

THE FATE OF TRACE METALS IN A ROTARY KILN INCINERATOR WITH A VENTURI/PACKED COLUMN SCRUBBER

Volume I — Technical Results

By

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FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Risk Reduction Engineering Laboratory is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible, engineering basis in support of the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

This report describes an extensive series of tests conducted at the EPA's Incineration Research Facility to evaluate the fate of trace metals fed to a rotary kiln incinerator. For further information, please contact the Waste Minimization, Destruction and Disposal Research Division of the Risk Reduction Engineering Laboratory.

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ABSTRACT

A 5-week series of pilot-scale incineration tests, employing a synthetic waste feed, was performed at the U.S. Environmental Protection Agency's (EPA) Incineration Research Facility (IRF) to evaluate the fate of trace metals fed to a rotary kiln incinerator equipped with a venturi scrubber/packed column scrubber. Completed were three tests to evaluate the valence state of chromium in emissions and discharges as a function of valence state in the feed and feed chlorine content, and eight tests to evaluate the fate of five hazardous constituent and four nonhazardous constituent trace metals as a function of incinerator operating temperatures and feed chlorine content.

Chromium test results indicated that when no chlorine was present in the feed, 95 percent of the measured chromium was discharged in the kiln ash, 1 to 2 percent in the scrubber exit flue gas, and 3 percent in the scrubber liquor. With chlorine in the feed, these fractions were 84, 4, and 11 percent, respectively. Kiln ash contained negligible Cr(+6) for all tests. Scrubber exit flue gas Cr(+6) as a fraction of total chromium was nominally 15 percent with no feed chlorine, increasing to about 50 percent with chlorine-containing feed. The scrubber liquor Cr(+6) fraction was 20 to 30 percent with Cr(+3) feed, increasing to about 60 percent with Cr(+6) feed.

Parametric trace metal test results confirm that cadmium, lead, and bismuth are relatively volatile metals, based on normalized discharge distribution data. Less than 32 percent of these metals were discharged in the kiln ash. Barium, copper, strontium, chromium, and magnesium are relatively nonvolatile, with greater than 75 percent of their discharge amounts present in kiln ash. Surprisingly, arsenic was found to be relatively nonvolatile. Apparent scrubber collection efficiencies generally correlated with observed volatility. Volatile metals exhibited collection efficiencies of 36 to 45 percent; nonvolatile metals, with the exception of copper, exhibited collection efficiencies of 49 to 88 percent. Feed chlorine content had a major effect on the observed volatility of cadmium, lead, and bismuth, with volatility increasing with increased feed chlorine content. Afterburner exit flue gas particle size distributions shifted to smaller size when chlorine was added to the feed.

The average mass balance closures around the kiln ash/scrubber discharge ranged from 48 to 96 percent for individual metals. Overall average closure was 71 percent. From past experience, trace metals mass balance closure results for combustion sources are in the 30 to 200 percent range.

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SECTION 1

INTRODUCTION

The RCRA hazardous waste incinerator performance standards promulgated by EPA in January 1981 established limits on incinerator particulate and HCl emissions, and on hazardous organic constituent emissions by requiring that at least 99.99 percent destruction and removal efficiency (DRE) be achieved for the principal organic hazardous constituents (POHCs) in wastes fed to an incinerator. Emissions of hazardous constituent trace metals were not regulated by these standards except in a global sense via the particulate standard. Risk assessments to date have suggested that, of the total risk to human health and the environment from properly operated incinerators, hazardous constituent trace metal emissions may represent the largest component.

Despite its importance, the data base on trace metal emissions from incinerators is currently very sparse. Data on the effects of waste composition and incinerator operation on trace metal emissions are particularly lacking. In response to these data needs, an extensive series of tests was conducted at EPA's Incineration Research Facility (IRF), with funding support from the Office of Solid Waste (OSW), to investigate the fate of trace metals fed to a rotary kiln incinerator equipped with a venturi scrubber/packed column scrubber for particulate and acid gas control.

A primary objective of the tests was to investigate the fate of five hazardous constituent trace metals fed in a solid waste matrix to a rotary kiln incinerator as a function of incinerator operating temperatures and feed chlorine content. Of interest were metal partitioning, particle size distribution, flue gas phase distribution, and scrubber efficiency for each of the metals. The trace metals investigated were arsenic, barium, cadmium, chromium, and lead.

In another OSW sponsored effort within EPA's Risk Reduction Engineering Laboratory (RREL) to support trace metal emission regulation development, a numerical model is being developed by another EPA contractor to aid in predicting the relative distributions of trace metals in emissions and discharges from incinerators. Thus, a second objective of this test program was to supply data to evaluate the predictive capabilities of this model, and to perhaps guide further model refinement. To support this objective, an additional four nonhazardous constituent trace metals were included in the test feed material. These were bismuth, copper, magnesium, and strontium.

Finally, there has been a continuing interest in establishing which valence state of chromium predominates in emissions and discharges from incinerators treating chromium-containing wastes. Of the two common chromium valence states, [the trivalent ($\text{Cr}(+3)$) and the hexavalent ($\text{Cr}(+6)$)], the hexavalent is much more toxic. In the absence of data showing which form is emitted from an incinerator, risk assessments have generally assumed that the entire amount emitted is in the hexavalent form. This assumption has resulted in specifying conservative emission limits in the regulatory development process. Thus, a third objective of

this test program was to develop data on the valence state distribution of chromium in emissions and discharges as a function of feed composition and chromium valence state fed.

The test program to address the above objectives was comprised of both a series of eight parametric tests in which the test waste feed contained nine trace metals, and a series of three tests in which the test waste feed contained either Cr(+3) or Cr(+6).

This report summarizes the results of the test program. Section 2 describes the rotary kiln incineration system at the IRF in which the tests were performed, outlines the test waste feed characteristics, and summarizes the incinerator operating conditions for each of the tests. Section 3 discusses the emissions and discharge stream sampling and analysis protocols. Section 4 presents test program results. Test conclusions are summarized in Section 5. Quality assurance and quality control aspects of the test program are discussed in Section 6.

SECTION 2

FACILITY DESCRIPTION AND TEST CONDITIONS

The pilot-scale rotary kiln incinerator system (RKS) at the IRF in Jefferson, Arkansas, was used in this test program. A description of the system is presented in Section 2.1, followed by a description of the synthetic waste mixture in Section 2.2. The test matrices and operating conditions are described in Section 2.3.

2.1 ROTARY KILN INCINERATOR SYSTEM DESCRIPTION

As indicated in Figure 1 and Table 1, the RKS consists of a primary combustion chamber, a transition section, and a fired afterburner chamber. The primary air pollution control system (APCS) consists of a quench section, a venturi scrubber, and a packed-column scrubber. Quench section installation was completed immediately prior to the initiation of this test series to allow the RKS to more closely reflect typical field installations of rotary kiln/venturi scrubber designs. The quench section modification is discussed further in Section 2.1.2. Downstream of the primary APCS a backup APCS, consisting of a carbon-bed adsorber and a high-efficiency particulate (HEPA) filter, is in place. The backup system is designed to ensure that organic compound emissions and particulate emissions to the atmosphere are negligible during less than optimal test conditions. The main components of the RKS and its APCS are discussed in more detail in the following subsections.

2.1.1 Incinerator Characteristics

The rotary kiln combustion chamber has an inside diameter of 0.95 m (37.5 in) and is 2.1 m (7 ft) long. The chamber consists of 13 cm (5 in) of refractory encased in a 6.3 mm (0.25 in) thick steel shell. The chamber volume, including the transition sections, is 1.74 m³ (61.4 ft³). Four steel rollers support the kiln barrel. A variable-speed DC motor coupled with a reducing gear transmission tumbles the rotary kiln. Typical rotation speeds range from 0.2 to 1.5 rpm.

The afterburner chamber has a 0.91 m (3 ft) inside diameter and is 2.74 m (9 ft) long. The afterburner chamber wall consists of a 15 cm (6 in) layer of refractory encased in a 6.3 mm (0.25 in) thick carbon steel shell. The volume of the afterburner chamber is 1.80 m³ (63.6 ft³).

An American Combustion, Inc. burner system was used in the RKS for these tests. The system consisted of propane-fired burners in the kiln and afterburner sections, with computer-based gas metering and process control systems designed to control burner flows of propane and air. A separate system was used to control scrubber system operation. Both control systems were interfaced with a personal computer for data acquisition at 20-second intervals.

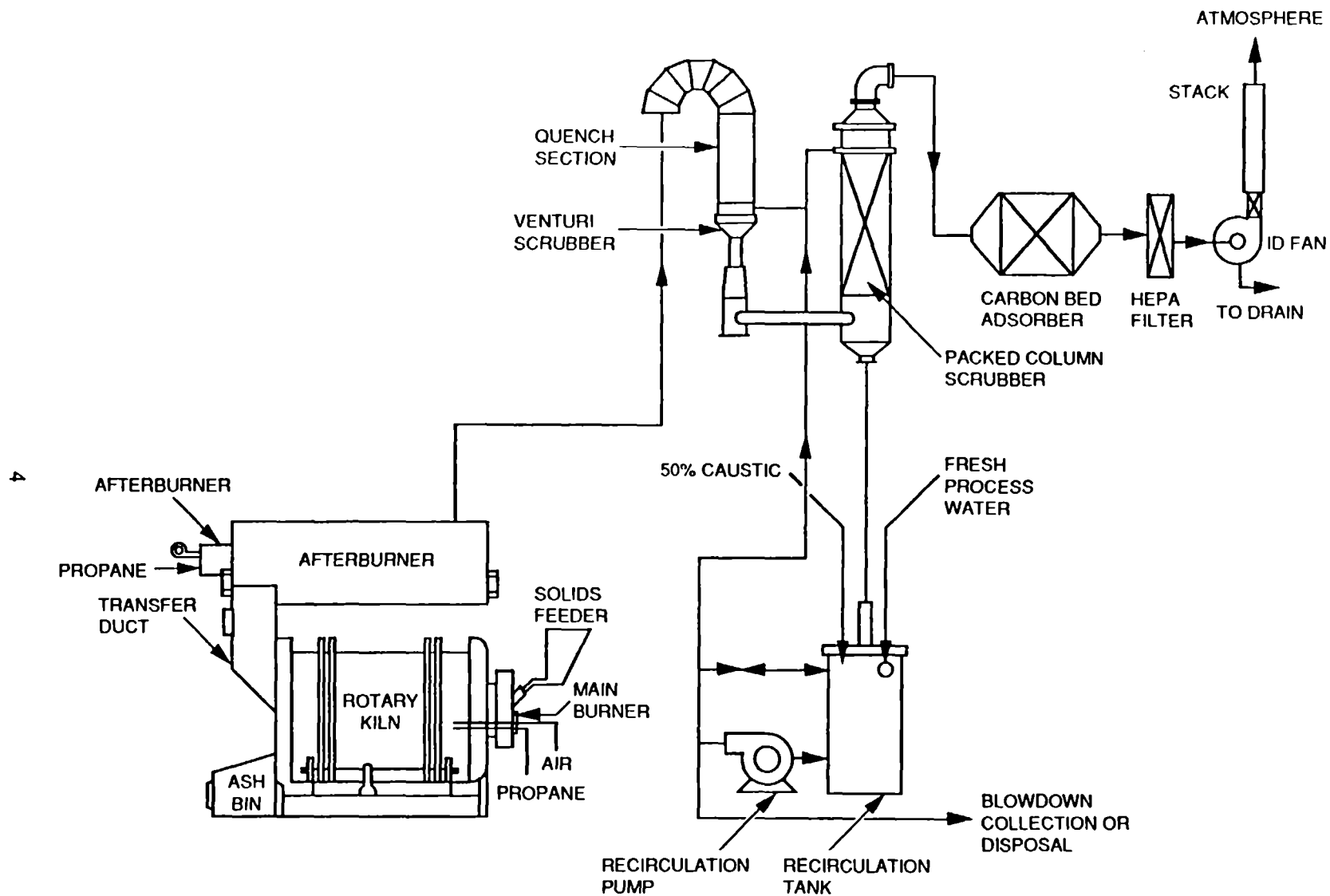


Figure 1. Schematic of the IRF RKS.

TABLE 1. DESIGN CHARACTERISTICS OF THE IRF ROTARY KILN SYSTEM

Characteristics of the Kiln Main Chamber

| | |
|-----------------------|--|
| Length, outside | 2.61 m (8 ft - 7 in) |
| Diameter, outside | 1.22 m (4 ft) |
| Length, inside | 2.13 m (7 ft) |
| Diameter, inside | 0.95 m (3 ft - 1-1/2 in) |
| Chamber volume | 1.74 m ³ (61.36 ft ³) |
| Construction | 0.63 cm (0.25 in) thick cold rolled steel |
| Refractory | 12.7 cm (5 in) thick high alumina castable refractory, variable depth to produce a frustroconical effect for moving solids |
| Rotation | Clockwise or counterclockwise 0.2 to 1.5 rpm |
| Solids retention time | 1 hr (at 0.2 rpm) |
| Burner | American Combustion Burner, rated at 880 kW (3.0 MMBtu/hr) with dynamic O ₂ enhancement capability |
| Primary fuel | Propane |
| Feed system | |
| Liquids | Positive displacement pump via water-cooled lance |
| Sludges | Moyno pump via front face, water-cooled lance |
| Solids | Metered twin-auger screw feeder or fiber pack ram feeder |
| Temperature (max) | 900°C (1,650°C) |

Characteristics of the Afterburner Chamber

| | |
|--------------------|--|
| Length, outside | 3.05 m (10 ft) |
| Diameter, outside | 1.22 m (4 ft) |
| Length, inside | 2.74 m (9 ft) |
| Diameter, inside | 0.91 m (3 ft) |
| Chamber volume | 1.80 m ³ (63.6 ft ³) |
| Construction | 0.63 cm (0.25 in) thick cold rolled steel |
| Refractory | 15.24 cm (6 in) thick high alumina castable refractory |
| Gas Residence Time | 1.2 to 2.5 seconds depending on temperature and excess air |
| Burner | American Combustion Burner rated at 440 kW (1.5 MMBtu/hr) with dynamic O ₂ enhancement capability |
| Primary fuel | Propane |
| Temperature (max) | 1,200°C (2,200°F) |

Characteristics of the Air Pollution Control System

| | |
|------------------|---|
| System capacity | |
| Inlet gas flow | 107 m ³ /min (3,773 acfm) at 1,200°C (2,200°F) and 101 kPa (14.7 psia) |
| Pressure drop | |
| Venturi scrubber | 7.5 kPa (30 in WC) |
| Packed column | 1.0 kPa (4 in WC) |
| Liquid flow | |
| Venturi scrubber | 77.2 L/min (20.4 gpm) at 69 kPa (10 psig) |
| Packed column | 116 L/min (30.6 gpm) at 69 kPa (10 psig) |
| pH control | Feed back control by NaOH solution addition |

2.1.2 Air Pollution Control System

As previously mentioned, combustion gas exiting the afterburner flowed through a primary APCS consisting of a quench section, a venturi scrubber, and a packed column scrubber, then through a secondary APCS consisting of a carbon bed adsorber followed by a HEPA filter. As originally installed, the afterburner exit flue gas was not quenched prior to passing through the venturi scrubber; the venturi scrubber itself was used to quench the flue gas. This is atypical of a conventional rotary kiln/venturi scrubber installation. In addition, since flue gas quenching occurred in the venturi scrubber, its particulate removal efficiency was adversely affected. To rectify this situation, the RKS APCS was modified prior to this test program by installing a quench section upstream of the venturi scrubber. With the quench section installed, the RKS more nearly reflects traditional rotary kiln/venturi scrubber installation designs; and particulate removal characteristics of the venturi scrubber more nearly reflect the capabilities of the technology.

The quench section reduces the temperature of the combustion gas to approximately 77°C (170°F). The cooled flue gas then enters the venturi scrubber which has an automatically adjustable throat. The scrubber is designed to operate at 7.5 kPa (30 in WC) differential pressure, with a maximum liquid flowrate of 77 L/min (20.4 gpm). The scrubber liquor enters at the top of the scrubber and contacts the gas to remove acid gases and entrained particulate.

Downstream of the venturi scrubber, the flue gas enters the packed-column scrubber where additional scrubbing occurs. The scrubber column is packed with 5.1 cm (2 in) diameter polypropylene ballast saddles at a depth of 2.1 m (82 in). It is designed to operate at 1.0 kPa (4 in WC) differential pressure, with a maximum liquid flowrate of 116 L/min (30.6 gpm). Ambient air is also drawn in at the packed-column liquor discharge port. Past experience indicates that induced ambient air comprises approximately 50 percent of the flue gas downstream of the scrubbers.

One liquor recirculation system supplies both the venturi and packed-column scrubbers. The liquor is a dilute NaOH solution and is monitored continuously by a pH sensor. An integral pH controller automatically meters the amount of NaOH needed to maintain the required pH for proper HCl removal.

At the exit of the packed-column scrubber, a demister removes most of the suspended liquid droplets. In a typical commercial incinerator system, the flue gas would be vented to the atmosphere at this point. However, a backup APCS is in place at the IRF. The flue gas passes through a bed of activated carbon designed to adsorb the remaining vapor phase organic compounds. Typically, the carbon bed operates at 77°C (170°F). Because the flue gas is saturated with moisture and is cooled as it flows through the ducts, condensate is continuously formed. The condensate accumulates in the carbon bed and drains through a bottom tap into the blowdown storage tanks.

A set of HEPA filters designed to remove remaining suspended particulate from the flue gas is located downstream of the carbon adsorption bed. An induced draft fan draws and vents the treated effluent gas to the atmosphere.

2.2 SYNTHETIC TEST MIXTURE

The synthetic waste fired during the test program was composed of a mixture of organic liquids added to a clay absorbent material. Trace metals were incorporated by spiking aqueous

mixtures of the metals of interest onto the organic liquid-containing solid material. This synthetic waste was fed to the rotary kiln via a screw feeder.

The organic-liquid base (which supplied the heat content and POHC concentrations to the test mixture) consisted of toluene, with varying amounts of tetrachloroethylene and chlorobenzene added to give a range of synthetic waste chlorine contents. Synthetic waste chlorine content was varied from zero (no chlorinated POHCs added) to nominally 8 percent by weight. The synthetic waste was prepared by mixing the organic liquid with the clay absorbent material to yield a mixture containing about 30 percent (weight) organic liquid. After mixing, the test mixture was poured into 55-gallon drums, each holding approximately 140 kg (300 lbs).

The trace metals of interest were prepared in aqueous solutions. These solutions were then metered into the clay/organic liquid matrix at the screw feeder introducing the waste into the kiln. For the parametric series of tests aimed at evaluating the distribution of the five hazardous constituent and four nonhazardous constituent metals in emissions and discharges, the aqueous trace metal feed solution was metered at a rate which would produce the final nominal synthetic waste feed concentrations noted in Table 2 (assuming no background metal concentration in the clay matrix). For the chromium valence state tests, the aqueous chromium feed solution was metered at a rate which would produce a synthetic waste feed concentration of about 50 ppm Cr(+3 or +6) (again assuming zero background clay chromium content).

2.3 TEST CONDITIONS

As noted in Section 1, the test program consisted of two test series: a three-test chromium valence state series, and an eight-test parametric trace metals series. For all tests, an auger was used to feed the clay/organic mixture at a nominal rate of 63 kg/hr (140 lbs/hr). The metals were premixed in an aqueous solution, then added to the clay/organic liquid feed just before introducing the mixture into the kiln. A gear pump continuously injected the trace metal aqueous solution spike into the feed at the auger to produce the desired spike concentrations. The nominal flowrate through the gear pump was 0.5 to 1 L/hr.

The test variables selected for the three chromium valence state tests were feed chlorine content and the chromium valence state in the waste feed. Two tests were performed with chromium fed in the trivalent state. One of these had a waste feed chlorine content of zero; the other had a waste feed chlorine content of nominally 8 percent. The third test was performed with the aqueous spike chromium fed in the hexavalent state with no chlorine in the feed. All three tests were performed under the same nominal incinerator operating conditions: kiln temperature of 871°C (1,600°F), kiln exit flue gas O₂ of 11.5 percent, afterburner temperature of 1,093°C (2,000°F), and afterburner exit flue gas O₂ of 7.5 percent. These conditions are considered typical of industrial hazardous waste incinerator operation.

The test variables chosen for the parametric test series were feed chlorine content, kiln temperature, and afterburner temperature. A factorial experimental matrix for three variables over three levels was chosen, giving eight target test conditions, as shown in Table 3. All tests were designed for the same nominal excess air levels. These were kiln exit flue gas O₂ of 11.5 percent and afterburner exit flue gas O₂ of 7.5 percent.

The actual kiln and afterburner operating conditions achieved for each test are summarized in Tables 4 and 5, respectively. Table 6 provides a similar summary of the air pollution control system operation for each test. In the tables, the chromium valence state tests are designated as Tests 1, 2, and 3; the parametric tests are designated as Tests 4 through 11.

**TABLE 2. TARGET TRACE METAL FEED CONCENTRATIONS
FOR THE PARAMETRIC TESTS**

| Metal | Synthetic solid hazardous waste concentration (ppm) |
|---|---|
| Hazardous constituent trace metals | |
| Arsenic | 50 |
| Barium | 40 |
| Cadmium | 10 |
| Chromium | 50 |
| Lead | 50 |
| Other trace metals | |
| Bismuth | 140 |
| Copper | 500 |
| Magnesium | 80 |
| Strontium | 250 |

TABLE 3. TARGET TEST CONDITIONS

| Test | Feed Cl content (%) | Kiln exit temperature, °C (°F) | Afterburner exit temperature, °C (°F) |
|---|---------------------|--------------------------------|---------------------------------------|
| Chromium Valence State Tests^a | | | |
| 1 | 0 | 871 (1600) | 1093 (2000) |
| 2 | 0 | 871 (1600) | 1093 (2000) |
| 3 | 8 | 871 (1600) | 1093 (2000) |
| Parametric Trace Metal Tests | | | |
| 4 | 0 | 871 (1600) | 1093 (2000) |
| 5 | 4 | 816 (1500) | 1093 (2000) |
| 6 | 4 | 927 (1700) | 1093 (2000) |
| 7 | 4 | 871 (1600) | 1093 (2000) |
| 8 | 4 | 871 (1600) | 1204 (2200) |
| 9 | 4 | 871 (1600) | 982 (1800) |
| 10 ^b | 4 | 871 (1600) | 1093 (2000) |
| 11 | 8 | 871 (1600) | 1093 (2000) |

^aTest: 1 performed with hexavalent chromium; tests 2 and 3 performed with trivalent chromium.

^bTest point 10 is a duplicate of test point 7.

TABLE 4. KILN OPERATING CONDITIONS

| Parameter | Test 1 (9-29-88) | Test 2 (9-28-88) | Test 3 (9-26-88) | Test 4 (9-14-88) | Test 5 (8-25-88) | Test 6 (9-16-88) |
|--|-------------------------------|-------------------------------|--------------------------------|-------------------------------|-------------------------------|--------------------------------|
| Propane feedrate range, scm/hr (scfh) | 5.1 to 6.8 (180 to 240) | 5.7 to 7.1 (200 to 250) | 7.4 to 9.1 (260 to 320) | 4.5 to 6.2 (160 to 220) | 1.1 to 6.5 (40 to 230) | 7.4 to 8.8 (260 to 310) |
| average, scm/hr (scfh) | 6.2 (217) | 6.4 (224) | 7.9 (280) | 5.0 (176) | 5.4 (190) | 7.8 (275) |
| Combustion air flowrate range, scm/hr (scfh) | 188 to 235 (6630 to 8310) | 201 to 251 (7110 to 8850) | 266 to 321 (9400 to 11,330) | 156 to 217 (5520 to 7650) | 9 to 190 (320 to 6700) | 257 to 304 (9090 to 10,720) |
| average, scm/hr (scfh) | 219 (7739) | 226 (7994) | 283 (9996) | 173 (6095) | 157 (5542) | 274 (9661) |
| Exit temperature range, °C (°F) | 856 to 893 (1572 to 1640) | 839 to 893 (1542 to 1639) | 854 to 886 (1569 to 1626) | 851 to 892 (1564 to 1638) | 793 to 843 (1459 to 1550) | 709 to 946 (1669 to 1735) |
| average, °C (°F) | 873 (1603) | 872 (1602) | 871 (1600) | 874 (1606) | 825 (1517) | 928 (1702) |
| Pressure range, Pa (in WC) | -3 to -25 (-0.01 to -0.18) | -5 to -12 (-0.02 to -0.05) | -3 to -45 (-0.01 to -0.18) | -5 to -16 (-0.02 to -0.07) | -5 to -12 (-0.02 to -0.05) | 0 to -8 (0.00 to -0.03) |
| average, Pa (in WC) | -10 (-0.03) | -6 (-0.02) | -7 (-0.03) | -9 (-0.04) | -8 (-0.03) | -6 (-0.02) |
| Exit O ₂ range, % average, % | 10.7 to 11.8 11.2 | 10.9 to 11.8 11.3 | 11.1 to 13.2 11.5 | 11.8 to 12.8 12.2 | 11.0 to 13.6 11.9 | 9.0 to 11.0 10.6 |
| Clay/organic mixture feedrate | | | | | | |
| total, kg/hr (lb/hr) | 62.6 138.0 | 62.0 136.7 | 61.3 135.1 | 59.4 131.0 | 63.5 140.0 | 62.6 138.0 |
| organic fraction, kg/hr (lb/hr) | 17.5 38.5 | 17.5 38.5 | 18.4 40.5 | 13.8 30.4 | 15.0 33.0 | 17.2 38.0 |

(continued)

TABLE 4. (concluded)

| Parameter | Test 7 (8-30-88) | Test 8 (9-7-88) | Test 9 (9-9-88) | Test 10 (9-20-88) | Test 11 (9-22-88) |
|-------------------------------|---------------------|--------------------|--------------------|----------------------|----------------------|
| Propane feedrate | | | | | |
| range, scm/hr | 6.2 to 7.9 | 7.1 to 10.2 | 5.4 to 6.8 | 4.8 to 7.1 | 7.1 to 9.3 |
| (scfh) | (220 to 280) | (250 to 360) | (190 to 240) | (170 to 250) | (250 to 330) |
| average, scm/hr | 6.9 | 9.2 | 6.1 | 6.4 | 7.5 |
| (scfh) | (244) | (325) | (216) | (225) | (263) |
| Combustion air flowrate | | | | | |
| range, scm/hr | 223 to 277 | 203 to 269 | 176 to 243 | 176 to 257 | 260 to 330 |
| (scfh) | (7880 to 9780) | (7180 to 9500) | (6230 to 8590) | (6230 to 9070) | (9170 to 11,670) |
| average, scm/hr | 246 | 245 | 213 | 233 | 271 |
| (scfh) | (8685) | (8643) | (7537) | (8226) | (9570) |
| Exit temperature | | | | | |
| range, °C | 869 to 886 | 832 to 916 | 836 to 891 | 841 to 894 | 852 to 885 |
| (°F) | (1597 to 1626) | (1530 to 1680) | (1536 to 1636) | (1546 to 1642) | (1565 to 1625) |
| average, °C | 878 | 871 | 875 | 873 | 870 |
| (°F) | (1612) | (1599) | (1607) | (1603) | (1599) |
| Pressure | | | | | |
| range, Pa | -5 to -11 | -5 to -8 | -8 to -20 | -3 to -5 | 0 to -45 |
| (in WC) | (-0.02 to -0.04) | (-0.02 to -0.03) | (-0.03 to -0.08) | (-0.01 to -0.02) | (0.00 to -0.18) |
| average, Pa | -8 | -6 | -10 | -5 | -10 |
| (in WC) | -0.03 | (-0.02) | (-0.04) | (-0.02) | (-0.04) |
| Exit O ₂ | | | | | |
| range, % | 11.7 to 13.7 | 12.8 to 14.5 | 12.1 to 13.0 | 10.6 to 12.7 | 10.8 to 11.7 |
| average, % | 12.4 | 13.6 | 12.7 | 11.4 | 11.4 |
| Clay/organic mixture feedrate | | | | | |
| total, kg/hr | 63.4 | 63.4 | 62.5 | 62.8 | 63.5 |
| (lb/hr) | 139.8 | 139.8 | 137.8 | 138.4 | 140.0 |
| organic fraction, | | | | | |
| kg/hr | 16.8 | 15.0 | 14.1 | 18.7 | 18.1 |
| (lb/hr) | 37.0 | 33.0 | 31.0 | 41.3 | 40.0 |

TABLE 5. AFTERBURNER OPERATING CONDITIONS

| Parameter | Test 1 (9-29-88) | Test 2 (9-28-88) | Test 3 (9-26-88) | Test 4 (9-14-88) | Test 5 (8-25-88) | Test 6 (9-16-88) |
|--|----------------------------------|----------------------------------|----------------------------------|--------------------------------|--------------------------------|--------------------------------|
| Propane feedrate range, scm/hr (scfh) | 17.0 to 17.8 (600 to 630) | 16.7 to 17.6 (590 to 620) | 17.0 to 17.8 (600 to 630) | 16.4 to 17.3 (580 to 610) | 17.8 (630) | 16.4 to 17.3 (580 to 610) |
| average, scm/hr (scfh) | 17.3 (609) | 17.0 (600) | 17.3 (611) | 16.7 (591) | 17.8 (630) | 17.1 (604) |
| Combustion air flowrate range, scm/hr (scfh) | 534 to 540 (18,860 to 19,060) | 510 to 526 (18,020 to 18,570) | 491 to 512 (17,330 to 18,070) | 540 (19,060) | 540 (19,060) | 540 (19,060) |
| average, scm/hr (scfh) | 540 (19,059) | 521 (18,392) | 497 (17,567) | 540 (19,060) | 540 (19,060) | 540 (19,060) |
| Exit temperature range, °C (°F) | 1090 to 1103 (1994 to 2018) | 1091 to 1103 (1996 to 2017) | 1091 to 1102 (1996 to 2016) | 1083 to 1100 (1982 to 2012) | 1052 to 1082 (1925 to 1979) | 1084 to 1112 (1984 to 2033) |
| average, °C (°F) | 1094 (2002) | 1095 (2003) | 1096 (2005) | 1093 (1999) | 1071 (1959) | 1092 (1998) |
| Pressure range, Pa (in WC) | -30 to -95 (-0.12 to -0.38) | -30 to -40 (-0.12 to -0.16) | -15 to -30 (-0.06 to -0.10) | -20 to -30 (-0.08 to -0.12) | -20 to -50 (-0.08 to -0.20) | -25 to -32 (0.10 to -0.13) |
| average, Pa (in WC) | -42 (-0.15) | -32 (-0.12) | -27 (-0.11) | -25 (-0.10) | -42 (-0.17) | -30 (-0.12) |
| Exit O ₂ range, % | 6.6 to 7.9 | 6.4 to 8.0 | 6.3 to 7.7 | 6.1 to 8.0 | 6.0 to 10.2 | 5.7 to 7.8 |
| average, % | 7.3 | 7.4 | 7.2 | 7.1 | 8.4 | 7.1 |
| Exit CO ₁ range, % | 8.9 to 10.1 | 8.9 to 10.2 | 9.0 to 10.2 | 8.6 to 10.2 | 6.7 to 10.0 | 8.7 to 10.4 |
| average, % | 9.5 | 9.4 | 9.5 | 9.5 | 8.2 | 9.4 |

(continued)

TABLE 5. (concluded)

| Parameter | Test 7 (8-30-88) | Test 8 (9-7-88) | Test 9 (9-9-88) | Test 10 (9-20-88) | Test 11 (9-22-88) |
|-------------------------|---------------------|--------------------|--------------------|----------------------|----------------------|
| Propane feedrate | | | | | |
| range, scm/hr | 17.8 | 17.6 to 17.8 | 13.0 to 13.3 | 17.3 to 17.8 | 17.3 to 17.8 |
| (scfh) | (620 to 630) | (620 to 630) | (460 to 470) | (610 to 630) | (610 to 630) |
| average, scm/hr | 17.8 | 17.8 | 13.2 | 17.5 | 17.5 |
| (scfh) | (630) | (630) | (466) | (619) | (619) |
| Combustion air flowrate | | | | | |
| range, scm/hr | 540 | 445 to 540 | 539 to 540 | 540 | 540 |
| (scfh) | (19,060) | (15,730 to 19,060) | (19,050 to 19,060) | (19,060) | (19,060) |
| average, scm/hr | 540 | 451 | 540 | 540 | 540 |
| (scfh) | (19,060) | (15,912) | (19,060) | (19,060) | (19,060) |
| Exit temperature | | | | | |
| range, °C | 1079 to 1095 | 1168 to 1213 | 967 to 991 | 1081 to 1103 | 1084 to 1105 |
| (°F) | (1974 to 2003) | (2135 to 2216) | (1791 to 1816) | (1981 to 2014) | (1984 to 2021) |
| average, °C | 1088 | 1196 | 983 | 1094 | 1092 |
| (°F) | (1991) | (2184) | (1803) | (2000) | (1998) |
| Pressure | | | | | |
| range, Pa | 0 to -55 | -30 to -37 | -15 to -37 | -25 | -25 to -95 |
| (in WC) | (0.00 to -0.22) | (-0.12 to -0.15) | (-0.06 to -0.15) | (-0.12) | (-0.10 to -0.38) |
| average, Pa | -52 | -30 | -22 | -25 | -35 |
| (in WC) | (-0.21) | (-0.12) | (-0.09) | (-0.10) | (-0.14) |
| Exit O ₂ | | | | | |
| range, % | 7.1 to 8.7 | 3.7 to 7.5 | 7.7 to 9.3 | 6.5 to 8.5 | 6.3 to 8.1 |
| average, % | 7.9 | 5.9 | 8.7 | 7.6 | 7.5 |
| Exit CO ₂ | | | | | |
| range, % | 7.6 to 9.5 | 8.9 to 11.2 | 7.7 to 9.0 | 8.5 to 10.0 | 4.8 to 6.8 |
| average, % | 8.8 | 10.3 | 8.3 | 9.2 | 6.1 |

TABLE 6. AIR POLLUTION CONTROL SYSTEM OPERATING CONDITIONS

| Parameter | Test 1 (9-29-88) | Test 2 (9-28-88) | Test 3 (9-26-88) | Test 4 (9-14-88) | Test 5 (8-25-88) | Test 6 (9-16-88) |
|--|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|
| Quench chamber scrubber liquor flowrate | | | | | | |
| range, L/min | 68 | 53 to 68 | 53 to 68 | 53 to 72 | 45 to 49 | 45 to 68 |
| (gpm) | (18.0) | (14.0 to 18.0) | (14.0 to 18.0) | (14.0 to 19.0) | (12.0 to 13.0) | (14.0 to 18.0) |
| average, L/min | 68 | 66 | 67 | 54 | 45 | 67 |
| (gpm) | (18.0) | (17.5) | (17.8) | (14.2) | (12.0) | (17.6) |
| Venturi scrubber liquor flowrate | | | | | | |
| range, L/min | 76 | 76 to 79 | 45 to 87 | 64 to 68 | 64 to 68 | 64 |
| (gpm) | (20.0) | (20.0 to 21.0) | (12.0 to 23.0) | (17.0 to 18.0) | (17.0 to 18.0) | (17.0) |
| average, L/min | 76 | 76 | 73 | 66 | 68 | 64 |
| (gpm) | (20.0) | (20.0) | (19.2) | (17.5) | (17.9) | (17.0) |
| Venturi scrubber pressure drop | | | | | | |
| range, kPa | 6.0 to 6.2 | 6.0 to 6.7 | 4.5 to 6.2 | 5.7 to 7.0 | 4.7 to 5.7 | 5.5 to 6.2 |
| (in WC) | (24 to 25) | (24 to 27) | (18 to 25) | (23 to 28) | (19 to 23) | (22 to 25) |
| average, kPa | 6.0 | 6.1 | 5.5 | 6.0 | 5.3 | 5.7 |
| (in WC) | (24.1) | (24.6) | (22.0) | (24.2) | (21.2) | (23.0) |
| Packed column scrubber liquor flowrate | | | | | | |
| range, L/min | 106 | 106 to 110 | 68 to 132 | 102 to 106 | 114 to 121 | 95 |
| (gpm) | (28.0) | (28.0 to 29.0) | (18.0 to 35.0) | (27.0 to 28.0) | (30.0 to 32.0) | (25.0) |
| average, L/min | 106 | 108 | 106 | 104 | 115 | 95 |
| (gpm) | (28.0) | (28.5) | (27.9) | (27.6) | (30.4) | (25.0) |
| Packed column pressure drop | | | | | | |
| range, kPa | 3.2 to 3.5 | 2.7 to 3.2 | 3.0 to 3.7 | 2.0 to 2.5 | 0.25 to 1.5 | 3.0 to 3.7 |
| (in WC) | (13 to 14) | (11 to 13) | (12 to 15) | (8 to 10) | (1 to 6) | (12 to 15) |
| average, kPa | 3.2 | 3.1 | 3.6 | 2.2 | 0.8 | 3.4 |
| (in WC) | (13.0) | (12.4) | (14.4) | (8.9) | (3.1) | (13.8) |
| Scrubber liquor pH | | | | | | |
| range | 6.5 to 7.1 | 6.5 to 7.2 | 6.0 to 9.6 | 7.0 to 7.3 | 3.3 to 8.8 | 6.4 to 8.2 |
| average | 7.0 | 7.0 | 7.6 | 7.2 | 6.8 | 7.1 |

(continued)

TABLE 6. (continued)

| Parameter | Test 1 (9-29-88) | Test 2 (9-28-88) | Test 3 (9-26-88) | Test 4 (9-14-88) | Test 5 (8-25-88) | Test 6 (9-16-88) |
|--------------------------------|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|
| Scrubber blowdown flowrate | | | | | | |
| range, L/min | 0.8 to 3.0 | 1.7 to 2.6 | 1.5 to 6.1 | 0.0 to 3.0 | 1.9 to 2.6 | 0.8 to 3.8 |
| (gpm) | (0.2 to 0.8) | (0.5 to 0.7) | (0.4 to 1.6) | (0.0 to 0.8) | (0.5 to 0.7) | (0.2 to 1.0) |
| average, L/min | 1.9 | 1.9 | 2.6 | 1.9 | 2.3 | 1.9 |
| (gpm) | (0.5) | (0.5) | (0.7) | (0.5) | (0.6) | (0.5) |
| Scrubber liquor temperature | | | | | | |
| range, (°C) | 68 to 76 | 74 to 75 | 43 to 76 | 73 to 75 | 72 to 75 | 68 to 75 |
| °F | (155 to 169) | (166 to 167) | (109 to 168) | (164 to 167) | (162 to 167) | (155 to 167) |
| average, (°C) | 74 | 75 | 73 | 74 | 73 | 74 |
| °F | (166) | (167) | (163) | (166) | (164) | (166) |
| ID fan inlet pressure | | | | | | |
| range, kPa | -6.0 to -9.0 | -8.5 to -8.7 | -8.5 to -8.7 | -7.5 to -8.5 | -5.5 to -5.7 | -8.7 |
| (in WC) | (-24 to -36) | (-34 to -35) | (-34 to -37) | (-30 to -34) | (-22 to -23) | (-35) |
| average, kPa | -8.3 | -8.6 | -8.6 | -7.6 | -5.5 | -8.7 |
| (in WC) | (-33.5) | (-34.4) | (-34.4) | (-30.6) | (-22.1) | (-35) |

(continued)

TABLE 6. (continued)

| Parameter | Test 7 (8-30-88) | Test 8 (9-7-88) | Test 9 (9-9-88) | Test 10 (9-20-88) | Test 11 (9-22-88) |
|--|---------------------|--------------------|--------------------|----------------------|----------------------|
| Quench chamber scrubber liquor flowrate | | | | | |
| range, L/min | 45 | 57 | 49 to 53 | 53 to 68 | 68 |
| (gpm) | (12.0) | (15.0) | (13.0 to 14.0) | (14.0 to 18.0) | (18.0) |
| average, L/min | 45 | 57 | 53 | 55 | 68 |
| (gpm) | (12.0) | (15.0) | (14.0) | (14.4) | (18.0) |
| Venturi scrubber liquor flowrate | | | | | |
| range, L/min | 53 to 72 | 68 | 68 to 72 | 76 | 72 to 76 |
| (gpm) | (14.0 to 19.0) | (18.0) | (18.0 to 19.0) | (20.0) | (19.0 to 20.0) |
| average, L/min | 69 | 68 | 69 | 76 | 73 |
| (gpm) | (18.1) | (18.0) | (18.3) | (20.0) | (19.3) |
| Venturi scrubber pressure drop | | | | | |
| range, kPa | 2.5 to 7.0 | 4.7 to 5.2 | 5.2 to 6.7 | 5.2 to 6.2 | 4.0 to 6.2 |
| (in WC) | (10 to 28) | (19 to 21) | (21 to 27) | (21 to 25) | (16 to 25) |
| average, kPa | 5.7 | 4.9 | 6.3 | 5.6 | 5.1 |
| (in WC) | (22.8) | (19.8) | (25.4) | (22.6) | (20.6) |
| Packed column scrubber liquor flowrate | | | | | |
| range, L/min | 110 to 114 | 110 to 114 | 106 to 110 | 110 to 114 | 110 to 114 |
| (gpm) | (29 to 30) | (29 to 30) | (28 to 29) | (29 to 30) | (29 to 30) |
| average, L/min | 111 | 112 | 108 | 113 | 111 |
| (gpm) | (29.3) | (29.6) | (28.4) | (29.8) | (29.3) |
| Packed column pressure drop | | | | | |
| range, kPa | 2.7 to 3.7 | 3.2 to 3.7 | 1.2 to 2.0 | 3.0 to 3.7 | 1.5 to 3.7 |
| (in WC) | (11 to 15) | (13 to 15) | (5 to 8) | (12 to 15) | (6 to 15) |
| average, kPa | 3.1 | 3.6 | 1.6 | 3.2 | 3.4 |
| (in WC) | (12.3) | (14.4) | (6.4) | (12.9) | (13.6) |
| Scrubber liquor pH | | | | | |
| range | 6.0 to 8.9 | 6.2 to 8.1 | 5.9 to 8.0 | 5.1 to 8.2 | 5.0 to 8.5 |
| average | 7.2 | 7.2 | 6.9 | 6.8 | 7.5 |

(continued)

TABLE 6. (concluded)

| Parameter | Test 7 (8-30-88) | Test 8 (9-7-88) | Test 9 (9-9-88) | Test 10 (9-20-88) | Test 11 (9-22-88) |
|--------------------------------|---------------------|--------------------|--------------------|----------------------|----------------------|
| Scrubber blowdown flowrate | | | | | |
| range, L/min | 1.5 to 5.7 | 0.8 to 9.5 | 0.0 to 1.9 | 1.5 to 5.7 | 1.5 to 3.0 |
| (gpm) | (0.4 to 1.5) | (0.2 to 2.5) | (0.0 to 0.5) | (0.4 to 1.5) | (0.4 to 0.8) |
| average, L/min | 2.3 | 1.9 | 1.5 | 2.3 | 2.3 |
| (gpm) | (0.6) | (0.5) | (0.4) | (0.6) | (0.6) |
| Scrubber liquor temperature | | | | | |
| range, (°C) | 72 to 74 | 74 to 76 | 72 to 74 | 74 to 76 | 69 to 77 |
| °F | (161 to 166) | (166 to 169) | (162 to 165) | (165 to 168) | (156 to 171) |
| average, (°C) | 73 | 76 | 73 | 75 | 75 |
| °F | (164) | (168) | (163) | (167) | (167) |
| ID fan inlet pressure | | | | | |
| range, kPa | -5.2 to -8.7 | -7.7 to -9.7 | -6.5 to -7.5 | -8.0 to -8.5 | -5.0 to -9.2 |
| (in WC) | (-21 to -35) | (-31 to -39) | (-26 to -30) | (-32 to -34) | (-32 to -37) |
| average, kPa | -7.9 | -8.1 | -7.4 | -8.2 | -8.1 |
| (in WC) | (-31.7) | (-32.6) | (-29.7) | (-32.8) | (-32.4) |

Control room records of the operating parameters recorded at 15-minute intervals are given in Appendix A. Appendix A also contains plots of the operating conditions for the kiln and afterburner recorded at 20-second intervals on the PC data acquisition system.

