aromatic ring	
1450-1100	W	aromatic ring, sharp bands	
820	W	aromatic subst.	
750, 720	S	aromatic subst.	

LC REPORT

SAMPLE: II CIF, particulates < 10, Ferromanganese

	TCO mg	GRAV mg	Total mg	Concentration ⁵ mg/M ³
Total Sample ¹	-	66.	66.4	48.8
Taken for LC ²	-	13.2	13.2	
Recovered ³	-	13.2	13.2	

Fraction	TCO ⁴ mg	GRAV ⁴ mg	Total ⁴ mg	Concentration ⁵ mg/M ³
1	-	ND	ND	ND
2	-	ND	ND	ND
3	-	32	32	23
4	-	14	14	10
5	-	ND	ND	ND
6	-	18	18	13
7	-	2.0	2.0	1.47

1. Quantity in entire sample, determined before LC
2. Portion of whole sample used for LC, actual mg
3. Quantity recovered from LC column, actual mg
4. Total mg computed back to total sample
5. Total mg divided by total volume
6. Not Detectable

LRMS REPORT

SAMPLE: Monsanto Ferro Alloy II - CIF-3

Major Categories

Intensity	Category	MW Range
100	Fused alternate/nonalternate hydrocarbons	> 216
1	Fused alternate/non-alternate hydrocarbons	< 216

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	Benzyrenes, etc.	252	C ₂₀ H ₁₂
100	Dibenzchrysenes, etc	276	C ₂₂ H ₁₂
100	Dibenzanthracene	278	C ₂₂ H ₁₄
10	Methyl-benzyrenes, etc.	266	C ₂₁ H ₁₄
10	Methyl-dibenzanthracene	292	C ₂₃ H ₁₆
10	Dibenzpyrene, etc	302	C ₂₄ H ₁₄
1	Benzantracene, etc.	228	C ₁₈ H ₁₂
1	Methyl benzantracene	242	C ₁₉ H ₁₄
1	pyrene / fluoranthene	202	C ₁₆ H ₁₀
1	anthracene / phenanthrene	178	C ₁₄ H ₁₀
1	Biphenyl / Acenaphthene	154	C ₁₂ H ₁₀

Other

1	fused alternate, nonalternate hydrocarbons	302 < m/e ≤ 452

LRMS REPORT

SAMPLE: Monsanto Ferro Alloy II - CIF-6

Major Categories

Intensity	Category	MW Range
100	Ketones	200-300+
100	Heterocyclic Nitrogen Compounds	200-300+
1	Esters	222
1	Carboxylic Acids	122

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100		280	
100	6-ring N	303	
100	6-ring O (Ketones)	304	
10	Dibenzacridine, etc	279	C ₂₁ H ₁₃ N
10	Fluorenone	180	C ₁₃ H ₈ O
10	Phenanthridone	195	C ₁₃ H ₉ NO
10	Methyl phenanthridone	209	C ₁₄ H ₁₀ NO
10		219	
10	Benzacridine, Anthraquinoline, etc	229	C ₁₇ H ₁₁ N
10		236	
10	5-ring-N (Heterocyclic N)	253	C ₁₉ H ₁₁ N
10	5-ring-O (Ketone)	254	C ₁₉ H ₁₀ O
10		270	
10		277	
1	Benzoic Acid	122	C ₇ H ₆ O ₂
1	Diethyl phthalate	222	C ₁₂ H ₁₄ O ₄
1		203	C ₁₅ H ₉ N
1		204	C ₁₅ H ₉ O

Other

1	polycyclics with 300 < m/e ≤ 500

IR REPORT

SAMPLE: ILCF-5, particulates < 1 μ, Ferromanganese

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3600-3100	w	OH, NH, broad	
3100-3000	w	CH, aromatic	
3000-2800	m	CH, aliphatic	
1700	m	C=O, ketone, acid	
1650, 1620	w	amide, ketone	
1600	w	amide, ketone, aromatic ring	
1250, 1060	m	ester, alcohol,	
1020			
750	s	aromatic subst.	

IR REPORT

SAMPLE: ILCF-6, particulates < 1 μ, Ferromanganese

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3600-2400	w	OH, NH, broad	
3100-3000	m	CH, aromatic	
3000-2800	s	CH, aliphatic	
1740, 1710	s	C=O, ester, ketone, lactone lactam, imide	
1670, 1650	s	ketone, amide, amidine,	
1630, 1610, 1600		nitrate, aromatic subst, C=C	
1230, 1170	s	ester	
1340, 1300	s	amine	
1120, 1020	m	alcohol,	
820, 750	s	aromatic subst.	

LC REPORT

SAMPLE: ILPW, Probe wash, Ferrumanganese

	TCO mg	GRAV mg	Total mg	Concentration ⁵ mg/M ³
Total Sample 1	—	51	51	37.
Taken for LC ²	—	25	25	
Recovered ³	—	32	32	

Fraction	TCO ⁴ mg	GRAV ⁴ mg	Total ⁴ mg	Concentration ⁵ mg/M ³
1	—	2.65	2.65	1.95
2	—	ND ⁶	ND	ND
3	—	39.	39.	28.
4	—	11.	11.	7.84
5	—	0.73	0.73	0.54
6	—	9.81	9.81	7.21
7	—	1.96	1.96	1.44

1. Quantity in entire sample, determined before LC
2. Portion of whole sample used for LC, actual mg
3. Quantity recovered from LC column, actual mg
4. Total mg computed back to total sample
5. Total mg divided by total volume
6. NOT Detectable.

LRMS REPORT

SAMPLE: Monsanto Ferro Alloy II - PW-3

Major Categories

Intensity	Category	MW Range
100	Fused Alternata / Non-alternata Hydrocarbons	< 216
100	Fused Alternata / Non-alternata Hydrocarbons	> 216

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	Pyrene / Fluoranthene	202	C ₁₆ H ₁₀
100	Benzoanthracene, etc.	228	C ₁₈ H ₁₂
100	Benzopyrenes, etc	252	C ₂₀ H ₁₂
10	Anthracene / Phenanthrene	178	C ₁₄ H ₁₀
10	Benzofluorene, etc	216	C ₁₇ H ₁₂
10		218	C ₁₇ H ₁₄
10	Methyl-benzanthracene	242	C ₁₉ H ₁₄
10	Dibenz chrysene	276	C ₂₂ H ₁₂
10	Dibenzanthracene	278	C ₂₂ H ₁₄
10	Dibenzopyrene	302	C ₂₄ H ₁₄
10		326	

Other

1	Polycyclics with 326 < m/e ≤ 450

LRMS REPORT

SAMPLE: Monsanto Ferro Alloy II-PW-4

Major Categories

Intensity	Category	MW Range
100	Fused alternate/non-alternate hydrocarbons	> 216
1	Fused alternate/non-alternate hydrocarbons	< 216
1	Heterocyclic Nitrogen compounds	217

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	Chrysene / Benzanthracene	228	C ₁₈ H ₁₂
100	Benzo pyrene, etc.	252	C ₂₀ H ₁₂
10	Dibenzchrysene	276	C ₂₂ H ₁₂
10	Dibenzanthracene	278	C ₂₂ H ₁₄
10	Dibenz pyrene	302	C ₂₄ H ₁₄
10		326	
1	Benzocarbazole	217	C ₁₆ H ₁₁ N
1		242	C ₁₉ H ₁₄
1		258	
1	Pyrene / Fluoranthene	202	C ₁₆ H ₁₀
1		376	

Other

< 1	polycyclics below 200
1	polycyclics 380 ≤ m/e ≤ 460

LRMS REPORT

SAMPLE: Monsanto Ferro Alloy II - PW-6

Major Categories

Intensity	Category	MW Range
100	Ketones	300+
10	Heterocyclic Nitrogen Compounds	200 - 300+
1	Esters	

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
100	Benzanthrone, etc.	230	C ₁₇ H ₁₀ O
10	phthalate		
10		200	
10		202	
10		203	C ₁₅ H ₉ N
10		243	
10		244	
10	5 ring - N	253	C ₁₉ H ₁₁ N
10	5 ring O (polycyclic Aromatic ketone)	254	C ₁₉ H ₁₀ O
10		258	
10	Dibenzacridine, etc.	279	C ₂₁ H ₁₃ N
10		280	
10		302	
10	6-ring N	303	
10	6-ring O (Ketone on 6-fused rings)	304	
1	Fluorenone, etc.	180	C ₁₃ H ₈ O

Other

1	polycyclics with 305 ≤ m/e < 460

LRMS REPORT

SAMPLE: Monsanto Ferro Alloy II - PW-7

Major Categories

Intensity	Category	MW Range
10	Ketones	200 - 300+
1	Heterocyclic Nitrogen Compounds	179 - 300+

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
10	Benzanthrones, etc.	230	C ₁₇ H ₁₀ O
1	Acridine	179	C ₁₃ H ₉ N
1		203	C ₁₅ H ₉ N
1		204	
1		229	C ₁₇ H ₁₁ N
1		253	C ₁₉ H ₁₁ N
1		254	
1		279	C ₂₁ H ₁₃ N
1		280	
1	6- ring N	303	
1		304	

Other

1	polycyclics up to m/e 350

IR REPORT

SAMPLE: IPW-3, LC3, Probe Wash, Ferromanganese

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3100-3000	S	CH, aromatic, olefinic	
1930	W	C=C=CH ₂ allene	
3000-2800	W	CH, aliphatic	
1600-1000	M	numerous sharp bands	
900		aromatic ring	
900-700	S	multiple bands: aromatic substitution, fused rings	

IR REPORT

SAMPLE: IPW-4, LC4, probe wash, Ferromanganese

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3500-3200	W	NH or OH	
3100-3000	M	CH, aromatic, olefinic	
3000-2800	W	CH, aliphatic	
1600	M	C=C, aromatic ring	
1500-1000	M-W	multiple bands, aromatic ring	
900-700	S	aromatic substitution	

IR REPORT

SAMPLE: II PW-5, L65, Probe wash, Ferromanganese

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3500-3200	m	OH, NH	
3000-3000	m	CH, aromatic, olefinic	
3000-2800	S	CH, aliphatic	
2210	W	C≡N nitrile	
1730	m	C=O ester	
1700	S	C=O, ketone, acid, carbamate imide, cyclic imide	
1650	W	C=C	
1600, 1580	m	aromatic substitution	
1450, 1350		Amines	
1520, 1350	m	Nitro aromatic	
1280, 1120	W	aromatic ester	
820, 750	S	aromatic substitution	

IR REPORT

SAMPLE: II PW-6, L66, Probe wash Ferromanganese

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3500-3200	W	OH, NH	
3100-3000	m	CH, aromatic, olefinic	
3000-2800	S	CH, aliphatic	
1720	S	C=O ester	
1700	S	C=O, acid, ketone	
1660, 1620	S	amide, nitrite	
1580, 1300	S	N-NO ₂ , nitramine	
1270, 1120	S	aromatic ester	
1230, 1210	S	ester	
1180, 1060		alcohol	
750	S	aromatic substitution, C-Cl	

LC REPORT

SAMPLE: COAL (CL)

	TCO mg	GRAV mg	Total mg	Concentration ⁵ mg/Kg
Total Sample 1	4.0	35	39	464
Taken for LC ²	2.8	25	28	
Recovered ³	1.0	30	31	

Fraction	TCO ⁴ mg	GRAV ⁴ mg	Total ⁴ mg	Concentration ⁵ mg/kg
1	0.36	24	24	286
2	ND ⁶	2.0	2.0	24
3	0.80	7.7	8.5	101
4	0.014	2.9	2.9	35
5	ND	1.1	1.1	13
6	0.29	4.9	5.2	62
7	-	0.86	0.86	10

1. Quantity in entire sample, determined before LC
2. Portion of whole sample used for LC, actual mg
3. Quantity recovered from LC column, actual mg
4. Total mg computed back to total sample
5. Total mg divided by total volume
6. NOT DETECTABLE

LRMS REPORT

SAMPLE: MONSANTO FERRO ALLOY COAL CL-4

Major Categories

Intensity	Category	MW Range
100	HETEROCYCLIC NITROGEN COMPOUNDS	167-323+
100	FUSED ALTERNATE, NON ALTERNATE H/C \geq m/e 216	252-~500
1	" " " " " " $<$ m/e 216	178-202
1	SULFUR	256
1	ESTERS	-

Sub-Categories, Specific Compounds

Intensity	Category	m/e	Composition
10	CARBAZOLE	167	$C_{12}H_9N$
10	ALKYL CARBAZOLES	181-231	$C_{12}H_{11}N - C_{17}H_{19}N$
10	BENZO CARBAZOLE	217	$C_{16}H_{11}N$
10	ALKYL BENZO CARBAZOLES	231-287	$C_{17}H_{19}N - C_{21}H_{21}N$
10	DIBENZO CARBAZOLE	267	$C_{20}H_{13}N$
10	ALKYL DIBENZO CARBAZOLES	281-323	$C_{21}H_{15}N - C_{24}H_{21}N$
10	CHRYSENE/BENZANTHRACENE, ETC	228	$C_{18}H_{12}$
10	BENZO PYRENES, ETC	252	$C_{20}H_{12}$
1	ANTHRACENE / PHENANTHRENE	178	$C_{14}H_{10}$
1	DI HYDRO ANTHRACENE / PHENANTHRENE	180	$C_{14}H_{12}$
1	PYRENE / FLUORANTHENE	202	$C_{16}H_{10}$
1	SULFUR	256	S_8
1	PHTHALATE		

Other

100	UNIDENTIFIED POLYCYCLICS (EXTENSIVE ALKYLATION PRESENT), OTHER THAN THOSE ABOVE, m/e 230 TO >500
-----	--

IR REPORT

SAMPLE: CL-1, coal

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3000-2800	m	CH, aliphatic	
1450, 1370	w	CH, "	

IR REPORT

SAMPLE: CL-2, coal

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3000-2800	m	CH, aliphatic	
1450, 1370	w	CH, "	

IR REPORT

SAMPLE: LC3, coal

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3100-3000	m	CH, aromatic, olefinic	
3000-2800	S	CH, aliphatic	
1600	m	aromatic ring	
1450	S	aliphatic CH ₂ , CH ₃	
1370	m	" " "	
1300, 1280		broad weak bands	
1250, 1020	W		
950			
870, 800	S	aromatic substitution	
740			

IR REPORT

SAMPLE: CL4, coal

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3450	W	OH or NH, broad	
3100-3000	m	CH, aromatic, olefinic	
3000-2800	S	CH, aliphatic	
1700	W	acid, ketone, lactam, imide	
1600, 1450	m	aromatic ring	
1400-1300	W	C-N, amines	
1020	W	alcohol	
800, 750	m	aromatic subst., pyridine	

IR REPORT

SAMPLE: CL-5, coal

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3600-3100	W	OH, NH	broad
3050	W	CH	aromatic
3000-2800	S	CH	aliphatic
1600, 1700	m	acid, ketone	aromatic
1450, 1370	m	CH ₂ , CH ₃	aromatic
870, 800	W	aromatic	pyridine
750	m	"	"

IR REPORT

SAMPLE: CL-6, coal

Frequency (cm ⁻¹)	Intensity	Assignment	Comments
3600-3100	m	OH or NH	broad
3100-3000	W	CH	aromatic
3000-2800	S	CH	aliphatic
2720	W	aldehyde	
1700	S	Ketone	acid
1780	W	aldehyde	
1660, 1610, 810	S	Nitrite	
1660, 1280, 870	S	Nitrate	
1020	S	alcohol	
750	m	aromatic subst	C-Cl

LC REPORT

SAMPLE: COKE (CK)

	TCO mg	GRAV mg	Total mg	Concentration ⁵ mg/kg
Total Sample ¹	0.30	17.	17.	270
Taken for LC ²	0.21	12.	12.	
Recovered ³	0.50	11.	12.	

Fraction	TCO ⁴ mg	GRAV ⁴ mg	Total ⁴ mg	Concentration ⁵ mg/kg
1	0.36	10.	10.	158
2	ND	ND	ND	ND
3	0.14	0.86	1.0	16
4	ND ⁶	0.86	0.86	14
5	ND	1.4	1.4	22
6	ND	0.6	0.6	10
7	ND	0.6	0.6	10

1. Quantity in entire sample, determined before LC
2. Portion of whole sample used for LC, actual mg
3. Quantity recovered from LC column, actual mg
4. Total mg computed back to total sample
5. Total mg divided by total volume
6. NOT DETECTABLE

LC Report

Sample: Solvent Blank, B, (ADL Methylene Chloride, 2500 mL)

	TCO, mg	GRAV, mg
Taken for LC	0.007	0
Recovered	0.02	2.4
Fraction 1	<< 0.01	< 0.1
2	<< 0.01	< 0.1
3	0.02	0.6
4	<< 0.01	< 0.1
5	<< 0.01	< 0.1
6	<< 0.01	0.8
7	<< 0.01	1.0

LC Report

Sample: Blank, Methylene Chloride (from field, 828 mL)