The ranges and averages presented in Tables 4 and 5 were developed using the computer recorded data obtained during periods of flue gas sampling for all tests except Test 6. During Test 6 a power outage disabled the data acquisition system midway through the test. The remaining test data were taken from the data log book. The kiln exit O₂ monitor also malfunctioned during this test. However, O₂ readings were obtained at 30-minute intervals using an Orsat analyzer; these values were used to develop the values presented in the tables. The APCS data in Table 6 were taken from the control room log. Only flue gas O₂ and CO₂ are given in Tables 4 and 5. Flue gas concentrations of other combustion gas species (e.g. CO) are presented in Section 4.2.

Table 7 summarizes the actual incinerator operating condition ranges and averages achieved for each test (temperatures and flue gas O₂ levels) and compares these to the respective target conditions. Figure 2 presents a graphical summary of the incinerator temperature data from Table 7.

Figure 2 illustrates that average test temperatures achieved were within 9°C (17°F) of target conditions for all tests except one. For this one test the afterburner temperature was 22°C (41°F) lower than the target. The data in Table 7 show that kiln exit flue gas O₂ was generally within 1 percent of the test program target of 11.5 percent, and that afterburner exit flue gas O₂ was generally within about 1 percent of the test program target of 7.5 percent for all tests except one. For this one test, fan capacity limitations resulted in afterburner exit flue gas O₂ being substantially below the target level, while kiln exit flue gas O₂ was high.

Brief flameouts in the kiln and afterburner did occur during Test 3, generally due to loss of signal from the flame sensor. Clay/organic liquid mixture and metal feeds were stopped while the system was restarted, and flue gas sampling had not yet begun. Therefore, this upset is not expected to affect the data.

TABLE 7. ACTUAL VERSUS TARGET OPERATING CONDITIONS FOR THE TRACE METAL TEST SERIES

| Test | Test date | Kiln exit | | | | | Afterburner exit | | | | |
|------------------------------|-----------|----------------------|------------|------------|------------|-----------------------------|----------------------|-------------|-------------|-------------|-----------------------------|
| | | Temperature, °C (°F) | | | | | Temperature, °C (°F) | | | | |
| | | Target | Actual | | | Flue gas O ₂ (%) | Target | Actual | | | Flue gas O ₂ (%) |
| | | | Minimum | Maximum | Average | | | Minimum | Maximum | Average | |
| Chromium valence state tests | | | | | | | | | | | |
| 1 | 9/29/88 | 871 (1600) | 856 (1572) | 893 (1640) | 873 (1603) | 11.1 | 1093 (2000) | 1090 (1994) | 1103 (2018) | 1094 (2003) | 7.3 |
| 2 | 9/28/88 | 871 (1600) | 839 (1542) | 893 (1639) | 872 (1602) | 11.2 | 1093 (2000) | 1091 (1996) | 1103 (2017) | 1095 (2003) | 7.3 |
| 3 | 9/26/88 | 871 (1600) | 854 (1569) | 866 (1626) | 871 (1600) | 11.5 | 1093 (2000) | 1091 (1996) | 1102 (2016) | 1096 (2005) | 7.2 |
| Parametric tests | | | | | | | | | | | |
| 4 | 9/14/88 | 871 (1600) | 851 (1564) | 892 (1638) | 874 (1606) | 12.2 | 1093 (2000) | 1083 (1982) | 1100 (2012) | 1093 (1999) | 7.1 |
| 5 | 8/25/88 | 816 (1500) | 793 (1459) | 843 (1550) | 825 (1517) | 11.9 | 1093 (2000) | 1052 (1925) | 1082 (1979) | 1071 (1959) | 8.4 |
| 6 | 9/26/88 | 927 (1700) | 909 (1669) | 946 (1735) | 928 (1701) | 10.6 ^a | 1093 (2000) | 1084 (1984) | 1112 (2033) | 1092 (1998) | 7.1 |
| 7 | 8/30/88 | 871 (1600) | 869 (1597) | 886 (1626) | 878 (1612) | 12.4 | 1093 (2000) | 1079 (1974) | 1095 (2003) | 1088 (1991) | 7.9 |
| 8 | 9/07/88 | 871 (1600) | 832 (1530) | 916 (1680) | 871 (1599) | 13.6 | 1204 (2200) | 1168 (2135) | 1213 (2216) | 1196 (2184) | 5.8 ^b |
| 9 | 9/09/88 | 871 (1600) | 836 (1536) | 891 (1636) | 875 (1607) | 12.7 | 982 (1800) | 967 (1791) | 991 (1816) | 983 (1803) | 8.7 |
| 10 | 9/20/88 | 871 (1600) | 841 (1546) | 894 (1642) | 873 (1603) | 11.4 | 1093 (2000) | 1081 (1981) | 1103 (2014) | 1094 (2000) | 7.6 |
| 11 | 9/22/88 | 871 (1600) | 852 (1565) | 885 (1625) | 870 (1599) | 11.4 | 1093 (2000) | 1084 (1984) | 1105 (2021) | 1092 (1998) | 7.4 |

^aMonitor not in service. Data obtained using Bacharach Fyrite O₂ analyzer.^bAfterburner air feedrate at fan capacity.

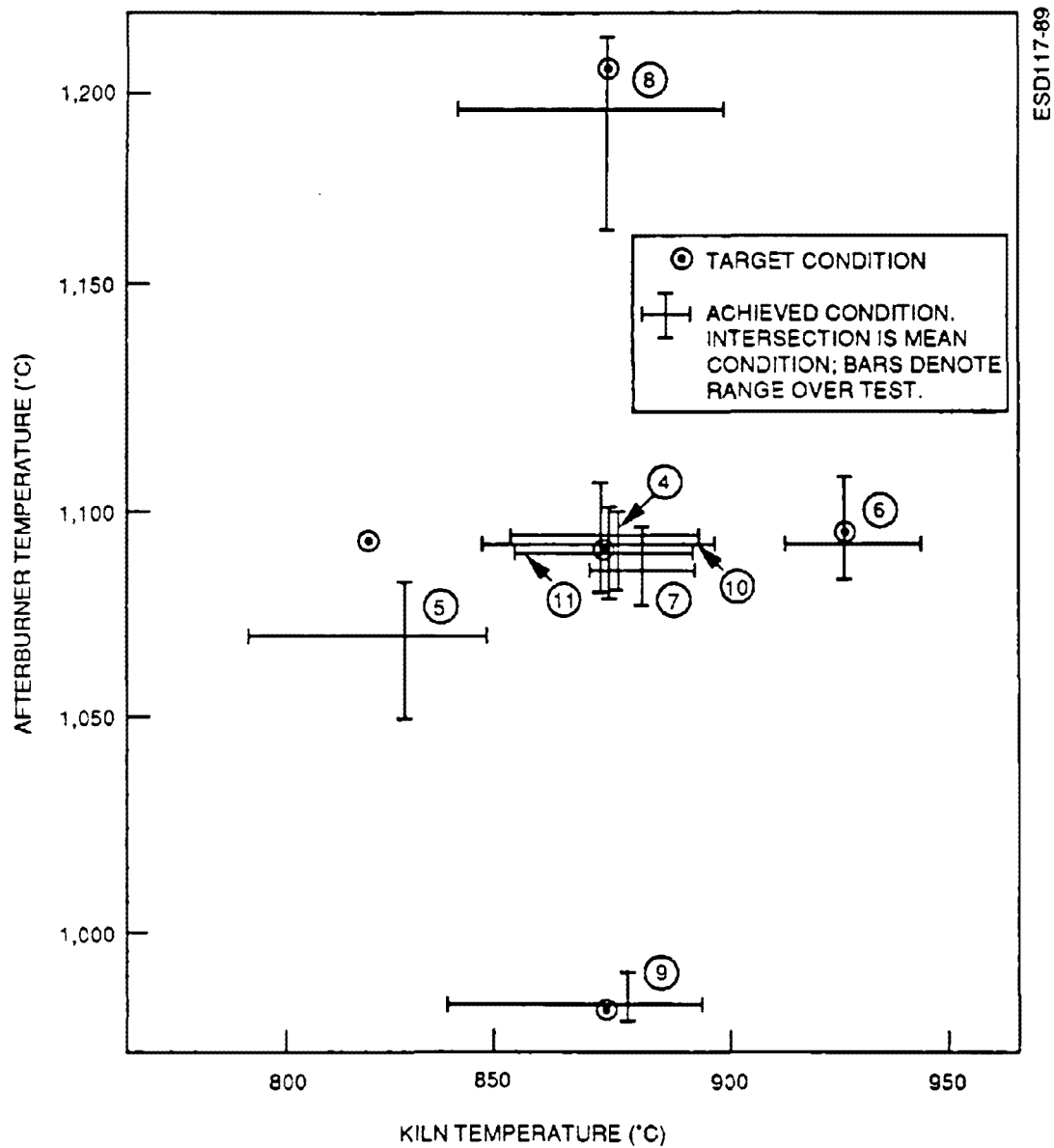


Figure 2. Actual versus target operating temperatures for the parametric trace metal tests.

SECTION 3

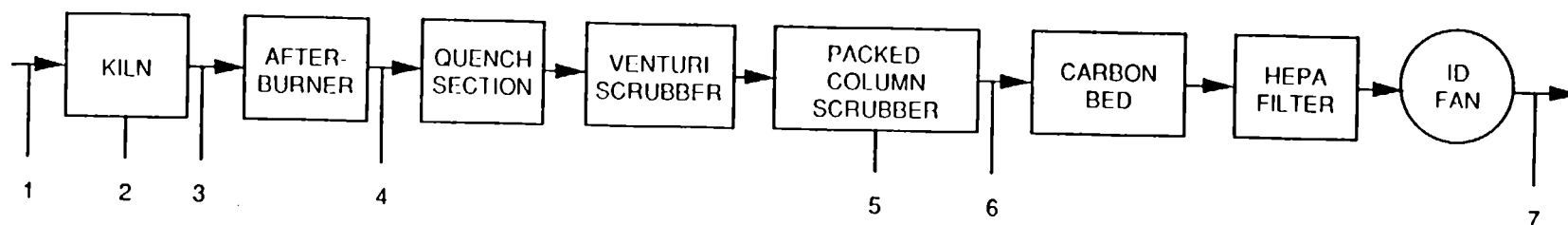
SAMPLING AND ANALYSIS PROCEDURES

Sampling and analysis for both the chromium valence state- and parametric test series were designed to meet research objectives and IRF permit-compliance requirements. Sampling locations were the same for both test series, but specific analysis methods were used for the different trace metals. Figure 3 identifies the sampling point locations. Stack sampling (sampling point 7) was primarily for permit compliance. In general, sampling for each test consisted the following:

- Obtaining a composite sample of the feed materials (clay/organic liquid mixture, and aqueous metal spike solution) and of the ash
- Obtaining several samples of the scrubber blowdown water over time
- Obtaining samples of the flue gas at the afterburner and scrubber exits for particulate and vapor phase metal analysis
- Obtaining samples of the flue gas at the afterburner and scrubber exits for volatile organic hazardous constituent analyses using a volatile organic sampling train (VOST)
- Obtaining continuous monitor sampling of various combinations of flue gas O₂, CO, CO₂, NO_x, and total unburned hydrocarbon (TUHC) at the kiln, afterburner, and scrubber exits and in the stack
- Obtaining samples of the flue gas at the afterburner and scrubber exits, and at the stack for total particulate and HCl

The continuous emission monitors (CEMs) available at the IRF and the locations they monitored for these tests are summarized in Table 8. This monitoring arrangement was employed in all tests. Figure 4 illustrates the generalized flue gas conditioning and flow distribution system at the IRF. Four independent systems, such as those illustrated in Figure 4, are in place so that appropriately conditioned sample gas from four separate sampling locations can be routed to any of the available monitors listed in Table 8. The CEM setup described in Table 8, with appropriate gas conditioning per Figure 4, was employed throughout this test program.

Details of the extractive sampling and analysis procedures are discussed in the following subsections.



SAMPLE LOCATIONS

| Sampling Point | Feeds and Residuals | | | | Continuous Monitors | | | | | Flue Gas | | | | | |
|----------------|---------------------|----------------------|----------|-------------------|---------------------|---------------------|-----------------|----------------|--------------|---|--|---|---|--------------------------|--------------------------------|
| | Clay Organic Liquid | Metal Spike Solution | Kiln Ash | Scrubber Blowdown | O ₂ | CO, CO ₂ | NO _x | Unheated TUIIC | Heated TUIIC | Method 5 (Tests 1-3) with multiple metals impingers, (particulate and Cr) | Simultaneous Method 5 (Tests 1-3) with no filter and caustic impingers (Cr(+6) and HCl) | Method 17 (Tests 4-11) with multiple metals impingers (particulate, metals and HCl) | Method 5 (Tests 4-11) with multiple metals impingers (particulate, metals, and HCl) | VOST (volatile organics) | Method 5 (particulate and HCl) |
| 1 | X | X | | | | | | | | | | | | | |
| 2 | | | X | | | | | | | | | | | | |
| 3 | | | | | X | | | | | | | | | | |
| 4 | | | | | X | X | | X | | X | X | X | | X ^a | |
| 5 | | | | X | | | | | | | | | | | |
| 6 | | | | | | X | X | X | X | X | X | | X | X ^a | |
| 7 | | | | | X | X | | | X | | | | | X ^a | X |

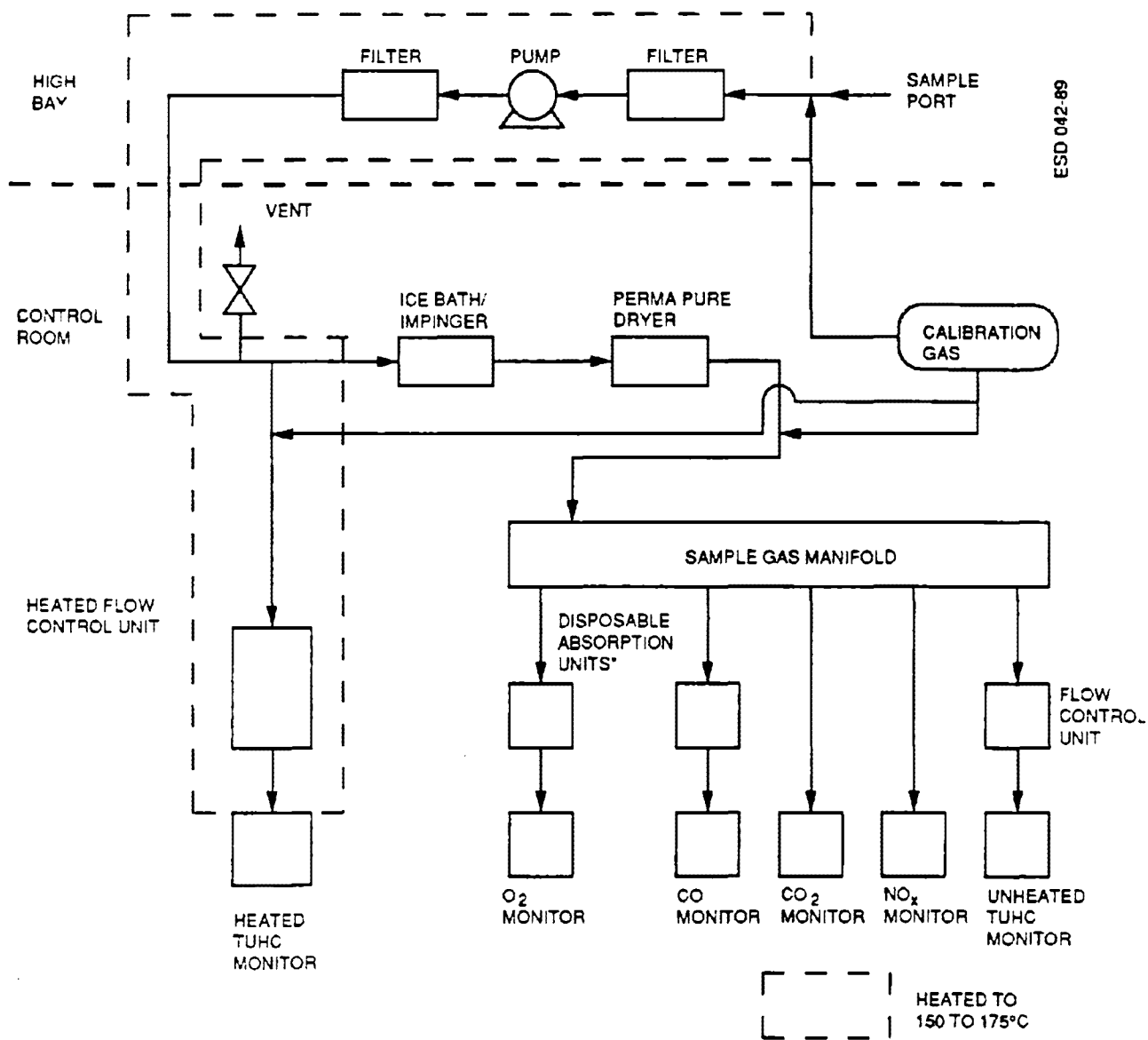
*Tests 4 through 11 only

*Tests 5, 8, and 11 only

Figure 3. Sampling summary for the chromium valence state and parametric trace metals test series.

TABLE 8. CONTINUOUS EMISSION MONITORS USED FOR THE TESTS

| Location | Constituent | Monitor | | | |
|------------------|--------------------|---------------------|----------|------------------|---|
| | | Manufacturer | Model | Principle | Range |
| Kiln exit | O ₂ | Beckman | 755 | Paramagnetic | 0-10 percent 0-25 percent 0-50 percent 0-100 percent |
| Afterburner exit | O ₂ | Beckman | 755 | Paramagnetic | 0-10 percent 0-25 percent 0-50 percent 0-100 percent |
| | CO | Horiba | VIA 500 | NDIR | 0-50 ppm 0-500 ppm |
| | CO ₂ | Horiba | PIR 2000 | NDIR | 0-20 percent 0-80 percent |
| | Unheated TUHC | Shimadzu | GC Mini | FID | 0-10 ppm to 0-2,000 ppm in multiples of 2 |
| Scrubber exit | CO | Horiba | VIA 500 | NDIR | 0-50 ppm 0-500 ppm |
| | CO ₂ | Horiba | PIR 2000 | NDIR | 0-20 percent 0-80 percent |
| | NO _x | Thermo Electron | 10AR | Chemiluminescent | 0-75 ppm to 0-10,000 ppm in multiples of 2 |
| | Unheated TUHC | Shimadzu | GC Mini | FID | 0-10 ppm to 0-2,000 ppm in multiples of 2 |
| | Heated TUHC | Shimadzu | GC Mini | FID | 0-10 ppm to 0-2,000 ppm in multiples of 2 |
| Stack | CO/CO ₂ | Infrared Industries | 7020 | NDIR | 0-20 percent CO ₂ 0-2,000 ppm CO |
| | Heated TUHC | Shimadzu | GC Mini | FID | 0-10 ppm to 0-2,000 ppm in multiples of 2 |
| | O ₂ | Teledyne | 326A | Fuel cell | 0-5 percent 0-10 percent 0-25 percent |



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* Mixed Na and Ca hydroxides for acid gas removal

Figure 4. Generalized CEM sample gas flow schematic.

3.1 CHROMIUM VALENCE TESTS

Table 9 summarizes the sampling and analysis matrix in addition to the CEM monitoring for the chromium valence state test series.

3.1.1 Sampling Procedures

As indicated in Table 9, the flue gas at two locations, the afterburner exit and the scrubber exit, was simultaneously sampled for particulate, total chromium (Cr), hexavalent chromium Cr(+6), and HCl (Test 3 only), using variations of a standard Method 5 sampling train. Two sampling trains were simultaneously operated at each location. One train at each location sampled for particulate load and total chromium levels. The other train at each location sampled for Cr(+6) and, during Test 3, HCl. Particulate load and HCl were also measured at the stack for all tests, using a standard Method 5 train.

The impingers in the train used for particulate and total chromium capture were specified for multiple metals sampling as noted in Table 10. After sampling, the train contents were collected as specified in Method 5. The probe wash (acetone) and filter were desiccated to constant weight to provide the particulate load measurement. After final weighing, the probe wash was resuspended in acetone for later metal analysis. The aqueous condensate from the first impinger was preserved to a pH of less than 2 with HNO_3 .

The impinger contents of the train used for Cr(+6) sampling were as noted in Table 11. This train did not contain a filter since Cr(+6) in flue gas samples is best preserved if it is collected in an alkaline solution as quickly as possible. A NaOH solution was used to wash the sampling probe. This solution was then combined with the solution in impinger 1. Aliquots of the impinger 1, 2, and 3 solutions were taken for HCl measurement for Test 3.

All Method 5 trains used glass-lined sampling probes. The trains at the afterburner and scrubber exits collected at least 2.8 m^3 (100 ft^3) of flue gas over about a 3-hour sampling period. This period began no less than 1/2 hour after the start of the test mixture feed. Test mixture feed continued until sampling was complete. The sampling trains at the stack collected at least 0.9 m^3 (32 ft^3).

Grab sampling of chromium spike solution (50 mL) and scrubber blowdown (100 mL) was performed at the beginning of Method 5 sampling. Additional blowdown grab samples were taken hourly until all Method 5 sampling was complete. The chromium spike solution and each blowdown sample were split into two samples, one for total chromium and one for Cr(+6) analysis. At the conclusion of each test, two composite samples of the kiln ash were collected from the ash pit, one for total chromium and one for Cr(+6) analysis. All spike solution and blowdown samples for total chromium analysis were preserved with HNO_3 to $\text{pH} < 2$. All blowdown samples for Cr(+6) analysis were preserved with NaOH solution to $\text{pH} > 12$. Kiln ash samples for Cr(+6) analysis were preserved with 3 percent Na_2CO_3 , 2 percent NaOH solution (the solution used for Cr(+6) digestion in Method 3060, Reference 2).

Composite feed samples from each drum were collected by trier sampling at three locations in the drum cross-section. Each of these composites was ultimately analyzed for toluene, tetrachloroethylene, chlorobenzene, and ultimate composition. Samples for volatile organic analysis were collected from each drum's composite sample when the contents of that drum were first fed into the kiln. These samples were sealed in 40 mL VOA vials at that time. An aliquot of each drum's composite sample was also submitted for ultimate analysis and for total chromium analysis.

TABLE 9. SAMPLING AND ANALYSIS MATRIX FOR THE CHROMIUM VALENCE STATE TESTS

| Sample | Location | Sampling procedure | Analysis | | |
|---------------------------------|---|-----------------------|---|--|-------------|
| | | | Parameter | Method | Frequency |
| Aqueous chromium spike solution | Kiln inlet | Composite | Total Cr | Digestion by Method 3010 analysis by Method 7191 ^a | 1/test |
| | | | Cr(+6) | Digestion by Method 3060 ^b analysis by Method 7197 ^c | 1/test |
| Feed clay | Waste preparation | Composite | Cr(+6) | Digestion by Method 3060 ^b analysis by Method 7197 ^c | 1 composite |
| Clay/organic liquid mixture | Preparation storage drum | Tner | C ₇ H ₈ , C ₂ Cl ₄ , C ₆ H ₅ Cl | Purge and trap by Method 5030, ^d GC/FID analysis | 1/test |
| | | | Ultimate analysis (C, H, O, Cl) | A003 ^e | 1/test |
| | | | Total Cr | Parr bomb ashing ^d , Digestion by Method 3050 ^e , analysis by Method 6010 ^e | 1/test |
| Kiln ash | Kiln ash pit | Composite | Total Cr | Digestion by Method 3050 analysis by Method 7191 ^a | 1/test |
| | | | Cr(+6) | Digestion by Method 3060 ^b , analysis by Method 7197 ^c | 4/test |
| Scrubber blow-down water | Blowdown discharge | Grab (tap) | Total Cr | Digestion by Method 3010 analysis by Method 7191 ^a | 4/test |
| | | | Cr(+6) | Digestion by Method 3060 ^b , analysis by 7197 ^c | 4/test |
| Flue gas | Afterburner and scrubber exits | Method 5 ^f | Particulate load | Method 5 ^f | 1/test |
| | | | Total Cr | Digestion by Method 3010 (impingers) or Method 3050 (particulate), analysis by Method 7191 ^a | 1/test |
| | | Method 5 ^f | HCl | Analysis of impinger solution for Cl by specific ion electrode | Test 3 only |
| | | | Cr(+6) | Digestion by Method 3060 ^b , analysis by Method 7197 ^c | 1/test |
| Stack gas | Stack downstream of carbon bed/ HEPA filter | Method 5 ^f | Particulate load | Method 5 ^f | 1/test |
| | | | HCl | Analysis of impinger solution for Cl by specific ion electrode | Test 3 only |

^a(1)

^b(2)

^c(3)

^d(4)

^e(5)

TABLE 10. MULTIPLE METALS TRAIN IMPINGER SYSTEM REAGENTS
FOR THE CHROMIUM VALENCE STATE TESTS

| Impinger Number | Reagent | Quantity |
|--------------------|---|----------|
| 1 | Empty | |
| 2 | 5 percent HNO ₃ and 10 percent H ₂ O ₂ | 100 mL |
| 3 | 5 percent HNO ₃ and 10 percent H ₂ O ₂ | 100 mL |
| 4 | Silica gel | 750g |

TABLE 11. Cr(+6) TRAIN IMPINGER
SYSTEM REAGENTS

| Impinger Number | Reagent | Quantity |
|--------------------|------------|----------|
| 1 | 0.1N NaOH | 100 mL |
| 2 | 0.1N NaOH | 100 mL |
| 3 | 0.1N NaOH | 100 mL |
| 4 | Silica gel | 750g |

One composite sample of the feed clay material was also taken and preserved for Cr(+6) analysis with 3 percent Na₂CO₃, 2 percent NaOH solution.

3.1.2 Analysis Procedures

Table 12 summarizes the number of samples collected over the three-test chromium valence state test series. Particulate load determinations from Method 5 train samples were performed at the IRF in accordance with Method 5 protocol. Chloride determinations for HCl measurement were performed on aliquots of appropriate impinger solutions via specific ion electrode analysis at the IRF.

All samples for chromium analyses, preserved as noted in Section 3.1.1, were shipped to the Acurex analytical laboratory in Mountain View, California. Total chromium analyses were performed using graphite furnace atomic absorption (AA) spectroscopy via Method 7191 for all samples except the composite feed samples (clay/organic liquid mixture). Inductively coupled argon plasma spectroscopy (ICAP) via Method 6010 was used for this sample matrix. Solid samples (probe wash, particulate, kiln ash) were digested by Method 3050 prior to analysis. Liquid samples (spike solution, blowdown) were digested by Method 3010 prior to analysis. The clay/organic liquid mixture samples were Parr bomb ashed, then digested by Method 3050 prior to analysis.

Samples for Cr(+6) analysis were digested by Method 3060. The solid samples (kiln ash and feed clay) had been preserved after collection in the method digestion solution. For these, digestion volumes were corrected and the method followed through. Digestates were analyzed by Method 7197 (chelation/extraction). Graphite furnace atomic absorption spectroscopy (in accordance with Method 7191) was used to analyze extracts from the Method 7197 procedure.

3.2 PARAMETRIC TRACE METAL TESTS

Table 13 summarizes the sampling and analysis matrix for the parametric trace metal tests.

3.2.1 Sampling Procedures

As indicated in Table 13, the incinerator flue gas was characterized at three locations: the afterburner exit, the scrubber exit, and the stack. Characterization at the afterburner and scrubber exit locations supported test objectives. Stack gas sampling was performed to ensure compliance with the IRF's operating permit.

The sampling protocols performed in the afterburner exit flue gas were designed to measure flue gas particulate load and size distribution, HCl, trace metal vapor phase and particulate emissions by particulate size range, and volatile organic hazardous constituent emissions.

Volatile organic hazardous constituent emissions were sampled using the VOST protocol. All other parameters listed above were measured in the afterburner exit flue gas using a variation of a Method 17 train. The impingers used for the afterburner exit Method 17 sampling train are noted in Table 14. This impinger train is the same as that used for total chromium in the chromium valence state tests, with the addition of a caustic impinger between the initially empty impinger and the first HNO₃/H₂O₂ impinger. After sampling, the contents of impingers 1 and 2 were combined and aliquots obtained for HCl analysis. The impinger

TABLE 12. SUMMARY OF CHROMIUM VALENCE STATE TEST SAMPLES

| Sample type | Analyte | Number of samples | |
|---------------------------------|---|-------------------|-------|
| | | Each test | Total |
| Feed clay | Cr(+6) | — | 1 |
| Clay/organic liquid mixture | C ₇ H ₈ , C ₂ Cl ₄ , C ₆ H ₅ Cl | 1 | 3 |
| | Ultimate analysis | 1 | 3 |
| | Total Cr | 1 | 3 |
| Aqueous chromium spike solution | Total Cr | 1 | 3 |
| | Cr(+6) | 1 | 3 |
| Kiln ash | Total Cr | 1 | 3 |
| | Cr(+6) | 1 | 3 |
| Scrubber blowdown | Total Cr | 4 | 12 |
| | Cr(+6) | 4 | 12 |
| Afterburner exit: | | | |
| Method 5 Train A | | | |
| Probe wash | Particulate, Total Cr | 1 | 3 |
| Filter | Particulate, Total Cr | 1 | 3 |
| 1st impinger | Total Cr | 1 | 3 |
| 2nd impinger | Total Cr | 1 | 3 |
| 3rd impinger | Total Cr | 1 | 3 |
| Method 5 Train B | | | |
| 1st impinger | Cr(+6), Cl ⁻ | 1 | 3 |
| 2nd impinger | Cr(+6), Cl ⁻ | 1 | 3 |
| 3rd impinger | Cr(+6), Cl ⁻ | 1 | 3 |
| Scrubber exit: | | | |
| Method 5 Train A | | | |
| Probe wash | Particulate, Total Cr | 1 | 3 |
| Filter | Particulate, Total Cr | 1 | 3 |
| 1st impinger | Total Cr | 1 | 3 |
| 2nd impinger | Total Cr | 1 | 3 |
| 3rd impinger | Total Cr | 1 | 3 |
| Method 5 Train B | | | |
| 1st impinger | Cr(+6), Cl ⁻ | 1 | 3 |
| 2nd impinger | Cr(+6), Cl ⁻ | 1 | 3 |
| 3rd impinger | Cr(+6), Cl ⁻ | 1 | 3 |
| Stack gas: | | | |
| Method 5 Train | | | |
| Probe wash + filter | Particulate | 1 | 3 |
| 1st and 2nd impinger | Cl ⁻ | Test 3 only | 1 |

**TABLE 13. SAMPLING AND ANALYSIS MATRIX FOR THE PARAMETRIC
TRACE METAL TESTS**

| Sample | Location | Sampling procedure | Analysis | | Frequency |
|------------------------------------|-----------------------------|-----------------------|--|--|------------------|
| | | | Parameter | Method | |
| Clay/organic liquid mixture | Preparation storage drum | Trier | C ₂ H ₆ , C ₂ Cl ₄ , C ₆ H ₆ , Cl | Purge and trap by Method 5030 ^a , GC/FID analysis | 1 composite/test |
| | | | Ultimate analysis (C, H, O, Cl) | A003 ^b | 1 composite/test |
| | | | Cr and Mg | Parr bomb ashing, digestion by Method 3050, ICAP analysis by Method 6010 ^c | 1 composite/test |
| | | | Pb | Parr bomb ashing, digestion by Method 3050, furnace AA by Method 7421 ^c | 1 composite/test |
| | | | Bi and Sr | Parr bomb ashing, digestion by Method 3050, AA analysis by 300 series methods ^c | 1 composite/test |
| Aqueous metal spike solution | Kiln inlet | Composite | As and Pb | Digestion by Method 3010, furnace AA by 7000 series methods ^a | 1 composite/test |
| | | | Ba, Cd, Cr, Cu, and Mg | Digestion by Method 3010, ICAP analysis by Method 6010 ^c | 1 composite/test |
| | | | Bi and Sr | Digestion by Method 3010 ^c , AA analysis by 300 series methods ^c | 1 composite/test |
| Kiln ash | Kiln ash pit | Composite | As and Pb | Digestion by Method 3050, furnace AA by 7000 series methods ^a | 1/test |
| | | | Ba, Cd, Cr, Cu, and Mg | Digestion by Method 3050, ICAP analysis by Method 6010 ^c | 1/test |
| | | | Bi and Sr | Digestion by Method 3050 ^c AA analysis by 300 series methods ^c | 1/test |

(continued)

^a(1)

^b(3)

^c(7)

TABLE 13. (continued)

| Sample | Location | Sampling procedure | Analysis | | Frequency |
|-------------------------|--------------------|--------------------------|---|--|-------------------|
| | | | Parameter | Method | |
| Scrubber blowdown water | Blowdown discharge | Grab/tap | As and Pb | Digestion by Method 3010, furnace AA by 7000 series methods ^a | At least 3/test |
| | | | Ba, Cd, Cr, Cu, and Mg | Digestion by Method 3010, ICAP analysis by Method 6010 ^b | At least 3/test |
| | | | Bi and Sr | Digestion by Method 3010 ^c , AA analysis by 300 series methods ^c | At least 3/test |
| Flue gas | Afterburner exit | Method 17 ^d | Particulate load | Method 17 ^d | 1/test |
| | | | Particulate size distribution | ASME PTC 28 ^e | 1/test |
| | | | HCl | Analysis of impinger solution for Cl by specific ion electrode | 1/test |
| | | | As and Pb | Digestion by Method 3010 or 3050, furnace AA by 7000 series methods ^a (impingers and particulate by size) | 1/test |
| | | | Ba, Cd, Cr, Cu, and Mg | Digestion by Method 3010 or 3050, ICAP analysis by Method 6010 ^b (impingers and particulate by size) | 1/test |
| | | | Bi and Sr | Digestion by Method 3010 or 3050 ^c , AA analysis by 300 series methods ^c (impingers and particulate by size) | 1/test |
| | | Method 0030 ^f | Volatile organic hazardous constituents | Thermal desorption, purge and trap by Method 5040 ^g , GC/FID analysis | 3 trap pairs/test |

(continued)

^a(1)^c(7)^e(5)^f(6)

TABLE 13. (concluded)

| Sample | Location | Sampling procedure | Analysis | | Frequency |
|-------------------------|--|--------------------------|---|--|--|
| | | | Parameter | Method | |
| Flue gas (continued) | Scrubber exit | Method 5 ^d | Particulate load | Method 5 ^d | 1/test |
| | | | HCl | Analysis of impinger solution for Cl by specific ion electrode | 1/test |
| | | | As and Pb | Digestion by Method 3010 or 3050, furnace AA by 7000 series methods ^e (impingers and particulate) | 1/test |
| | | | Ba, Cd, Cr, Cu, and Mg | Digestion by Method 3010 or 3050, ICAP analysis by Method 6010 ^e (impingers and particulate) | 1/test |
| | | | Bi and Sr | Digestion by Method 3010 or 3050 ^e , AA analysis by 300 series methods ^e (impingers and particulate) | 1/test |
| | | Method 0030 ^f | Volatile organic hazardous constituents | Thermal desorption, purge and trap by Method 5040 ^f , GC/FID analysis | 3 trap pairs/test |
| | | | | Thermal desorption and trap GC/MS analysis by Method 5040 ^f | 3 Trap pairs, Test 4 only |
| Stack gas | Stack downstream of carbon bed/HEPA filter | Method 5 ^d | Particulate load | Method 5 ^d | 1/test |
| | | | HCl | Analysis of impinger solution for Cl by specific ion electrode | |
| | | Method 0030 ^f | Volatile organic hazardous constituents | Thermal desorption, purge and trap by Method 5040 ^f , GC/FID analysis | 3 trap pairs/test, Tests 8, 9, and 11 only |

^a(1)^c(7)^a(5)

**TABLE 14. MULTIPLE METALS TRAIN IMPINGER SYSTEM
REAGENTS FOR THE PARAMETRIC TRACE
METAL TESTS**

| Impinger Number | Reagent | Quantity |
|--------------------|---|----------|
| 1 | Empty | |
| 2 | 0.1 N NaOH | 100 mL |
| 3 | 5 percent HNO ₃ and 10 percent H ₂ O ₂ | 100 mL |
| 4 | 5 percent HNO ₃ and 10 percent H ₂ O ₂ | 100 mL |
| 5 | Silica gel | 750g |

contents were preserved to a pH of less than 2 with HNO₃ for later metal analysis. The sampling probe was washed with acetone. The probe wash was desiccated and weighed, then resuspended in acetone for later metal analysis.

The modified Method 17 train at the afterburner exit also collected particulate in an oversized alundum thimble placed within the sampling probe. After obtaining the total particulate weight, the particulate in these samples was divided according to the terminal velocity in air using a centrifugal classifier. This device is described in the ASME Power Test Code 28 (6). Weights of the resulting eight size cuts were recorded. These samples were later recombined into four size cuts and subjected to metals analysis to provide data on metal distribution by particulate size.

The sampling protocol performed in the scrubber exit flue gas was designed to measure the same parameters measured in the afterburner exit flue gas, with the exception that particle sizing and metals analysis by particle size were not performed. Accordingly, VOST (Method 0030) sampling of the scrubber exit was performed. However, instead of a modified Method 17 train, a Method 5 train was run in the scrubber exit. The particulate levels in the scrubber exit were expected to be significantly lower than in the afterburner exit, making the collection of sufficient particulate difficult. In addition, the high water content of the scrubber exit flue gas would cause a moist and agglomerated particulate catch. Classification of this catch would likely not be representative of the actual particulate size distribution in the scrubber exit flue gas.

The impingers used in the scrubber exit Method 5 train were the same as those used in the afterburner exit Method 17 train. After sampling, the filter and probe wash (acetone) from the Method 5 train were recovered and desiccated to constant weight per the Method 5 procedure. The probe wash was then resuspended in acetone for later metal analysis. Impinger collection, aliquoting, combining, and preservation for the scrubber exit Method 5 train were exactly the same as for the afterburner exit Method 17 train discussed above.

A Method 5 train was used to sample the stack gas for each test to ensure compliance with the IRF operating permit. Particulate load and HCl levels were measured. A Method 5 train with impinger contents as noted in Table 11 was used at this location. In addition, VOST (Method 0030) sampling was performed at the stack location for Tests 8, 9, and 11 to supply continuing data on carbon bed organic component removal efficiency.

As for the chromium valence state tests, all scrubber exit Method 5 train sampling collected at least 2.8 m³ (100 ft³) of flue gas over a nominal 3-hour sampling period. Afterburner exit Method 17 train sampling collected at least 19.8 m³ (700 ft³) of flue gas over the same nominal 3-hour sampling period. This period began no less than 1/2 hour after the start of test mixture feed. Test mixture feed continued until all sampling was completed.

In addition to the flue gas sampling described above, samples of the clay/organic liquid feed mixture, the aqueous trace metal spike solution, the kiln ash, and the scrubber blowdown were collected for analysis. Composite feed samples from each drum were collected by trier sampling at three locations in the drum cross-section. Each of these composites were ultimately analyzed for toluene, tetrachloroethylene, chlorobenzene, bismuth, chromium, lead, magnesium, strontium, and ultimate composition. Samples for volatile organic analysis were collected from each drum's composite sample when the contents of that drum were first fed into the kiln. These samples were sealed in 40 mL VOA vials at that time. An aliquot of each drum's composite sample was also submitted for ultimate analysis and bismuth, chromium, lead, magnesium, and strontium analysis. Previous data suggested that clay background levels of chromium and lead might be greater than 10 percent of the final feed material (clay/organic liquid plus aqueous metal spike solution) concentration. Thus, the contribution of the clay to final concentrations of chromium and lead was measured. No data on bismuth, magnesium, and strontium concentrations in the clay existed, so analyses of the clay/organic liquid for these were also included in the analysis protocol.

A composite sample of each test's aqueous metal spike solution was also collected for trace metal analysis. These samples were preserved with HNO₃ to pH < 2.