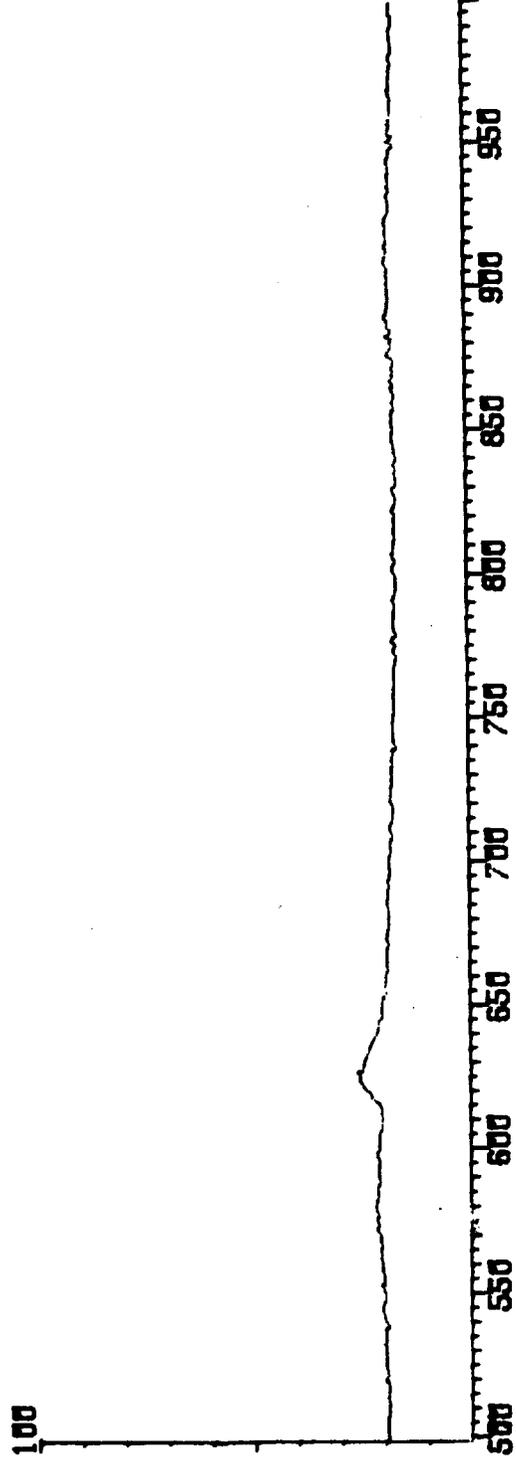
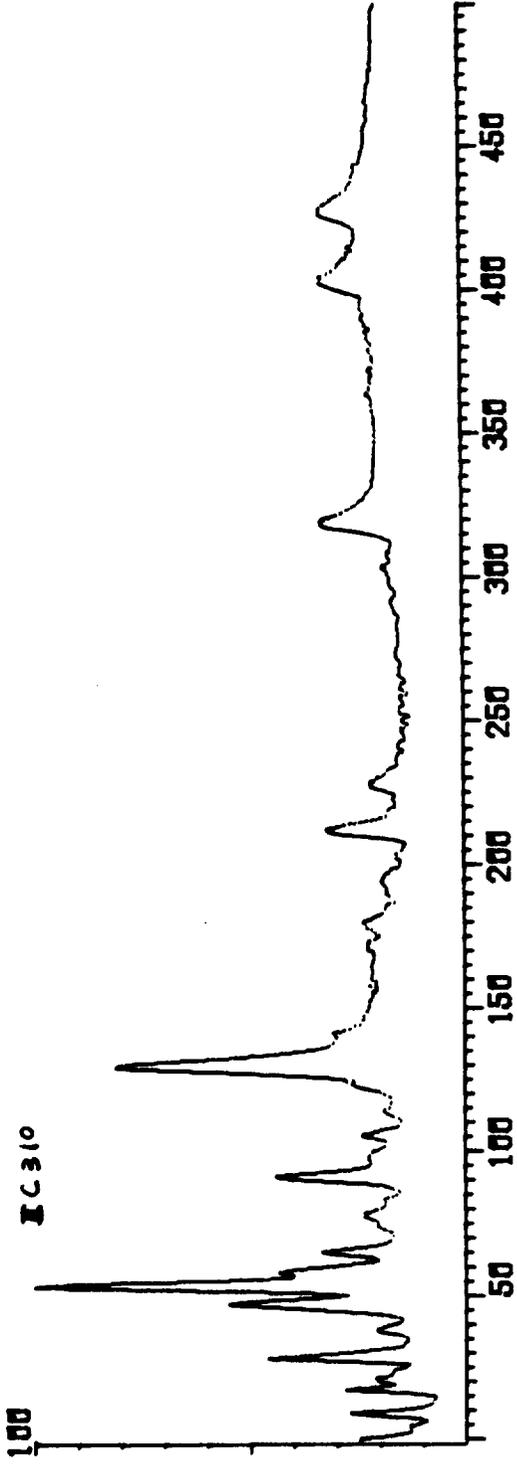
		TCO, mg	GRAV, mg
Taken for LC		0.15	2.1
Recovered		0.14	2.1
Fraction	1	<< 0.01	0.5
	2	<< 0.01	< 0.1
	3	0.01	0.4
	4	<< 0.01	0.6
	5	0.02	< 0.1
	6	0.01	< 0.1
	7	0.1	0.6

LC Report

Sample: Blank, Methylene Chloride/Methanol (from field, 541 mL)

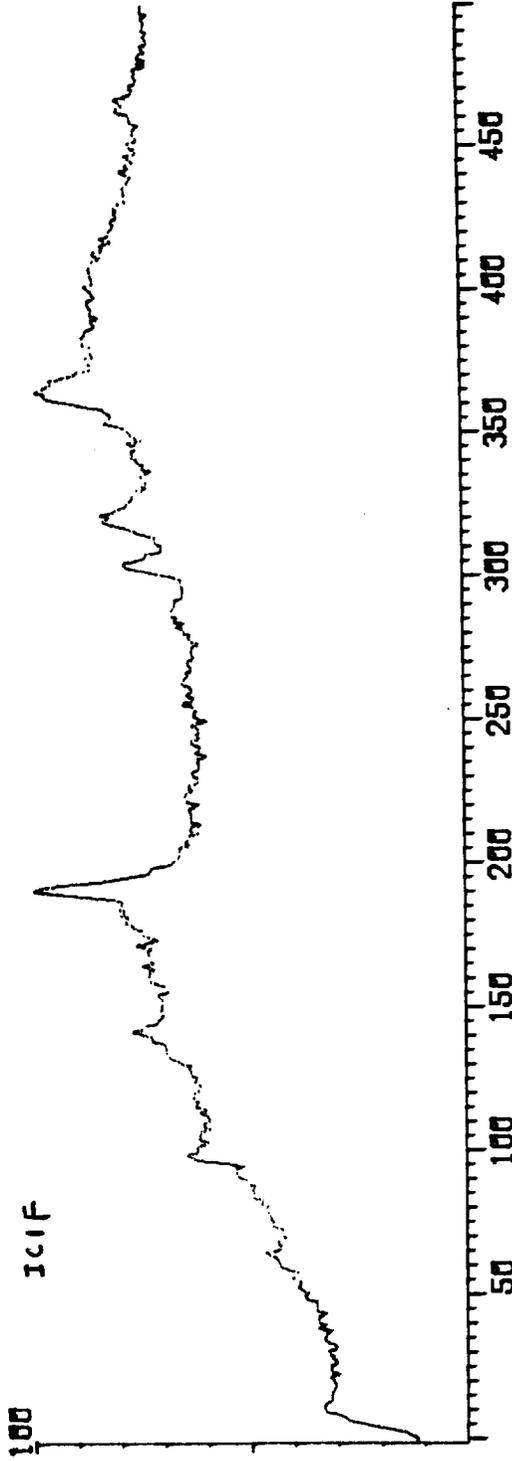
		GRAV, mg
Taken for LC		2.1
Recovered		2.1
Fraction	1	0.25
	2	< 0.1
	3	< 0.1
	4	0.2
	5	< 0.1
	6	< 0.1
	7	1.6