Blowdown sampling was performed as discussed in Section 3.1.1 for the chromium valence state tests. To reiterate, a grab (100 mL) sample was taken at the start of Method 17/5 sampling and hourly thereafter until sampling was completed. A final blowdown sample was taken at the end of the Method 17/5 sampling period. Each individual blowdown sample was preserved with HNO₃ to pH < 2.

A composite kiln ash sample was collected from the ash pit after the completion of each test. The ash pit contents were weighed and a clean, empty ash collection drum was placed in the collection pit for the subsequent test.

3.2.2 Analysis Procedures

Table 15 summarizes the number of samples collected over the eight-test parametric trace metal program. Particulate load determinations from Methods 17 and 5 samples were performed at the IRF, in accordance with respective method procedures, prior to size classification and combination for trace metal analysis as discussed in Section 3.2.1. Chloride analyses for determining HCl emissions were performed on aliquots of appropriate impinger solution combinations via specific ion electrode analysis at the IRF.

Analysis of VOST (Method 0030) traps was performed at the IRF by thermal desorption/purge and trap GC/FID analysis. Thermal desorption/purge and trap was in

TABLE 15. SUMMARY OF PARAMETRIC TRACE METAL TEST SAMPLES

| Sample type | Analyte | Number of samples | |
|--|---|-------------------|-------|
| | | Each test | Total |
| Clay/organic liquid feed | C ₇ H ₈ , C ₂ Cl ₄ , C ₆ H ₅ Cl | 1 to 3 | 26 |
| | Ultimate analysis (C, H, O, Cl) | 1 to 3 | 23 |
| | Bi, Cr, Pb, Mg, Sr | 1 | 8 |
| Aqueous metal spike solution | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| Kiln ash | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| Scrubber blowdown | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 3 to 5 | 39 |
| Afterburner exit flue gas: VOST (Method 0030) Sample trap pair | Volatile organic hazardous constituents | 3 | 24 |
| Field blank trap pair | Volatile organic hazardous constituents | 1 | 8 |
| Method 17 train <2 μ m particulate | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| 2 to 4 μ m particulate | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| 4 to 10 μ m particulate | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| >10 μ m particulate | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| 1st and 2nd impingers | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr, Cl ⁻ | 1 | 8 |

(continued)

TABLE 15. (concluded)

| Sample type | Analyte | Number of samples | |
|---|--|----------------------------|-------|
| | | Each test | Total |
| 3rd impinger | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| 4th impinger | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| Scrubber Exit Flue gas: VOST (Method 0030) Sample trap pair | Volatile organic hazardous constituents | 3 | 24 |
| Field blank trap pair | Volatile organic hazardous constituents | 1 | 8 |
| Method 5 train: Probe wash | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| Filter | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| 1st and 2nd impinger | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr, Cl ⁻ | 1 | 8 |
| 3rd impinger | As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| 4th impinger | As, Ba, Cu, Cr, Pb, Bi, Cu, Mg, Sr | 1 | 8 |
| Stack gas: VOST (Method 0030) Sample trap pair | Volatile organic hazardous constituents | 3 (Tests 5, 8, 11 only) | 9 |
| Field blank trap pairs | Volatile organic hazardous constituents | 1 (Tests 5, 8, 11 only) | 3 |
| Method 5 train | | | |
| 1st and 2nd impinger | Cl ⁻ | 1 | 8 |
| 3rd impinger | Cl ⁻ | 1 | 8 |

accordance with Method 5040. Analysis was by capillary column GC/FID. The 22 volatile organic compounds routinely determined via this method at the IRF were analyzed. These compounds are listed in Table 16.

The clay/organic liquid feed composite samples were also analyzed at the IRF for toluene, tetrachloroethylene, and chlorobenzene via purge and trap GC/FID. Purge and trap was in accordance with Method 5030. Composite clay/organic liquid feed samples were sent to Galbraith Laboratories in Knoxville, Tennessee, for ultimate analysis.

All samples for trace metal analysis were preserved as noted in Section 3.2.1, and shipped to the Acurex analytical laboratory in Mountain View, California for analysis. Arsenic and lead analyses were performed via graphite furnace atomic absorption (AA) spectroscopy: arsenic by Method 7060 and lead by Method 7421. Barium, cadmium, chromium, copper, and magnesium analyses were performed by ICAP via Method 6010. Bismuth and strontium analyses were performed by flame AA: bismuth by Method 303a and strontium by Method 326a (7). Samples were digested appropriately prior to analyses. Method 3050 was used for solid samples; Method 3010 was used for aqueous liquid samples. Clay/organic liquid samples were Parr bomb ashed (4) prior to digestion.

TABLE 16. VOLATILE ORGANIC COMPOUNDS ROUTINELY ANALYZED BY GC/FID AT THE IRF

| | |
|-------------------------|---|
| Methylene chloride | Benzene |
| 1,1-Dichloroethane | 1,1,2-Trichloroethane |
| t-1,2-Dichloroethylene | Hexane |
| Chloroform | Bromoform |
| 1,2-Dichloroethane | Tetrachloroethylene + Tetrachloroethane |
| 1,1,1-Trichloroethane | Toluene |
| Carbon tetrachloride | Chlorobenzene |
| Bromodichloromethane | Ethyl benzene |
| 1,2-Dichloropropane | 1,3-Dichlorobenzene |
| t-1,3-Dichloropropylene | 1,2-Dichlorobenzene |
| Trichloroethylene | 1,4-Dichlorobenzene |

SECTION 4

TEST RESULTS

Results from the test program are discussed in this section. The discussion is subdivided as follows: measured synthetic waste feed composition, continuous emission monitoring data, particulate and HCl emissions, chromium valence state test results, parametric trace metals test results, POHC DREs, and PIC emissions.

4.1 SYNTHETIC WASTE FEED COMPOSITION

Section 2 discussed the targeted synthetic waste feed composition planned for each test so that test program objectives could be attained. Section 3 noted that the various waste feed components were sampled during each test and analyzed to verify the actual composition of the feeds prepared. This section discusses results of the actual feed analyses.

Table 17 summarizes the clay/organic liquid mixture POHC concentrations measured for each test's composite sample and compares these to target values. The data in Table 17 confirm the close agreement between measured and target composition for all tests.

Table 18 summarizes the analyzed aqueous spike solution concentrations for Tests 1 through 3, the chromium valence state tests. Table 19 provides a similar summary for Tests 4 through 11, the parametric trace metal tests. As indicated, with one possible exception, analyzed concentrations were comparable to prepared concentrations. The exception was Test 5 in which the analyzed aqueous spike metal concentrations were lower than the prepared concentrations for all the metals. The reason for this exception is not known.

Available data on the clay matrix composition from a previous analysis indicated that the clay material contained less than 1 mg/kg of arsenic and cadmium and about 4 mg/kg copper. These values were negligible compared to the target integrated feed concentrations noted in Section 2 for these elements. These same data also indicated that the clay matrix contained about 20 mg/kg barium, 25 mg/kg lead, and 50 mg/kg chromium. These values are sufficiently high that the background clay matrix contribution to integrated feed concentrations would not be negligible. No previous data on bismuth, strontium, and magnesium concentrations were available. As a consequence, the feed clay/organic liquid mixtures for the parametric trace metals tests were analyzed for bismuth, chromium, lead, magnesium, and strontium, and a composite of the Test 1 and 2 clay/organic liquid mixture and the Test 3 mixture were analyzed for chromium to clearly determine the clay contribution to integrated feed metal concentrations. Results of these analyses are summarized in Table 20.

As indicated in Table 20, the analysis results for 10 separate feed samples were very comparable. The average values noted in the table were, thus, used to calculate the clay contribution to the integrated feed concentration for each metal. Bismuth was only analyzed for in one feed matrix sample. At the 12 ppm level noted for this sample in Table 20, the clay

TABLE 17. POHC CONCENTRATIONS IN CLAY/ORGANIC LIQUID FEED SAMPLES

| Test | Test date | Weight percent in mixture | | | |
|----------------------|-----------|---------------------------|---------------------|---------------|-------------------|
| | | Toluene | Tetrachloroethylene | Chlorobenzene | Chlorine content* |
| Mixture 1 | | | | | |
| Target Composition | | 28.6 | 0 | 0 | 0 |
| Measured composition | | | | | |
| 1 | 9/29/88 | 27.9 | 0 | 0 | 0 |
| 2 | 9/28/88 | 27.9 | 0 | 0 | 0 |
| 4 | 9/14/88 | 23.2 | 0 | 0 | 0 |
| Mixture 2 | | | | | |
| Target Composition | | 21.7 | 3.4 | 3.4 | 4 |
| Measured composition | | | | | |
| 5 | 8/25/88 | 16.7 | 3.0 | 3.6 | 3.7 |
| 6 | 9/16/88 | 20.5 | 3.6 | 3.5 | 4.2 |
| 7 | 8/30/88 | 19.7 | 3.2 | 3.2 | 3.8 |
| 8 | 9/07/88 | 17.1 | 3.1 | 3.0 | 3.6 |
| 9 | 9/09/88 | 16.5 | 2.9 | 2.9 | 3.4 |
| 10 | 9/20/88 | 22.5 | 3.9 | 3.8 | 4.6 |
| Mixture 3 | | | | | |
| Target Composition | | 14.9 | 6.9 | 6.9 | 8 |
| Measured composition | | | | | |
| 3 | 9/26/88 | 15.9 | 7.5 | 6.7 | 8.5 |
| 11 | 9/22/88 | 14.6 | 7.1 | 6.9 | 8.3 |

*Based on measured tetrachloroethylene and chlorobenzene concentrations.

TABLE 18. AQUEOUS SPIKE SOLUTION CHROMIUM CONCENTRATIONS FOR THE CHROMIUM VALENCE STATE TESTS.

| Cr valence state | Compound | Aqueous spike solution concentration (ppm Cr) | | | | | |
|------------------|---|---|----------|------------------|----------|------------------|----------|
| | | Test 1 (9-29-88) | | Test 2 (9-28-88) | | Test 3 (9-26-88) | |
| | | Prepared | Analyzed | Prepared | Analyzed | Prepared | Analyzed |
| Cr(+3) | Cr(NO ₃) ₃ · 9H ₂ O | — | — | 3400 | — | 3400 | — |
| Cr(+6) | CrO ₃ | 3400 | 2700 | — | 0.93 | — | 2.6 |
| Total Cr | | 3400 | 3000 | 3400 | 3100 | 3400 | 3200 |

**TABLE 19. AQUEOUS SPIKE SOLUTION METALS CONCENTRATIONS FOR THE
PARAMETRIC TRACE METALS TEST SERIES**

| Metal | Compound | Prepared spike solution concentration (ppm as metal) | Analyzed spike solution concentration (ppm metal) | | | | | | | |
|-----------|---|---|---|---------------------|---------------------|---------------------|---------------------|---------------------|----------------------|----------------------|
| | | | Test 4 (9/14/88) | Test 5 (8/25/88) | Test 6 (9/16/88) | Test 7 (8/30/88) | Test 8 (9/07/88) | Test 9 (9/09/88) | Test 10 (9/20/88) | Test 11 (9/22/88) |
| Arsenic | As ₂ O ₃ | 3,230 | 2,800 | 2,300 | 2,900 | 2,400 | 2,400 | 2,300 | 3,600 | 3,300 |
| Barium | Ba(NO ₃) ₂ | 2,580 | 2,300 | 1,800 | 2,400 | 2,400 | 2,500 | 2,500 | 2,500 | 2,300 |
| Bismuth | Bi(NO ₃) ₃ · 5H ₂ O | 9,040 | 10,000 | 6,600 | 11,000 | 7,400 | 9,400 | 11,000 | 11,000 | 11,000 |
| Cadmium | Cd(NO ₃) ₂ | 650 | 605 | 410 | 540 | 530 | 520 | 540 | 640 | 540 |
| Chromium | Cr(NO ₃) ₃ · 9H ₂ O | 3,230 | 3,100 | 2,300 | 2,900 | 2,900 | 3,100 | 3,000 | 3,300 | 2,900 |
| Copper | Cu(NO ₃) ₂ · 3H ₂ O | 32,300 | 29,000 | 23,000 | 30,000 | 30,000 | 32,000 | 31,000 | 30,000 | 30,000 |
| Lead | Pb(NO ₃) ₂ | 3,230 | 3,400 | 2,400 | 3,000 | 3,100 | 3,200 | 3,100 | 3,500 | 3,000 |
| Magnesium | Mg(NO ₃) ₂ · 6H ₂ O | 5,040 | 4,600 | 3,400 | 4,500 | 4,300 | 4,400 | 4,600 | 4,900 | 4,300 |
| Strontium | Sr(NO ₃) ₂ | 16,100 | 18,000 | 11,000 | 15,000 | 14,000 | 15,000 | 16,000 | 19,000 | 17,000 |

TABLE 20. CLAY MATRIX METALS CONCENTRATIONS

| Metal | Concentration (mg/kg clay)* | | | | | | | | | | Average |
|-----------|---------------------------------------|---------------------|---------------------|---------------------|---------------------|---------------------|--------------------|--------------------|----------------------|----------------------|---------|
| | Test 1/2 composite (9-28/29-88) | Test 3 (9-26-88) | Test 4 (9-14-88) | Test 5 (8-25-88) | Test 6 (9-16-88) | Test 7 (8-30-88) | Test 8 (9-7-88) | Test 9 (9-9-88) | Test 10 (9-20-88) | Test 11 (9-22-88) | |
| Bismuth | NA ^b | NA | NA | 12 | NA | NA | NA | NA | NA | NA | 12 |
| Chromium | 54 | 52 | 55 | 55 | 54 | 53 | 54 | 55 | 50 | 50 | 53 |
| Lead | NA | NA | 3.2 | 3.7 | 3.5 | 2.9 | 3.1 | 2.3 | 3.0 | 2.5 | 3.0 |
| Magnesium | NA | NA | 23,000 | 23,000 | 22,000 | 22,000 | 22,000 | 22,000 | 20,000 | 22,000 | 22,000 |
| Strontium | NA | NA | 33 | 37 | 36 | 32 | 33 | 30 | 33 | 35 | 34 |

*Clay matrix residue after Parr bomb ashing.

^bNA: not analyzed.

matrix would have a minor effect on the integrated (clay/organic liquid plus aqueous spike solution) feed bismuth concentration. Thus, other clay/organic liquid samples were not analyzed for bismuth. Barium analyses of the feed samples were inadvertently not performed. The previous analysis data (20 ppm Ba) was used to calculate the clay contribution to the integrated feed concentration for barium. A composite clay sample was analyzed for Cr(+6). The result was a Cr(+6) concentration of 2.3 ppm.

Table 21 combines the clay matrix analysis results from Table 20 with the aqueous spike solution data from Table 18 to give the chromium concentration in the integrated feed for the chromium valence state tests (Tests 1 through 3). Table 22 provides a similar summary (from the data in Tables 19 and 20) for the metals concentrations in the integrated feeds for the parametric trace metal tests (Tests 4 through 11). The clay/organic liquid feedrate data in Table 4 combined with the aqueous spike solution feedrate for each test were used to ratio individual component concentrations to give the integrated feed concentrations in Tables 21 and 22.

4.2 CONTINUOUS EMISSION MONITORING DATA

Table 23 summarizes the continuous emission monitor (CEM) data obtained for each of the tests performed. The values in the table were developed from the data recorded by the PC data acquisition system. The data in the table show that throughout the test program CO levels at the afterburner and scrubber exits remained below the detection limit of the monitors (5 ppm). CO spikes of up to 200 ppm accompanied a few of the flameouts experienced. However, as noted above, sampling was suspended during these periods. The CO/CO₂ monitor at the stack operated continuously; however, it performed erratically and had excessive drift. Reliable stack CO and CO₂ data were, therefore, not obtained. The CO/CO₂ monitor at the scrubber exit functioned well, providing reliable data for both CO and CO₂ emissions. Flue gas dilution by inleaking air between the afterburner exit and scrubber exit is evidenced by the reduction in CO₂ concentration. Finally, average NO_x concentrations at the scrubber exit ranged from 35 to 76 ppm.

TABLE 21. INTEGRATED FEED CHROMIUM CONCENTRATIONS FOR THE CHROMIUM VALENCE STATE TESTS

| | Test 1 (9-29-88) | Test 2 (9-28-88) | Test 3 (9-26-88) |
|---------------------------------|-----------------------------|-----------------------------|-----------------------------|
| Total Cr Concentration (ppm) | 79 | 84 | 85 |
| Cr(+6) Concentration (ppm) | 41 | 1.6 | 1.6 |
| Percent of total Cr | 52 | 2 | 2 |

TABLE 22. INTEGRATED FEED METAL CONCENTRATIONS FOR THE PARAMETRIC TRACE METALS TEST SERIES

| Metal | Metal concentration (ppm) | | | | | | | |
|--------------|----------------------------------|-----------------------------|-----------------------------|-----------------------------|----------------------------|----------------------------|------------------------------|------------------------------|
| | Test 4 (9/14/88) | Test 5 (8/25/88) | Test 6 (9/16/88) | Test 7 (8/30/88) | Test 8 (9/7/88) | Test 9 (9/9/88) | Test 10 (9/20/88) | Test 11 (9/22/88) |
| Arsenic | 25 | 23 | 23 | 24 | 19 | 19 | 32 | 33 |
| Barium | 36 | 33 | 33 | 39 | 36 | 36 | 36 | 37 |
| Bismuth | 98 | 75 | 95 | 84 | 85 | 99 | 110 | 120 |
| Cadmium | 5 | 4 | 4 | 5 | 4 | 4 | 6 | 5 |
| Chromium | 68 | 64 | 61 | 68 | 66 | 66 | 66 | 67 |
| Copper | 260 | 230 | 240 | 310 | 260 | 260 | 270 | 310 |
| Lead | 33 | 26 | 26 | 34 | 28 | 28 | 33 | 32 |
| Magnesium | 17,700 | 17,700 | 16,700 | 17,000 | 17,700 | 17,900 | 16,100 | 16,500 |
| Strontium | 190 | 140 | 140 | 170 | 150 | 160 | 200 | 200 |

TABLE 23. CEM DATA

| Parameter | Test 1 (9/29/88) | Test 2 (9/28/88) | Test 3 (9/26/88) | Test 4 (9/14/88) | Test 5 (8/25/88) |
|----------------------------------|---------------------|---------------------|---------------------|---------------------|---------------------|
| Kiln exit | | | | | |
| O ₂ , range, percent | 10.7-11.8 | 10.9-11.5 | 11.5-13.2 | 11.8-12.8 | 11.0-13.6 |
| average, percent | 11.2 | 11.2 | 11.5 | 12.2 | 11.9 |
| Afterburner exit | | | | | |
| O ₂ , range, percent | 6.6-7.9 | 6.4-7.7 | 6.3-7.7 | 6.1-8.0 | 6.0-10.2 |
| average, percent | 7.3 | 7.3 | 7.2 | 7.1 | 8.4 |
| CO ₂ , range, percent | 8.9-10.1 | 9.1-10.2 | 9.0-10.2 | 8.6-10.2 | 6.7-10.0 |
| average, percent | 9.5 | 9.5 | 9.5 | 9.5 | 8.2 |
| CO, ppm | <5 | <5 | <5 | <5 | <5 |
| Scrubber exit | | | | | |
| CO ₂ , range, percent | 5.8-6.7 | 5.8-6.7 | 4.8-6.7 | 4.5-6.9 | 4.5-6.6 |
| average, percent | 6.3 | 6.2 | 6.3 | 6.3 | 5.7 |
| CO, ppm | <5 | <5 | <5 | <5 | <5 |
| NO _x , range, ppm | 33-41 | 32-37 | 33-45 | 33-56 | 40-57 |
| average, ppm | 37 | 35 | 41 | 44 | 49 |
| Stack | | | | | |
| O ₂ , range, percent | 11.5-12.3 | 11.5-12.1 | 11.4-12.6 | 11.4-13.1 | 11.6-13.5 |
| average, percent | 11.9 | 11.8 | 11.9 | 11.7 | 12.1 |

(continued)

TABLE 23. (concluded)

| Parameter | Test 6 (9/16/88) | Test 7 (8/30/88) | Test 8 (9/7/88) | Test 9 (9/9/88) | Test 10 (9/20/88) | Test 11 (9/22/88) |
|----------------------------------|-----------------------|---------------------|--------------------|--------------------|----------------------|----------------------|
| Kiln exit | | | | | | |
| O ₂ , range, percent | 9.0-11.0 ^a | 11.7-13.7 | 12.8-14.5 | 12.1-13.0 | 10.6-12.7 | 10.8-11.7 |
| average, percent | 10.6 | 12.4 | 13.6 | 12.6 | 11.4 | 11.4 |
| Afterburner exit | | | | | | |
| O ₂ , range, percent | 5.7-7.8 | 7.0-8.7 | 3.7-7.5 | 6.3-7.7 | 6.5-8.5 | 6.3-8.1 |
| average, percent | 7.1 | 7.9 | 5.9 ^b | 7.6 | 7.6 | 7.5 |
| CO ₂ , range, percent | 8.7-10.4 | 7.6-9.4 | 8.9-11.8 | 7.7-9.0 | 8.5-10.0 | 8.5-10.0 |
| average, percent | 9.4 | 8.8 | 10.2 | 8.3 | 9.2 | 9.1 |
| CO, ppm | <5 | <5 | <5 | <5 | <5 | <5 |
| Scrubber exit | | | | | | |
| CO ₂ , range, percent | 4.6-5.9 | 4.1-6.4 | 5.1-7.2 | 4.5-6.1 | 5.3-7.1 | 4.8-6.8 |
| average, percent | 5.5 | 5.7 | 6.7 | 5.8 | 6.5 | 6.1 |
| CO, ppm | <5 | <5 | <5 | <5 | <5 | <5 |
| NO _x , range, ppm | 45-63 | 35-58 | 59-89 | 32-48 | 43-64 | 39-56 |
| average, ppm | 56 | 50 | 76 | 42 | 55 | 48 |
| Stack | | | | | | |
| O ₂ , range, percent | 11.8-13.0 | 11.5-14.0 | 10.6-11.9 | 12.3-13.4 | 13.1-14.1 | 11.1-12.8 |
| average, percent | 12.2 | 12.6 | 11.1 | 12.6 | 13.7 | 11.9 |

^aCEM not in service. Data obtained using a Bacharach Fryrite O₂ analyzer.^bAfterburner air feedrate at fan capacity.

As mentioned in Section 1, a secondary test objective was to compare heated and unheated hydrocarbon analyzers. However, hydrocarbon emissions throughout the test program remained below the instrument detection limit of 1 ppm (except for brief periods just after a flameout). Thus, useful information for comparing analyzer performance was not obtained.

4.3 FLUE GAS PARTICULATE AND HCl

Flue gas particulate emissions were measured at the afterburner exit, scrubber exit, and stack. Particulate data are given in Table 24. HCl data are given in Table 25.

As shown in Table 24, stack particulate concentrations ranged from 29 to 58 mg/dscm at 7 percent O₂. All levels were below the hazardous waste incinerator performance standard of 180 mg/dscm at 7 percent O₂. Tests 1, 2, and 3 particulate data indicate the levels were lower at the afterburner exit than at the scrubber exit. Low afterburner values are likely a sampling artifact related to the difficulty in achieving isokinetic sampling at the afterburner exit due to the limited distance between flow disruptions. Samples taken at the afterburner exit that depend on isokinetic sampling must, therefore, be interpreted cautiously. Stack particulate emissions are generally greater than scrubber exit levels, most likely due to particulate entrainment as the gas passes through the carbon bed absorber. The greatest confidence is placed in the scrubber exit particulate concentrations where the requirements for isokinetic sampling are met.

As shown in Table 25, stack HCl levels were nondetectable at detection limits up to 6.6 mg/dscm for Tests 1 to 10. Corresponding HCl emission rates were less than 12.4 g/hr for these tests. Stack HCl levels were measurable at 12.0 mg/dscm (24.1 g/hr) for Test 11, one of the two tests at the highest feed chlorine content. All HCl stack emissions were substantially below the hazardous waste incinerator waste incinerator performance limit of 1.8 kg/hr.

Table 25 also includes information on chlorine feedrates and afterburner and scrubber exit HCl emission rates. For four of the nine tests in which flue gas HCl levels were measured, afterburner exit discharge rates agreed well with chlorine feedrates, as expected. However, for five tests, agreement was poor. The lack of correlation between chlorine feedrate and afterburner exit flue gas HCl emission rate for these five tests may be due to sampling problems associated with the flow distribution at the afterburner exit. The nonzero value measured in the afterburner exit for Test 4 (0 percent chlorine feed) may be due to hysteresis emissions of chlorides deposited during earlier tests. In any event, the afterburner exit HCl level for Test 4 is significantly lower than the values measured for the other tests.

It is interesting to note that the scrubber exit HCl levels for the two tests at the highest feed chlorine concentration (Tests 3 and 11) were above detection limits and comparable. Scrubber exit HCl levels for all but one of the other tests at lower feed chlorine concentrations were nondetectable.

4.4 TRACE METAL DISCHARGE DATA

This section discusses the distributions of the trace metals fed to the RKS in each test among the discharge streams sampled and analyzed. Distributions discussed are based on the mass flowrate of a given element in a specified discharge. This mass flowrate is calculated from the analyzed concentration of each metal in a given stream (e.g., kiln ash, flue gas particulate) and the total mass flowrate for the given stream. Appendix B includes all the laboratory analysis reports which serve as the basis for the given stream concentrations. Appendix C contains the flue gas sampling train data which includes flue gas stream flowrate information.

TABLE 24. FLUE GAS PARTICULATE DATA

| Test | Test date | Particulate concentration (mg/dscm at 7 percent O ₂) | | |
|------|-----------|---|---------------|-------|
| | | Afterburner exit | Scrubber exit | Stack |
| 1 | 9/29/88 | 21 | 31 | 58 |
| 2 | 9/28/88 | 23 | 32 | 42 |
| 3 | 9/26/88 | 36 | 37 | 39 |
| 4 | 9/14/88 | 273 | 7 | 44 |
| 5 | 8/25/88 | 360 | 7 | 31 |
| 6 | 9/16/88 | 162 | 31 | 27 |
| 7 | 8/30/88 | 146 | 23 | 29 |
| 8 | 9/07/88 | 50 | 40 | 40 |
| 9 | 9/09/88 | 152 | 12 | 33 |
| 10 | 9/20/88 | 414 | 21 | 47 |
| 11 | 9/22/88 | 240 | 51 | 38 |

TABLE 25. HCl EMISSIONS DATA

| Test | Test date | Cl feedrate (kg/hr) | HCl concentration and emission rate | | | | | |
|------|-----------|------------------------|-------------------------------------|---------|---------------|--------|-----------|--------|
| | | | Afterburner exit | | Scrubber exit | | Stack | |
| | | | (g/dscm) | (kg/hr) | (mg/dscm) | (g/hr) | (mg/dscm) | (g/hr) |
| 1 | 9/29/88 | 0 | | | | | <5.8 | <11.9 |
| 2 | 9/28/88 | 0 | | | | | <4.7 | <10.5 |
| 3 | 9/26/88 | 5.23 | 10.07 | 11.42 | 7.6 | 17.8 | <5.6 | <12.4 |
| 4 | 9/14/88 | 0 | 0.13 | 0.12 | <3.9 | <7.1 | <6.6 | <8.9 |
| 5 | 8/25/88 | 2.35 | 8.93 | 8.30 | <3.4 | <6.4 | <5.7 | <8.0 |
| 6 | 9/16/88 | 2.64 | 2.37 | 2.18 | <3.9 | <7.9 | <5.8 | <12.2 |
| 7 | 8/30/88 | 2.40 | 2.51 | 2.35 | <4.2 | <8.4 | <5.8 | <8.9 |
| 8 | 9/07/88 | 2.28 | 0.54 | 0.49 | 7.6 | 17.7 | <6.6 | <9.8 |
| 9 | 9/09/88 | 2.12 | 1.16 | 1.11 | <4.1 | <7.7 | <6.4 | <9.1 |
| 10 | 9/20/88 | 2.86 | 2.72 | 2.52 | <2.3 | <5.1 | <5.8 | <11.8 |
| 11 | 9/22/88 | 5.24 | 2.91 | 2.64 | 7.5 | 15.5 | 12.0 | 24.1 |

Table 26 summarizes the synthetic waste feedrate and kiln ash discharge flowrate data. It is interesting to note in Table 26 that the fraction of the clay matrix mass fed to the kiln accounted for by the kiln ash discharge was relatively constant at about 80 percent (range of 73 to 83 percent) for all tests. The remaining 20 percent of the clay fed represents ash entrained in the kiln exit flue gas and carried out of the kiln into the afterburner in addition to the moisture content of the clay.

The mass flowrate of a given metal removed by the venturi/packed column scrubber deserves some discussion. Prior to each test, the RKS was operated for a minimum of 18 hours fired on propane alone. Following this period, synthetic waste feed was started. Flue gas sampling was begun only after at least half an hour of waste feeding had elapsed. At the conclusion of flue gas sampling, synthetic waste feeding was stopped and the RKS was returned to propane firing. Kiln rotation continued until all ash was discharged from the kiln into the ash collection pit.

During propane firing, scrubber blowdown continued with fresh water scrubber makeup introduced to keep the scrubber liquor loop "full". As a consequence, the scrubber loop was "purged" of a previous test's metal buildup during propane-only firing such that, at the beginning of a subsequent test, the scrubber liquor was clean; it contained very little to no test trace metal content. Thus, over the time span of a given test, the recirculating scrubber liquor metal concentrations increased from near zero to some nonzero value. Further, at the end of a test, a significant portion of the metal fed to the RKS during the test which was removed by the scrubber remained in the 760 L (200 gal) scrubber liquor loop.

To account for the nonsteady scrubber loop operation over a test resulting from the above mode of operation, the scrubber blowdown sampling protocol specifically called for obtaining a scrubber blowdown sample at the beginning of a test, hourly thereafter, and at the end of a test. This allowed the buildup of metal concentrations in the scrubber liquor to be determined.

The mass flowrate of metal removed by the scrubber was then calculated as the sum of two terms. The first was that removed in the scrubber blowdown. This term was calculated from the blowdown rate noted in Table 6 and the average blowdown concentration measured from the several blowdown samples analyzed. The second term was that remaining in the 760 L (200 gal) scrubber recirculation loop at the end of a test. This term was based on the metal concentration in the last blowdown sample taken.

The following subsections summarize trace metal distributions among discharge streams for the chromium valence state and parametric trace metal tests, respectively.

4.4.1 Chromium Valence State Tests

As noted in Section 1, the objectives of this particular test series were to identify the relative distributions of chromium in the RKS discharges and to determine the fraction discharged as hexavalent chromium ($\text{Cr}(+6)$). The parameters varied were feed chromium valence state and feed chlorine content.

Table 27 summarizes the total chromium discharge distributions expressed as a fraction of the amount of chromium present in the feed. Almost half of the feed chromium was present as background in the feed clay absorbent. Overall mass balance closure around the system for Tests 1 and 2 determined at the two locations where the flue gas was sampled was

TABLE 26. SYNTHETIC WASTE FEEDRATES AND KILN ASH DISCHARGE RATES

| Test | Test data | Synthetic waste feedrate, kg/hr (lb/hr) | | Kiln Ash | |
|------|-----------|--|------------------|----------------------------------|----------------------------------|
| | | Clay/organic liquid | Clay fraction | Discharge rate, kg/hr (lb/hr) | Fraction of clay feedrate (%) |
| 1 | 9-29-88 | 62.6 (138.0) | 45.1 (99.5) | 37.4 (82.6) | 83 |
| 2 | 9-28-88 | 62.0 (136.7) | 44.7 (98.5) | 34.9 (76.8) | 78 |
| 3 | 9-26-88 | 61.3 (135.1) | 42.9 (94.5) | 31.3 (69.0) | 73 |
| 4 | 9-14-88 | 59.4 (131.0) | 45.6 (100.6) | 34.7 (76.5) | 76 |
| 5 | 8-25-88 | 63.5 (140.0) | 48.5 (107.0) | 37.8 (83.5) | 78 |
| 6 | 9-16-88 | 62.6 (138.0) | 45.4 (100.0) | 33.6 (74.0) | 74 |
| 7 | 8-30-88 | 63.4 (139.8) | 46.7 (103.0) | 37.8 (83.4) | 81 |
| 8 | 9-7-88 | 63.4 (139.8) | 48.5 (106.9) | 38.3 (84.5) | 79 |
| 9 | 9-9-88 | 62.5 (137.8) | 48.5 (106.9) | 39.8 (87.7) | 82 |
| 10 | 9-20-88 | 62.8 (138.4) | 43.9 (96.7) | 34.7 (76.4) | 79 |
| 11 | 9-22-88 | 63.5 (140.0) | 45.4 (100.0) | 34.5 (76.0) | 76 |

TABLE 27. TOTAL CHROMIUM DISCHARGE DISTRIBUTIONS AND MASS BALANCE FOR THE CHROMIUM VALENCE STATE TEST SERIES

| | Total Cr fraction (percent of feed) | | |
|-----------------------------|-------------------------------------|---------------------------------|--|
| | Test 1 Cr(+6) feed, no Cl | Test 2 Cr(+3) feed, no Cl | Test 3 Cr(+3) feed, 8.5% feed Cl |
| Feed: Clay mixture | 48.4 | 45.4 | 43.8 |
| Aqueous spike | <u>51.6</u> | <u>54.6</u> | <u>56.2</u> |
| Total | 100.0 | 100.0 | 100.0 |
| Kiln ash | 74.8 | 73.6 | 46.7 |
| Afterburner exit flue gas | <u>0.4</u> | <u>0.5</u> | <u>0.8</u> |
| Around kiln and afterburner | 75.2 | 74.1 | 47.5 |
| Kiln ash | 74.8 | 73.6 | 46.7 |
| Scrubber exit flue gas | 1.1 | 1.3 | 2.3 |
| Scrubber liquor | <u>2.5</u> | <u>2.3</u> | <u>6.4</u> |
| Around kiln and scrubber | 78.4 | 77.2 | 55.4 |

approximately 75 percent. Closure for Test 3 was poorer, 47 percent around the system with an afterburner flue gas discharge and approximately 55 percent around the system with the scrubber discharges.

Most of the chromium fed to the RKS is accounted for by the kiln ash discharge. A relatively constant, and very small, amount on percentage of feed basis was measured in the afterburner exit discharge. However, as noted in several instances in the preceding discussion, the afterburner exit flue gas sampling location is very near a major flow disturbance. Thus, it is very difficult to obtain a representative flue gas sample at this location. The lower total chromium fractions measured in the tests are probably influenced by the poor sampling characteristics of the available sampling location.

The fractions of total chromium feed accounted for in the scrubber exit flue gas and in the scrubber liquor were comparable for Tests 1 and 2 in which the feed contained no chlorine. However, these fractions were increased for Test 3 with chlorine-containing feed. This increase occurs in absolute terms despite a decrease in the overall mass balance around the kiln and scrubber for this test. This is consistent with expectations if the presence of chlorine gives rise to more volatile chromium species and more water soluble chromium species.

Table 28 summarizes the normalized total chromium discharge distributions around the RKS closed around the afterburner exit and closed around the scrubber discharges. Two discharge streams are considered with closure around the afterburner exit: the kiln ash discharge and the afterburner exit flue gas. Three discharges are considered with closure around the scrubber discharges: the kiln ash discharge, the scrubber liquor, and the scrubber exit flue gas. The distributions have been normalized with the total amount of chromium measured in the sum of the discharge streams considered for each closure case. This has the effect of normalizing discharge distributions to what they would have been if mass balance closure were 100 percent for each closure case.

The data in Table 28 emphasize the point noted above that the kiln ash discharge accounts for most of the chromium fed. In addition, as also noted above, the fraction of total chromium in the scrubber exit flue gas and the scrubber liquor were comparable for Tests 1 and 2 in which the feed contained no chlorine. No change with chromium feed valence state is seen. However, these fractions increase for Test 3 with chlorine-containing feed. As noted above, this is consistent with expectation if the presence of chlorine in the incinerator gives rise to more volatile chromium species.

Interestingly, the apparent scrubber chromium collection efficiency (scrubber liquor fraction/(scrubber liquor fraction + scrubber exit flue gas fraction)) is relatively constant at 63 to 73 percent.

Table 29 shows the distribution of total chromium between the particulate phase and the vapor/dissolved phase in the flue gas at the two locations sampled. The particulate phase fraction represents that accounted for in the probe wash and filter catch of the sampling train. The vapor/dissolved phase fraction represents that accounted for in the impingers of the sampling train. Presumably, any chromium present in the vapor phase at the sampling train probe exit would be captured and accounted for in the impingers. In addition, much of the chromium present as water soluble salts would also be collected in the impinger. The flue gas at both sampling locations contains significant water vapor. Thus, water soluble salts can be "washed" through the glass fiber filter in the sampling train and be captured in the impingers.

**TABLE 28. TOTAL CHROMIUM DISCHARGE DISTRIBUTIONS FOR THE CHROMIUM VALENCE
VALENCE STATE TEST SERIES**

| | Total Cr fraction (percent of measured) | | |
|--|---|------------------------------|-------------------------------------|
| | Test 1 Cr(+6) feed, no Cl | Test 2 Cr(+3) feed, no Cl | Test 3 Cr(+3) feed, 8.5% feed Cl |
| Around kiln and afterburner | | | |
| Kiln ash | 99.6 | 99.3 | 98.4 |
| Afterburner exit flue gas | <u>0.4</u> | <u>0.7</u> | <u>1.6</u> |
| Total | 100.0 | 100.0 | 100.0 |
| Around kiln and scrubber | | | |
| Kiln ash | 95.5 | 95.2 | 84.4 |
| Scrubber exit flue gas | 1.3 | 1.8 | 4.2 |
| Scrubber liquor | <u>3.2</u> | <u>3.0</u> | <u>11.4</u> |
| Total | 100.0 | 100.0 | 100.0 |
| Apparent scrubber Cr removal efficiency | 71 | 63 | 73 |

**TABLE 29. TOTAL CHROMIUM PARTICULATE/VAPOR-DISSOLVED PHASE FLUE GAS DISTRIBUTIONS
FOR THE CHROMIUM VALENCE STATE TEST SERIES**

| | Percent of flue gas total Cr | | |
|---------------------------|------------------------------|------------------------------|-------------------------------------|
| | Test 1 Cr(+6) feed, no Cl | Test 2 Cr(+3) feed, no Cl | Test 3 Cr(+3) feed, 8.5% feed Cl |
| Afterburner exit flue gas | | | |
| Particulate | 33 | 68 | 56 |
| Vapor/dissolved phase | <u>67</u> | <u>32</u> | <u>44</u> |
| Total | 100 | 100 | 100 |
| Scrubber exit flue gas | | | |
| Particulate | 57 | 49 | 79 |
| Vapor/dissolved phase | <u>43</u> | <u>51</u> | <u>21</u> |
| Total | 100 | 100 | 100 |

The afterburner exit data in Table 29 show that the particulate phase total chromium was highest for Test 2, the test with Cr(+3) feed and no feed chlorine; intermediate for Test 3, with the Cr(+3) feed and chlorine in the feed; and lowest for Test 1, with Cr(+6) in the feed and no feed chlorine. This would be as expected if a significant fraction of the chromium in the afterburner exit flue gas was present as soluble Cr(+6) species (e.g., CrO_4^{2-} , $\text{Cr}_2\text{O}_7^{2-}$) for the Cr(+6) feed case or as soluble chlorides in the feed chlorine case, as opposed to insoluble Cr_2O_3 in the no chlorine/Cr(+3) feed case. However, given the difficulty in obtaining a representative flue gas sample at the afterburner exit, this result should be treated with caution.

If the soluble species are more effectively removed in the scrubber, one would expect the fraction accounted for in the flue gas vapor/dissolved phase to decrease in the scrubber exit flue gas for Tests 1 and 3. This is indeed the case.

Table 30 summarizes the fractions of the total chromium analyzed as Cr(+6) in feed and discharge streams. As shown, the feed was analyzed as 52 percent Cr(+6) for Test 1, and as 2 percent Cr(+6) for Tests 2 and 3. For all tests, the kiln ash chromium was comprised of negligible amounts of Cr(+6).

The scrubber exit flue gas Cr(+6) fraction was the same (12 to 16 percent) regardless of whether or not Cr(+6) was present in the feed, in the two cases with no feed chlorine. In contrast, for the case in which the feed contained chlorine, roughly half the scrubber exit flue gas chromium was Cr(+6). This would be as expected if entrained particulate chromium from the kiln vaporized in the hotter afterburner and reacted with the flue gas chlorine to form chromyl chloride (CrO_2Cl_2), a relatively stable compound with chromium as Cr(+6).

The scrubber liquor Cr(+6) fraction for Test 1 with Cr(+6) in the feed was significantly higher than for the other two tests with only Cr(+3) in the feed. This is as expected if some of the chromium in the scrubber inlet flue gas were present as soluble Cr(+6) species (CrO_4^{2-} and $\text{Cr}_2\text{O}_7^{2-}$ as noted above).

4.4.2 Parametric Trace Metals Tests

Experimental and laboratory data were reduced and analyzed to address the experimental objectives discussed in Section 1. Data obtained during this testing program provided information on the following:

- Distribution of metals among the kiln ash, flue gas, and scrubber liquor streams
- Afterburner exit and scrubber exit flue gas metal distributions between solid and vapor/dissolved phase
- Afterburner exit flue gas metal particulate distributions by size
- Apparent scrubber collection efficiency for each metal
- Effects of the primary test variables of kiln exit temperature, afterburner exit temperature, and feed chlorine content on metal distributions

The test results listed above are summarized and reviewed in the following subsections. To facilitate the discussion, results from all eight parametric tests are first summarized and discussed independently of the RKS operating conditions, in Section 4.4.2.1. The intent is to provide a concise summary of the relative distributions of each metal during the test program.