FA12:FER.AL-IIC-310,4/10/78,1N61S,D400
R6C

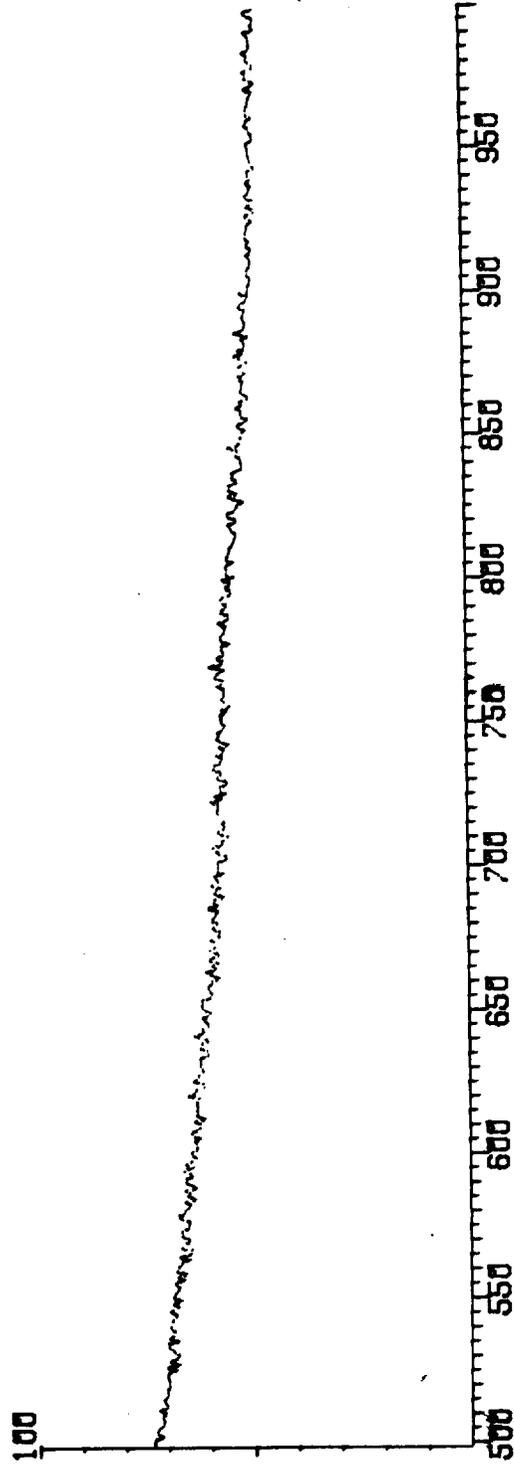


FA28:2(IC1F+1NGIS).D400.170-300/15.1800V.4/14

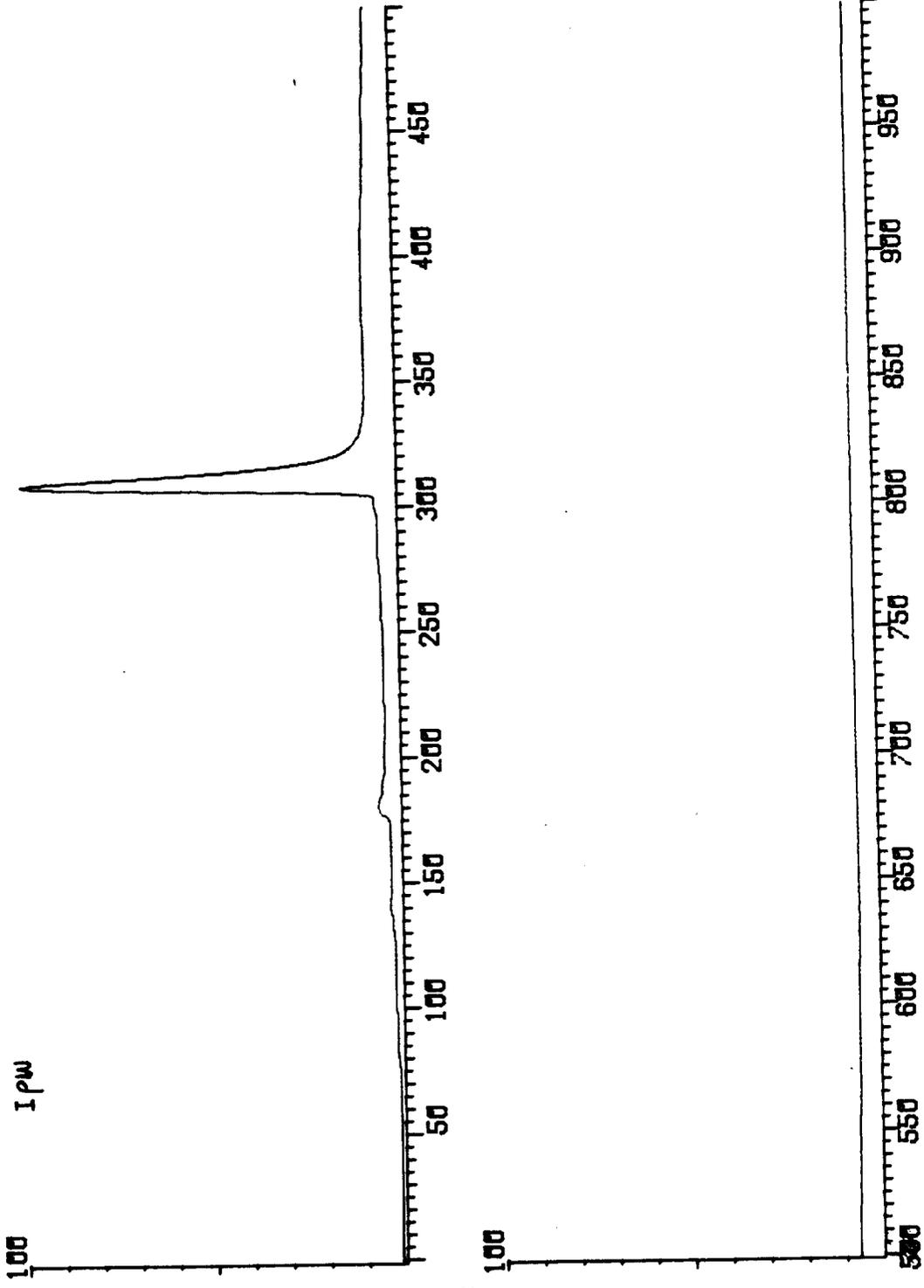
RSC



A83

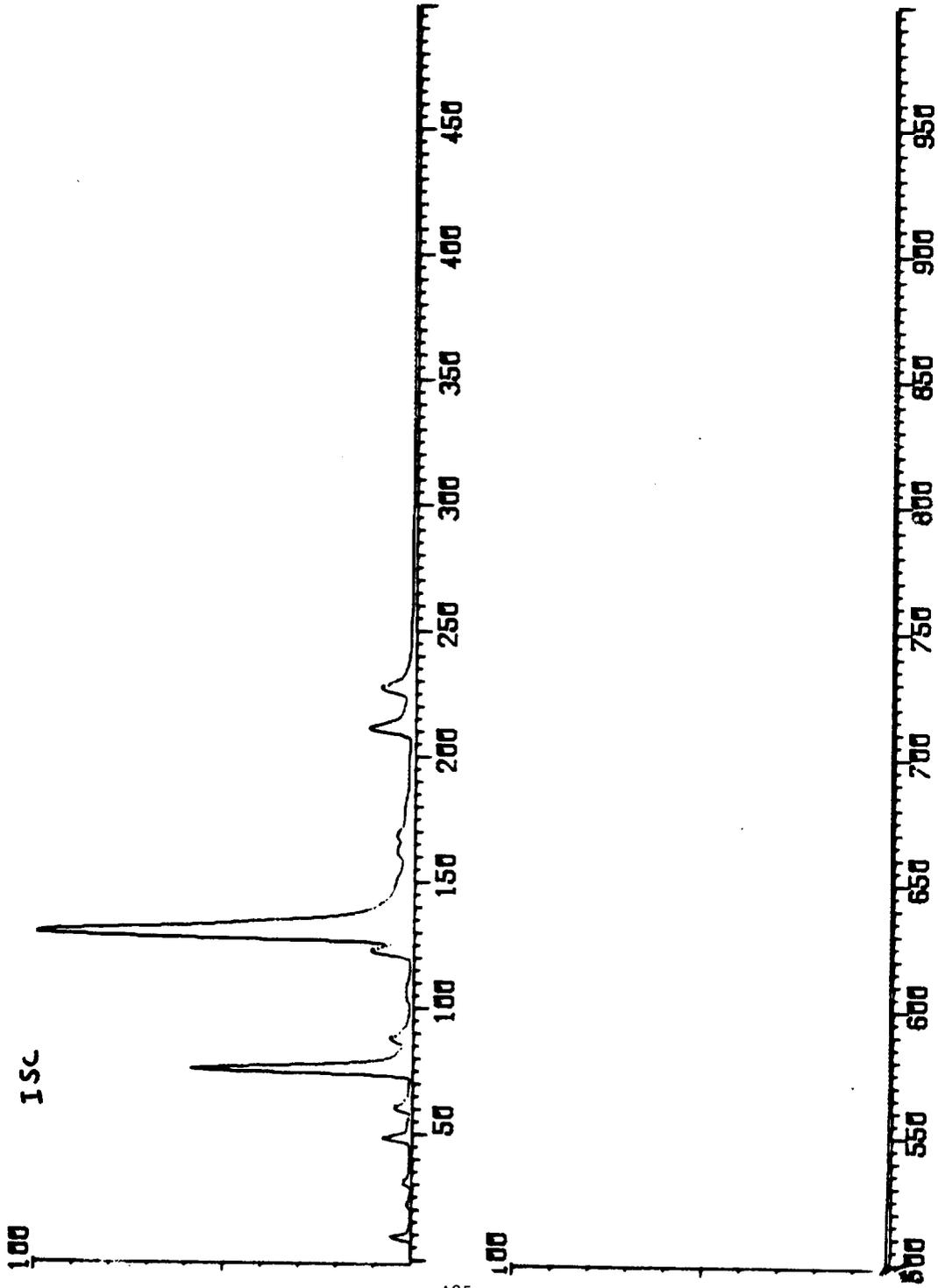


FR22: IPW+1NGIS, D400, 170-300/15, 1800V, 4/13/78
R6C



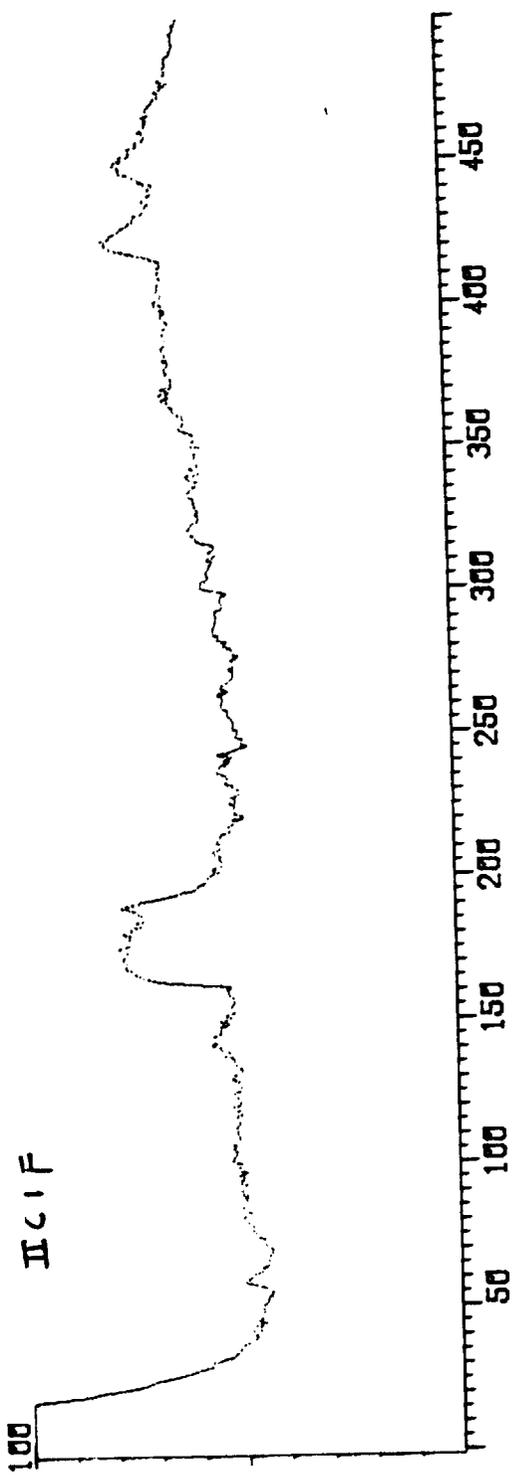
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R6C