TABLE 30. HEXAVALENT CHROMIUM FRACTIONS FOR THE CHROMIUM VALENCE STATE TEST SERIES

| | Cr(+6)/total Cr (%) | | |
|---------------------------|--------------------------------------|--------------------------------------|---|
| | Test 1 Cr(+6) feed, no Cl | Test 2 Cr(+3) feed, no Cl | Test 3 Cr(+3) feed, 8.5% feed Cl |
| Composite feed | 52 | 2 | 2 |
| Kiln ash | 0.3 | 0.2 | 0.1 |
| Afterburner exit flue gas | 102 | 35 | 4 |
| Scrubber exit flue gas | 12 | 16 | 48 |
| Scrubber liquor | 57 | 21 | 28 |

The relationships between the discharge distributions of the metals and the RKS operating conditions are then discussed in Section 4.4.2.2. The tables discussed in Section 4.4.2.1, therefore, contain only ranges and averages of the test results. The individual values obtained for each test are contained in the tables and figures discussed in Section 4.4.2.2.

For many samples, laboratory analysis results showed that several metals were not detected at a certain detection limit. In the data evaluation, two calculations were performed: one with the analysis result assumed to be zero, and one with the analysis result assumed to be the detection limit. In many cases, distribution conclusions were not affected. In those cases where distribution conclusions were affected, however, distributions are reported as ranges.

4.4.2.1 Average Trace Metal Discharge Distributions

As previously noted, a major objective of this test series was to identify the relative distribution of each metal among the several RKS discharge streams. Table 31 summarizes the relative distributions between the kiln ash and afterburner exit flue gas. Table 32 is a similar summary among the kiln ash, scrubber exit flue gas, and scrubber liquor. In both tables the first set of columns represents the fraction of the metal feed accounted for by the noted discharge (e.g., kiln ash, afterburner exit flue gas). The range (low, high) exhibited over the 8 tests performed and the average for all 8 tests are noted. The second set of columns represents fractions normalized to the total amount measured in the discharge streams analyzed. These normalized values represent fractions which would have resulted had mass balance closure been 100 percent. Note that the sum of the normalized values for each element for the discharge streams in each table is indeed 100.

Since the first set of columns are percent of metal feed, the kiln ash fractions are the same in both tables. However, normalized kiln ash values change between the two tables because mass balance closures experienced around the kiln ash/afterburner exit flue gas discharge differed from those experienced around the kiln ash/scrubber system discharges.

The metals are ordered in the tables by increasing average normalized kiln ash fraction, an ordering equivalent to decreasing experimentally observed average volatility. Those metals with low average normalized kiln ash fractions were more volatile (found in kiln ash at lower percentages) than metals with higher average normalized kiln ash fractions.

Cadmium was not detected in any kiln ash sample. The kiln ash fractions noted correspond to the kiln ash detection limits. The normalized cadmium fractions for the other discharge streams in the tables assume cadmium was present in kiln ash at the detection limit.

Chromium is not included in Table 31. The afterburner exit flue gas sampling train contained a stainless steel condenser between the alundum thimble filter, employed to collect the large quantities of particulate needed for size fractionation, and the impinger train. As a consequence, all afterburner exit sampling train impinger solutions contained significant amounts of chromium.

The data in Tables 31 and 32 show that there are two distinct groupings of metals. Cadmium, lead, and bismuth (Table 32) tend to be relatively volatile and are not found to a major degree in the kiln ash. Barium, copper, strontium, arsenic, chromium, and magnesium were found to be more refractory; kiln ash accounted for the major fraction of these metals.

The last column in Tables 31 and 32 contains a value termed volatility temperature (8). This is the temperature at which the vapor pressure of the most volatile principal species of each

TABLE 31. SUMMARY OF METAL DISCHARGE DISTRIBUTIONS IN THE KILN ASH AND AFTERBURNER EXIT FLUE GAS FOR THE TRACE METALS PARAMETRIC TEST SERIES

| Metal | Distribution (% of metal fed) | | | Normalized distribution (% of total measured) | | | Volatility temperature*, °C (°F) |
|---------------------------|----------------------------------|-------|---------|--|-------|---------|--|
| | Kiln ash | | | | | | |
| | Low | High | Average | Low | High | Average | |
| Cadmium | <9.7 | <17.1 | <12.7 | <13.7 | <43.6 | <28.9 | 216(420) |
| Lead | 4.7 | 12.6 | 8.3 | 9.5 | 61.7 | 35.5 | 627(1160) |
| Bismuth | 8.4 | 24.4 | 16.2 | 53.4 | 89.6 | 70.0 | 621(1150) |
| Copper | 34.8 | 64.2 | 49.0 | 63.6 | 94.1 | 84.8 | 1116(2040) |
| Barium | 39.2 | 127.7 | 67.0 | 83.3 | 96.7 | 88.6 | 849(1560) |
| Arsenic | 36.3 | 72.0 | 54.8 | 88.1 | 97.4 | 94.6 | 32(90) |
| Strontium | 25.5 | 64.5 | 43.2 | 99.5 | 99.8 | 99.7 | 1454(2650) |
| Magnesium | 69.2 | 133.1 | 91.7 | 99.7 | 99.9 | 99.8 | 1549(2820) |
| Afterburner exit flue gas | | | | | | | |
| Metal | Low | High | Average | Low | High | Average | |
| Cadmium | 14.2 | 70.9 | 29.6 | 56.4 | 86.3 | 71.1 | |
| Lead | 4.7 | 12.6 | 8.3 | 38.3 | 90.5 | 64.5 | |
| Bismuth | 2.5 | 19.3 | 6.2 | 10.4 | 46.6 | 30.0 | |
| Copper | 2.8 | 19.5 | 6.6 | 5.9 | 36.4 | 15.2 | |
| Barium | 1.4 | 15.4 | 7.3 | 3.3 | 16.7 | 11.4 | |
| Arsenic | 1.6 | 5.5 | 2.1 | 2.6 | 11.9 | 5.4 | |
| Strontium | 0.07 | 0.3 | 0.2 | 0.2 | 0.5 | 0.3 | |
| Magnesium | 0.1 | 0.3 | 0.2 | 0.1 | 0.3 | 0.2 | |

*Temperature at which the vapor pressure of a principal vapor species of the metal under oxidizing conditions is 10^{-6} atm (8).

TABLE 32. SUMMARY OF METAL DISCHARGE DISTRIBUTIONS IN THE KILN ASH SCRUBBER EXIT FLUE GAS, AND SCRUBBER LIQUOR FOR THE TRACE METALS PARAMETRIC TEST SERIES

| Distribution (% of metal fed) | | | Normalized distribution (% of total measured) | | | Volility temperature*, °C(°F) | |
|----------------------------------|------|-------|--|------|-------|-------------------------------------|------------|
| Kiln ash | | | | | | | |
| Metal | Low | High | Average | Low | High | Average | |
| Cadmium | <9.7 | <17.1 | <12.7 | <9.3 | <29.3 | <14.5 | 216(420) |
| Lead | 4.7 | 12.6 | 8.3 | 5.8 | 83.7 | 20.1 | 627(1160) |
| Bismuth | 8.4 | 24.4 | 16.2 | 20.9 | 64.8 | 31.6 | 621(1150) |
| Barium | 39.2 | 127.7 | 67.0 | 68.8 | 86.9 | 76.6 | 849(1560) |
| Copper | 34.0 | 64.2 | 49.0 | 58.0 | 97.6 | 78.6 | 1116(2040) |
| Strontium | 25.5 | 64.5 | 43.2 | 81.9 | 94.3 | 89.3 | 1454(2650) |
| Arsenic | 36.3 | 72.0 | 54.8 | 84.0 | 94.4 | 91.0 | 32(90) |
| Chromium | 54.2 | 88.6 | 68.4 | 85.9 | 95.7 | 92.8 | 1613(2935) |
| Magnesium | 69.2 | 133.1 | 91.7 | 99.2 | 99.6 | 99.4 | 1549(2820) |
| Scrubber exit flue gas | | | | | | | |
| Metal | Low | High | Average | Low | High | Average | |
| Cadmium | 15.7 | 74.4 | 53.2 | 41.9 | 67.5 | 53.5 | |
| Lead | 0.9 | 70.6 | 39.7 | 11.6 | 73.6 | 51.0 | |
| Bismuth | 6.0 | 30.4 | 20.6 | 15.7 | 50.7 | 38.4 | |
| Barium | 1.3 | 4.3 | 2.2 | 1.6 | 5.5 | 2.7 | |
| Copper | 0.3 | 19.5 | 9.9 | 0.8 | 33.2 | 15.6 | |
| Strontium | 0.5 | 1.6 | 1.0 | 1.1 | 3.7 | 2.1 | |
| Arsenic | 1.4 | 4.0 | 2.6 | 2.2 | 8.4 | 4.5 | |
| Chromium | 0.7 | 2.7 | 1.6 | 1.1 | 4.2 | 2.3 | |
| Magnesium | 0.03 | 0.3 | 0.1 | 0.03 | 0.2 | 0.1 | |

(continued)

*Temperature at which the vapor pressure of a principal vapor species of the metal under oxidizing conditions is 10^{-6} atm (8).

TABLE 32. (concluded)

| Metal | Distribution (% of metal fed) | | | Normalized distribution (% of total measured) | | | Volility temperature*, °C(°F) |
|-----------|----------------------------------|------|---------|--|------|---------|-------------------------------------|
| | Scrubber liquor | | | | | | |
| | Low | High | Average | Low | High | Average | |
| Cadmium | 10.8 | 41.1 | 30.4 | 23.2 | 44.9 | 32.0 | 216(420) |
| Bismuth | 7.4 | 27.1 | 16.2 | 19.5 | 36.7 | 30.0 | 621(1150) |
| Lead | 0.4 | 37.2 | 22.3 | 4.7 | 41.1 | 28.9 | 627(1160) |
| Barium | 11.2 | 31.7 | 17.0 | 11.5 | 28.8 | 20.7 | 849(1560) |
| Copper | 0.7 | 6.4 | 4.1 | 1.6 | 9.1 | 5.8 | 1116(2040) |
| Strontium | 1.5 | 6.3 | 3.9 | 4.4 | 16.0 | 8.6 | 1454(2650) |
| Arsenic | 1.4 | 3.8 | 2.5 | 2.6 | 8.2 | 4.5 | 32(90) |
| Chromium | 1.9 | 7.7 | 3.4 | 2.3 | 12.2 | 4.9 | 1613(2935) |
| Magnesium | 0.3 | 0.6 | 0.5 | 0.3 | 0.7 | 0.5 | 1549(2820) |

*Temperature at which the vapor pressure of a principal vapor species of the metal under oxidizing conditions is 10^{-6} atm (8).

metal under oxidizing conditions is 10^{-6} atm. The lower a metal's volatility temperature is, the more volatile it is expected to be. The data in Tables 31 and 32 show a remarkable correspondence between the observed volatility and volatility temperature for all the metals tested except arsenic. That is, except for arsenic, average normalized kiln ash fraction increases more or less directly with increasing volatility temperature.

Based on volatility temperature, arsenic was expected to have been the most volatile element, and the one found at lowest percentage in the kiln ash. However, the data clearly show arsenic to be apparently refractory, remaining largely with the kiln ash. The volatility temperature for arsenic is based on the vapor pressure of As_2O_3 , the most volatile arsenic species under oxidizing combustion conditions. The fact that arsenic is significantly less volatile than would be expected if As_2O_3 were the predominant arsenic species suggests that either some other, more refractory arsenic compound is preferred, or that some other chemical interaction, for example strong adsorption to the clay, occurred.

Table 33 summarizes the range and average of the percentage mass balance closures achieved for all the tests performed. Again, achieved closures around the kiln ash discharge and the afterburner exit flue gas, and around the kiln ash and scrubber discharges (flue gas and scrubber liquor), are tabulated. The data in Table 33 show that average mass balance closure ranged from 25 percent for bismuth to 88 percent for magnesium around the kiln ash/afterburner exit flue gas, with an overall average of 55 percent. Individual metal closures ranged from 11 to 140 percent.

TABLE 33. SUMMARY OF ACHIEVED METAL MASS BALANCE CLOSURE FOR THE TRACE METALS PARAMETRIC TEST SERIES

| Metal | Mass balance closure ^a (% of metal fed) | | | | | |
|-----------|--|------|---------|--|------|---------|
| | Closed around kiln ash discharge and afterburner exit flue gas | | | Closed around kiln ash discharge and scrubber discharges | | |
| | Low | High | Average | Low | High | Average |
| Arsenic | 42 | 74 | 58 | 39 | 77 | 60 |
| Barium | 41 | 140 | 83 | 57 | 147 | 86 |
| Bismuth | 11 | 44 | 25 | 36 | 74 | 53 |
| Cadmium | 25 | 84 | 52 | 37 | 120 | 96 |
| Chromium | — | — | — | 61 | 94 | 73 |
| Copper | 47 | 76 | 57 | 46 | 79 | 63 |
| Lead | 12 | 61 | 31 | 8 | 96 | 70 |
| Magnesium | 69 | 98 | 88 | 70 | 134 | 92 |
| Strontium | 26 | 65 | 47 | 28 | 71 | 48 |

^a(Sum of total metal discharge rate over all discharges)/(metal feedrate).

Achieved mass balance closures were better around the kiln ash/scrubber discharge, with average metal closures ranging from 48 to 96 percent. Overall average closure was 71 percent. Individual metal closures ranged from 8 to 147 percent. Previous discussion stated that the afterburner exit flue gas sampling location is a poor one and likely does not allow a representative flue gas sample to be collected. This is the most likely explanation for the lesser closures achieved around the kiln ash/afterburner exit flue gas.

Almost all achieved mass balance closures were less than 100 percent. That is, the total amount of metal which could be accounted for in the sum of discharge streams was almost always less than the amount fed. The most likely explanation for this is that some quantity of the metal fed was deposited in slag buildup in the afterburner chamber of the RKS. A gradual accumulation of slag in the afterburner was observed over the duration of the test program. This slag was no doubt a source of metal loss.

Despite the above, the mass balance closures achieved (averaging about 65 percent) are considered good. Typical trace metal mass balance closure results from past experience for combustion sources are in the 30 to 200 percent range.

Table 34 summarizes the ranges and averages of the relative distribution of each of the metals between particulate and vapor/dissolved phases in the afterburner and scrubber exit flue gas. Again, the particulate phase fraction is defined as that analyzed in the probe wash and filter catches. The vapor/dissolved phase fraction is defined as that analyzed in the sampling train impingers. The values in Table 34 assume a metal is present in a sample at the detection limit for sample concentrations reported as less than the detection limit.

The data in Table 34 show that barium was found largely in the vapor/dissolved phase in both the afterburner exit and the scrubber exit flue gas. This suggests that the specific barium species predominant in the flue gas is quite water soluble. Bismuth and strontium were found predominantly in the vapor/dissolved phase in the afterburner exit flue gas, but were found predominantly in the particulate phase in the scrubber exit flue gas. All other metals, on average, were found predominantly in the particulate phase at both locations, although individual test conditions had opposite distributions for a few of these other metals.

Table 35 summarizes the observed range in, and average of, apparent scrubber efficiencies in removing each metal from the flue gas stream. As defined in Section 4.4.1, the apparent scrubber efficiency represents the ratio of the normalized metal fraction measured in the scrubber liquor to the sum of the normalized metal fractions measured in the scrubber liquor and scrubber exit flue gas.

The data in Table 35 show that average removal efficiencies ranged from 31 to 88 percent; the overall average for all metals was 57 percent. The metals are listed in the table in order of increasing apparent scrubber efficiency. With two exceptions, the order is generally the same as the observed metal volatility order in Tables 31 and 32. The volatile metals (those found to a lesser extent in the kiln ash, such as lead, cadmium, and bismuth) have lower average apparent scrubber collection efficiencies than do the less volatile metals (those found in the kiln ash to a greater extent, such as arsenic, chromium, strontium, and magnesium).

This is as expected. The more volatile metals vaporize in the incinerator. When the flue gas is cooled or quenched, these metals subsequently condense into either condensation nuclei, resulting in very fine fume, or onto available existing particulate. The result of this volatilization/condensation occurrence is that volatile metals become enriched in fine particulate.

TABLE 34. SUMMARY OF FLUE GAS METAL PARTICULATE/VAPOR-DISSOLVED PHASE DISTRIBUTIONS FOR THE TRACE METALS PARAMETRIC TEST SERIES

| | Afterburner exit flue gas | | | | | | Scrubber exit flue gas | | | | | |
|-----------|--|------|---------|--------------------------------------|------|---------|--|------|---------|--------------------------------------|------|---------|
| | Vapor/dissolved phase fraction (percent) | | | Particulate phase fraction (percent) | | | Vapor/dissolved phase fraction (percent) | | | Particulate phase fraction (percent) | | |
| | | | | | | | | | | | | |
| | Low | High | Average | Low | High | Average | Low | High | Average | Low | High | Average |
| Arsenic | 3 | 53 | 19 | 47 | 97 | 81 | 19 | 68 | 32 | 32 | 81 | 68 |
| Barium | 90 | 99 | 97 | 1 | 10 | 3 | 45 | 90 | 70 | 10 | 55 | 30 |
| Bismuth | 63 | 91 | 77 | 9 | 37 | 23 | 7 | 30 | 18 | 70 | 93 | 82 |
| Cadmium | 8 | 43 | 24 | 57 | 92 | 76 | 5 | 18 | 10 | 82 | 95 | 90 |
| Chromium | -- | -- | -- | -- | -- | -- | 13 | 33 | 21 | 67 | 87 | 79 |
| Copper | 20 | 71 | 38 | 29 | 80 | 62 | 1 | 55 | 19 | 45 | 99 | 81 |
| Lead | 24 | 84 | 47 | 26 | 76 | 53 | 0.1 | 10 | 4 | 90 | 99.9 | 96 |
| Magnesium | 9 | 43 | 25 | 57 | 91 | 75 | 7 | 75 | 32 | 25 | 93 | 68 |
| Strontium | 42 | 75 | 62 | 25 | 58 | 38 | 9 | 68 | 29 | 32 | 99 | 71 |

**TABLE 35. SUMMARY OF APPARENT SCRUBBER EFFICIENCY
RANGES AND AVERAGES FOR THE TRACE
METALS PARAMETRIC TEST SERIES**

| Metal | Apparent scrubber removal efficiency (%) | | |
|-----------|---|------|---------|
| | Low | High | Average |
| Copper | 19 | 67 | 31 |
| Lead | 22 | 48 | 36 |
| Cadmium | 26 | 52 | 38 |
| Bismuth | 34 | 55 | 45 |
| Arsenic | 36 | 64 | 49 |
| Chromium | 44 | 87 | 65 |
| Strontium | 65 | 93 | 78 |
| Magnesium | 63 | 92 | 84 |
| Barium | 82 | 95 | 88 |

That is, their concentration on a mass percent basis ($\mu\text{g/g}$ of particulate) is higher in fine particulate than in coarse particulate. This is so because:

- Metal condensing into condensation nuclei form fume particles which are very fine
- Metal condensing onto available existing particulate are mass enriched in the available fine particulate, since condensation rates per unit particulate surface area are constant, and since the available surface area to mass is higher for fine particulate

Since the more volatile metals are expected to become enriched in fine particulate, the observation that the apparent scrubber efficiencies for the more volatile metals are lower is understandable, given that a venturi scrubber is significantly more efficient at collecting coarse particulate than fine particulate.

The two exceptions to the volatility/apparent scrubber efficiency relationship noted in Table 35 were copper and barium. The average apparent collection efficiency for barium is higher than might have been expected based on its relative volatility. However, it was noted above that barium was found predominantly in the vapor/dissolved phase in both the afterburner exit and scrubber exit flue gas streams. It was suggested that this observation would be explained if the barium compound predominating in the flue gas was quite water soluble. The observation of higher-than-expected apparent scrubber collection efficiency would be similarly explained.

An explanation is being sought as to why copper has a lower apparent scrubber collection efficiency than might be expected based on its relative volatility.

4.4.2.2 Effects of Incinerator Operating Conditions on Metal Distributions

A major objective of this test series was to identify the effects of the RKS operating conditions on the metal distributions among the incinerator discharges. As discussed in Section 2, the conditions varied in the test program were kiln exit temperature, afterburner exit temperature, and waste feed chlorine content. Previous discussion in Section 4.4.2.1 considered the 8 parameter tests performed as one set of tests, without mention of variations in test conditions. In this subsection, the data from each test are presented in a format that facilitates interpretation with respect to the primary test variables.

Table 36 summarizes the metal discharge distributions and mass balances, with the discharge distributions expressed in terms of the fraction of the metal fed. Table 37 summarizes the normalized distributions in both the kiln ash and afterburner exit flue gas discharges, and in the kiln ash and scrubber discharges. As before, in cases where some samples were reported as not containing a given metal, the distributions are reported as ranges. Limits of the ranges correspond to an assumed sample concentration of zero and the detection limit, respectively. No afterburner exit data are available for tests 5 and 10, since the alundum thimble particulate sample was compromised by excessive moisture. Also, as discussed in Section 4.4.2.1, afterburner exit flue gas chromium data are not reported.

Three groupings of tests appear in Tables 36 and 37. Each grouping corresponds to a series of tests in which one test variable (kiln exit temperature, afterburner exit temperature, or feed chlorine content) varied with the other two variables held nominally constant. The center point, which was tested in duplicate (Tests 7 and 10), is included in all groupings.

The data in Table 36 represent the individual test data comprising the ranges and averages noted in Tables 31, 32, and 33. The data in Table 37 represent the individual test data comprising the ranges and averages noted in Tables 31, 32 and 35.

Figures 5 through 13 show the variation in the normalized discharge distributions among the kiln ash, scrubber exit flue gas, and scrubber liquor for each of the test metals. Three bar charts are shown in each figure corresponding to the three groupings of tests in Tables 36 and 37. Thus, each bar chart in Figures 5 through 13 corresponds to a series of tests in which one test variable was varied, with the other two variables held nominally constant. Each figure shows the normalized discharge distributions for one of the nine test trace metals. The figures are ordered by decreasing observed metal volatility; this is the ordering of the metals used in Table 32. Thus, Figure 5 shows cadmium distribution (the observed most volatile metal) data; Figure 13 shows magnesium distribution (the observed most refractory metal) data.

Normalized discharge distributions have been used in Figures 5 through 13, and will be used in the following discussion, because they remove one significant source for test to test data variability, and thereby allow clearer data interpretation. Normalized distributions would be equivalent to distributions expressed as percent of metal fed had mass balance closure been 100 percent in all cases. Use of normalized distributions, therefore, "corrects" for data variability introduced by less than perfect mass balance closure. Mass balance closure varied significantly from test to test in this test program, as it has in all past experience in measuring trace metal discharges from combustion sources. Thus, variations in metal discharge distributions expressed as percent of metal fed would be affected by both actual variations due to changes in test variables and by individual test mass balance closure. Variations in normalized distributions more closely reflect those due to changes in test variables by "forcing" 100 percent mass balance closure for each test.

TABLE 36. METAL DISCHARGE DISTRIBUTIONS AND MASS BALANCE CLOSURE FOR THE TRACE METALS PARAMETRIC TEST SERIES

| | | | | | | | | | | | | |
|-------------------|---------------------------------|------|------|------|-----------------------------------|------|------|------|-------------------------------------|------|------|------|
| Test: | 5 | 7 | 10 | 6 | 9 | 7 | 10 | 8 | 4 | 7 | 10 | 11 |
| Primary variable: | Kiln exit temperature (°C) | | | | Afterburner exit temperature (°C) | | | | Feed chlorine content (wt %) | | | |
| Target: | 816 | 871 | 871 | 927 | 982 | 1093 | 1093 | 1204 | 0 | 4 | 4 | 8 |
| Test average: | 825 | 878 | 873 | 928 | 983 | 1088 | 1094 | 1196 | 0.0 | 3.8 | 4.6 | 8.3 |
| Held constant: | AB exit = 1093°C; chlorine = 4% | | | | Kiln exit = 871°C; chlorine = 4% | | | | Kiln exit = 871°C; AB exit = 1093°C | | | |
| Test average: | 1071 | 1088 | 1094 | 1092 | 875 | 878 | 873 | 871 | 874 | 878 | 873 | 870 |
| Test average: | 3.7 | 3.8 | 4.6 | 4.2 | 3.4 | 3.8 | 4.6 | 3.6 | 1093 | 1088 | 1094 | 1092 |

| Discharge distribution (% of metal fed) | | | | | | | | | | | | |
|--|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Arsenic | | | | | | | | | | | | |
| Kiln ash | 71.9 | 39.5 | 36.3 | 40.8 | 72.0 | 39.5 | 36.3 | 66.6 | 63.5 | 39.5 | 36.3 | 47.6 |
| Afterburner exit flue gas | — | 2.0 | — | 5.5 | 1.9 | 2.0 | — | 2.2 | 1.6 | 2.0 | — | 3.8 |
| Mass balance closure around kiln/afterburner | — | 41.5 | — | 46.3 | 73.9 | 41.5 | — | 68.8 | 65.1 | 41.5 | — | 51.4 |
| Scrubber exit flue gas | 1.9-2.3 | 2.1-2.7 | 1.1-1.5 | 3.6-4.0 | 2.5-3.0 | 2.1-2.7 | 1.1-1.5 | 2.7-3.2 | 1.2-1.4 | 2.1-2.7 | 1.1-1.5 | 2.2-2.5 |
| Scrubber liquor | 2.1 | 3.8 | 1.4 | 3.7 | 1.9 | 3.8 | 1.4 | 3.3 | 2.7 | 3.8 | 1.4 | 1.4 |
| Mass balance closure around kiln/scrubber | 76.3 | 46.0 | 39.2 | 48.5 | 76.9 | 46.0 | 39.2 | 73.1 | 67.6 | 46.0 | 39.2 | 51.5 |
| Barium | | | | | | | | | | | | |
| Kiln ash | 43.0 | 64.3 | 55.0 | 76.6 | 76.7 | 64.3 | 55.0 | 127.7 | 39.2 | 64.3 | 55.0 | 52.4 |
| Afterburner exit flue gas | — | 11.7 | — | 15.4 | 10.0 | 11.7 | — | 12.1 | 1.4 | 11.7 | — | 7.7 |
| Mass balance closure around kiln/afterburner | — | 76.0 | — | 92.0 | 86.7 | 76.0 | — | 139.0 | 40.6 | 76.0 | — | 60.1 |
| Scrubber exit flue gas | 2.3 | 1.7 | 4.3 | 1.8 | 2.0 | 1.7 | 4.3 | 2.4 | 1.3 | 1.7 | 4.3 | 1.7 |
| Scrubber liquor | 12.9 | 14.7 | 19.4 | 31.7 | 11.2 | 14.7 | 19.4 | 17.1 | 16.5 | 14.7 | 19.4 | 12.7 |
| Mass balance closure around kiln/scrubber | 59.0 | 80.7 | 78.7 | 110.1 | 89.9 | 80.7 | 78.7 | 147.2 | 57.0 | 80.7 | 78.7 | 66.8 |
| Bismuth | | | | | | | | | | | | |
| Kiln ash | 16.2 | 16.5 | 10.9 | 9.2 | 8.4 | 16.5 | 10.9 | 20.7 | 24.4 | 16.5 | 10.9 | 23.3 |
| Afterburner exit flue gas | — | 3.5 | — | 8.0 | 2.5 | 3.5 | — | 2.5 | 19.3 | 3.5 | — | 14.0 |
| Mass balance closure around kiln/afterburner | — | 20.0 | — | 17.2 | 10.9 | 20.0 | — | 23.2 | 43.7 | 20.0 | — | 37.3 |
| Scrubber exit flue gas | 26.1 | 30.4 | 12.7 | 20.3 | 19.1 | 30.4 | 12.7 | 25.6 | 6.0 | 30.4 | 12.7 | 24.7 |
| Scrubber liquor | 20.4 | 27.1 | 12.7 | 10.5 | 12.7 | 27.1 | 12.7 | 22.5 | 7.4 | 27.1 | 12.7 | 16.3 |
| Mass balance closure around kiln/scrubber | 62.7 | 74.0 | 36.3 | 40.0 | 40.2 | 74.0 | 36.3 | 68.8 | 37.8 | 74.0 | 36.3 | 64.3 |

(continued)

TABLE 36. (continued)

| | | | | | | | | | | | | |
|-------------------|---------------------------------|------|------|------|-----------------------------------|------|------|------|-------------------------------------|------|------|------|
| Test: | 5 | 7 | 10 | 6 | 9 | 7 | 10 | 8 | 4 | 7 | 10 | 11 |
| Primary variable: | Kiln exit temperature (°C) | | | | Afterburner exit temperature (°C) | | | | Feed chlorine content (wt %) | | | |
| Target: | 816 | 871 | 871 | 927 | 982 | 1093 | 1093 | 1204 | 0 | 4 | 4 | 8 |
| Test average: | 825 | 878 | 873 | 928 | 983 | 1088 | 1094 | 1196 | 0.0 | 3.8 | 4.6 | 8.3 |
| Held constant: | AB exit = 1093°C; chlorine = 4% | | | | Kiln exit = 871°C; chlorine = 4% | | | | Kiln exit = 871°C; AB exit = 1093°C | | | |
| Test average: | 1071 | 1088 | 1094 | 1092 | 875 | 878 | 873 | 871 | 874 | 878 | 873 | 870 |
| Test average: | 3.7 | 3.8 | 4.6 | 4.2 | 3.4 | 3.8 | 4.6 | 3.6 | 1093 | 1088 | 1094 | 1092 |

| Discharge distribution (% of metal fed) | | | | | | | | | | | | |
|---|-------|--------|-------|---------|--------|--------|-------|---------|-------|--------|-------|--------|
| Cadmium | | | | | | | | | | | | |
| Kiln ash | <14.9 | <11.2 | <9.7 | <12.9 | <14.5 | <11.2 | <9.7 | <17.1 | <10.9 | <11.2 | <9.7 | <10.0 |
| Afterburner exit flue gas | — | 20.8 | — | 70.9 | 43.3 | 20.8 | — | 24.9 | 14.2 | 20.8 | — | 62.8 |
| Mass balance closure around kiln/afterburner | — | 21-32 | — | 71-83 | 43-57 | 21-32 | — | 25-42 | 14-25 | 21-32 | — | 63-73 |
| Scrubber exit flue gas | 42.1 | 61.6 | 31.1 | 74.4 | 63.4 | 61.6 | 31.1 | 64.7 | 15.7 | 61.6 | 31.1 | 72.8 |
| Scrubber liquor | 41.1 | 36.7 | 33.7 | 37.7 | 26.6 | 36.7 | 33.7 | 36.2 | 10.8 | 36.7 | 33.7 | 25.0 |
| Mass balance closure around kiln/scrubber | 83-98 | 98-110 | 65-75 | 107-120 | 90-105 | 98-110 | 65-75 | 101-118 | 27-37 | 98-110 | 65-75 | 98-108 |
| Chromium | | | | | | | | | | | | |
| Kiln ash | 88.6 | 68.9 | 54.9 | 75.3 | 79.9 | 68.9 | 54.9 | 54.2 | 64.2 | 68.9 | 54.9 | 60.9 |
| Afterburner exit flue gas | — | — | — | — | — | — | — | — | — | — | — | — |
| Mass balance closure around kiln/afterburner | — | — | — | — | — | — | — | — | — | — | — | — |
| Scrubber exit flue gas | 2.7 | 1.5 | 1.1 | 1.7 | 0.9 | 1.5 | 1.1 | 2.6 | 0.7 | 1.5 | 1.1 | 1.8 |
| Scrubber liquor | 2.2 | 2.9 | 7.9 | 2.1 | 2.8 | 2.9 | 7.9 | 4.0 | 1.9 | 2.9 | 7.9 | 3.4 |
| Mass balance closure around kiln/scrubber | 93.5 | 73.3 | 63.9 | 79.1 | 83.6 | 73.3 | 63.9 | 60.8 | 66.8 | 73.3 | 63.9 | 66.1 |
| Copper | | | | | | | | | | | | |
| Kiln ash | 63.0 | 43.0 | 41.1 | 64.2 | 52.7 | 43.0 | 41.1 | 49.3 | 44.8 | 43.0 | 41.1 | 34.0 |
| Afterburner exit flue gas | — | 4.0 | — | 11.7 | 8.5 | 4.0 | — | 5.9 | 2.8 | 4.0 | — | 19.5 |
| Mass balance closure around kiln/afterburner | — | 47.0 | — | 75.9 | 61.2 | 47.0 | — | 55.2 | 47.6 | 47.0 | — | 53.5 |
| Scrubber exit flue gas | 9.6 | 8.6 | 9.5 | 11.0 | 10.1 | 8.6 | 9.5 | 10.5 | 0.3 | 8.6 | 9.5 | 19.5 |
| Scrubber liquor | 6.4 | 5.1 | 3.3 | 2.7 | 3.6 | 5.1 | 3.3 | 5.9 | 0.7 | 5.1 | 3.3 | 5.2 |
| Mass balance closure around kiln/scrubber | 79.0 | 56.7 | 53.9 | 77.9 | 66.4 | 56.7 | 53.9 | 65.7 | 45.8 | 56.7 | 53.9 | 58.7 |

(continued)

TABLE 36. (concluded)

| | | | | | | | | | | | | |
|-------------------|---------------------------------|------|------|------|-----------------------------------|------|------|------|-------------------------------------|------|------|------|
| Test: | 5 | 7 | 10 | 6 | 9 | 7 | 10 | 8 | 4 | 7 | 10 | 11 |
| Primary variable: | Kiln exit temperature (°C) | | | | Afterburner exit temperature (°C) | | | | Feed chlorine content (wt %) | | | |
| Target: | 816 | 871 | 871 | 927 | 982 | 1093 | 1093 | 1204 | 0 | 4 | 4 | 8 |
| Test average: | 825 | 878 | 873 | 928 | 983 | 1088 | 1094 | 1196 | 0.0 | 3.8 | 4.6 | 8.3 |
| Held constant: | AB exit = 1093°C; chlorine = 4% | | | | Kiln exit = 871°C; chlorine = 4% | | | | Kiln exit = 871°C; AB exit = 1093°C | | | |
| Test average: | 1071 | 1088 | 1094 | 1092 | 875 | 878 | 873 | 871 | 874 | 878 | 873 | 870 |
| Test average: | 3.7 | 3.8 | 4.6 | 4.2 | 3.4 | 3.8 | 4.6 | 3.6 | 1093 | 1088 | 1094 | 1092 |

Discharge distribution (% of metal fed)

Lead

| | | | | | | | | | | | | |
|---|------|------|------|------|------|------|------|------|------|------|------|------|
| Kiln ash | 11.4 | 12.5 | 5.0 | 8.1 | 4.7 | 12.5 | 5.0 | 12.6 | 6.7 | 12.5 | 5.0 | 5.7 |
| Afterburner exit flue gas | — | 7.7 | — | 31.4 | 26.2 | 7.7 | — | 11.2 | 5.7 | 7.7 | — | 55.1 |
| Mass balance closure around kiln/afterburner | — | 20.2 | — | 39.5 | 30.9 | 20.2 | — | 23.8 | 12.4 | 20.2 | — | 60.8 |
| Scrubber exit flue gas | 45.5 | 40.9 | 18.4 | 51.6 | 48.8 | 40.9 | 18.4 | 41.1 | 0.9 | 40.9 | 18.4 | 70.6 |
| Scrubber liquor | 33.3 | 30.3 | 13.2 | 17.2 | 27.0 | 30.3 | 13.2 | 37.2 | 0.4 | 30.3 | 13.2 | 19.6 |
| Mass balance closure around kiln/scrubber | 90.2 | 83.7 | 36.6 | 76.9 | 80.5 | 83.7 | 36.6 | 90.9 | 8.0 | 83.7 | 36.6 | 95.9 |

Magnesium

| | | | | | | | | | | | | |
|---|-------|------|------|------|------|------|------|------|------|------|------|------|
| Kiln ash | 133.1 | 97.9 | 75.3 | 90.4 | 96.2 | 97.9 | 75.3 | 69.2 | 89.1 | 97.9 | 75.3 | 82.6 |
| Afterburner exit flue gas | — | 0.1 | — | 0.3 | 0.3 | 0.1 | — | 0.1 | 0.1 | 0.1 | — | 0.2 |
| Mass balance closure around kiln/afterburner | — | 98.0 | — | 90.7 | 96.5 | 98.0 | — | 69.3 | 89.2 | 98.0 | — | 82.8 |
| Scrubber exit flue gas | 0.3 | 0.05 | 0.1 | 0.05 | 0.06 | 0.05 | 0.1 | 0.07 | 0.03 | 0.05 | 0.1 | 0.07 |
| Scrubber liquor | 0.5 | 0.6 | 0.4 | 0.4 | 0.6 | 0.6 | 0.4 | 0.5 | 0.3 | 0.6 | 0.4 | 0.4 |
| Mass balance closure around kiln/scrubber | 133.9 | 98.6 | 75.8 | 90.9 | 96.9 | 98.6 | 75.8 | 69.8 | 89.4 | 98.6 | 75.8 | 83.1 |

Strontium

| | | | | | | | | | | | | |
|---|------|------|------|------|------|------|------|------|------|------|------|------|
| Kiln ash | 32.6 | 56.7 | 34.1 | 64.1 | 64.5 | 56.7 | 34.1 | 34.8 | 25.5 | 56.7 | 34.1 | 33.1 |
| Afterburner exit flue gas | — | 0.3 | — | 0.2 | 0.1 | 0.3 | — | 0.1 | 0.07 | 0.3 | — | 0.1 |
| Mass balance closure around kiln/afterburner | — | 57.0 | — | 64.3 | 64.6 | 57.0 | — | 34.9 | 25.6 | 57.0 | — | 33.2 |
| Scrubber exit flue gas | 0.5 | 1.0 | 1.3 | 1.2 | 1.0 | 1.0 | 1.3 | 1.6 | 0.6 | 1.0 | 1.3 | 0.7 |
| Scrubber liquor | 6.3 | 3.3 | 2.5 | 5.9 | 3.1 | 3.3 | 2.5 | 6.1 | 1.5 | 3.3 | 2.5 | 2.8 |
| Mass balance closure around kiln/scrubber | 39.4 | 61.0 | 37.9 | 71.2 | 68.6 | 61.0 | 37.9 | 42.5 | 27.6 | 61.0 | 37.0 | 36.6 |

TABLE 37. NORMALIZED METAL DISCHARGE DISTRIBUTIONS AND APPARENT SCRUBBER COLLECTION EFFICIENCY FOR THE TRACE METALS PARAMETRIC TEST SERIES

| | | | | | | | | | | | | |
|---------------------------------------|---------------------------------|---------|---------|---------|-----------------------------------|---------|---------|---------|-------------------------------------|---------|---------|---------|
| Test: | 5 | 7 | 10 | 6 | 9 | 7 | 10 | 8 | 4 | 7 | 10 | 11 |
| Primary variable: | Kiln exit temperature (°C) | | | | Afterburner exit temperature (°C) | | | | Feed chlorine content (wt %) | | | |
| Target: | 816 | 871 | 871 | 927 | 982 | 1093 | 1093 | 1204 | 0 | 4 | 4 | 8 |
| Test average: | 825 | 878 | 873 | 928 | 983 | 1088 | 1094 | 1196 | 0.0 | 3.8 | 4.6 | 8.3 |
| Held constant: | AB exit = 1093°C; chlorine = 4% | | | | Kiln exit = 871°C; chlorine = 4% | | | | Kiln exit = 871°C; AB exit = 1093°C | | | |
| Test average: | 1071 | 1088 | 1094 | 1092 | 875 | 878 | 873 | 871 | 874 | 878 | 873 | 870 |
| Test average: | 3.7 | 3.8 | 4.6 | 4.2 | 3.4 | 3.8 | 4.6 | 3.6 | 1093 | 1088 | 1094 | 1092 |
| Normalized discharge distribution (%) | | | | | | | | | | | | |
| Arsenic | | | | | | | | | | | | |
| Kiln ash | — | 95.1 | — | 88.1 | 97.3 | 95.1 | — | 96.9 | 97.4 | 95.1 | — | 92.7 |
| Afterburner exit flue gas | — | 4.9 | — | 11.9 | 2.7 | 4.9 | — | 3.1 | 2.6 | 4.9 | — | 7.3 |
| Total | — | 100 | — | 100 | 100 | 100 | — | 100 | 100 | 100 | — | 100 |
| Kiln ash | 94.4 | 86.1 | 92.3 | 84.0 | 93.6 | 86.1 | 92.3 | 91.2 | 93.9 | 86.1 | 92.3 | 92.4 |
| Scrubber exit flue gas | 2.1-2.9 | 3.8-5.8 | 2.3-4.1 | 6.8-8.4 | 2.6-3.8 | 3.8-5.8 | 2.3-4.1 | 3.0-4.3 | 1.7-2.2 | 3.8-5.8 | 2.3-4.1 | 4.0-4.8 |
| Scrubber liquor | 2.7 | 8.2 | 3.6 | 7.6 | 2.6 | 8.2 | 3.6 | 4.6 | 3.9 | 8.2 | 3.6 | 2.7 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Apparent scrubber efficiency | 48-57 | 59-69 | 47-62 | 47-53 | 40-50 | 59-69 | 47-62 | 52-61 | 64-70 | 59-69 | 47-62 | 36-41 |
| Barium | | | | | | | | | | | | |
| Kiln ash | — | 84.6 | — | 83.3 | 88.4 | 84.6 | — | 91.6 | 96.7 | 84.6 | — | 87.2 |
| Afterburner exit flue gas | — | 15.4 | — | 16.7 | 11.6 | 15.4 | — | 8.4 | 3.3 | 15.4 | — | 12.8 |
| Total | — | 100 | — | 100 | 100 | 100 | — | 100 | 100 | 100 | — | 100 |
| Kiln ash | 74.3 | 79.6 | 69.9 | 69.6 | 85.2 | 79.6 | 69.9 | 86.9 | 68.8 | 79.6 | 69.9 | 78.6 |
| Scrubber exit flue gas | 3.8 | 2.2 | 5.5 | 1.6 | 2.2 | 2.2 | 5.5 | 1.6 | 1.6-2.4 | 2.2 | 5.5 | 2.4 |
| Scrubber liquor | 21.9 | 18.2 | 24.7 | 28.8 | 12.5 | 18.2 | 24.7 | 11.5 | 28.8 | 18.2 | 24.7 | 19.0 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Apparent scrubber efficiency | 85 | 89 | 82 | 95 | 85 | 89 | 82 | 88 | 92 | 89 | 82 | 89 |
| Bismuth | | | | | | | | | | | | |
| Kiln ash | — | 82.6 | — | 53.4 | 76.3 | 82.6 | — | 89.6 | 56.0 | 82.6 | — | 62.4 |
| Afterburner exit flue gas | — | 17.4 | — | 46.6 | 23.7 | 17.4 | — | 10.4 | 44.0 | 17.4 | — | 37.6 |
| Total | — | 100 | — | 100 | 100 | 100 | — | 100 | 100 | 100 | — | 100 |
| Kiln ash | 25.8 | 22.2 | 30.0 | 22.9 | 20.9 | 22.2 | 30.0 | 30.1 | 64.8 | 22.2 | 30.0 | 36.3 |
| Scrubber exit flue gas | 41.5 | 41.1 | 35.2 | 50.7 | 47.4 | 41.1 | 35.2 | 37.1 | 15.7 | 41.1 | 35.2 | 38.4 |
| Scrubber liquor | 32.6 | 36.7 | 34.7 | 26.3 | 31.6 | 36.7 | 34.7 | 32.8 | 19.5 | 36.7 | 34.7 | 25.4 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Apparent scrubber efficiency | 44 | 47 | 50 | 34 | 40 | 47 | 50 | 47 | 55 | 47 | 50 | 40 |