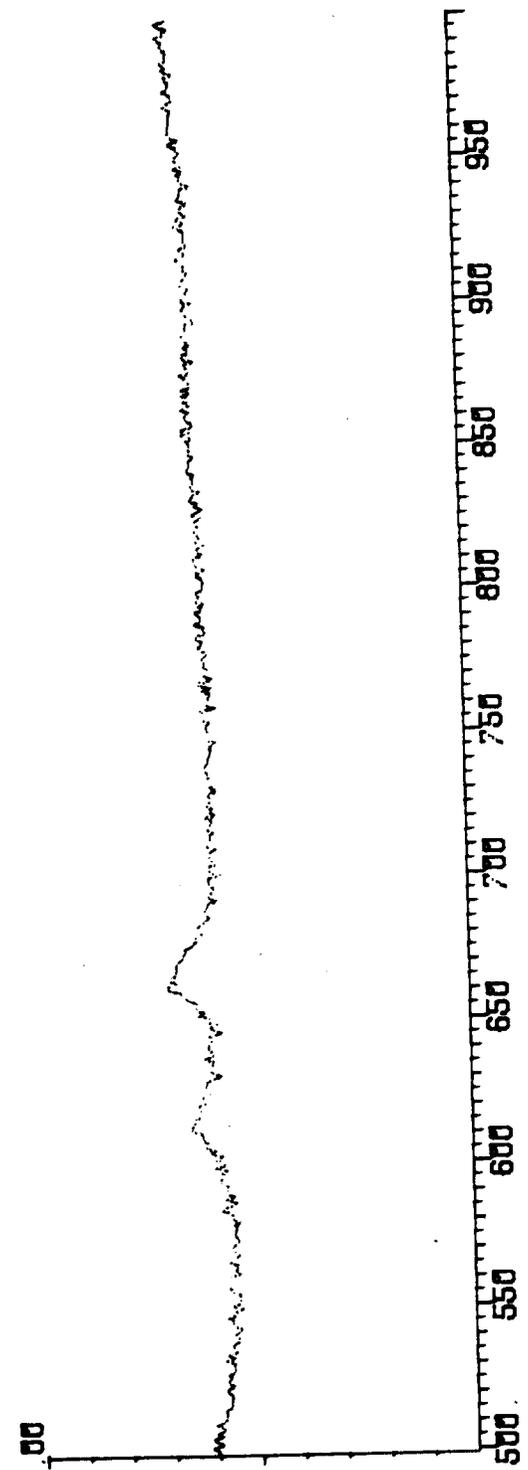


A85

FR26: IIC1F+1NG1S.D400.170-300/15.1800V.4/14/78
P30.PAGE1

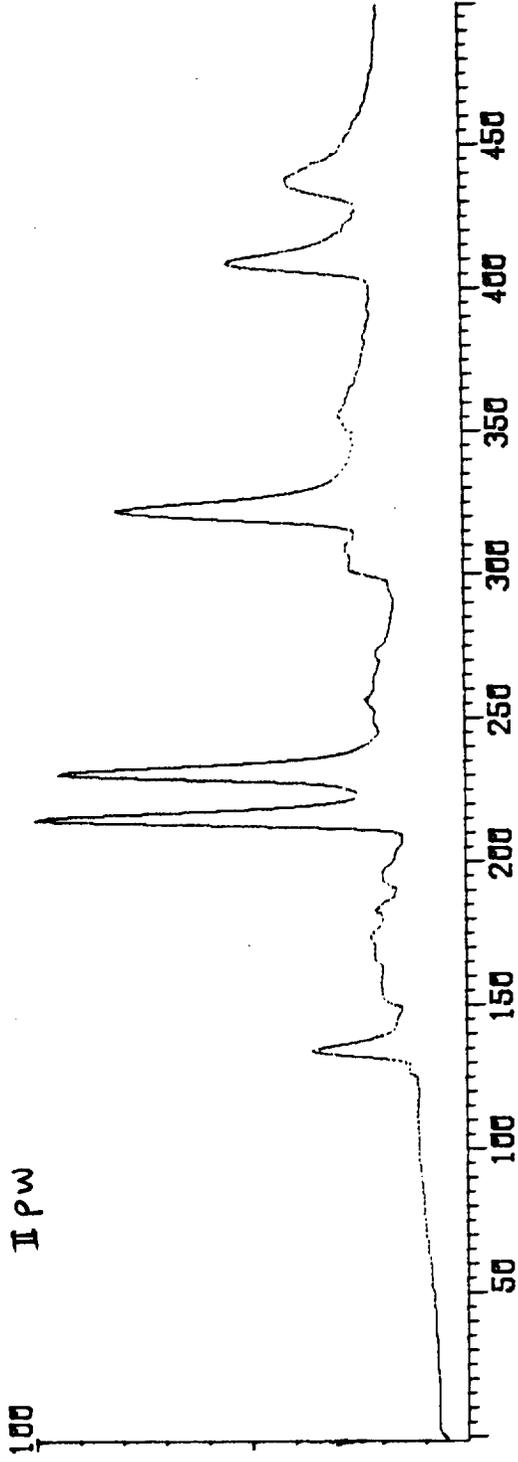


A86

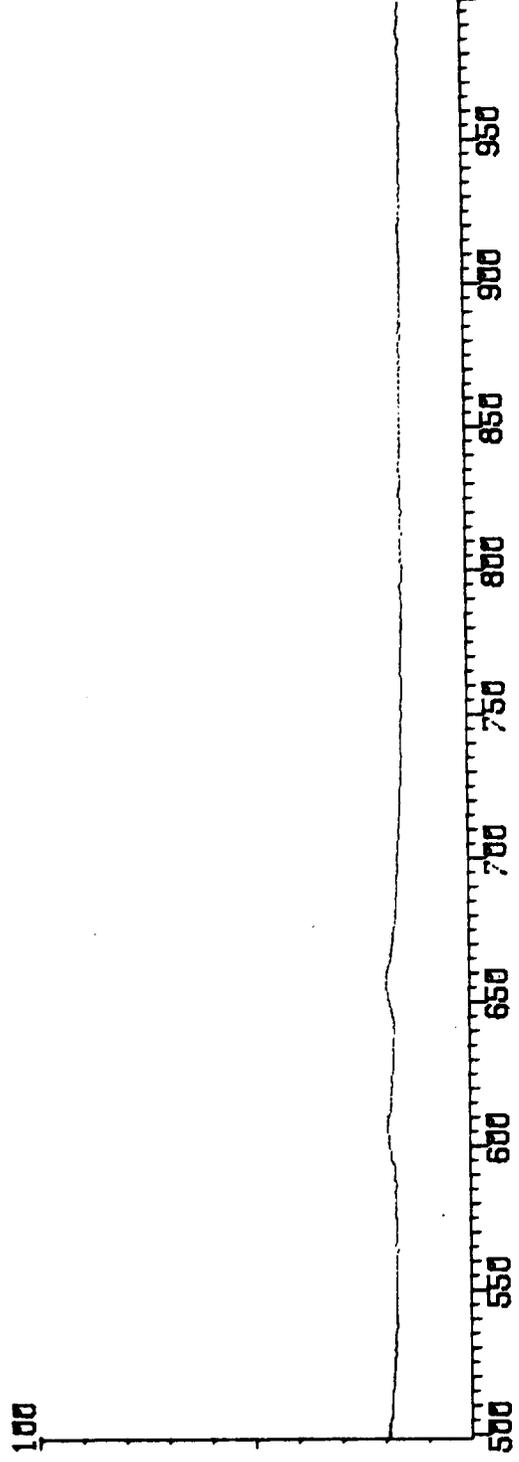


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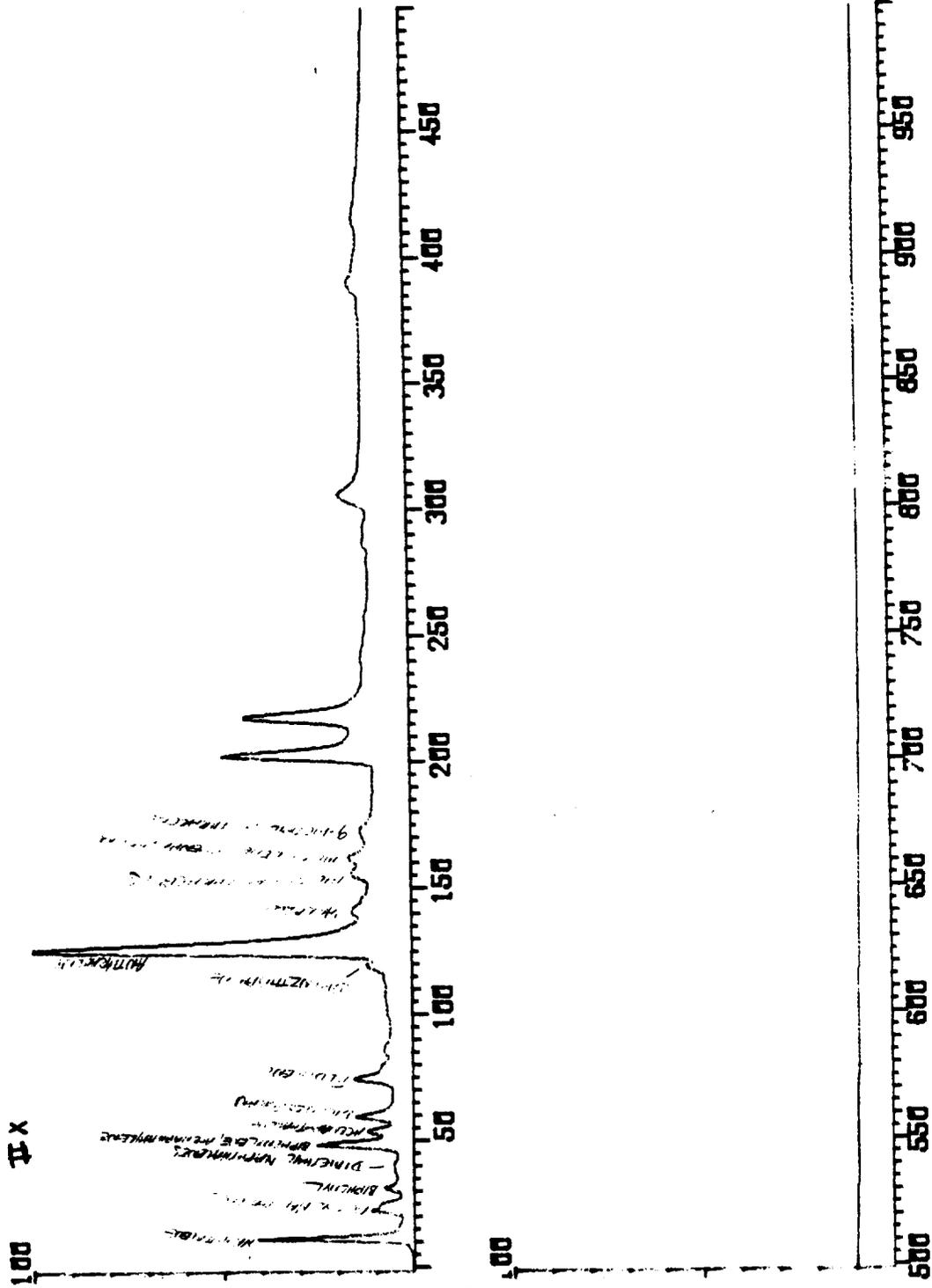
RSC,PAGE1