(continued)

TABLE 37. (continued)

| | | | | | | | | | | | | |
|---------------------------------------|---------------------------------|-------|-------|-------|-----------------------------------|-------|-------|-------|-------------------------------------|-------|-------|-------|
| Test: | 5 | 7 | 10 | 6 | 9 | 7 | 10 | 8 | 4 | 7 | 10 | 11 |
| Primary variable: | Kiln exit temperature (°C) | | | | Afterburner exit temperature (°C) | | | | Feed chlorine content (wt %) | | | |
| Target: | 816 | 871 | 871 | 927 | 982 | 1093 | 1093 | 1204 | 0 | 4 | 4 | 8 |
| Test average: | 825 | 878 | 873 | 928 | 983 | 1088 | 1094 | 1196 | 0.0 | 3.8 | 4.6 | 8.3 |
| Held constant: | AB exit = 1093°C; chlorine = 4% | | | | Kiln exit = 871°C; chlorine = 4% | | | | Kiln exit = 871°C; AB exit = 1093°C | | | |
| Test average: | 1071 | 1088 | 1094 | 1092 | 875 | 878 | 873 | 871 | 874 | 878 | 873 | 870 |
| Test average: | 3.7 | 3.8 | 4.6 | 4.2 | 3.4 | 3.8 | 4.6 | 3.6 | 1093 | 1088 | 1094 | 1092 |
| Normalized discharge distribution (%) | | | | | | | | | | | | |
| Cadmium | | | | | | | | | | | | |
| Kiln ash | — | <35.1 | — | <15.3 | <25.1 | <35.1 | — | <40.7 | <43.6 | <35.1 | — | <13.7 |
| Afterburner exit flue gas | — | >64.9 | — | >84.7 | >74.9 | >64.9 | — | >59.3 | >56.4 | >64.9 | — | >86.3 |
| Total | — | 100 | — | 100 | 100 | 100 | — | 100 | 100 | 100 | — | 100 |
| Kiln ash | <15.2 | <10.3 | <12.9 | <10.7 | <13.9 | <10.3 | <12.9 | <14.5 | <29.3 | <10.3 | <12.9 | <9.3 |
| Scrubber exit flue gas | 43-49 | 56-61 | 42-45 | 62-69 | 61-69 | 56-61 | 42-45 | 55-62 | 42-54 | 56-61 | 42-45 | 68-74 |
| Scrubber liquor | 42-51 | 34-39 | 45-55 | 27-31 | 25-31 | 34-39 | 45-55 | 31-38 | 29-46 | 34-39 | 45-55 | 23-26 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Apparent scrubber efficiency | 51 | 39 | 52 | 31 | 31 | 39 | 52 | 38 | 46 | 39 | 52 | 26 |
| Chromium | | | | | | | | | | | | |
| Kiln ash | — | — | — | — | — | — | — | — | — | — | — | — |
| Afterburner exit flue gas | — | — | — | — | — | — | — | — | — | — | — | — |
| Total | — | — | — | — | — | — | — | — | — | — | — | — |
| Kiln ash | 94.7 | 94.1 | 85.9 | 95.3 | 95.5 | 94.1 | 85.9 | 89.3 | 95.7 | 94.1 | 85.9 | 92.1 |
| Scrubber exit flue gas | 3.0 | 2.0 | 1.9 | 2.1 | 1.1 | 2.0 | 1.9 | 4.2 | 1.4 | 2.0 | 1.9 | 2.8 |
| Scrubber liquor | 2.3 | 3.9 | 12.2 | 2.6 | 3.4 | 3.9 | 12.2 | 6.6 | 2.8 | 3.9 | 12.2 | 5.1 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Apparent scrubber efficiency | 44 | 66 | 87 | 56 | 75 | 66 | 87 | 61 | 67 | 66 | 87 | 64 |
| Copper | | | | | | | | | | | | |
| Kiln ash | — | 90.8 | — | 84.7 | 86.0 | 90.8 | — | 89.4 | 94.1 | 90.8 | — | 63.6 |
| Afterburner exit flue gas | — | 9.2 | — | 15.3 | 14.0 | 9.2 | — | 10.6 | 5.9 | 9.2 | — | 36.4 |
| Total | — | 100 | — | 100 | 100 | 100 | — | 100 | 100 | 100 | — | 100 |
| Kiln ash | 84.2 | 75.8 | 76.2 | 82.3 | 79.2 | 75.8 | 76.2 | 75.1 | 97.6 | 75.8 | 76.2 | 58.0 |
| Scrubber exit flue gas | 12.9 | 15.1 | 17.8 | 14.1 | 15.2 | 15.1 | 17.8 | 16.0 | 0.8 | 15.1 | 17.8 | 33.2 |
| Scrubber liquor | 3.0 | 9.1 | 5.9 | 3.6 | 5.6 | 9.1 | 5.9 | 8.9 | 1.6 | 9.1 | 5.9 | 8.8 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Apparent scrubber efficiency | 19 | 38 | 25 | 21 | 27 | 38 | 25 | 36 | 67 | 38 | 25 | 21 |

(continued)

TABLE 37. (concluded)

| | | | | | | | | | | | | |
|---------------------------------------|---------------------------------|------|------|------|-----------------------------------|------|------|------|-------------------------------------|------|------|------|
| Test: | 5 | 7 | 10 | 6 | 9 | 7 | 10 | 8 | 4 | 7 | 10 | 11 |
| Primary variable: | Kiln exit temperature (°C) | | | | Afterburner exit temperature (°C) | | | | Feed chlorine content (wt %) | | | |
| Target: | 816 | 871 | 871 | 927 | 982 | 1093 | 1093 | 1204 | 0 | 4 | 4 | 8 |
| Test average: | 825 | 878 | 873 | 928 | 983 | 1088 | 1094 | 1196 | 0.0 | 3.8 | 4.6 | 8.3 |
| Held constant: | AB exit = 1093°C; chlorine = 4% | | | | Kiln exit = 871°C; chlorine = 4% | | | | Kiln exit = 871°C; AB exit = 1093°C | | | |
| Test average: | 1071 | 1088 | 1094 | 1092 | 875 | 878 | 873 | 871 | 874 | 878 | 873 | 870 |
| Test average: | 3.7 | 3.8 | 4.6 | 4.2 | 3.4 | 3.8 | 4.6 | 3.6 | 1093 | 1088 | 1094 | 1092 |
| Normalized discharge distribution (%) | | | | | | | | | | | | |
| Lead | | | | | | | | | | | | |
| Kiln ash | — | 61.7 | — | 20.3 | 15.2 | 61.7 | — | 52.8 | 53.7 | 61.7 | — | 9.5 |
| Afterburner exit flue gas | — | 38.3 | — | 79.7 | 84.8 | 38.3 | — | 47.2 | 46.3 | 38.3 | — | 90.5 |
| Total | — | 100 | — | 100 | 100 | 100 | — | 100 | 100 | 100 | — | 100 |
| Kiln ash | 12.6 | 15.0 | 13.7 | 10.4 | 5.8 | 15.0 | 13.7 | 13.8 | 83.7 | 15.0 | 13.7 | 6.0 |
| Scrubber exit flue gas | 50.4 | 48.9 | 50.2 | 67.2 | 60.6 | 48.9 | 50.2 | 45.0 | 11.6 | 48.9 | 50.2 | 73.6 |
| Scrubber liquor | 37.0 | 36.1 | 36.0 | 22.4 | 33.6 | 36.1 | 36.0 | 41.1 | 4.7 | 36.1 | 36.0 | 20.3 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Apparent scrubber efficiency | 42 | 42 | 42 | 25 | 36 | 42 | 42 | 48 | 29 | 42 | 42 | 22 |
| Magnesium | | | | | | | | | | | | |
| Kiln ash | — | 99.9 | — | 99.7 | 99.7 | 99.9 | — | 99.9 | 99.9 | 99.9 | — | 99.8 |
| Afterburner exit flue gas | — | 0.1 | — | 0.3 | 0.3 | 0.1 | — | 0.1 | 0.01 | 0.1 | — | 0.2 |
| Total | — | 100 | — | 100 | 100 | 100 | — | 100 | 100 | 100 | — | 100 |
| Kiln ash | 99.4 | 99.3 | 99.3 | 99.5 | 99.3 | 99.3 | 99.3 | 99.2 | 99.6 | 99.3 | 99.3 | 99.4 |
| Scrubber exit flue gas | 0.2 | 0.1 | 0.2 | 0.1 | 0.1 | 0.1 | 0.2 | 0.1 | 0.03 | 0.1 | 0.2 | 0.1 |
| Scrubber liquor | 0.4 | 0.6 | 0.5 | 0.4 | 0.6 | 0.6 | 0.5 | 0.7 | 0.36 | 0.6 | 0.5 | 0.5 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Apparent scrubber efficiency | 63 | 92 | 75 | 89 | 91 | 92 | 75 | 88 | 91 | 92 | 75 | 85 |
| Strontium | | | | | | | | | | | | |
| Kiln ash | — | 99.5 | — | 99.7 | 99.8 | 99.5 | — | 99.6 | 99.7 | 99.5 | — | 99.7 |
| Afterburner exit flue gas | — | 0.5 | — | 0.3 | 0.2 | 0.5 | — | 0.4 | 0.3 | 0.5 | — | 0.3 |
| Total | — | 100 | — | 100 | 100 | 100 | — | 100 | 100 | 100 | — | 100 |
| Kiln ash | 82.9 | 93.0 | 89.8 | 90.1 | 94.3 | 93.0 | 89.8 | 81.9 | 91.8 | 93.0 | 89.8 | 90.7 |
| Scrubber exit flue gas | 1.1 | 1.7 | 3.5 | 1.6 | 1.3 | 1.7 | 3.5 | 3.7 | 2.5 | 1.7 | 3.5 | 1.6 |
| Scrubber liquor | 16.0 | 5.3 | 6.6 | 8.3 | 4.4 | 5.3 | 6.6 | 14.4 | 5.7 | 5.3 | 6.6 | 7.7 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Apparent scrubber efficiency | 93 | 76 | 65 | 84 | 77 | 76 | 65 | 80 | 69 | 76 | 65 | 83 |

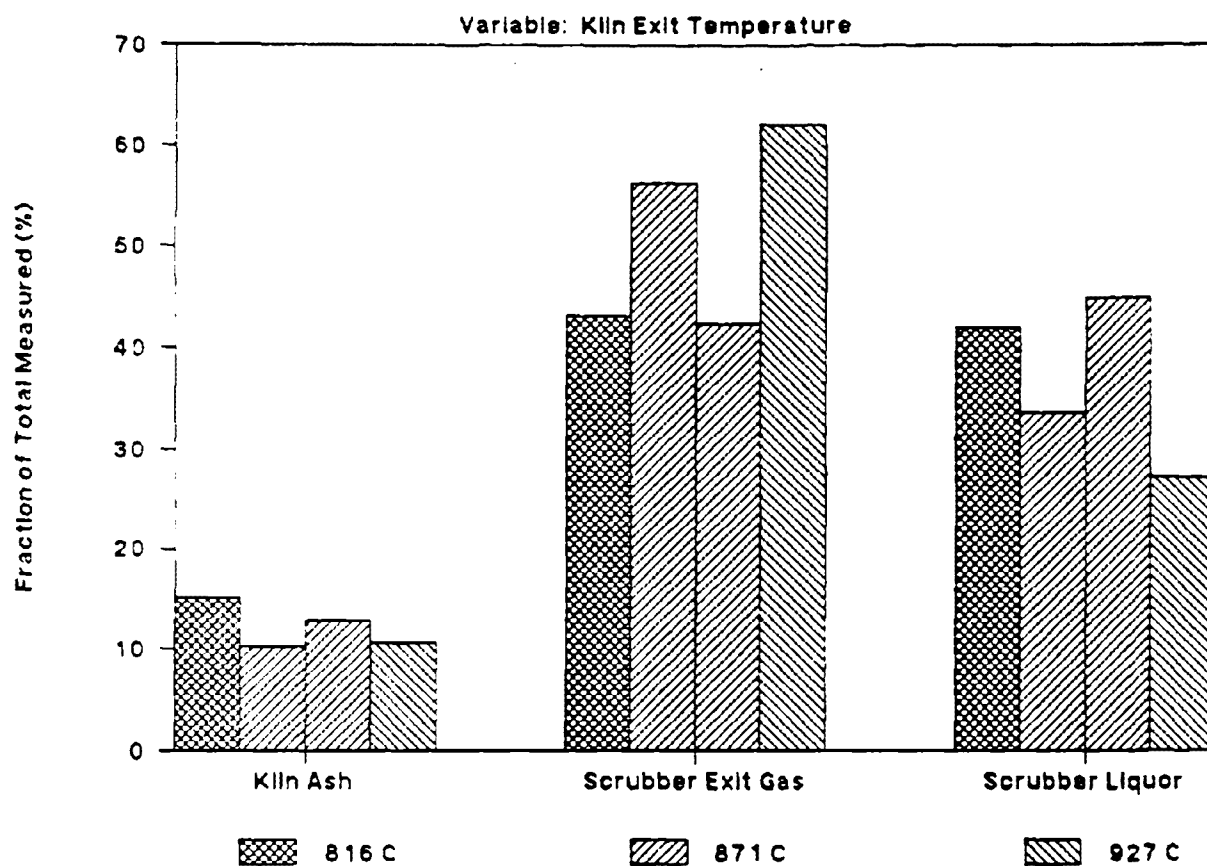


Figure 5a. Cadmium discharge distributions for the parametric trace metal tests: effect of kiln temperature.

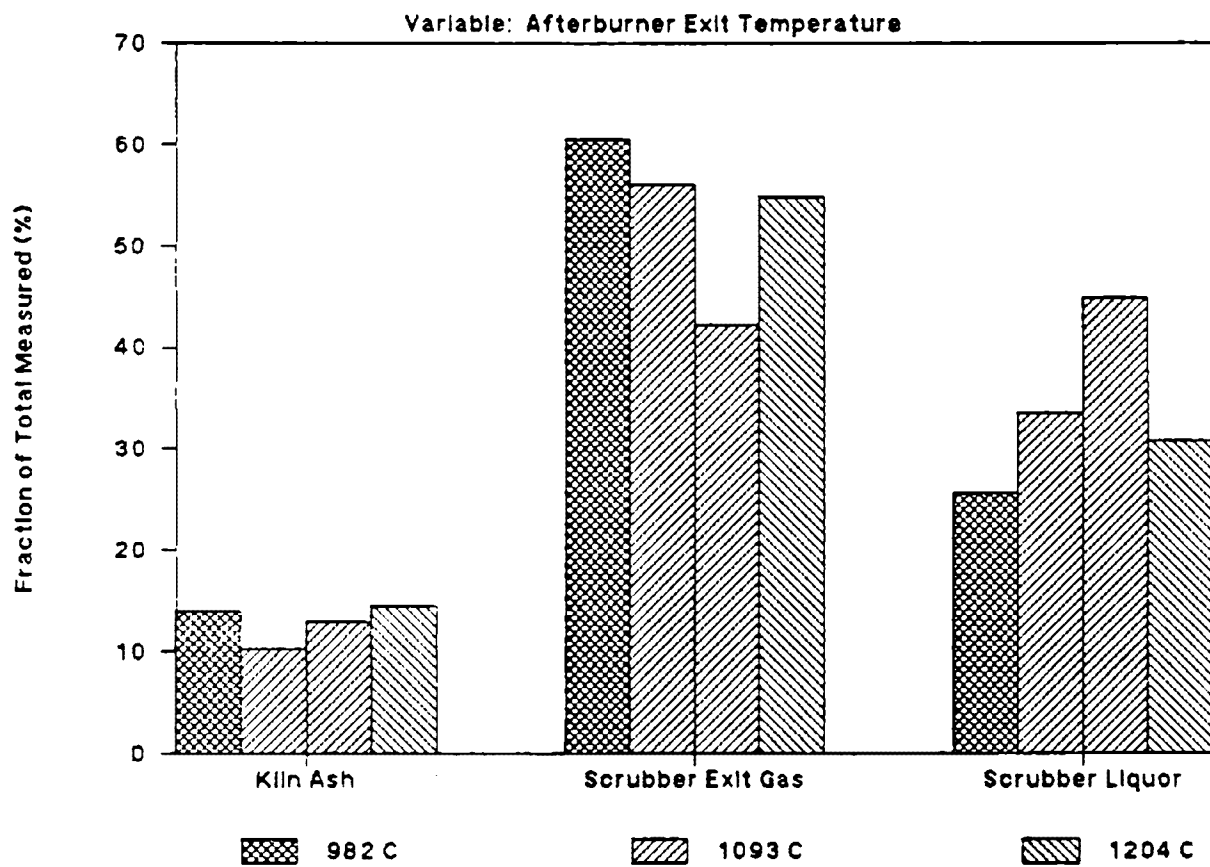


Figure 5b. Cadmium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

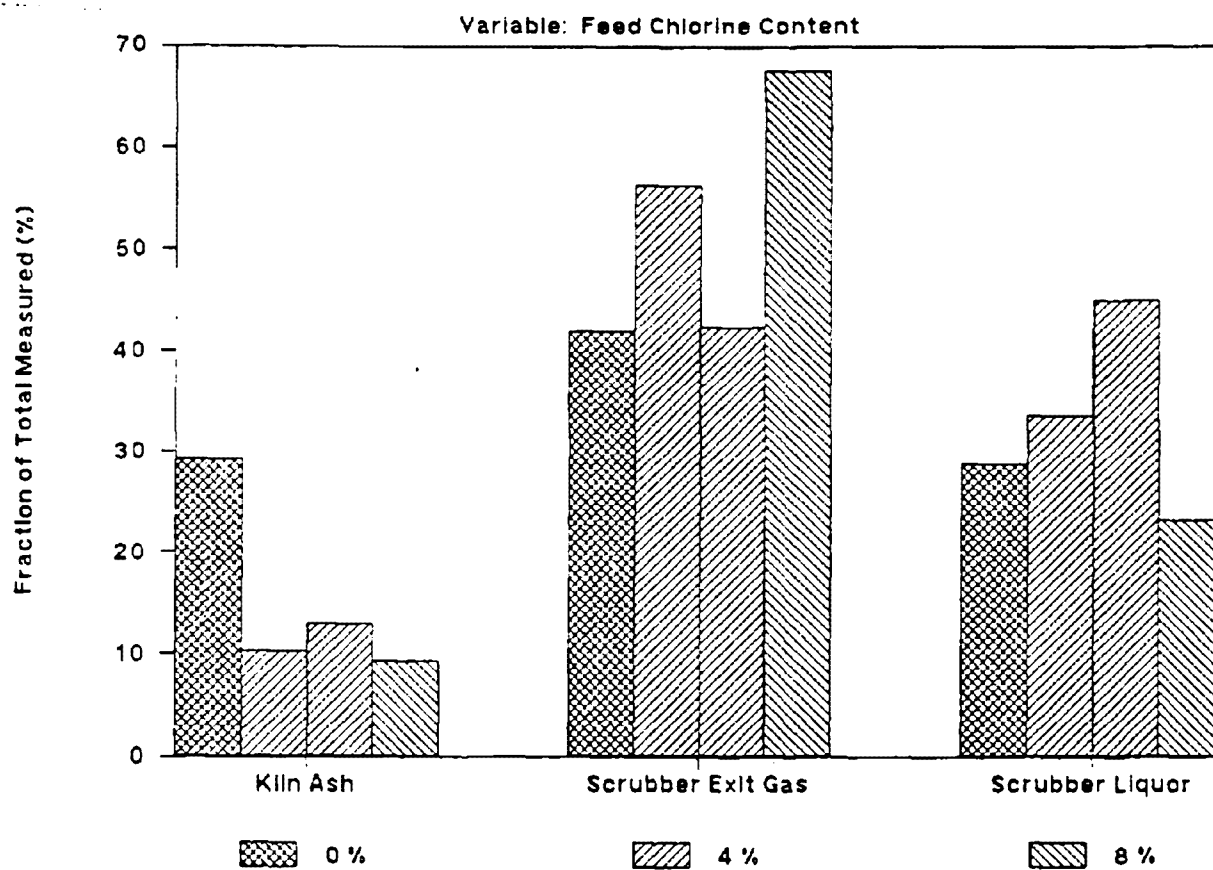


Figure 5c. Cadmium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

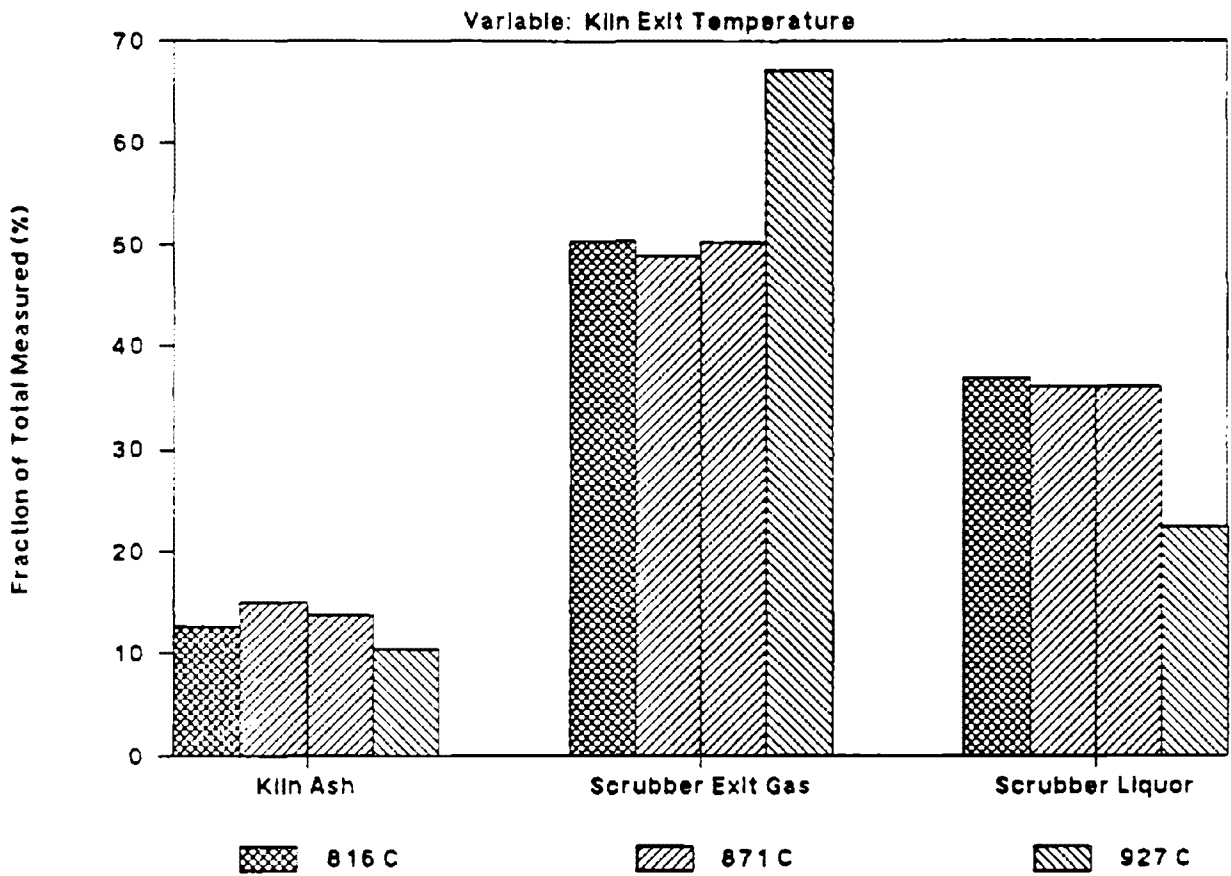


Figure 6a. Lead discharge distributions for the parametric trace metal tests: effect of kiln temperature.

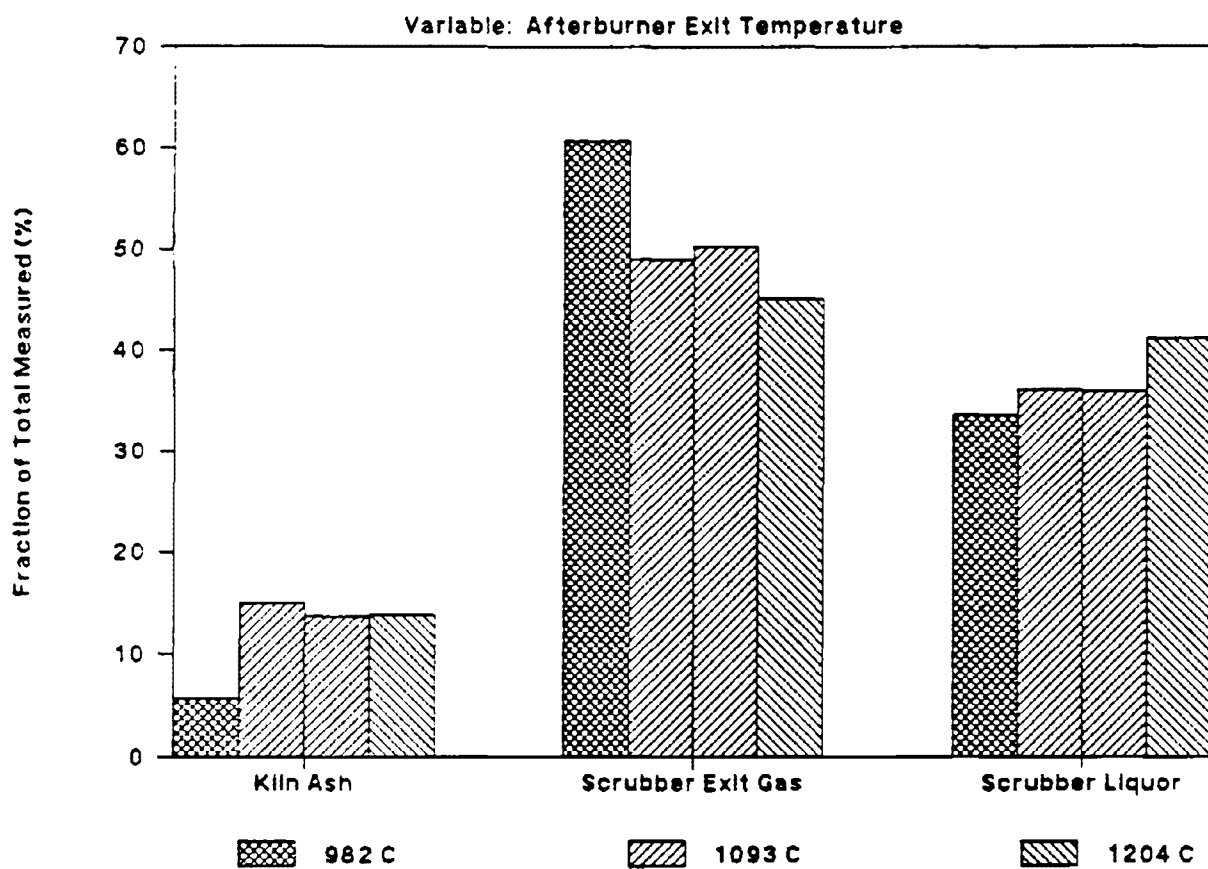


Figure 6b. Lead discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

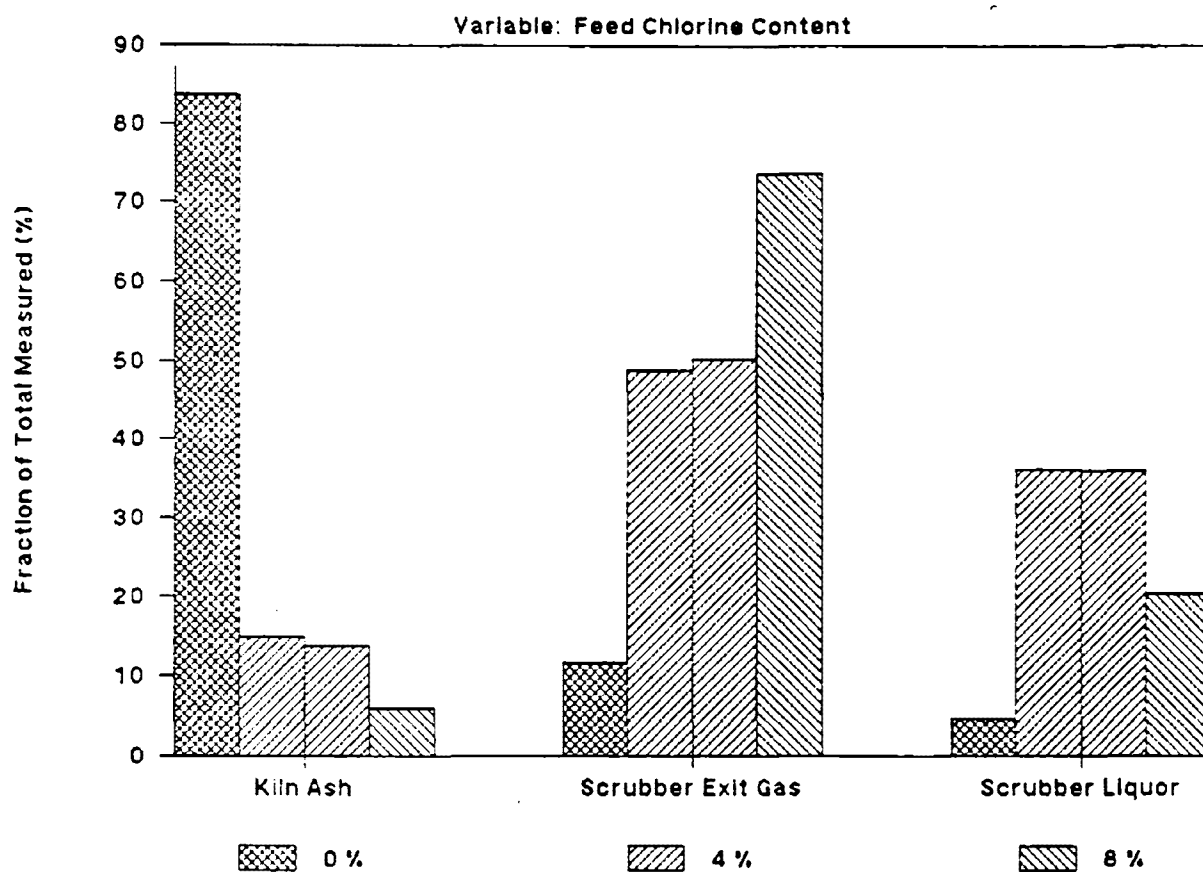


Figure 6c. Lead discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

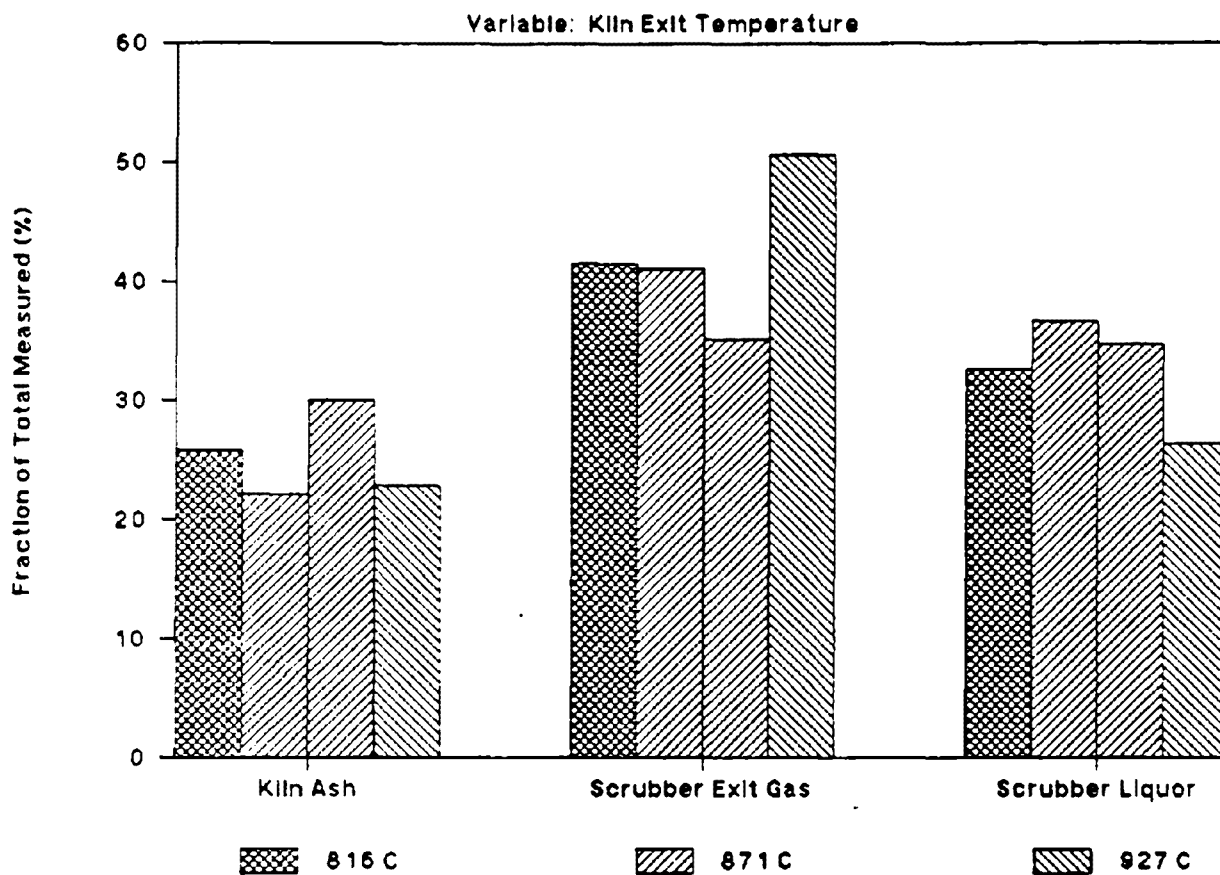


Figure 7a. Bismuth discharge distributions for the parametric trace metal tests: effect of kiln temperature.

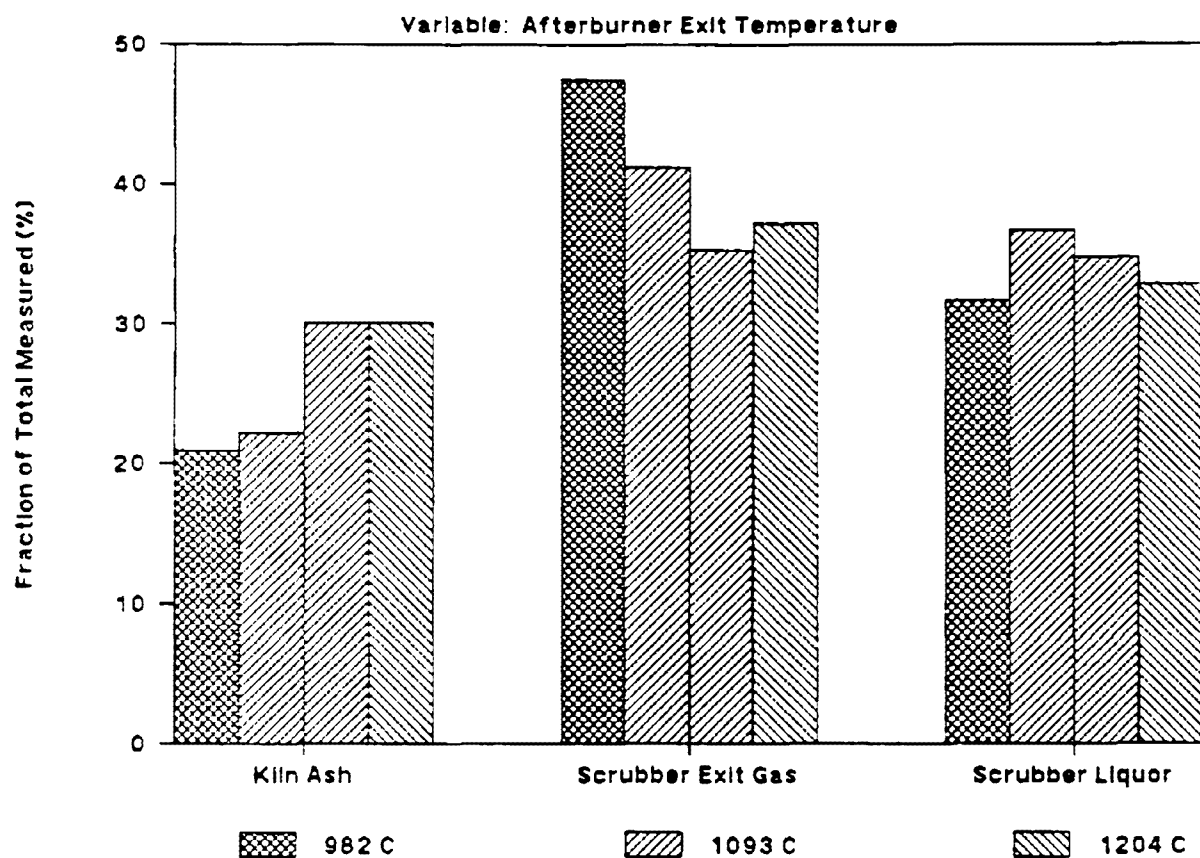


Figure 7b. Bismuth discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

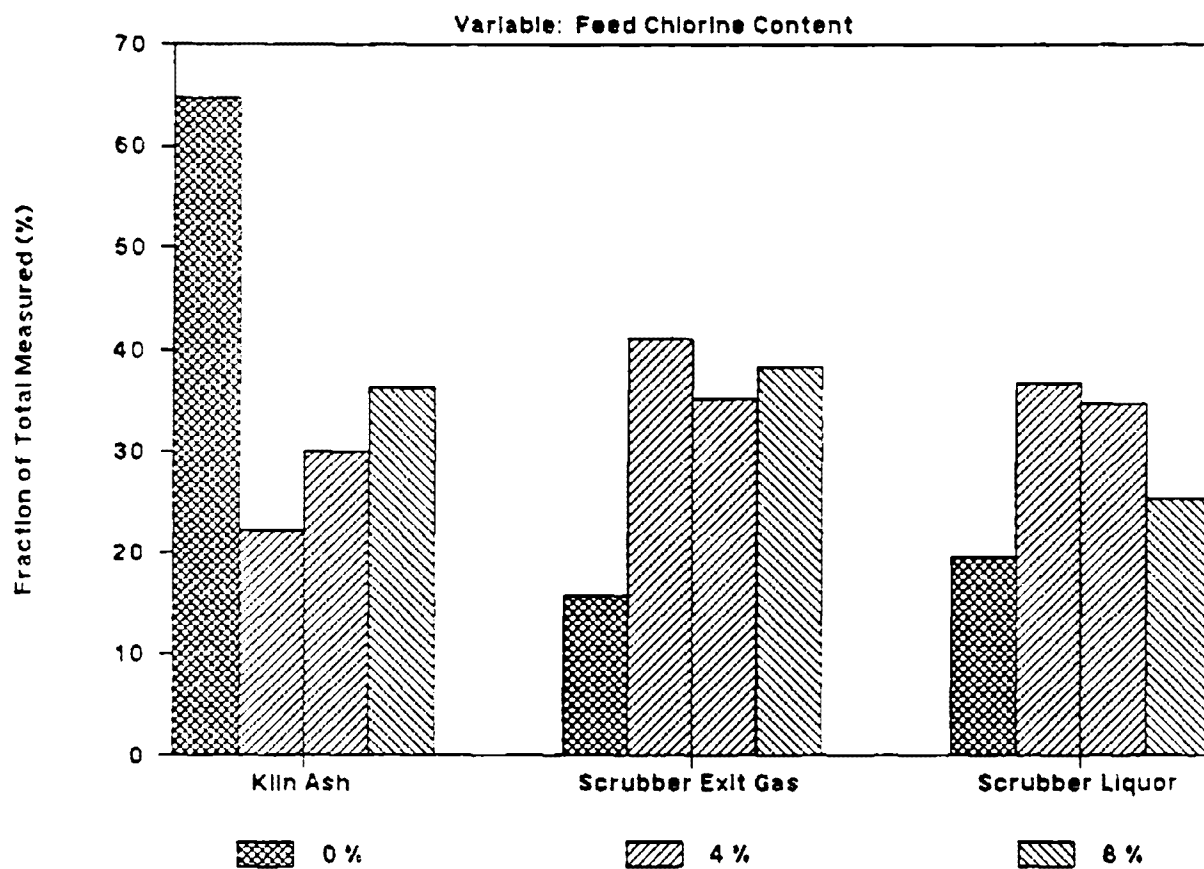


Figure 7c. Bismuth discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

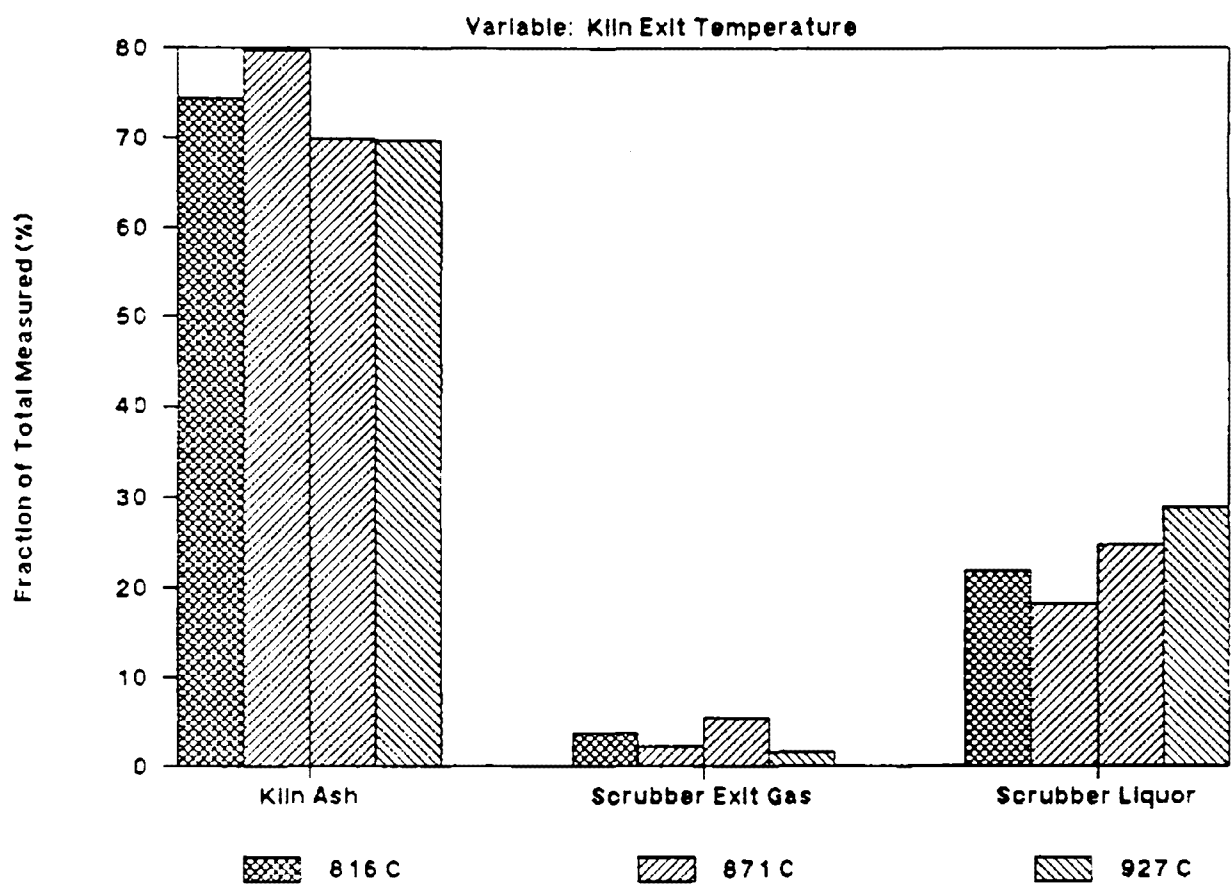


Figure 8a. Barium discharge distributions for the parametric trace metal tests: effect of kiln temperature.