A87

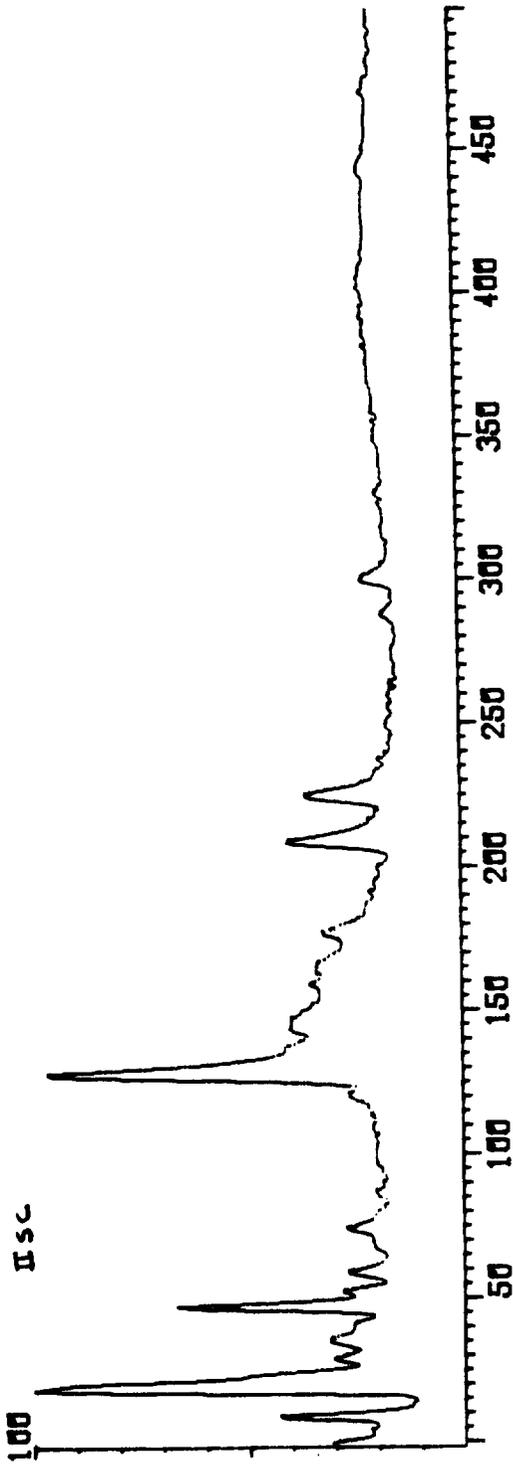


FR4:FER.ALL.II-X.300:1.1ULDEX400.170-300/15.1800
TOTAL RGC 4/4/78



FR14:FER.AL-IJSC.4/10/78.1N61S.D400 170-300/15

150



A89

100

500

APPENDIX B

INORGANIC ANALYSIS DATA

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IIX	B9
II imp 1	B10
XAD-2 Blank	B11
Imp. Blank	B12
Coal	B13
Coke	B14

COMMERCIAL TESTING & ENGINEERING CO.

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Analyst: S. Sweeney

P. O. No.: 540530

Sample No.: I C10 + 3

IAD No.: 97-A981-110-12

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	39	Terbium	1	Ruthenium		Vanadium	320
Thorium	29	Gadolinium	4	Molybdenum	42	Titanium	MC
Bismuth	3	Europium	2	Niobium	43	Scandium	1
Lead	150	Samarium	18	Zirconium	360	Calcium	MC
Thallium	20	Neodymium	42	Yttrium	110	Potassium	MC
Mercury	NR	Praseodymium	35	Strontium	MC	Chlorine	MC
Gold		Cerium	50	Rubidium	290	Sulfur	MC
Platinum		Lanthanum	40	Bromine	2	Phosphorus	MC
Iridium		Barium	MC	Selenium	8	Silicon	MC
Osmium		Cesium	25	Arsenic	390	Aluminum	MC
Rhenium	<0.5	Iodine	0.4	Germanium	19	Magnesium	MC
Tungsten	2	Tellurium	0.6	Gallium	110	Sodium	MC
Tantalum	<0.7	Antimony	7	Zinc	220	Fluorine	MC
Hafnium	5	Tin	15	Copper	280	Oxygen	NR
Lutetium	0.4	Indium	STD	Nickel	50	Nitrogen	NR
Ytterbium	2	Cadmium	140	Cobalt	13	Carbon	NR
Thulium	0.5	Silver	1	Iron	MC	Boron	18
Erbium	3	Palladium		Manganese	MC	Beryllium	0.9
Holmium	3	Rhodium		Chromium	380	Lithium	>240
Dysprosium	5					Hydrogen	NR

NR - Not Reported
 All elements not reported <0.1 ppm weight
 MC - Major Component

51

Approved: 

COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 · AREA CODE 312 728-8434
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 Arthur D. Little Company
 25 Acorn Park
 Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

IAD No.: 97-A981-110-12

Sample No.: I C 1 + F

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	48	Terbium	2	Ruthenium		Vanadium	310
Thorium	59	Gadolinium	5	Molybdenum	51	Titanium	MC
Bismuth	4	Europium	3	Niobium	24	Scandium	15
Lead	250	Samarium	21	Zirconium	270	Calcium	MC
Thallium	22	Neodymium	46	Yttrium	85	Potassium	MC
Mercury	NR	Praseodymium	21	Strontium	661	Chlorine	140
Gold		Cerium	240	Rubidium	360	Sulfur	MC
Platinum		Lanthanum	110	Bromine	2	Phosphorus	MC
Iridium		Barium	MC	Selenium	3	Silicon	MC
Osmium		Cesium	15	Arsenic	860	Aluminum	MC
Rhenium	≤0.2	Iodine	0.5	Germanium	10	Magnesium	MC
Tungsten	5	Tellurium	≤0.3	Gallium	230	Sodium	MC
Tantalum	≤0.9	Antimony	11	Zinc	MC	Fluorine	MC
Hafnium	6	Tin	9	Copper	180	Oxygen	NR
Lutetium	1	Indium	STD	Nickel	0.3	Nitrogen	NR
Ytterbium	5	Cadmium	MC	Cobalt	52	Carbon	NR
Thulium	0.7	Silver	3	Iron	MC	Boron	97
Erbium	4	Palladium		Manganese	MC	Beryllium	4
Holmium	6	Rhodium		Chromium	780	Lithium	>290
Dysprosium	9					Hydrogen	NR

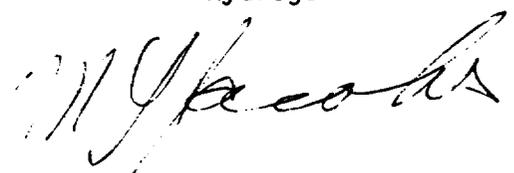
NR - Not Reported

All elements not reported <0.1 ppm weight

MC - Major Component

Approved:

82



COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 80801 · AREA CODE 312 726-8434
INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
Arthur D. Little Company
25 Acorn Park
Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

Sample No.: I PW

IAD No.: 97-A981-110-12

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	43	Terbium	2	Ruthenium		Vanadium	250
Thorium	52	Gadolinium	6	Molybdenum	680	Titanium	MC
Bismuth	5	Europium	4	Niobium	31	Scandium	9
Lead	280	Samarium	14	Zirconium	130	Calcium	MC
Thallium	16	Neodymium	42	Yttrium	50	Potassium	MC
Mercury	NR	Praseodymium	18	Strontium	610	Chlorine	MC
Gold	0.2	Cerium	88	Rubidium	88	Sulfur	MC
Platinum		Lanthanum	130	Bromine	530	Phosphorus	MC
Iridium		Barium	MC	Selenium	83	Silicon	MC
Osmium		Cesium	9	Arsenic	MC	Aluminum	MC
Rhenium	<0.4	Iodine	3	Germanium	14	Magnesium	MC
Tungsten	15	Tellurium	0.7	Gallium	50	Sodium	MC
Tantalum		Antimony	24	Zinc	MC	Fluorine	MC
Hafnium	4	Tin	14	Copper	MC	Oxygen	NR
Lutetium	0.8	Indium	STD	Nickel	MC	Nitrogen	NR
Ytterbium	4	Cadmium	MC	Cobalt	77	Carbon	NR
Thulium	0.5	Silver	140	Iron	MC	Boron	32
Erbium	4	Palladium		Manganese	MC	Beryllium	2
Holmium	5	Rhodium		Chromium	MC	Lithium	430
Dysprosium	7					Hydrogen	NR

NR - Not Reported
All elements not reported <0.2 ppm weight
MC - Major Component

B3

Approved:



COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 • AREA CODE 312 726-8434
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 Arthur D. Little Company
 25 Acorn Park
 Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