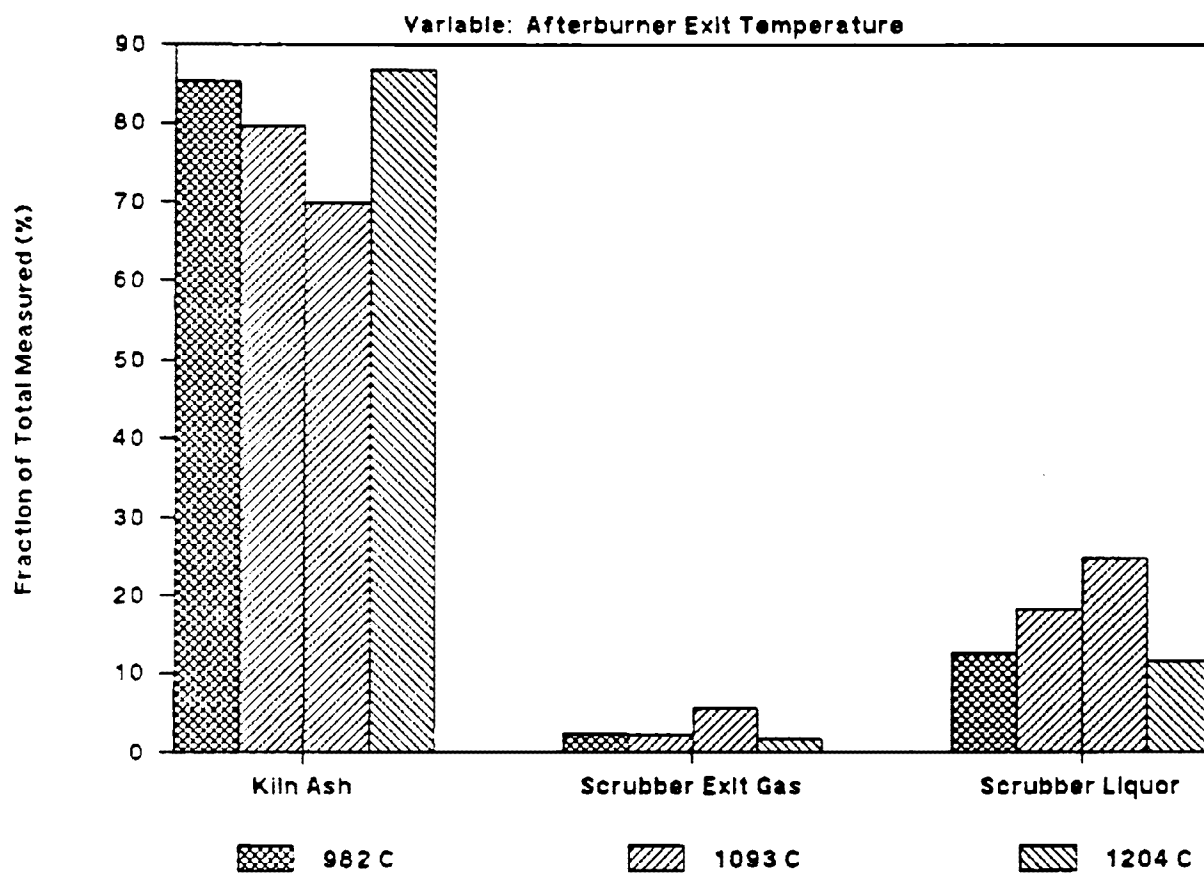


Figure 8b. Barium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

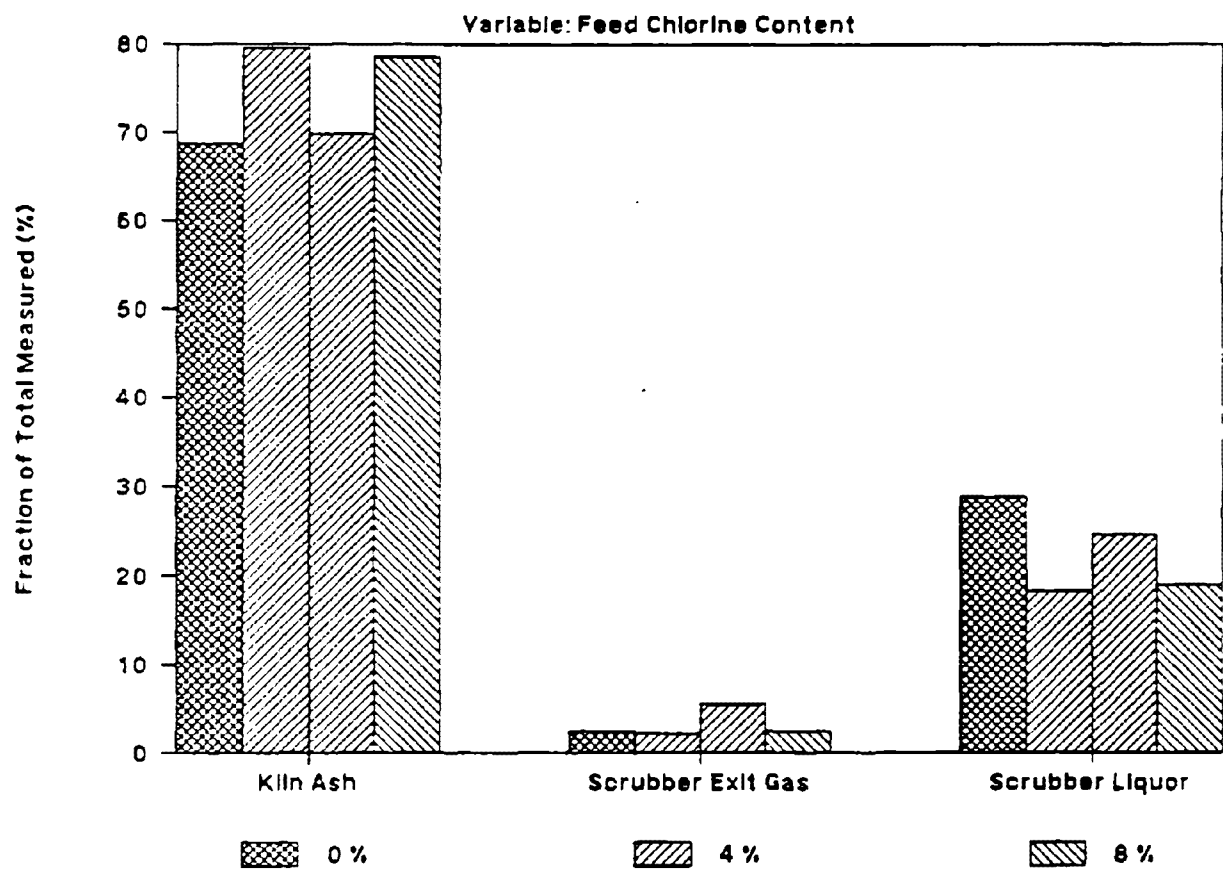


Figure 8c. Barium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

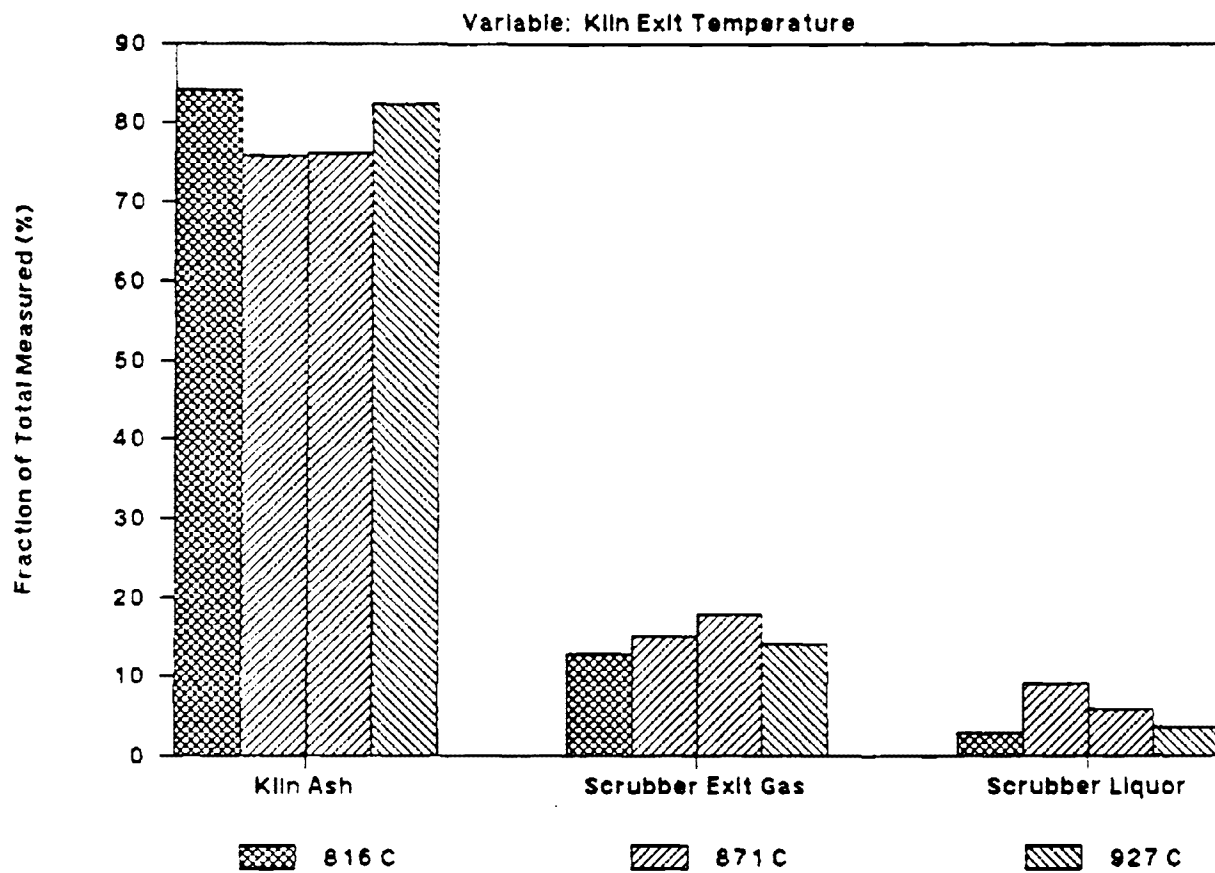


Figure 9a. Copper discharge distributions for the parametric trace metal tests: effect of kiln temperature.

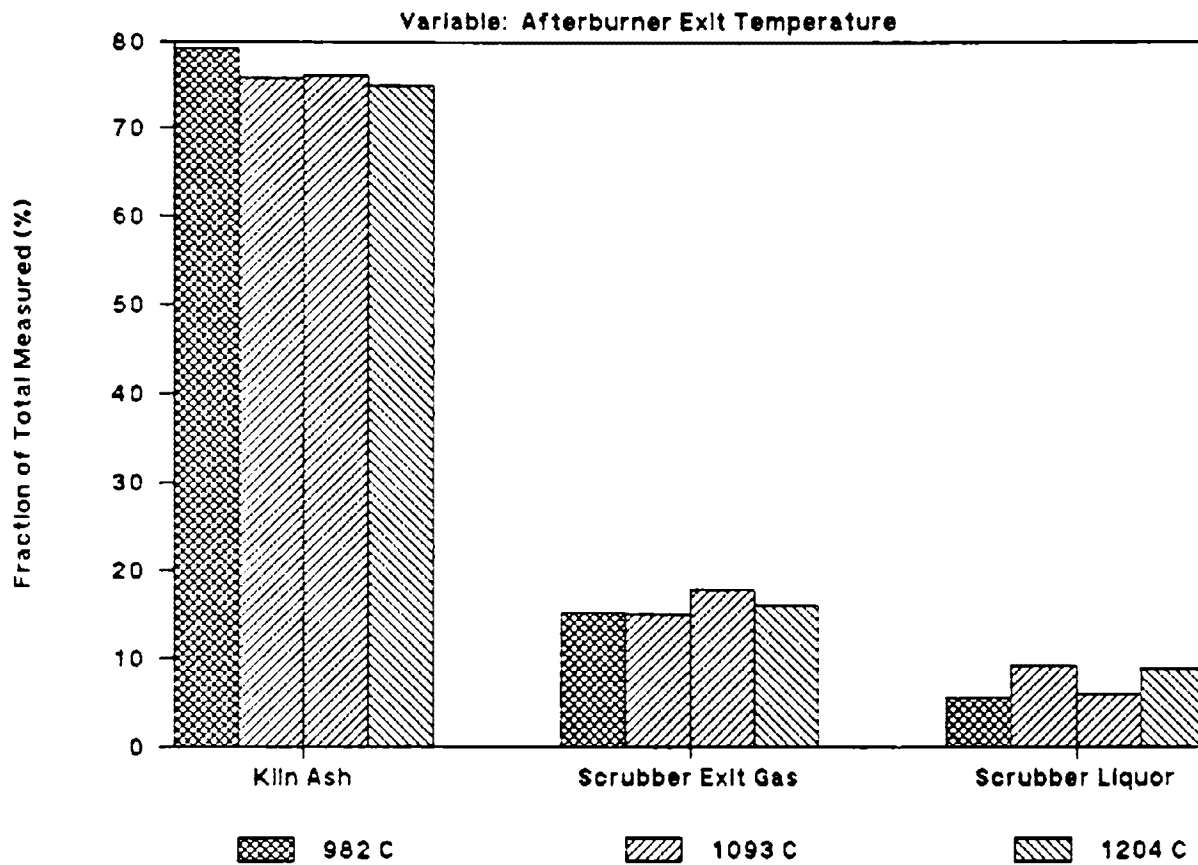


Figure 9b. Copper discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

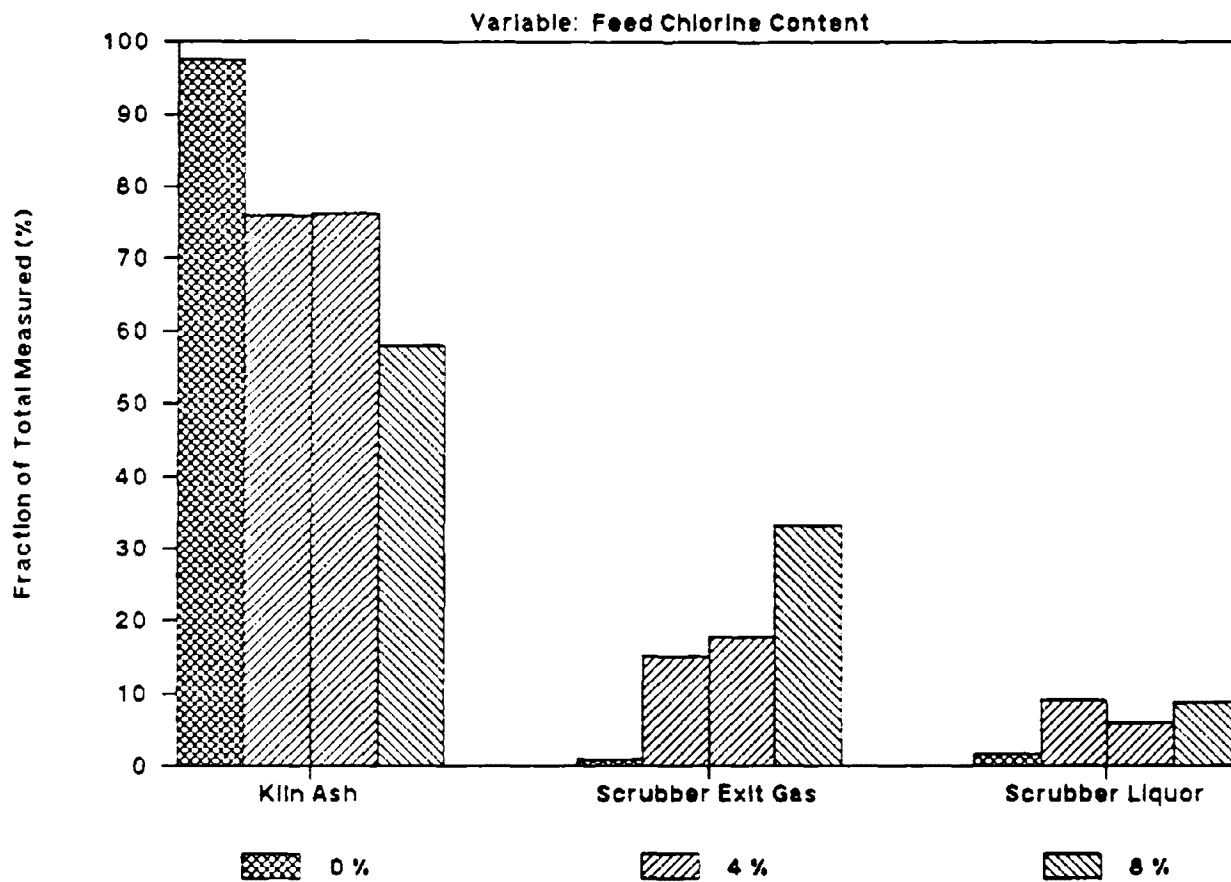


Figure 9c. Copper discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

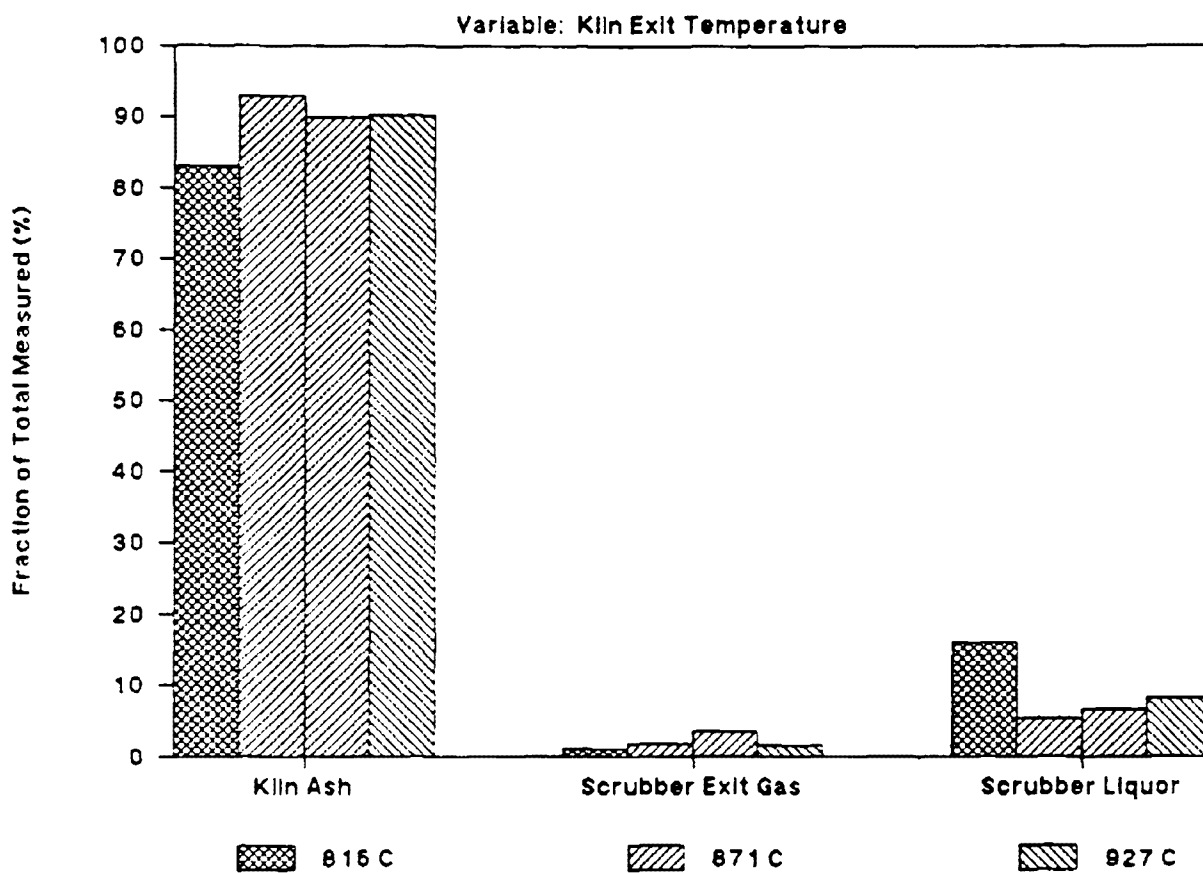


Figure 10a. Strontium discharge distributions for the parametric trace metal tests: effect of kiln temperature.

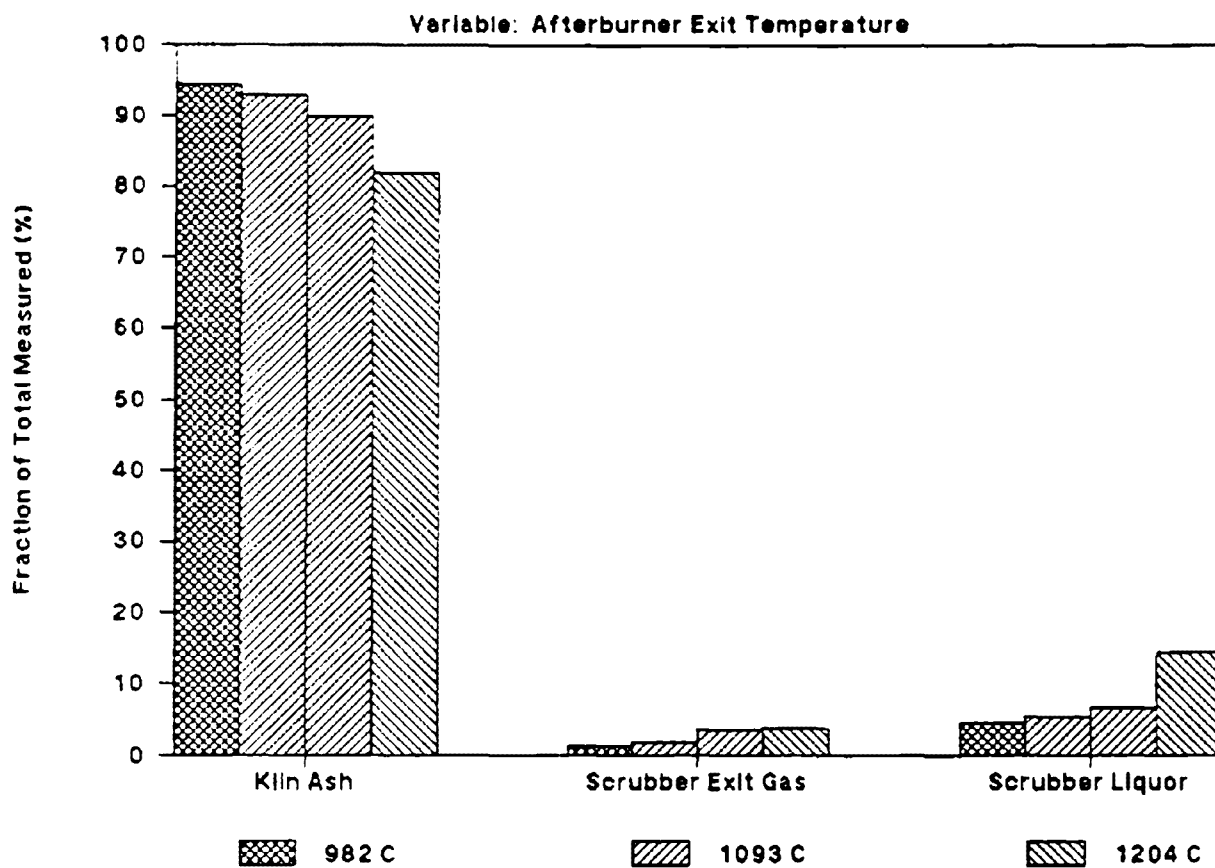


Figure 10b. Strontium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

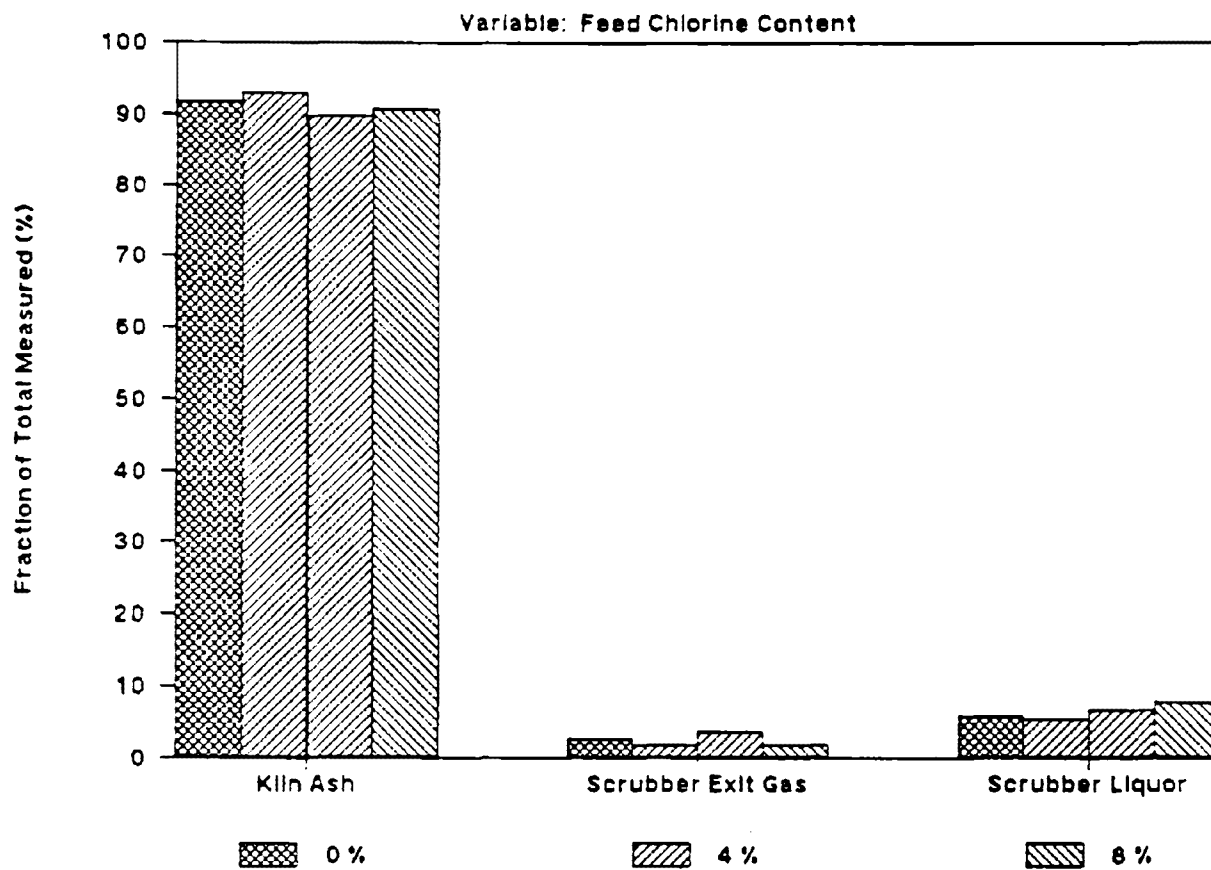


Figure 10c. Strontium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

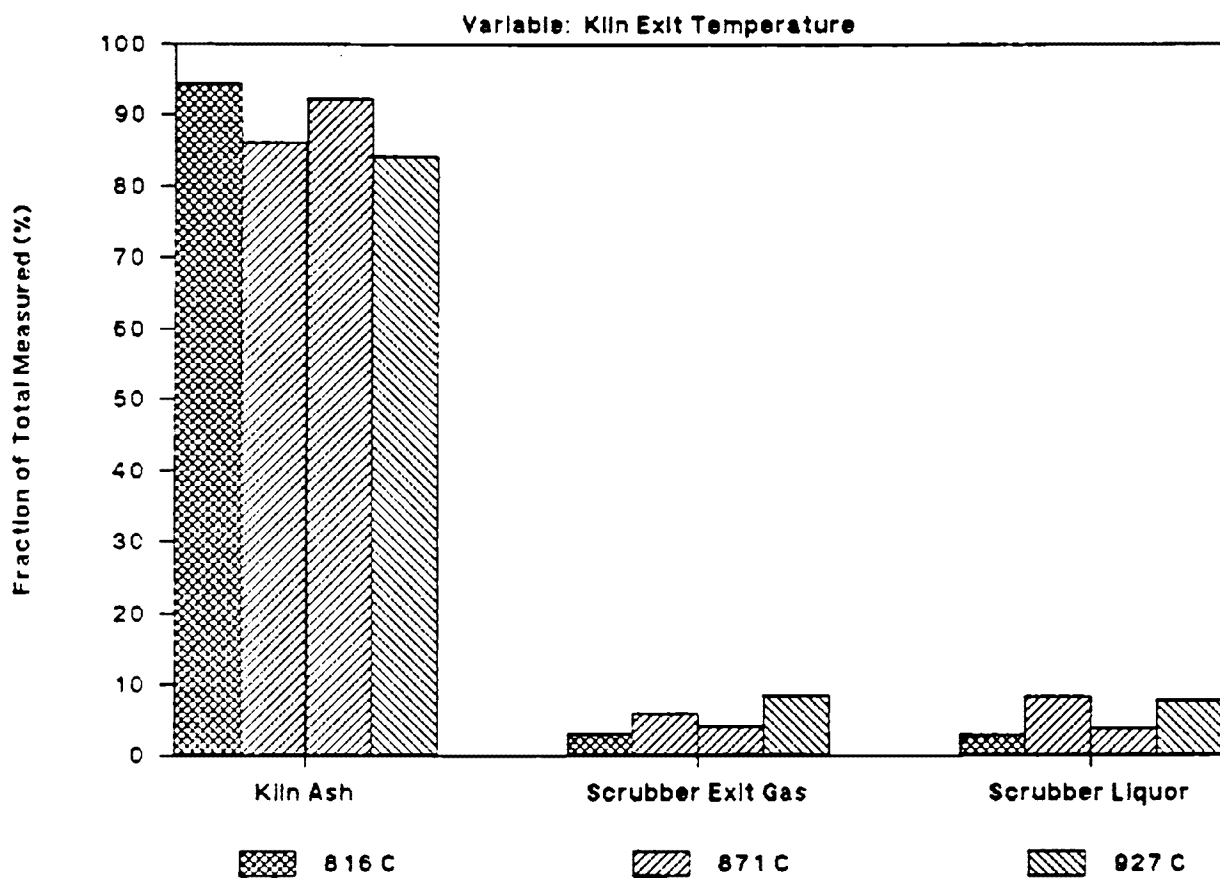


Figure 11a. Arsenic discharge distributions for the parametric trace metal tests: effect of kiln temperature.

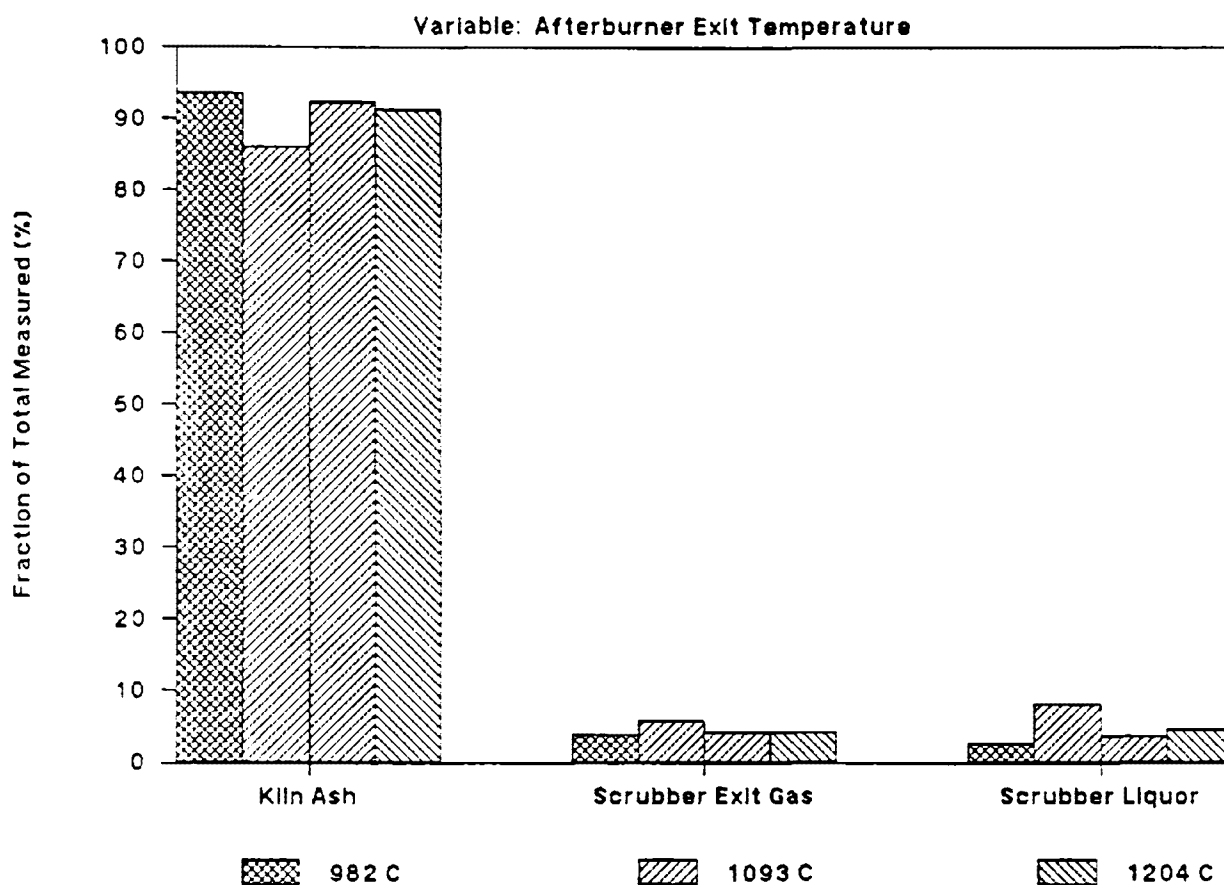


Figure 11b. Arsenic discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

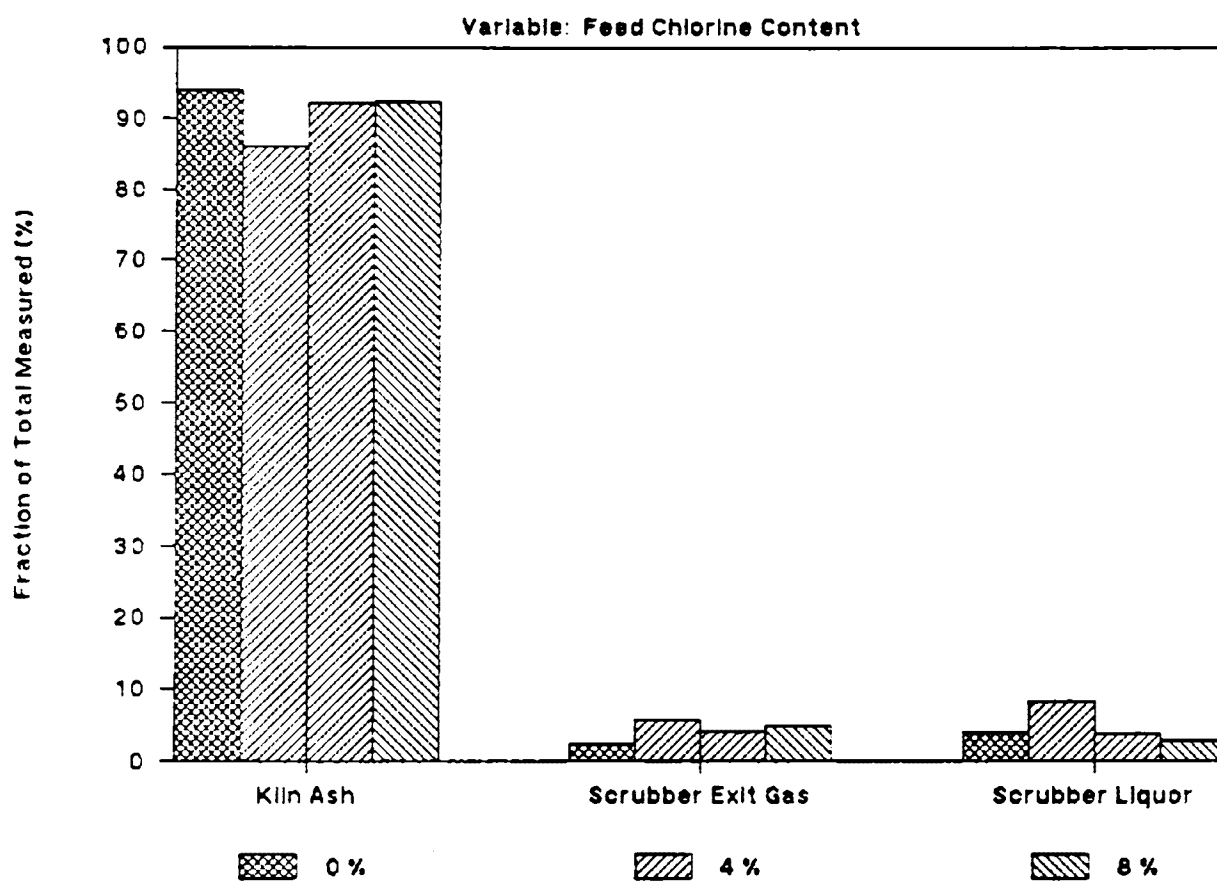


Figure 11c. Arsenic discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

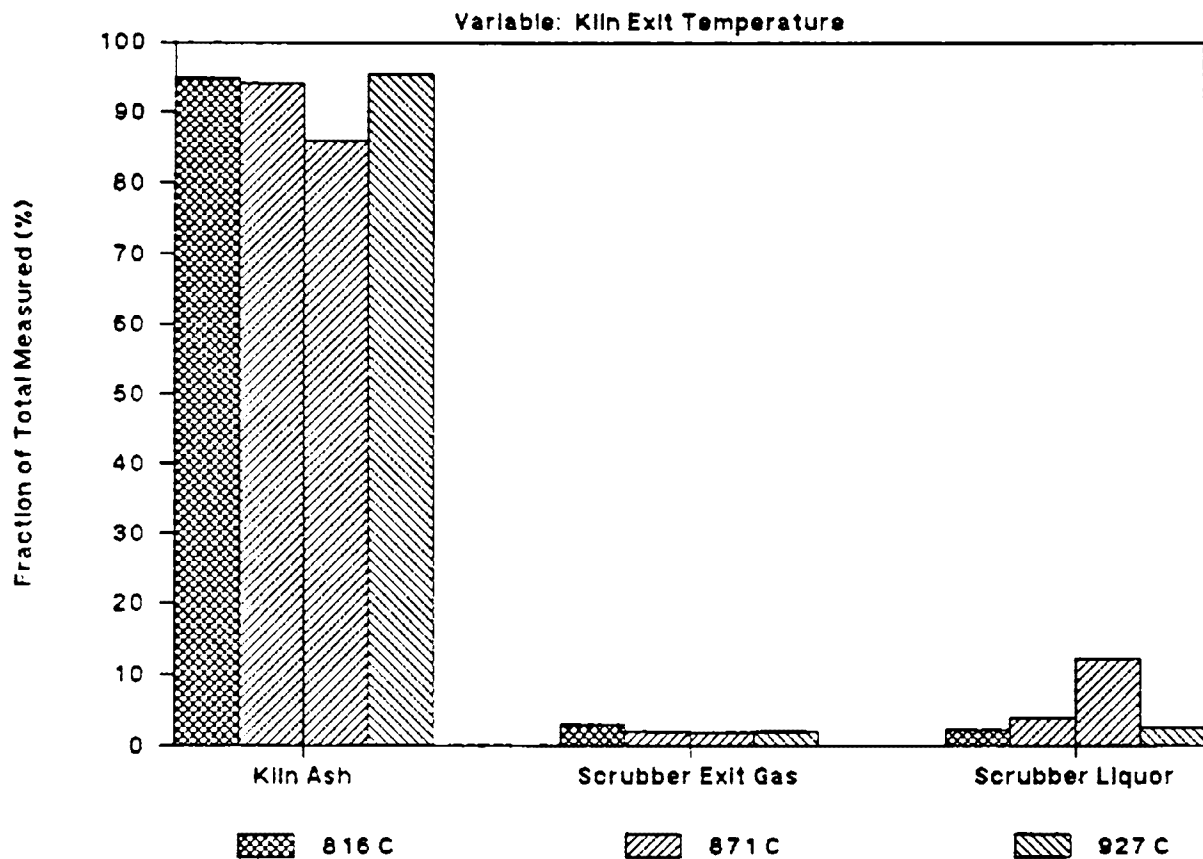


Figure 12a. Chromium discharge distributions for the parametric trace metal tests: effect of kiln temperature.

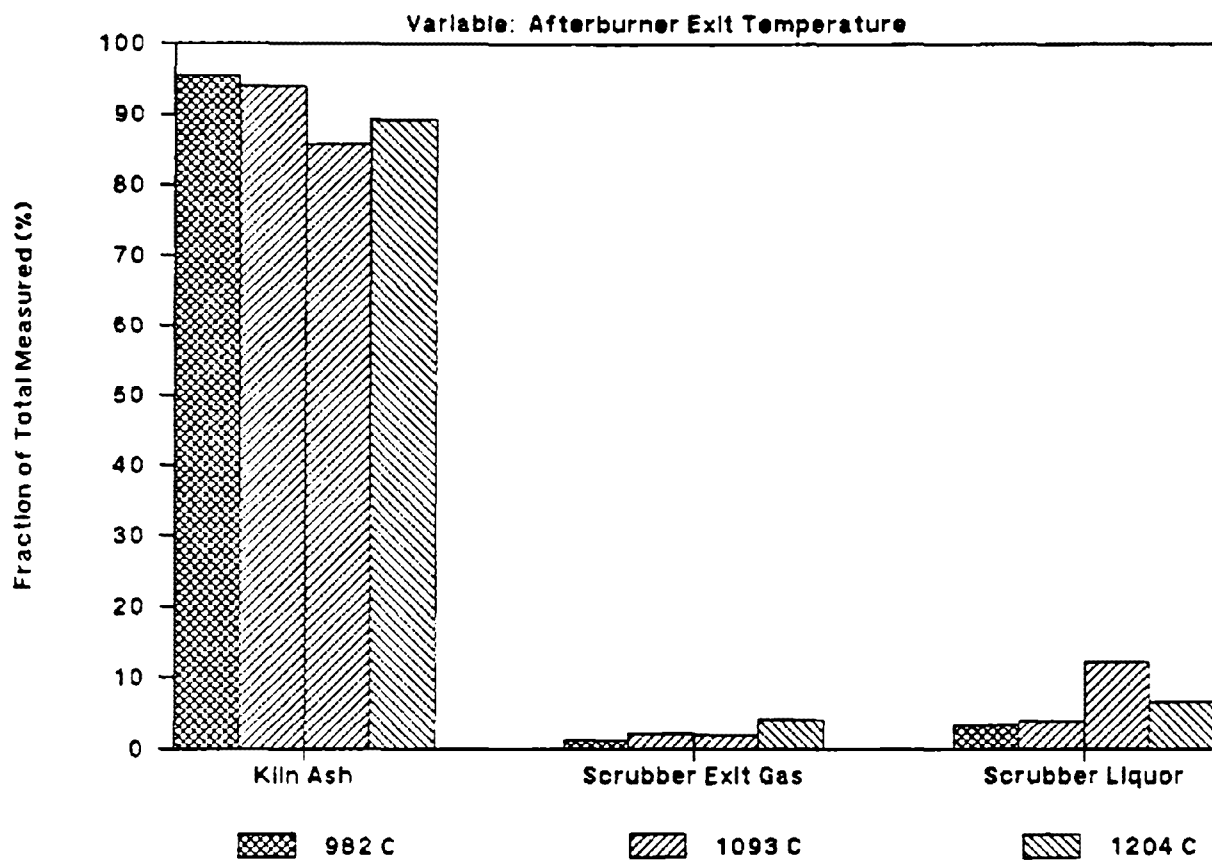


Figure 12b. Chromium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

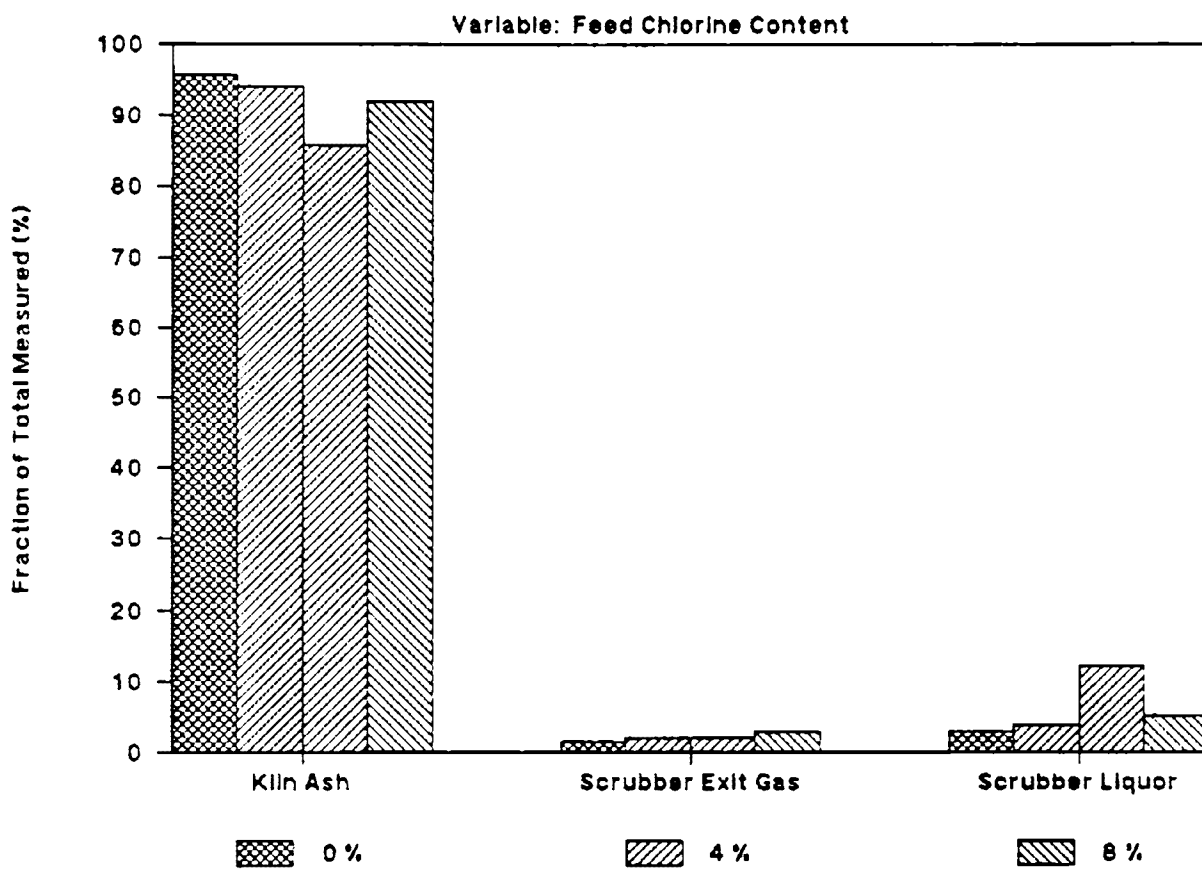


Figure 12c. Chromium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

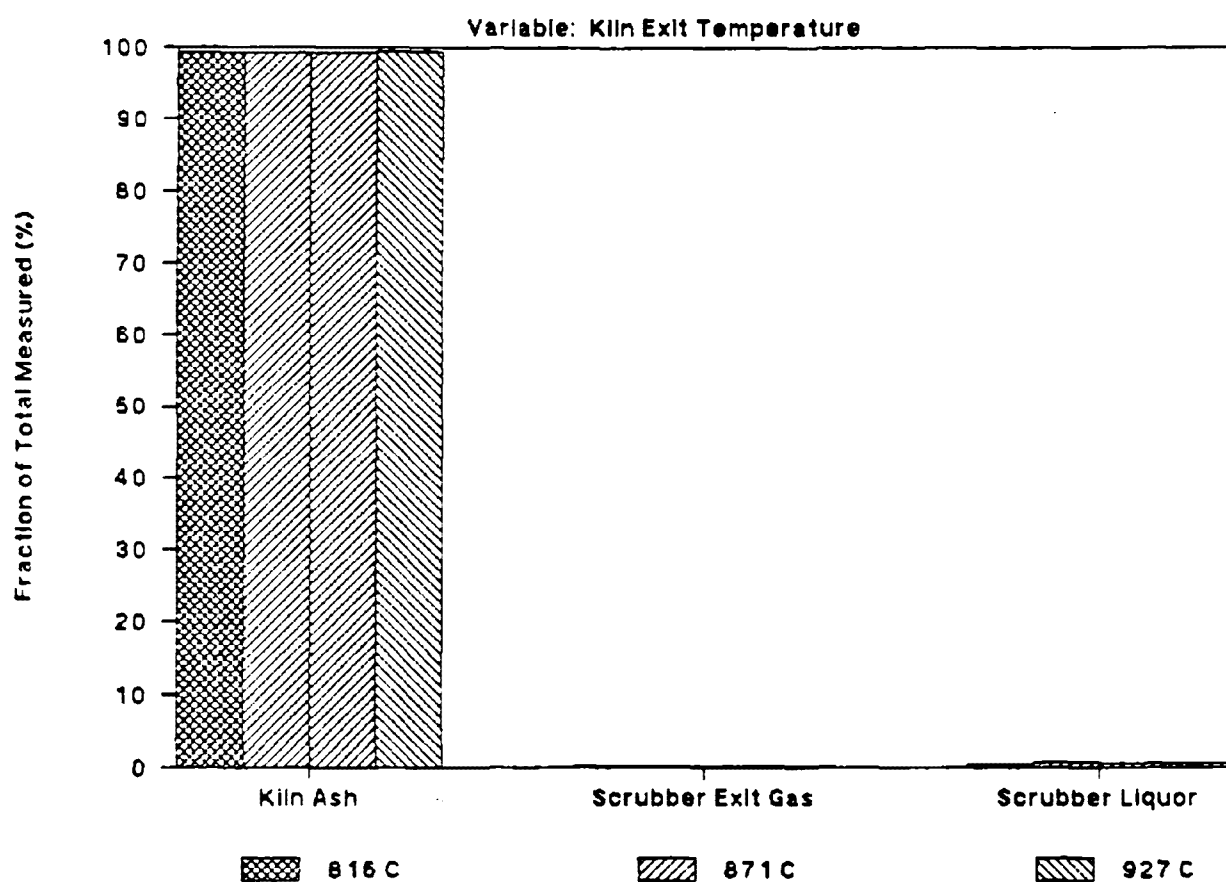


Figure 13a. Magnesium discharge distributions for the parametric trace metal tests: effect of kiln temperature.

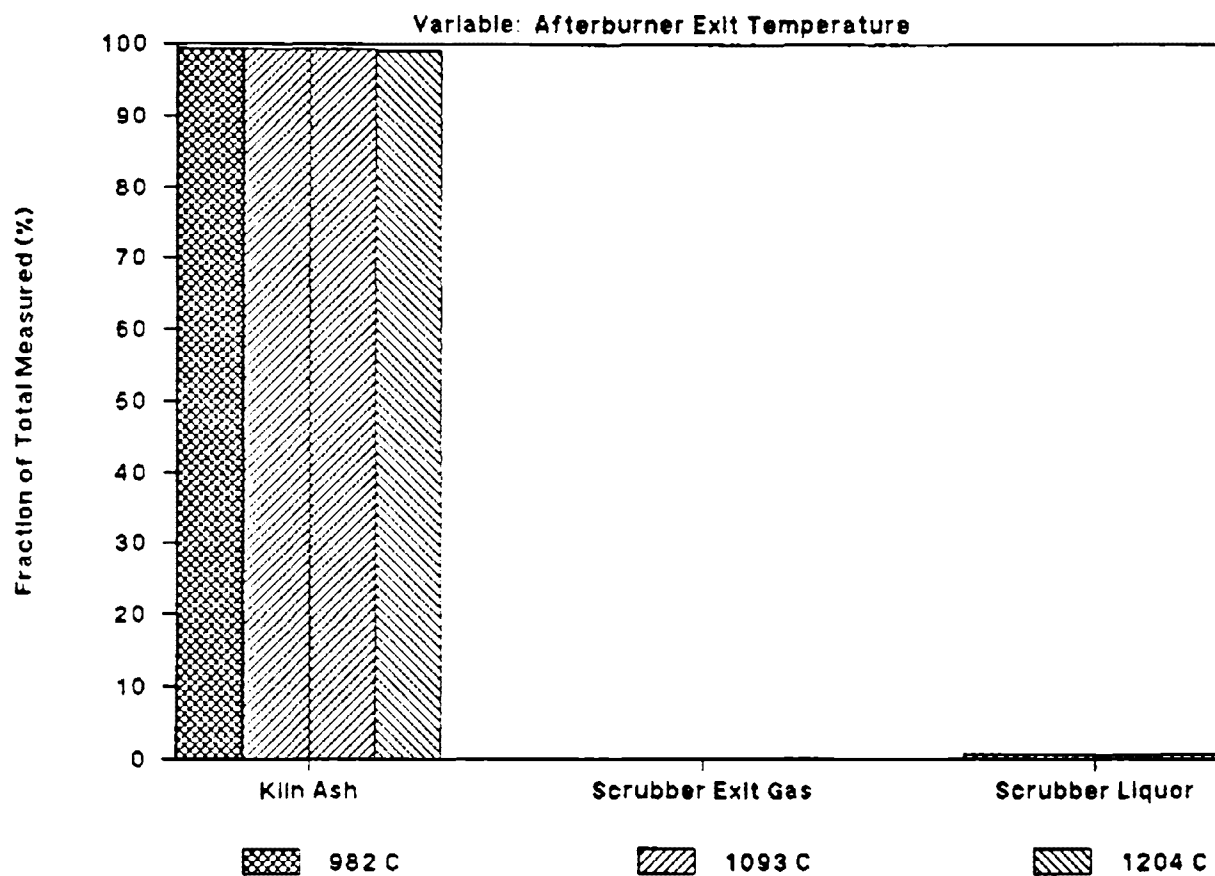


Figure 13b. Magnesium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

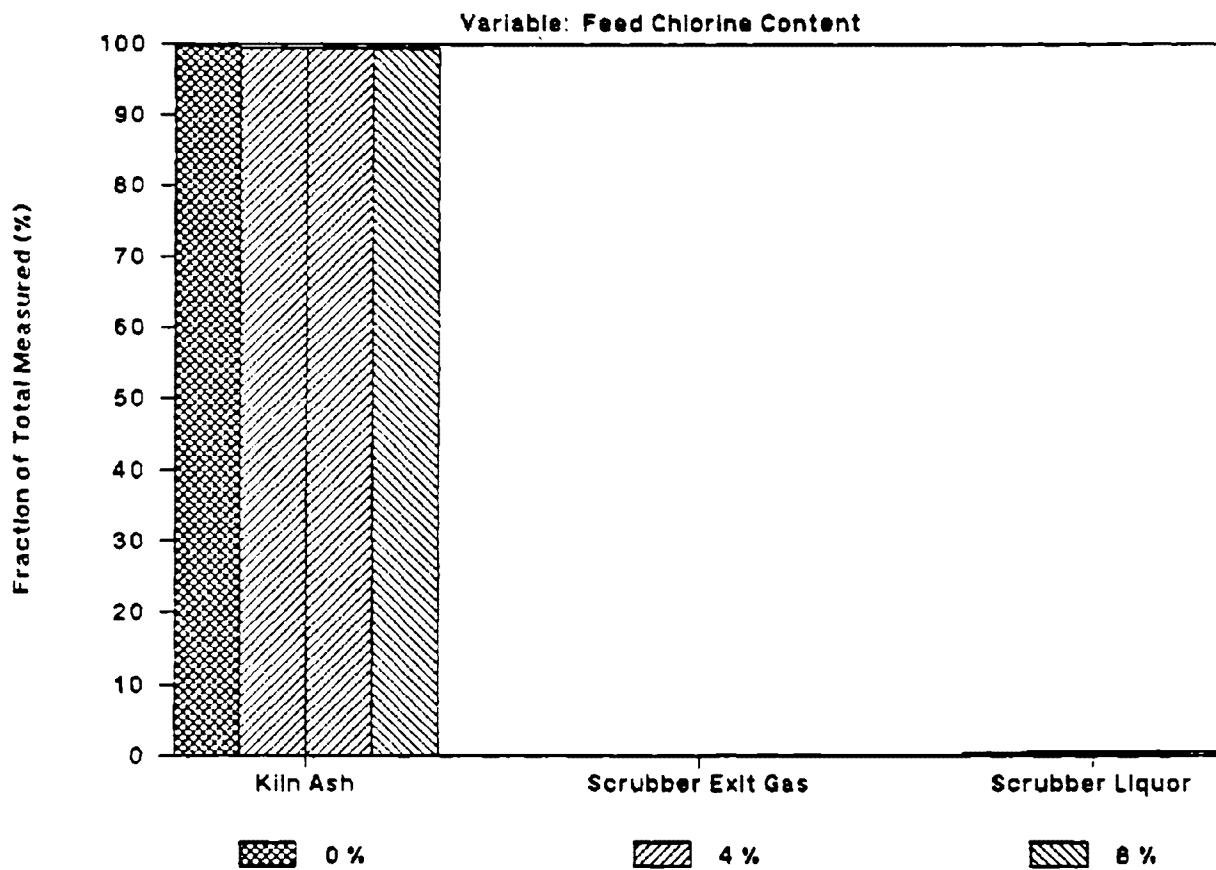


Figure 13c. Magnesium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

The following discussion focuses on each metal's discharge distribution data in turn.

Cadmium

The data in Figure 5 show that the kiln ash accounted for about 10 percent of the measured discharge cadmium provided there was chlorine in the feed mixture. The data suggest that the kiln ash fraction decreases slightly with increasing kiln temperature. A corresponding increase in the scrubber exit flue gas with a decrease in the scrubber liquor cadmium fraction seems to have occurred with increasing temperature, although the data show some variability. This behavior would be consistent with expectations. Increasing kiln temperature would be conducive to volatilizing more cadmium in the kiln. Any cadmium vaporized in the kiln would remain in the vapor phase through the afterburner, then condense as the flue gas was quenched. In accordance with the volatilization/condensation discussion in Section 4.4.2.1, scrubber efficiency would be expected to decrease. This would give rise to a greater scrubber exit flue gas fraction and a smaller scrubber liquor fraction.

No clear trend in any discharge stream's cadmium fraction, within the apparent data variability, is seen with varying afterburner temperatures.

The kiln ash cadmium fraction is clearly greater when no chlorine is in the feed than when chlorine is present. This would be expected if cadmium chlorides were more volatile than cadmium oxides.

Lead

The data in Figure 6 show that, as for cadmium, the kiln ash accounted for slightly greater than 10 percent of the measured discharge lead, provided there was chlorine in the feed. Again, there appears to be a slight decrease in kiln ash lead fraction with increasing kiln temperature, with a corresponding increase in scrubber exit flue gas fraction and a decrease in scrubber liquor fraction.

Interestingly, there appeared to be a decrease in scrubber exit flue gas lead fraction and an increase in scrubber liquor fraction with increasing afterburner temperature. This seems counter to expectations.

As with cadmium, the kiln ash lead fraction was substantially greater for the test with no feed chlorine than for the tests with 4 percent chlorine in the feed. This is consistent with expectations. The principal vapor phase lead species in a chlorine-free excess oxygen environment is expected to be lead metal with a volatility temperature of 627°C (1160°F). However, in a chlorine-containing environment, the principal vapor phase species becomes PbCl_4 with a volatility temperature of -15°C (5°F).

The data in Figure 6 show a further decrease in kiln ash lead fraction when feed chlorine content was further increased from 4 to 8 percent. Scrubber exit flue gas lead fraction monotonically increases with increased feed chlorine content. Scrubber efficiency correspondingly decreases.

Bismuth

The data in Figure 7 show bismuth behaves in a manner similar to cadmium and lead with respect to feed chlorine content. The kiln ash bismuth fraction was in the 20 to 35 percent range for all tests in which the feed contained chlorine. A marked increase in kiln ash bismuth

fraction (a decrease in bismuth volatility) occurred when chlorine was removed from the feed. A modest increase in scrubber efficiency for bismuth was also observed when feed chlorine was removed.

The data show no significant variation in any discharge stream bismuth fraction with changes in kiln or afterburner temperature.

Barium

The data in Figure 8 suggest that no test variable had a significant effect on barium discharge distributions.

Copper

The data in Figure 9 show that neither kiln nor afterburner temperatures had measurable effects on copper discharge distributions. However, copper kiln ash fractions clearly decreased monotonically with increasing feed chlorine content. Scrubber exit flue gas copper fractions showed a corresponding monotonic increase with increasing feed chlorine content. Both of these observations would be consistent with expectations if copper chlorides were more volatile than corresponding oxides.

Interestingly, scrubber liquor copper fractions may have increased slightly with increasing feed chlorine content. This suggests that the increased solubility of copper chlorides may have somewhat offset the partitioning of copper to finer particle size with increased copper volatilization, and thereby offset to some degree the expected decrease in scrubber collection efficiency.

Strontium

The data in Figure 10 show that neither kiln temperature nor feed chlorine content affect strontium discharge distributions. There was an apparent steady decrease in kiln ash strontium fraction with increasing afterburner temperature. This is coincidental, since afterburner temperature can have no effect on kiln ash fraction. The apparent steady increase in scrubber liquor strontium fraction is most likely similarly coincidental.

Scrubber exit flue gas strontium fraction appeared to increase slightly with increasing afterburner temperature. Although this may also have been coincidental, it would be expected if higher afterburner temperature caused an increase in the amount of strontium in entrained flyash from the kiln which vaporized in the afterburner.

Arsenic

The data in Figure 11 show no effect of afterburner temperature on arsenic discharge distributions. A slight decrease in kiln ash fraction, accompanied by a slight increase in scrubber exit flue gas fraction, with increasing kiln temperature appears to have occurred. A decrease in scrubber efficiency was observed with increasing feed chlorine content.

Chromium and Magnesium

The data in Figures 12 and 13 show that no test variable measurably affected chromium or magnesium discharge distributions.

Table 38 summarizes the distribution of each metal between the particulate phase and the vapor/dissolved phase in the afterburner and scrubber exit flue gas for each test. The data in Table 38 represent the individual test data comprising the ranges and averages noted in Table 34. Again, distributions affected by samples with nondetectable levels of a metal are represented by ranges. The range boundaries reflect the assumption that the samples in question contained zero and the detection limit of the metal, respectively. The data in Table 38 show no consistent relationship between particulate/vapor-dissolved phase ratios and test variables.

4.4.2.3 Afterburner Exit Particle Size Distributions

Recall from the sampling and analysis protocol discussion in Section 3, that the flue gas at the afterburner exit was sampled with a sampling train designed to collect a large (~1g) sample of particulate so that this particulate could be size fractionated and trace metal concentrations as a function of particle size could be determined. Sufficient afterburner exit flue gas particulate was collected by the sampling train to allow particle size fractionation to be performed for six of the eight tests performed. Size fractionation was not possible for the particulate catch from tests 5 and 10.

Figure 14 shows a log-probability plot of the total particulate particle size distribution data for the six tests for which size fractionation was performed. As indicated, the size distributions for all tests were roughly log normal. The distributions were roughly comparable for the five tests in which the test waste feed contained chlorine. The size distribution of total particulate was shifted to larger particle size for the one test with no feed chlorine. This would be as expected if the presence of chlorine in the feed served to increase the volatility of feed inorganic constituents. In such a case the recondensation of volatilized inorganic would tend to result in a finer particle size distribution for the chlorine containing feed cases.

Particle size fractions were combined to give four size fraction samples for trace metal analysis. These size fractions were nominally <2, 2 to 4, 4 to 10, and >10 μm respectively. From the analyses of these size fractions, size distributions of each of the test trace metals were obtained.

Figure 15 shows the average size distribution for the five hazardous constituent trace metals included in the test waste mixture (arsenic, barium, cadmium, chromium, and lead) and compares these to the total particulate size distribution. The size distributions shown in Figure 15 represent averages over the six tests for which size distribution analyses were performed. Figure 16 shows analogous size distributions for the four non-hazardous trace metals included in the test waste mixture (bismuth, copper, magnesium, and strontium).

The data in Figures 15 and 16 show that the average size distributions for chromium, copper, magnesium, and strontium were coarser (shifted to larger particle sizes) than the overall particulate size distribution. These are all relatively nonvolatile metals with high volatility temperatures (See Tables 31 and 32). The average size distributions for barium and bismuth were finer (shifted to smaller particle sizes) than the overall particulate. These two are more volatile, having lower volatility temperature than chromium, copper, magnesium, and strontium.

Figures 17 through 22 show individual test metal size distributions for both the hazardous constituent and non-hazardous constituent trace metals.

TABLE 38. PHASE DISTRIBUTION OF FLUE GAS METALS IN THE AFTERBURNER AND SCRUBBER EXIT FLUE GAS FOR THE TRACE METALS PARAMETRIC TEST SERIES

| | | | | | | | | | | | | |
|-----------------------|---------------------------------|-------|-------|-------|-----------------------------------|-------|-------|-------|-------------------------------------|-------|-------|-------|
| Test: | 5 | 7 | 10 | 6 | 9 | 7 | 10 | 8 | 4 | 7 | 10 | 11 |
| Primary variable: | Kiln exit temperature (°C) | | | | Afterburner exit temperature (°C) | | | | Feed chlorine content (wt %) | | | |
| Target: | 816 | 871 | 871 | 927 | 982 | 1093 | 1093 | 1204 | 0 | 4 | 4 | 8 |
| Test average: | 825 | 878 | 873 | 928 | 983 | 1088 | 1094 | 1196 | 0.0 | 3.8 | 4.6 | 8.3 |
| Held constant: | AB exit = 1093°C; chlorine = 4% | | | | Kiln exit = 871°C; chlorine = 4% | | | | Kiln exit = 871°C; AB exit = 1093°C | | | |
| Test average: | 1071 | 1088 | 1094 | 1092 | 875 | 878 | 873 | 871 | 874 | 878 | 873 | 870 |
| Test average: | 3.7 | 3.8 | 4.6 | 4.2 | 3.4 | 3.8 | 4.6 | 3.6 | 1093 | 1088 | 1094 | 1092 |
| % | | | | | | | | | | | | |
| Arsenic | | | | | | | | | | | | |
| Afterburner exit: | | | | | | | | | | | | |
| Particulate | -- | 91 | -- | 91 | >97 | 91 | -- | 94 | 47 | 91 | -- | 65 |
| Vapor/dissolved phase | -- | 9 | -- | 9 | <3 | 9 | -- | 6 | 53 | 9 | -- | 35 |
| Scrubber exit: | | | | | | | | | | | | |
| Particulate | >76 | >72 | >53 | 81-95 | >74 | >72 | >53 | >74 | 32 | >72 | >53 | >78 |
| Vapor/dissolved phase | <24 | <28 | <47 | 5-19 | <26 | <28 | <47 | <26 | 68 | <28 | <47 | <22 |
| Barium | | | | | | | | | | | | |
| Afterburner exit: | | | | | | | | | | | | |
| Particulate | -- | 3 | -- | 1 | 3 | 3 | -- | 1 | 6-10 | 3 | -- | 3 |
| Vapor/dissolved phase | -- | 97 | -- | 99 | 97 | 97 | -- | 99 | 90-94 | 97 | -- | 97 |
| Scrubber exit: | | | | | | | | | | | | |
| Particulate | 55 | 26-29 | 10-27 | 31-33 | 25-29 | 26-29 | 10-27 | 32-35 | 25 | 26-29 | 10-27 | 27 |
| Vapor/dissolved phase | 45 | 71-74 | 73-90 | 67-69 | 71-75 | 71-74 | 73-90 | 65-68 | 75 | 71-74 | 73-90 | 73 |
| Bismuth | | | | | | | | | | | | |
| Afterburner exit: | | | | | | | | | | | | |
| Particulate | -- | 17 | -- | 31 | 37 | 17 | -- | 12-20 | 23 | 17 | -- | 9 |
| Vapor/dissolved phase | -- | 83 | -- | 69 | 63 | 83 | -- | 80-88 | 77 | 83 | -- | 91 |
| Scrubber exit: | | | | | | | | | | | | |
| Particulate | 90-94 | 83 | 70-76 | 86-93 | 73 | 83 | 70-76 | 86 | >75 | 83 | 70-76 | 93-99 |
| Vapor/dissolved phase | 6-10 | 17 | 24-30 | 7-14 | 27 | 17 | 24-30 | 14 | <25 | 17 | 24-30 | 1-7 |

(continued)

TABLE 38. (continued)

| | | | | | | | | | | | | |
|-----------------------|---------------------------------|------|-------|-------|-----------------------------------|------|-------|------|-------------------------------------|------|-------|------|
| Test: | 5 | 7 | 10 | 6 | 9 | 7 | 10 | 8 | 4 | 7 | 10 | 11 |
| Primary variable: | Kiln exit temperature (°C) | | | | Afterburner exit temperature (°C) | | | | Feed chlorine content (wt %) | | | |
| Target: | 816 | 871 | 871 | 927 | 982 | 1093 | 1093 | 1204 | 0 | 4 | 4 | 8 |
| Test average: | 825 | 878 | 873 | 928 | 983 | 1088 | 1094 | 1196 | 0.0 | 3.8 | 4.6 | 8.3 |
| Held constant: | AB exit = 1093°C; chlorine = 4% | | | | Kiln exit = 871°C; chlorine = 4% | | | | Kiln exit = 871°C; AB exit = 1093°C | | | |
| Test average: | 1071 | 1088 | 1094 | 1092 | 875 | 878 | 873 | 871 | 874 | 878 | 873 | 870 |
| Test average: | 3.7 | 3.8 | 4.6 | 4.2 | 3.4 | 3.8 | 4.6 | 3.6 | 1093 | 1088 | 1094 | 1092 |
| % | | | | | | | | | | | | |
| Cadmium | | | | | | | | | | | | |
| Afterburner exit: | | | | | | | | | | | | |
| Particulate | -- | 57 | -- | 92 | 73 | 57 | -- | 82 | 68 | 57 | -- | 82 |
| Vapor/dissolved phase | -- | 43 | -- | 8 | 27 | 43 | -- | 18 | 32 | 43 | -- | 18 |
| Scrubber exit: | | | | | | | | | | | | |
| Particulate | 84-89 | > 94 | > 86 | 87 | 95-99 | > 94 | > 86 | > 94 | > 82 | > 94 | > 86 | > 95 |
| Vapor/dissolved phase | 10-16 | < 6 | < 14 | 13 | 1-5 | < 6 | < 14 | < 6 | < 18 | < 6 | < 14 | < 5 |
| Chromium | | | | | | | | | | | | |
| Afterburner exit: | | | | | | | | | | | | |
| Particulate | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| Vapor/dissolved phase | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| Scrubber exit: | | | | | | | | | | | | |
| Particulate | 79-84 | > 82 | 68-90 | 81-92 | 70 | > 82 | 68-90 | 87 | 77-94 | > 82 | 68-90 | > 85 |
| Vapor/dissolved phase | 16-21 | < 18 | 10-32 | 8-19 | 30 | < 18 | 10-32 | 13 | 6-23 | < 18 | 10-32 | < 15 |
| Copper | | | | | | | | | | | | |
| Afterburner exit: | | | | | | | | | | | | |
| Particulate | -- | 58 | -- | 80 | 55 | 58 | -- | 77 | 29 | 58 | -- | 72 |
| Vapor/dissolved phase | -- | 42 | -- | 20 | 45 | 42 | -- | 23 | 71 | 42 | -- | 28 |
| Scrubber exit: | | | | | | | | | | | | |
| Particulate | 97 | 97 | 45 | 87 | 80 | 97 | 45 | 89 | 51-57 | 97 | 45 | 99 |
| Vapor/dissolved phase | 3 | 3 | 55 | 13 | 20 | 3 | 55 | 11 | 43-49 | 3 | 55 | 1 |

(continued)

TABLE 38. (concluded)

| | | | | | | | | | | | | |
|-----------------------|---------------------------------|-------|------|-------|-----------------------------------|-------|------|-------|-------------------------------------|-------|------|-------|
| Test: | 5 | 7 | 10 | 6 | 9 | 7 | 10 | 8 | 4 | 7 | 10 | 11 |
| Primary variable: | Kiln exit temperature (°C) | | | | Afterburner exit temperature (°C) | | | | Feed chlorine content (wt %) | | | |
| Target: | 816 | 871 | 871 | 927 | 982 | 1093 | 1093 | 1204 | 0 | 4 | 4 | 8 |
| Test average: | 825 | 878 | 873 | 928 | 983 | 1088 | 1094 | 1196 | 0.0 | 3.8 | 4.6 | 8.3 |
| Held constant: | AB exit = 1093°C; chlorine = 4% | | | | Kiln exit = 871°C; chlorine = 4% | | | | Kiln exit = 871°C; AB exit = 1093°C | | | |
| Test average: | 1071 | 1088 | 1094 | 1092 | 875 | 878 | 873 | 871 | 874 | 878 | 873 | 870 |
| Test average: | 3.7 | 3.8 | 4.6 | 4.2 | 3.4 | 3.8 | 4.6 | 3.6 | 1093 | 1088 | 1094 | 1092 |
| % | | | | | | | | | | | | |
| Lead | | | | | | | | | | | | |
| Afterburner exit: | | | | | | | | | | | | |
| Particulate | — | 28 | — | 76 | 54 | 28 | — | 66 | 26 | 28 | — | 71 |
| Vapor/dissolved phase | — | 72 | — | 24 | 46 | 72 | — | 34 | 74 | 72 | — | 29 |
| Scrubber exit: | | | | | | | | | | | | |
| Particulate | 90 | > 99 | 93 | 90 | > 99 | > 99 | 93 | > 99 | > 95 | > 99 | 93 | > 99 |
| Vapor/dissolved phase | 10 | < 1 | 7 | 10 | < 1 | < 1 | 7 | < 1 | < 5 | < 1 | 7 | < 1 |
| Magnesium | | | | | | | | | | | | |
| Afterburner exit: | | | | | | | | | | | | |
| Particulate | — | 69 | — | 70 | 91 | 69 | — | 84 | 57 | 69 | — | 76 |
| Vapor/dissolved phase | — | 31 | — | 30 | 9 | 31 | — | 16 | 43 | 31 | — | 24 |
| Scrubber exit: | | | | | | | | | | | | |
| Particulate | 93 | 73 | 25 | 82-88 | 62 | 73 | 25 | 71 | 65 | 73 | 25 | 72 |
| Vapor/dissolved phase | 7 | 27 | 75 | 12-18 | 38 | 27 | 75 | 29 | 35 | 27 | 75 | 28 |
| Strontium | | | | | | | | | | | | |
| Afterburner exit: | | | | | | | | | | | | |
| Particulate | — | 41 | — | 41 | 58 | 41 | — | 19-25 | 8-34 | 41 | — | 25-32 |
| Vapor/dissolved phase | — | 59 | — | 59 | 42 | 59 | — | 75-81 | 66-92 | 59 | — | 68-75 |
| Scrubber exit: | | | | | | | | | | | | |
| Particulate | 32 | 79-85 | 47 | 89-98 | > 91 | 79-85 | 47 | 85-89 | 74-80 | 79-85 | 47 | 76-86 |
| Vapor/dissolved phase | 68 | 15-21 | 53 | 2-11 | < 9 | 15-21 | 53 | 11-15 | 20-26 | 15-21 | 53 | 14-24 |

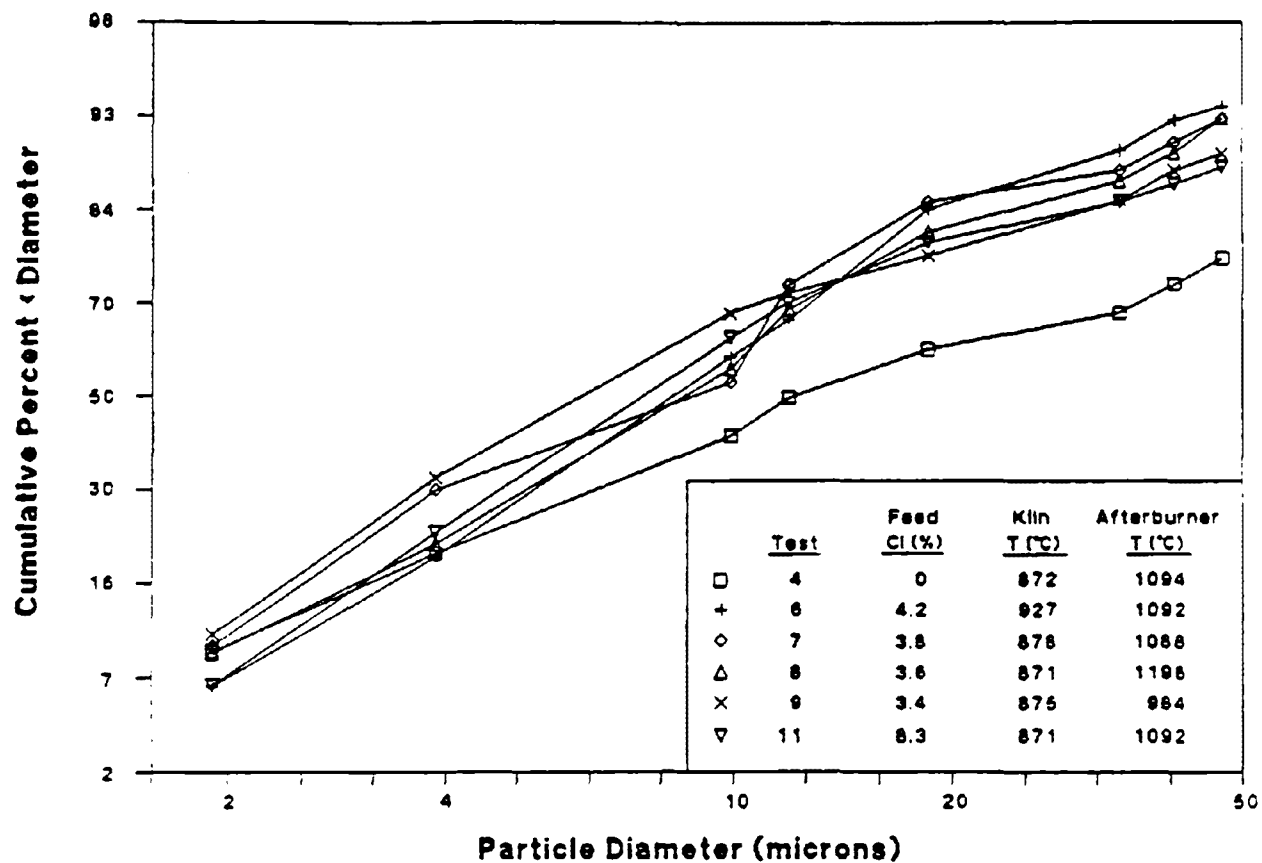


Figure 14. Afterburner exit flue gas particulate size distributions for the parametric trace metal tests.

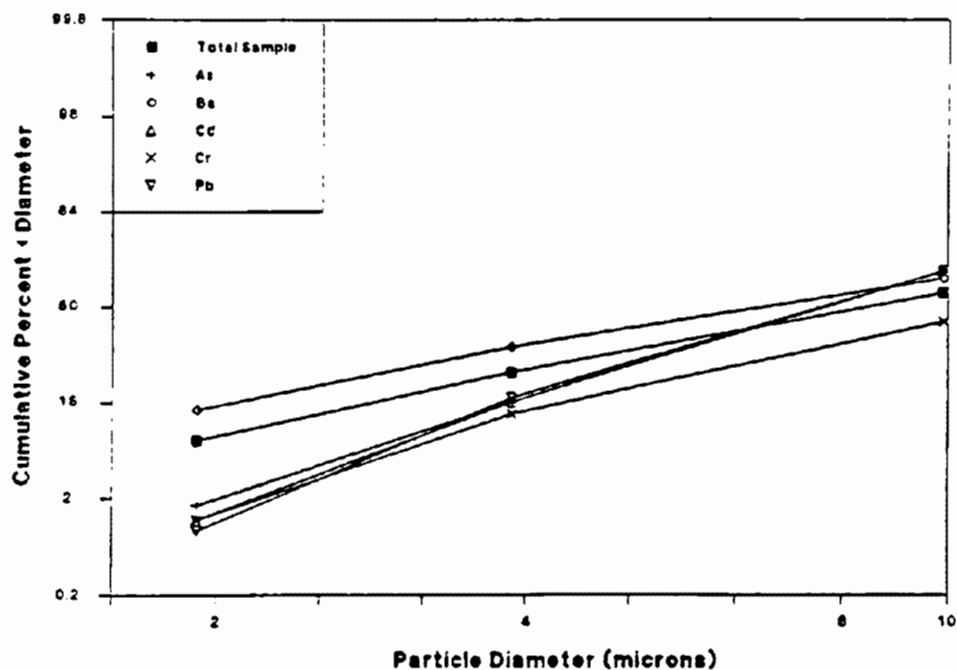


Figure 15. Hazardous constituent trace metal size distributions in afterburner exit flue gas particulate for the parametric trace metal tests.