Sample No.: I XAD Parr Bombed

IAD No.: 97-A981-110-12

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	<3	Terbium		Ruthenium		Vanadium	0.4
Thorium		Gadolinium		Molybdenum	4	Titanium	59
Bismuth	3	Europium		Niobium		Scandium	≤0.4
Lead	3	Samarium		Zirconium	82	Calcium	210
Thallium		Neodymium		Yttrium	3	Potassium	520
Mercury	NR	Praseodymium		Strontium	4	Chlorine	CONT
Gold		Cerium	2	Rubidium	0.3	Sulfur	24
Platinum	4	Lanthanum	2	Bromine	4	Phosphorus	29
Iridium		Barium	9	Selenium	0.7	Silicon	52
Osmium		Cesium		Arsenic	8	Aluminum	470
Rhenium		Iodine	0.4	Germanium		Magnesium	17
Tungsten		Tellurium		Gallium	0.3	Sodium	>970
Tantalum		Antimony	2	Zinc	100	Fluorine	CONT
Hafnium		Tin	3	Copper	10	Oxygen	NR
Lutetium		Indium	STD	Nickel	16	Nitrogen	NR
Ytterbium		Cadmium	6	Cobalt	≤0.6	Carbon	NR
Thulium		Silver	0.4	Iron	73	Boron	CONT
Erbium		Palladium		Manganese	5	Beryllium	
Holmium		Rhodium		Chromium	4	Lithium	
Dysprosium						Hydrogen	NR

NR - Not Reported
 All elements not reported <0.4ppm weight
 MC - Major Component CONT - Contamination

Approved: 
 BH

COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 · AREA CODE 312 728-8434
INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
Arthur D. Little Inc.
25 Acorn Park
Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

Sample No.: Impinger I

IAD No.: 97-A981-110-12

CONCENTRATION IN $\mu\text{g/ml}$

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	0.1	Terbium		Ruthenium		Vanadium	0.005
Thorium		Gadolinium		Molybdenum	2	Titanium	0.3
Bismuth		Europium		Niobium	0.004	Scandium	<0.001
Lead	0.03	Samarium		Zirconium	0.02	Calcium	2
Thallium		Neodymium		Yttrium		Potassium	4
Mercury	NR	Praseodymium		Strontium	0.04	Chlorine	0.4
Gold		Cerium		Rubidium	0.003	Sulfur	5
Platinum		Lanthanum		Bromine	0.06	Phosphorus	0.2
Iridium		Barium	3	Selenium	0.2	Silicon	1
Osmium		Cesium		Arsenic	0.07	Aluminum	0.3
Rhenium		Iodine	0.02	Germanium		Magnesium	0.7
Tungsten		Tellurium		Gallium		Sodium	0.9
Tantalum		Antimony		Zinc	0.2	Fluorine	≈0.5
Hafnium		Tin	0.01	Copper	0.1	Oxygen	NR
Lutetium		Indium	STD	Nickel	2	Nitrogen	NR
Ytterbium		Cadmium	0.02	Cobalt	0.04	Carbon	NR
Thulium		Silver	0.006	Iron	2	Boron	0.01
Erbium		Palladium		Manganese	0.2	Beryllium	
Holmium		Rhodium		Chromium	0.7	Lithium	0.02
Dysprosium						Hydrogen	NR

NR - Not Reported

All elements not reported <0.003 $\mu\text{g/ml}$

MC - Major Component

Approved: 

B5

COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 · AREA CODE 312 726-8434
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 Arthur D. Little Company
 25 Acorn Park
 Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

Sample No.: II C10 + 3

IAD No.: 97-A981-110-12

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	2	Terbium	0.2	Ruthenium		Vanadium	16
Thorium	2	Gadolinium	0.6	Molybdenum	26	Titanium	130
Bismuth	8	Europium	0.3	Niobium	1	Scandium	0.7
Lead	380	Samarium	2	Zirconium	8	Calcium	MC
Thallium	55	Neodymium	3	Yttrium	2	Potassium	MC
Mercury	NR	Praseodymium	1	Strontium	110	Chlorine	MC
Gold		Cerium	8	Rubidium	MC	Sulfur	MC
Platinum		Lanthanum	5	Bromine	300	Phosphorus	MC
Iridium		Barium	MC	Selenium	14	Silicon	MC
Osmium		Cesium	17	Arsenic	MC	Aluminum	MC
Rhenium		Iodine	54	Germanium	5	Magnesium	MC
Tungsten	15	Tellurium	4	Gallium	43	Sodium	MC
Tantalum		Antimony	18	Zinc	MC	Fluorine	MC
Hafnium		Tin	5	Copper	460	Oxygen	NR
Lutetium	0.1	Indium	STD	Nickel	85	Nitrogen	NR
Ytterbium	0.5	Cadmium	75	Cobalt	200	Carbon	NR
Thulium	0.1	Silver	9	Iron	MC	Boron	10
Erbium	0.4	Palladium		Manganese	MC	Beryllium	0.1
Holmium	0.5	Rhodium		Chromium	130	Lithium	29
Dysprosium	0.8					Hydrogen	NR

NR - Not Reported
 All elements not reported <0.1 ppm weight
 MC - Major Component

Approved: 

COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 · AREA CODE 312 726-8494
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 Arthur D. Little, Incorporated
 25 Acorn Park
 Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

IAD No.: 97-A981-110-12

Sample No.: II C1 + F

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	2	Terbium	0.5	Ruthenium		Vanadium	8
Thorium		Gadolinium	1	Molybdenum	60	Titanium	130
Bismuth	5	Europium	0.7	Niobium	0.3	Scandium	1
Lead	940	Samarium	2	Zirconium	7	Calcium	MC
Thallium	120	Neodymium	2	Yttrium	2	Potassium	MC
Mercury	NR	Praseodymium	1	Strontium	330	Chlorine	MC
Gold		Cerium	8	Rubidium	450	Sulfur	MC
Platinum		Lanthanum	5	Bromine	300	Phosphorus	MC
Iridium		Barium	MC	Selenium	16	Silicon	MC
Osmium		Cesium	25	Arsenic	MC	Aluminum	MC
Rhenium		Iodine	160	Germanium	2	Magnesium	MC
Tungsten	23	Tellurium	4	Gallium	68	Sodium	MC
Tantalum		Antimony	40	Zinc	MC	Fluorine	MC
Hafnium		Tin	4	Copper	460	Oxygen	NR
Lutetium	<0.1	Indium	STD	Nickel	3	Nitrogen	NR
Ytterbium	0.3	Cadmium	130	Cobalt	80	Carbon	NR
Thulium	<0.1	Silver	7	Iron	MC	Boron	19
Erbium	0.3	Palladium		Manganese	MC	Beryllium	0.2
Holmium	0.4	Rhodium		Chromium	6	Lithium	30
Dysprosium	1					Hydrogen	NR

NR - Not Reported
 All elements not reported <0.1 ppm weight
 MC - Major Component

Approved: 
 B7

COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 - AREA CODE 312 726-8434
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 Arthur D. Little, Inc.
 25 Acorn Park
 Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

Sample No.: II PW

IAD No.: 97-A981-110-12

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	8	Terbium	0.2	Ruthenium		Vanadium	8
Thorium	2	Gadolinium	0.6	Molybdenum	67	Titanium	80
Bismuth	17	Europium	0.6	Niobium	0.9	Scandium	0.3
Lead	MC	Samarium	3	Zirconium	12	Calcium	MC
Thallium	65	Neodymium	3	Yttrium	2	Potassium	MC
Mercury	NR	Praseodymium	1	Strontium	60	Chlorine	MC
Gold		Cerium	16	Rubidium	590	Sulfur	MC
Platinum		Lanthanum	11	Bromine	230	Phosphorus	MC
Iridium		Barium	MC	Selenium	27	Silicon	MC
Osmium		Cesium	16	Arsenic	MC	Aluminum	MC
Rhenium		Iodine	50	Germanium	6	Magnesium	MC
Tungsten	15	Tellurium	5	Gallium	60	Sodium	MC
Tantalum		Antimony	47	Zinc	MC	Fluorine	≈630
Hafnium	0.2	Tin	2	Copper	810	Oxygen	NR
Lutetium	<0.1	Indium	STD	Nickel	50	Nitrogen	NR
Ytterbium	0.3	Cadmium	130	Cobalt	80	Carbon	NR
Thulium	<0.1	Silver	11	Iron	MC	Boron	10
Erbium	0.3	Palladium		Manganese	MC	Beryllium	0.1
Holmium	0.3	Rhodium		Chromium	20	Lithium	25
Dysprosium	0.5					Hydrogen	NR

NR - Not Reported
 All elements not reported <0.1 ppm weight
 MC - Major Component

38

Approved:



COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 · AREA CODE 312 726-8434
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 Arthur D. Little Company
 25 Acorn Park
 Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

Sample No.: II XAD Parr Bombed

IAD No.: 97-A981-110-12

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	2	Terbium		Ruthenium		Vanadium	0.3
Thorium		Gadolinium		Molybdenum	6	Titanium	27
Bismuth	8	Europium		Niobium		Scandium	≤0.3
Lead	3	Samarium		Zirconium	59	Calcium	260
Thallium		Neodymium		Yttrium	0.3	Potassium	140
Mercury	NR	Praseodymium	0.3	Strontium	5	Chlorine	CONT
Gold		Cerium	0.9	Rubidium	0.1	Sulfur	7
Platinum	1	Lanthanum	1	Bromine	2	Phosphorus	16
Iridium		Barium	5	Selenium	1	Silicon	310
Osmium		Cesium		Arsenic	5	Aluminum	≥230
Rhenium		Iodine	0.4	Germanium	0.1	Magnesium	26
Tungsten		Tellurium		Gallium	0.2	Sodium	MC
Tantalum		Antimony		Zinc	12	Fluorine	CONT
Hafnium		Tin	0.9	Copper	3	Oxygen	NR
Lutetium		Indium	STD	Nickel	13	Nitrogen	NR
Ytterbium		Cadmium	≤0.9	Cobalt	0.2	Carbon	NR
Thulium		Silver	0.4	Iron	44	Boron	CONT
Erbium		Palladium		Manganese	5	Beryllium	
Holmium		Rhodium		Chromium	5	Lithium	
Dysprosium						Hydrogen	NR

NR - Not Reported

All elements not reported <0.2 ppm weight
 MC - Major Component CONT - Contamination

Approved:

M. J. Jacobs
 B-9

COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 • AREA CODE 312 726-8434
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 Arthur D. Little Inc.
 25 Acorn Park
 Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

Sample No.: Impinger II

IAD No.: 97-A981-110-12

CONCENTRATION IN $\mu\text{g}/\text{ml}$

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium		Terbium		Ruthenium		Vanadium	0.001
Thorium		Gadolinium		Molybdenum	2	Titanium	0.04
Bismuth	0.006	Europium		Niobium	0.02	Scandium	≤ 0.002
Lead	0.03	Samarium		Zirconium	0.02	Calcium	5
Thallium		Neodymium		Yttrium		Potassium	MC
Mercury	NR	Praseodymium		Strontium	0.01	Chlorine	0.3
Gold		Cerium		Rubidium	0.003	Sulfur	0.8
Platinum		Lanthanum		Bromine	0.008	Phosphorus	0.1
Iridium		Barium	0.03	Selenium	0.2	Silicon	0.3
Osmium		Cesium		Arsenic	0.03	Aluminum	0.1
Rhenium		Iodine		Germanium		Magnesium	0.2
Tungsten		Tellurium		Gallium	0.003	Sodium	2
Tantalum		Antimony		Zinc	0.1	Fluorine	≈ 2
Hafnium		Tin		Copper	0.1	Oxygen	NR
Lutetium		Indium	STD	Nickel	0.3	Nitrogen	NR
Ytterbium		Cadmium		Cobalt	0.006	Carbon	NR
Thulium		Silver	0.2	Iron	0.9	Boron	0.002
Erbium		Palladium		Manganese	0.4	Beryllium	
Holmium		Rhodium		Chromium	1	Lithium	0.001
Dysprosium						Hydrogen	NR

NR - Not Reported
 All elements not reported $< 0.004 \mu\text{g}/\text{ml}$
 MC - Major Component

Approved:



B-10

COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 · AREA CODE 312 726-8434
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 A. D. Little, Inc.
 25 Acorn Park
 Cambridge, MA 02140

Date: April 4, 1978

Analyst: S. Sweeney

P. O. No.:

Sample No.: Parr Bombed XAD Resin Blank

IAD No.: 97-B085-110-01

(Sample was received broken) CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	3	Terbium		Ruthenium		Vanadium	0.4
Thorium	≤2	Gadolinium		Molybdenum	3	Titanium	13
Bismuth		Europium		Niobium		Scandium	≤0.1
Lead	90	Samarium		Zirconium	72	Calcium	210
Thallium		Neodymium		Yttrium	≤0.7	Potassium	170
Mercury	NR	Praseodymium	≤0.1	Strontium	5	Chlorine	CONT
Gold		Cerium	1	Rubidium	0.2	Sulfur	23
Platinum	780	Lanthanum	0.5	Bromine	4	Phosphorus	8
Iridium		Barium	79	Selenium	≤0.7	Silicon	95
Osmium		Cesium		Arsenic	1	Aluminum	>110
Rhenium		Iodine		Germanium		Magnesium	51
Tungsten		Tellurium		Gallium	0.3	Sodium	>280
Tantalum		Antimony	0.4	Zinc	7	Fluorine	CONT
Hafnium		Tin	1	Copper	37	Oxygen	NR
Lutetium		Indium	STD	Nickel	10	Nitrogen	NR
Ytterbium		Cadmium	≤0.4	Cobalt	0.2	Carbon	NR
Thulium		Silver	0.5	Iron	180	Boron	CONT
Erbium		Palladium		Manganese	2	Beryllium	
Holmium		Rhodium		Chromium	25	Lithium	0.6
Dysprosium						Hydrogen	NR

NR - Not Reported

All elements not reported <0.1 ppm weight

MC - Major Component

CONT-Contamination

Approved: *M. L. Jacobs* (SS)

B-11

COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 · AREA CODE 312 726-8434
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 A. D. Little, Inc.
 20 Acorn Park
 Cambridge, MA 02140

Date: April 4, 1978

Analyst: S. Sweeney

P. O. No.:

Sample No.: Blank Imp.

IAD No.: 97-B089-110-01

CONCENTRATION IN $\mu\text{g/ml}$

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	0.09	Terbium		Ruthenium		Vanadium	0.003
Thorium		Gadolinium		Molybdenum	0.1	Titanium	0.03
Bismuth		Europium		Niobium		Scandium	<0.002
Lead	0.04	Samarium		Zirconium	0.004	Calcium	2
Thallium		Neodymium		Yttrium		Potassium	1
Mercury	NR	Praseodymium		Strontium	0.04	Chlorine	0.4
Gold		Cerium	0.02	Rubidium	<0.002	Sulfur	0.08
Platinum		Lanthanum	0.01	Bromine	0.02	Phosphorus	0.09
Iridium		Barium	0.03	Selenium	<0.004	Silicon	0.9
Osmium		Cesium		Arsenic	<0.002	Aluminum	0.09
Rhenium		Iodine		Germanium		Magnesium	0.3
Tungsten		Tellurium		Gallium		Sodium	>7
Tantalum		Antimony		Zinc	0.08	Fluorine	≈0.2
Hafnium		Tin		Copper	0.04	Oxygen	NR
Lutetium		Indium	STD	Nickel	0.01	Nitrogen	NR
Ytterbium		Cadmium		Cobalt	<0.002	Carbon	NR
Thulium		Silver		Iron	0.2	Boron	0.05
Erbium		Palladium		Manganese	0.006	Beryllium	
Holmium		Rhodium		Chromium	0.007	Lithium	<0.002
Dysprosium						Hydrogen	NR

NR – Not Reported

All elements not reported < 0.002 $\mu\text{g/ml}$

MC – Major Component

Approved: *M.L. Jacobs* (SS)

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COMMERCIAL TESTING & ENGINEERING CO.

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INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
Arthur D. Little Company
25 Acorn Park
Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

Sample No.: Coal Parr Bomb

IAD No.: 97-A981-110-12

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	<0.8	Terbium	0.1	Ruthenium		Vanadium	9
Thorium	<1	Gadolinium	0.3	Molybdenum	6	Titanium	300
Bismuth	220	Europium	0.2	Niobium	1	Scandium	1
Lead	9	Samarium	0.8	Zirconium	74	Calcium	860
Thallium		Neodymium	1	Yttrium	4	Potassium	MC
Mercury	NR	Praseodymium	1	Strontium	37	Chlorine	CONT
Gold		Cerium	7	Rubidium	1	Sulfur	MC
Platinum	120	Lanthanum	5	Bromine	2	Phosphorus	780
Iridium		Barium	810	Selenium	3	Silicon	39
Osmium		Cesium	0.1	Arsenic	11	Aluminum	>110
Rhenium		Iodine	0.2	Germanium	<2	Magnesium	350
Tungsten		Tellurium		Gallium	2	Sodium	MC
Tantalum		Antimony	0.9	Zinc	33	Fluorine	CONT
Hafnium		Tin	3	Copper	12	Oxygen	NR
Lutetium		Indium	STD	Nickel	12	Nitrogen	NR
Ytterbium		Cadmium	2	Cobalt	2	Carbon	NR
Thulium		Silver	1	Iron	MC	Boron	CONT
Erbium		Palladium		Manganese	MC	Beryllium	0.1
Holmium		Rhodium		Chromium	26	Lithium	40
Dysprosium						Hydrogen	NR

NR - Not Reported

All elements not reported <0.1 ppm weight

MC - Major Component CONT-Contamination

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Approved:



COMMERCIAL TESTING & ENGINEERING CO.

GENERAL OFFICES: 228 NORTH LA SALLE STREET, CHICAGO, ILLINOIS 60601 · AREA CODE 312 726-8434
 INSTRUMENTAL ANALYSIS DIVISION, 14335 WEST 44TH AVENUE, GOLDEN, COLORADO 80401, PHONE: 303-278-9521

Reply to

To: Ms. Julie Rudolph
 Arthur D. Little Company
 25 Acorn Park
 Cambridge, MA 02140

Date: March 9, 1978

Analyst: S. Sweeney

P. O. No.: 540530

IAD No.: 97-A981-110-12

Sample No.: Coke Parr Bombed

CONCENTRATION IN PPM WEIGHT

ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.	ELEMENT	CONC.
Uranium	4	Terbium	0.1	Ruthenium		Vanadium	41
Thorium	3	Gadolinium	0.5	Molybdenum	12	Titanium	MC
Bismuth	3	Europium	0.3	Niobium	7	Scandium	4
Lead	7	Samarium	2	Zirconium	210	Calcium	MC
Thallium		Neodymium	4	Yttrium	5	Potassium	MC
Mercury	NR	Praseodymium	2	Strontium	110	Chlorine	CONT
Gold		Cerium	10	Rubidium	14	Sulfur	MC
Platinum	0.8	Lanthanum	14	Bromine	6	Phosphorus	710
Iridium		Barium	240	Selenium	1	Silicon	MC
Osmium		Cesium	1	Arsenic	14	Aluminum	MC
Rhenium		Iodine	0.3	Germanium	2	Magnesium	MC
Tungsten		Tellurium	≤0.8	Gallium	5	Sodium	MC
Tantalum		Antimony	1	Zinc	110	Fluorine	CONT
Hafnium		Tin	5	Copper	30	Oxygen	NR
Lutetium		Indium	STD	Nickel	17	Nitrogen	NR
Ytterbium		Cadmium	3	Cobalt	10	Carbon	NR
Thulium		Silver	3	Iron	MC	Boron	CONT
Erbium		Palladium		Manganese	560	Beryllium	0.5
Holmium		Rhodium		Chromium	38	Lithium	46
Dysprosium						Hydrogen	NR

NR - Not Reported

All elements not reported <0.1 ppm weight

MC - Major Component CONT - Contamination

Approved:



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Cincinnati, Ohio 45268

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