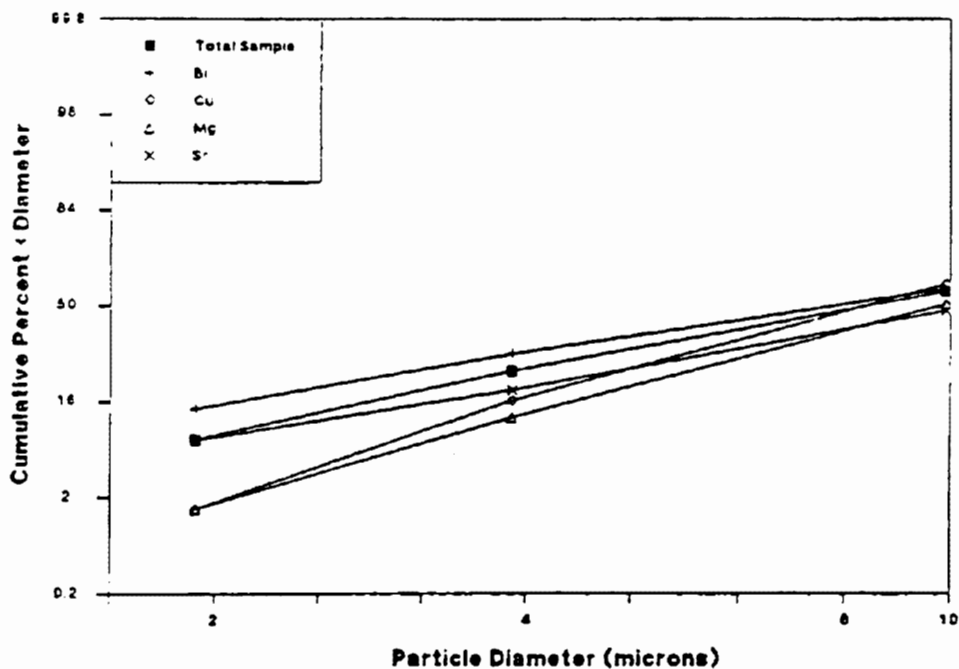


Figure 16. Nonhazardous trace metal size distributions in afterburner exit flue gas particulate for the parametric trace metal tests.

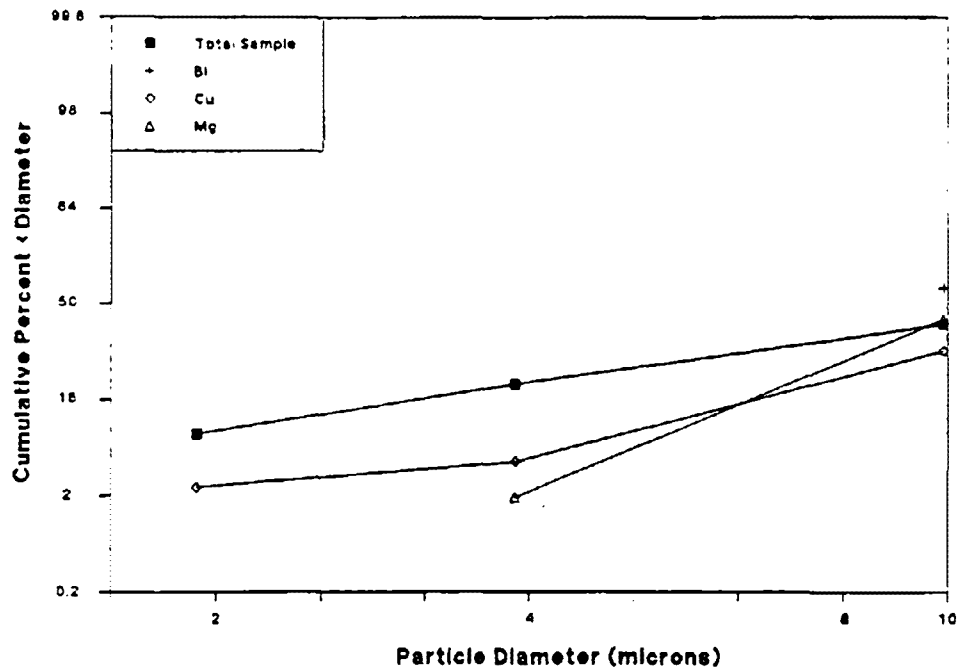
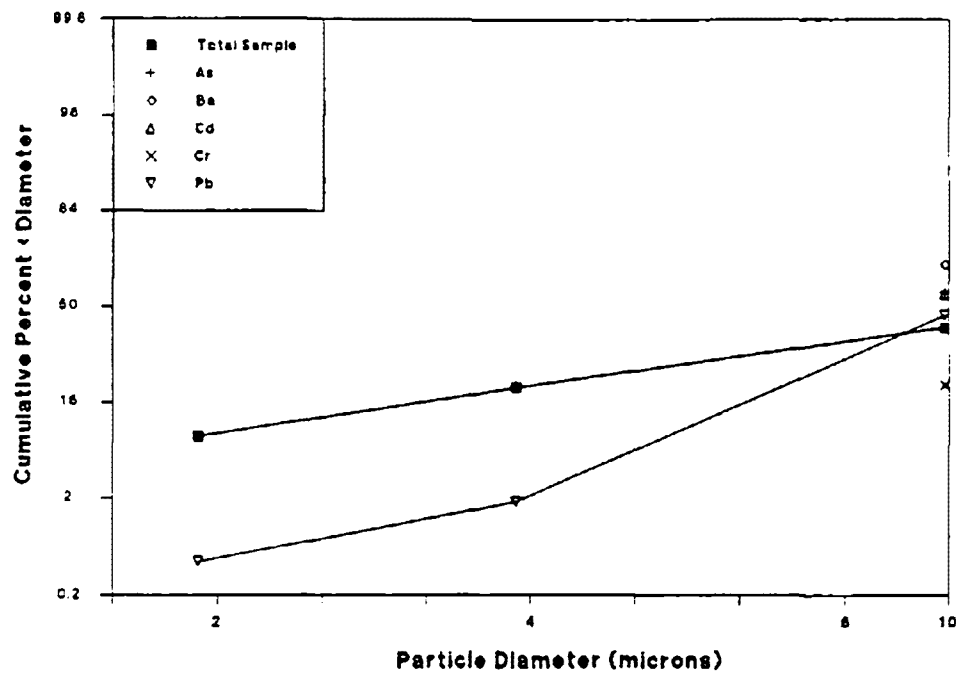


Figure 17. Trace metal size distributions in afterburner exit flue gas for Test 4.

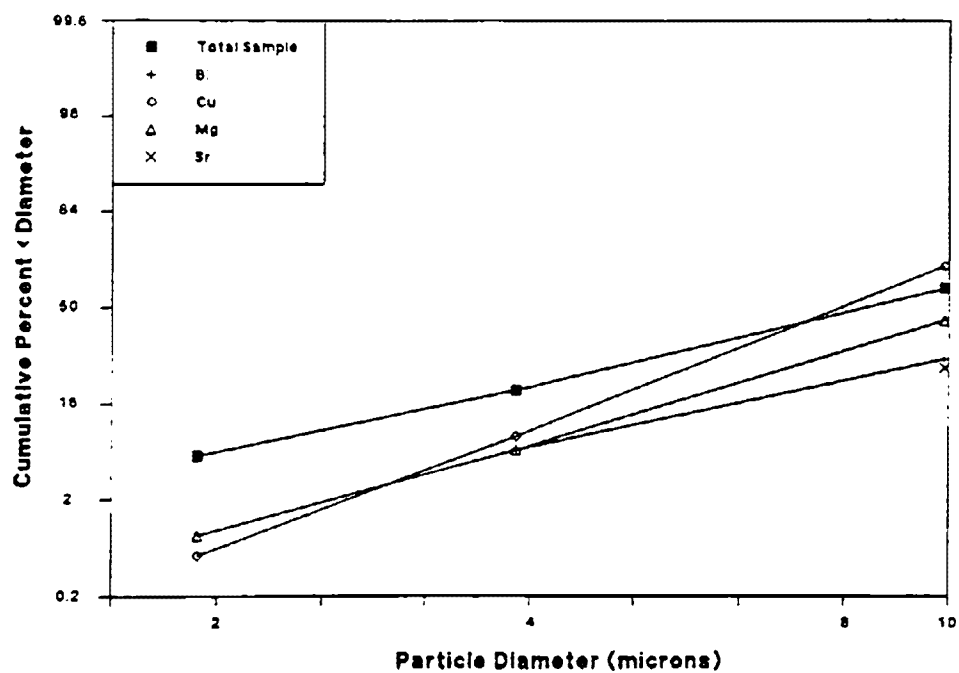
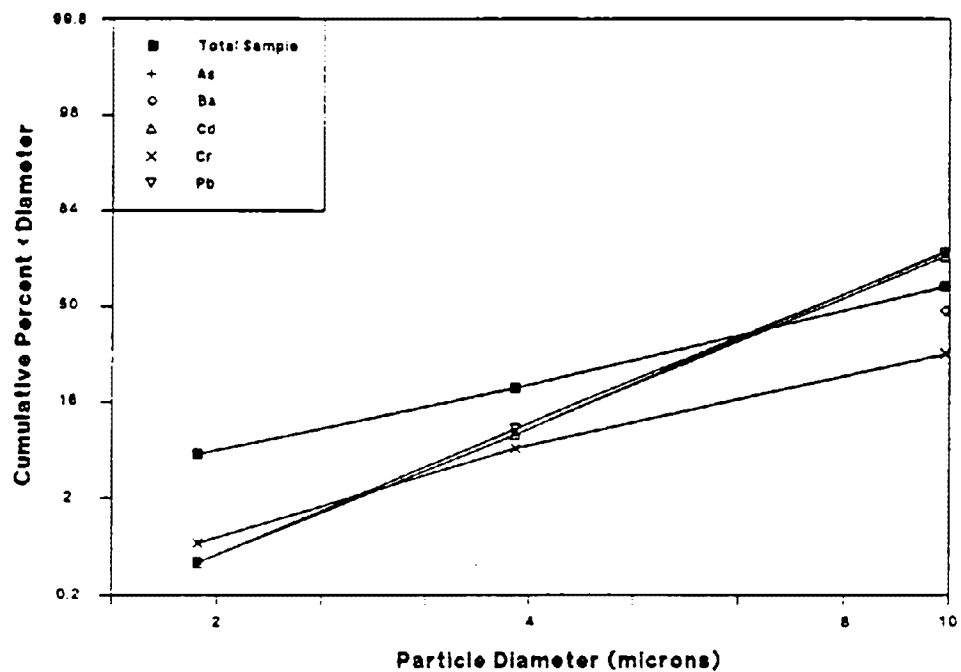


Figure 18. Trace metal size distributions in afterburner exit flue gas for Test 6.

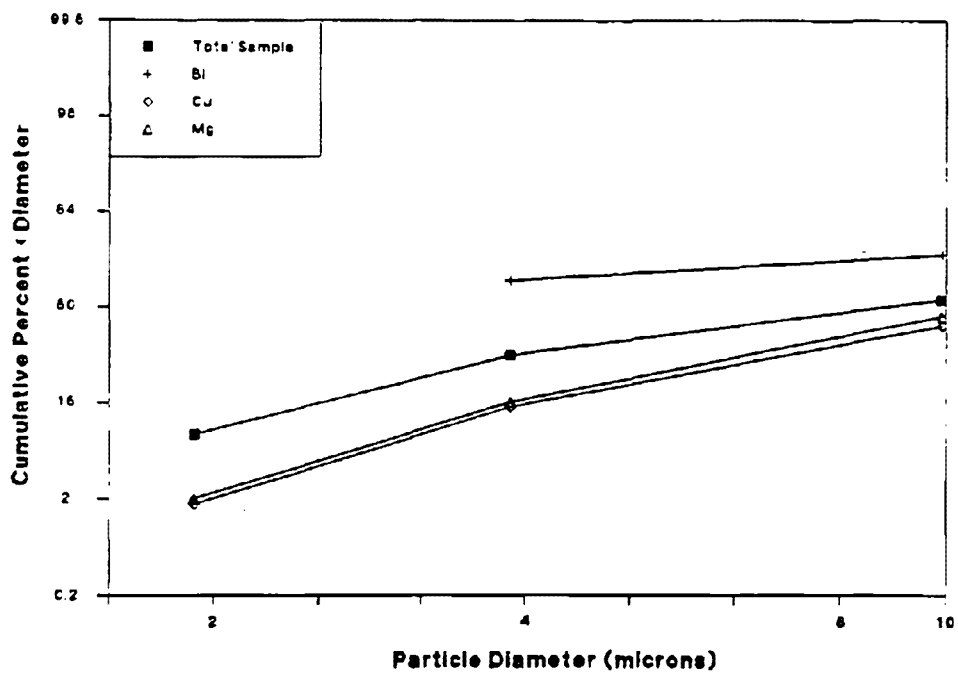
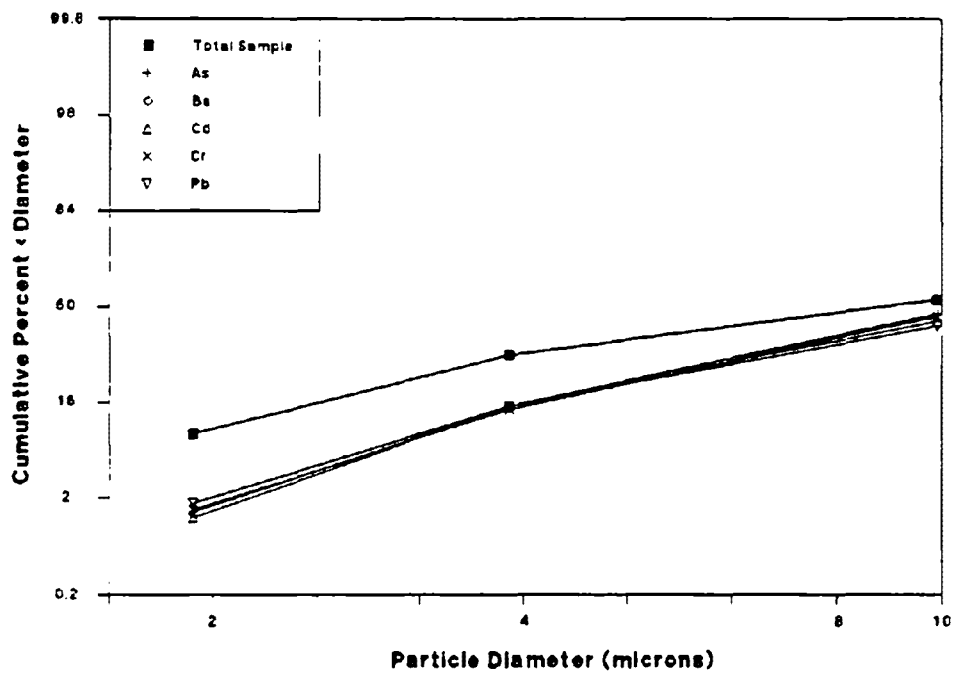


Figure 19. Trace metal size distributions in afterburner exit flue gas for Test 7.

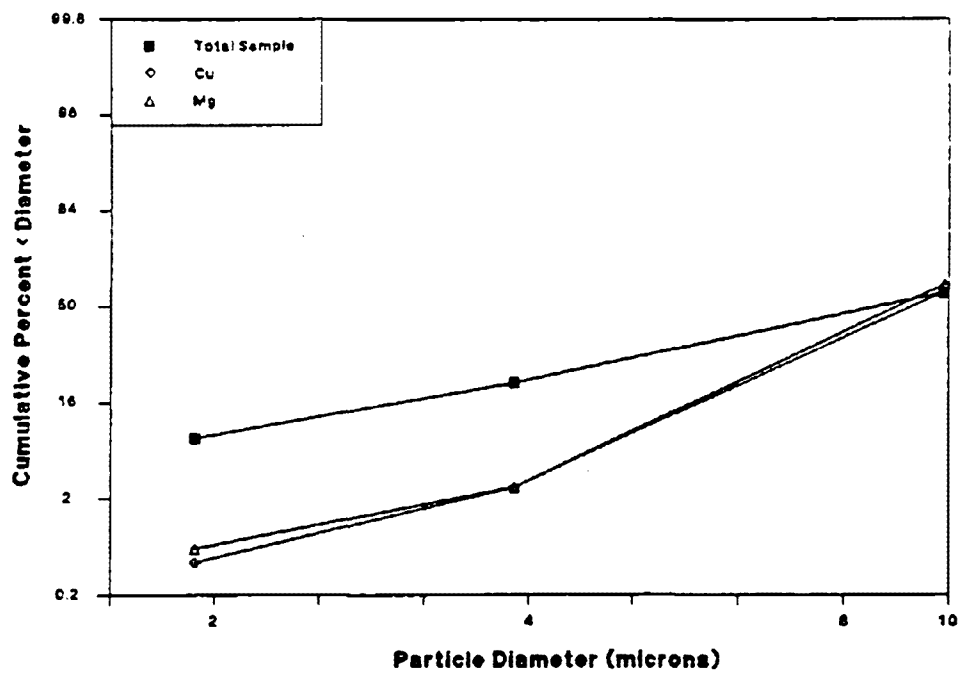
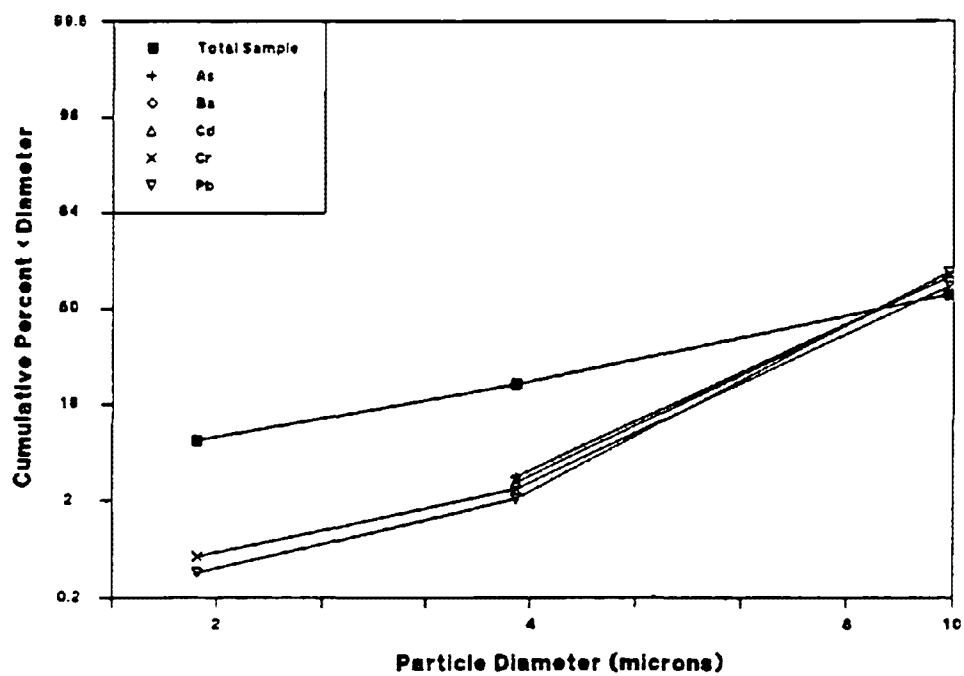


Figure 20. Trace metal size distributions in afterburner exit flue gas for Test 8.

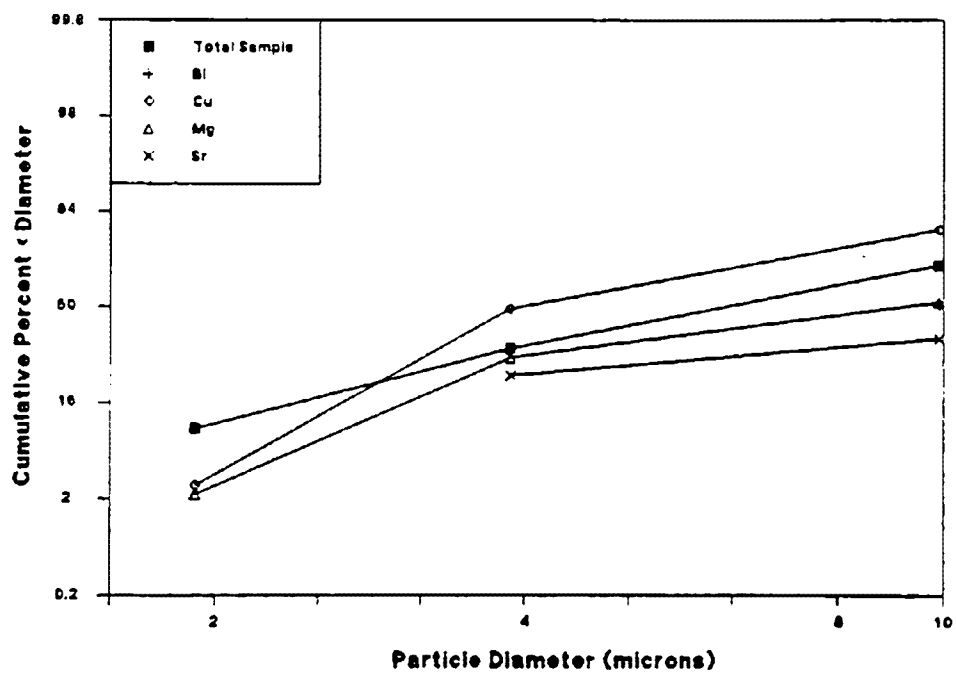
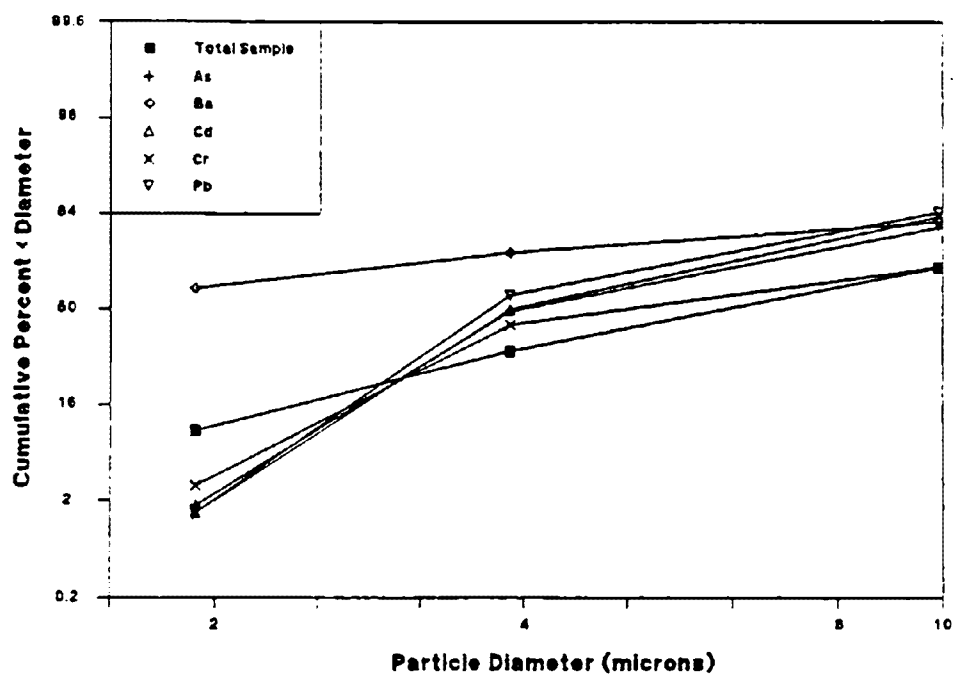


Figure 21. Trace metal size distributions in afterburner exit flue gas for Test 9.

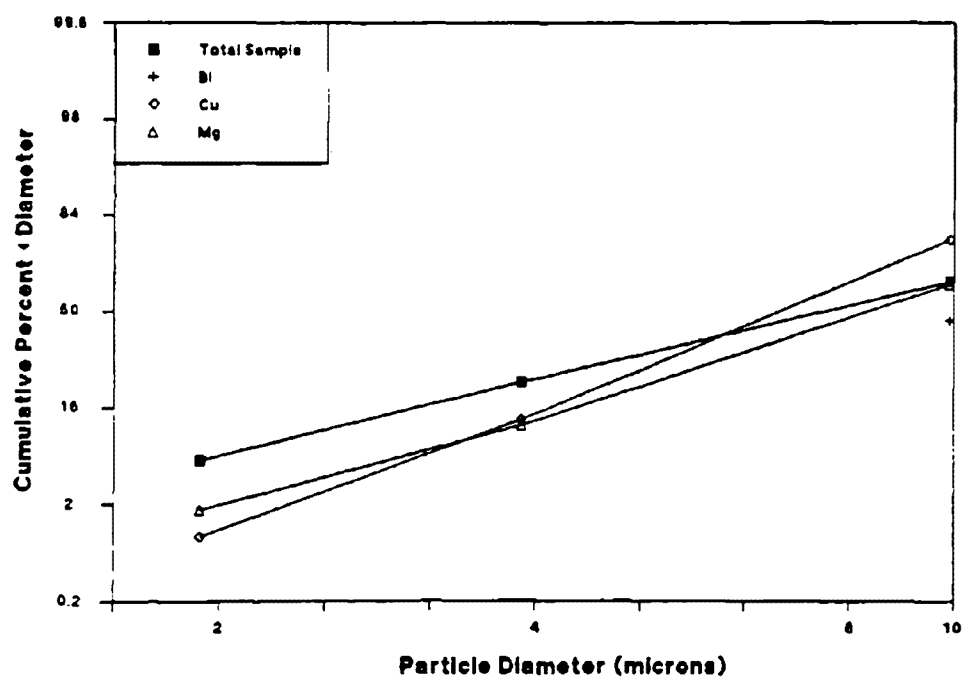
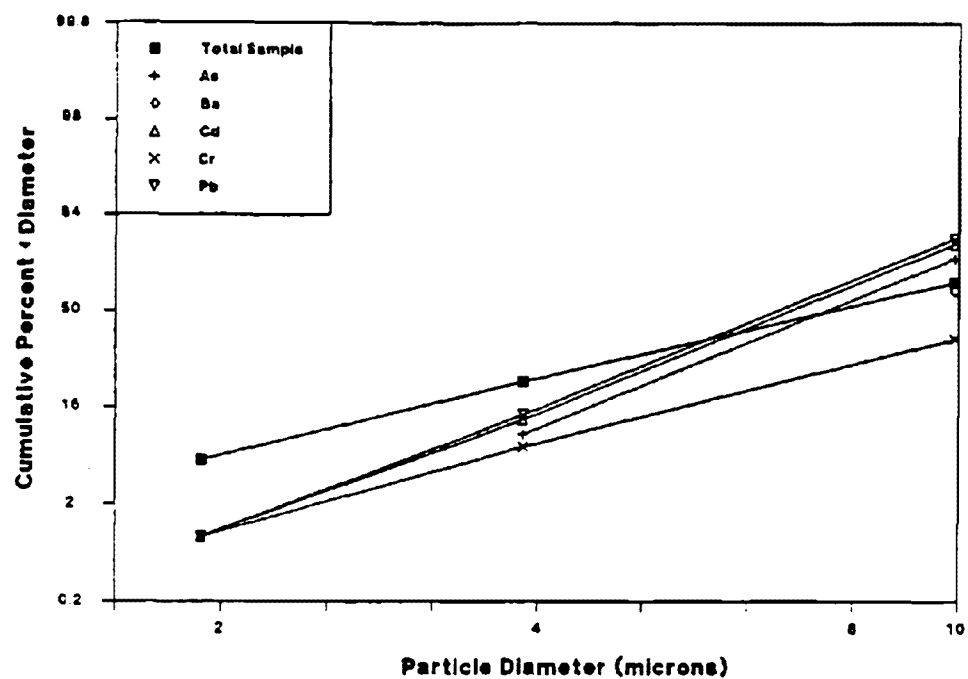


Figure 22. Trace metal size distributions in afterburner exit flue gas for Test 11.

4.5 POHC DESTRUCTION AND REMOVAL EFFICIENCIES

The three compounds introduced in the synthetic waste feed as principal organic hazardous constituents (POHCs) for these tests were toluene, chlorobenzene, and tetrachloroethylene. Flue gas concentrations of the compounds measured at the three locations sampled (the afterburner exit, the scrubber system exit, and the stack (three tests)) during the parametric trace metal tests are summarized in Table 39. The data in the table show that POHC concentrations were generally decreased in the scrubber exit flue gas compared to the afterburner exit flue gas; although toluene and chlorobenzene concentrations increased for Tests 5, 6, and 9. Reduced concentrations are expected due to some combination of flue gas dilution via air inleakage and POHC removal in the scrubber system. The increased concentrations observed for Tests 5, 6, and 9 are possibly related to a sampling artifact in either the afterburner or scrubber exit sampling trains.

Table 40 combines the flue gas concentration data from Table 39 with the flue gas flowrate data in the sampling sheets in Appendix C, the synthetic liquid waste feed composition data from Table 17, and the liquid waste feedrate data from Table 4, to give the POHC DREs at the three locations sampled. As shown in the table, POHC DREs in the stack ranged from 99.9975 to 99.9994 percent. With the exception of the afterburner exit for Test 11, POHC DREs were above 99.99 percent at all locations for all tests. For Test 11 with 8.4 percent waste chlorine feed, the POHC DREs were uniformly lower than the other tests (with lower feed chlorine contents) at all three sampling locations.

4.6 VOLATILE PRODUCTS OF INCOMPLETE COMBUSTION (PICs)

As noted in Section 3, all VOST traps were analyzed by thermal desorption, purge and trap, GC/FID for the volatile organic constituents listed in Table 16. This list includes the POHCs discussed in Section 4.5. However, several other potential volatile PICs are included in this analyte list as well. Table 41 summarizes the flue gas concentrations of the non-POHC volatile organic constituents detected in the afterburner exit flue gas for one or more of the tests performed. Tabulated values represent the average of the three VOST trap pairs operated during each test. For determining the average emission rate, practical quantitation limits (PQLs) were used for trains containing concentrations less than the PQL. Table 42 presents an analogous summary for the scrubber system exit flue gas.

The data in Table 41 show that the major PICs detected in the afterburner exit flue gas were chloroform, carbon tetrachloride, and benzene. Concentrations ranged from about 1 to 12 $\mu\text{g}/\text{dscm}$. Note that for Test 4 (0 percent chlorine feed), the chlorinated PICs are present at significantly reduced concentrations, as expected. Also for Test 4, both tetrachloroethylene and chlorobenzene were detected, and are reported as PICs since neither was a POHC for this test.

Table 42 shows that chloroform levels in the scrubber exit flue gas were above those at the afterburner exit for five of the eight tests. The origin of the increased chloroform is likely the scrubber makeup water. The scrubber water source is a chlorine treated potable supply in which the trihalomethane (THM) compounds are typically found. These compounds volatilize readily; the scrubber essentially acts as a purger, releasing the THM into the flue gas stream with the entrained water vapor.

For all tests, carbon tetrachloride levels in the scrubber exit flue gas were less than the levels in the afterburner exit flue gas. Benzene concentrations increased in the four tests, decreased in three tests, and remained the same in the one test. No correlations between PIC levels in the flue gas at either location and corresponding POHC DRE are apparent.

**TABLE 39. FLUE GAS POHC CONCENTRATIONS FOR THE PARAMETRIC TRACE
METAL TESTS**

| Test | Test date | POHC concentration ($\mu\text{g}/\text{dscm}$) | | | | | | | | |
|------|-----------|--|---------------|---------------------|---------------|---------------|---------------------|---------|---------------|---------------------|
| | | Afterburner exit | | | Scrubber exit | | | Stack | | |
| | | Toluene | Chlorobenzene | Tetrachloroethylene | Toluene | Chlorobenzene | Tetrachloroethylene | Toluene | Chlorobenzene | Tetrachloroethylene |
| 4 | 9/14/88 | 85 | — | — | 48 | — | — | — | — | — |
| 5 | 8/25/88 | 10 | 2.1 | 7.5 | 40 | 3.3 | 5.6 | — | — | — |
| 6 | 9/16/88 | 14 | 1.8 | 13 | 73 | 7.4 | 10 | — | — | — |
| 7 | 8/30/88 | 140 | 17 | 35 | 21 | 2.4 | 3.1 | — | — | — |
| 8 | 9/07/88 | 240 | 29 | 43 | 40 | 4.2 | 5.1 | 99 | 13 | 14 |
| 9 | 9/09/88 | 19 | 2.2 | 5.0 | 27 | 3.3 | 4.2 | 85 | 14 | 7.9 |
| 10 | 9/20/88 | 510 | 86 | 55 | 21 | 1.9 | 4.0 | — | — | — |
| 11 | 9/22/88 | 1300 | 550 | 750 | 62 | 18 | 24 | 120 | 22 | 29 |

TABLE 40. POHC DREs FOR THE PARAMETRIC TRACE METAL TESTS

| Test | Test date | DRE (%) | | | | | | | | |
|------|-----------|------------------|---------------|---------------------|---------------|---------------|---------------------|---------|---------------|---------------------|
| | | Afterburner exit | | | Scrubber exit | | | Stack | | |
| | | Toluene | Chlorobenzene | Tetrachloroethylene | Toluene | Chlorobenzene | Tetrachloroethylene | Toluene | Chlorobenzene | Tetrachloroethylene |
| 4 | 9/14/88 | 99.99943 | — | — | 99.99936 | — | — | — | — | — |
| 5 | 8/25/88 | 99.999910 | 99.999913 | 99.99966 | 99.99930 | 99.99973 | 99.99945 | — | — | — |
| 6 | 9/16/88 | 99.999903 | 99.999923 | 99.99948 | 99.9988 | 99.99932 | 99.99907 | — | — | — |
| 7 | 8/30/88 | 99.9990 | 99.99919 | 99.9984 | 99.99967 | 99.99976 | 99.99969 | — | — | — |
| 8 | 9/07/88 | 99.9980 | 99.9986 | 99.9980 | 99.99915 | 99.99949 | 99.99940 | 99.9986 | 99.9990 | 99.9990 |
| 9 | 9/09/88 | 99.99982 | 99.99988 | 99.99974 | 99.99951 | 99.99966 | 99.99957 | 99.9988 | 99.9990 | 99.9994 |
| 10 | 9/20/88 | 99.9967 | 99.9967 | 99.9979 | 99.99967 | 99.99987 | 99.99964 | — | — | — |
| 11 | 9/22/88 | 99.998 | 99.9989 | 99.9985 | 99.9986 | 99.99918 | 99.9989 | 99.9975 | 99.9990 | 99.9987 |

TABLE 41. AFTERBURNER EXIT FLUE GAS VOLATILE PIC CONCENTRATIONS

| Test Test date | 4 (9-14-88) | 5 (8-25-88) | 6 (9-16-88) | 7 (8-30-88) | 8 (9-07-88) | 9 (9-09-88) | 10 (9-20-88) | 11 (9-22-88) |
|----------------------|---|----------------|----------------|----------------|----------------|----------------|-----------------|-----------------|
| Compound | Afterburner exit flue gas concentration (µg/dscm) | | | | | | | |
| Chloroform | 2.4 | 9.1 | 5.3 | 12.6 | 5.4 | 9.0 | 6.4 | 4.9 |
| Carbon tetrachloride | 1.6 | 8.4 | 5.3 | 3.1 | 5.5 | 5.5 | 11.9 | <1.5 |
| Benzene | 7.8 | 2.7 | <1.6 | 2.2 | 9.9 | 10.6 | 7.5 | 11.7 |
| Trichloroethylene | <1.7 | 1.7 | <1.7 | <1.7 | <1.7 | 1.9 | 1.9 | <1.7 |
| 1,3-Dichlorobenzene | <1.6 | 2.0 | <1.6 | <1.6 | <1.6 | <1.6 | <1.6 | <1.6 |
| 1,4-Dichlorobenzene | <1.6 | 2.1 | <1.6 | 1.7 | 1.6 | 1.6 | <1.6 | <1.6 |
| 1,2-Dichlorobenzene | 1.9 | <1.5 | <1.5 | <1.5 | 1.7 | <1.5 | 2.2 | <1.5 |
| Tetrachlorethylene | 16.8 | P ^a | P | P | P | P | P | P |
| Chlorobenzene | 2.0 | P | P | P | P | P | P | P |

^aP: Compound was a POHC for these tests.

TABLE 42. SCRUBBER EXIT FLUE GAS VOLATILE PIC CONCENTRATIONS

| Test Test date | 4 (9-14-88) | 5 (8-25-88) | 6 (9-16-88) | 7 (8-30-88) | 8 (9-07-88) | 9 (9-09-88) | 10 (9-20-88) | 11 (9-22-88) |
|----------------------|--|----------------|----------------|----------------|----------------|----------------|-----------------|-----------------|
| Compound | Scrubber exit flue gas concentration (µg/dscm) | | | | | | | |
| Chloroform | 4.3 | 10.0 | 8.4 | 3.3 | 14.7 | 5.6 | 17.9 | 27.7 |
| Carbon tetrachloride | <1.5 | 2.1 | 2.1 | 1.9 | 4.7 | 2.7 | 3.6 | 2.3 |
| Benzene | <1.6 | 8.5 | 4.8 | <1.6 | 10.0 | 16.7 | 15.7 | 5.8 |
| 1,3-Dichlorobenzene | <1.6 | 1.7 | 1.6 | <1.6 | 1.9 | 1.9 | <1.6 | <1.6 |
| 1,4-Dichlorobenzene | <1.6 | 1.6 | 1.5 | <1.6 | 1.8 | 2.2 | 2.5 | <1.6 |
| 1,2-Dichlorobenzene | 2.3 | <1.5 | 3.0 | <1.5 | <1.5 | 2.4 | 4.6 | 1.6 |
| Tetrachlorethane | <1.8 | <1.8 | <1.8 | <1.8 | 7.8 | 14.1 | <1.8 | <1.8 |
| 1,2-Dichloroethane | <1.5 | <1.5 | <1.5 | 2.1 | <1.5 | <1.5 | 2.3 | <1.5 |
| Ethylbenzene | 6.4 | 6.3 | 6.0 | 3.2 | <1.5 | 10.0 | 7.5 | 3.1 |

SECTION 5

CONCLUSIONS

A five-week series of pilot-scale incineration tests was performed to evaluate the fate of trace metals fed to a rotary kiln incinerator equipped with a venturi scrubber/packed column scrubber for particulate/acid gas control. Three tests focused on determining the valence state (trivalent/hexavalent) of chromium in emissions and discharges from the incinerator, and investigating whether valence state distributions were affected by the chromium valence state in the waste feed to the incinerator or the feed chlorine content. Eight tests focused on a parametric evaluation of the fate of five hazardous constituent trace metals (arsenic, barium, cadmium, chromium, and lead), and four nonhazardous constituent trace metals (bismuth, copper, magnesium, and strontium) fed to the incinerator. Test variables for the parametric evaluation were kiln temperature, afterburner temperature, and feed chlorine content. A factorial experimental matrix was tested in which kiln temperature was varied from 816 to 927°C (1500 to 1700°F), afterburner temperature was varied from 982 to 1204°C (1800 to 2200°F), and feed chlorine content was varied from 0 to 8 percent.

For all tests, the waste feed consisted of a synthetic mixture prepared by combining an organic liquid mixture with a clay absorbent material. The organic liquid mixture consisted of toluene with varying amounts of tetrachloroethylene and chlorobenzene added to give desired feed chlorine content. This synthetic solid waste material contained nominally 0.4 kg of organic liquid to 1 kg of clay absorbent. This mixture was introduced to the kiln via a screw feeder. Test trace metals were prepared in an aqueous solution. This solution was metered into the clay/organic liquid mixture in the screw feeder.

For the chromium valence state tests, total chromium and hexavalent chromium (Cr(+6)) concentrations were measured in the kiln ash and scrubber blowdown discharges as well as in the incinerator flue gas at two locations: the afterburner exit and downstream of the scrubber system. For the parametric trace metal tests, trace metal concentrations were measured in the same emission/discharge streams. In addition, the distribution of flue gas trace metal between particulate and vapor/dissolved phases was measured, and the particulate size distribution of trace metals in the afterburner exit flue gas was determined.

Conclusions from the chromium valence state tests include:

- Most of the chromium fed was discharged in the kiln ash. Kiln ash accounted for 95 percent of the discharge amount with no chlorine in the feed, regardless of the chromium valence state in the feed. The kiln ash fraction decreased to 84 percent when chlorine was present in the feed.
- Scrubber exit flue gas accounted for 1 to 2 percent of the chromium discharged with no chlorine in the feed, again regardless of the feed valence state. Scrubber exit flue gas fraction increased to 4 percent when chlorine was present in the feed.

- Scrubber liquor accounted for 3 percent of the discharged chromium with no feed chlorine. The scrubber liquor fraction increased to 11 percent with chlorine-containing feed.
- The kiln ash contained negligible Cr(+6) regardless of feed chromium valence state or chlorine content.
- Nominally 15 percent of the chromium in the scrubber exit flue gas was Cr(+6) with no feed chlorine. The Cr(+6) fraction increased to almost 50 percent of the scrubber exit flue gas chromium with chlorine present in the feed. This result is consistent with the formation of chromyl chloride from entrained chromium vaporized in the afterburner.
- Scrubber liquor chromium was 20 to 30 percent Cr(+6) with Cr(+3) feed; scrubber liquor chromium increased to about 60 percent Cr(+6) with Cr(+6) in the feed.

Conclusions from the parametric trace metal tests include:

- Based on normalized discharge distribution data, cadmium, lead, and bismuth were relatively volatile metals with an average of less than about 30 percent of the discharged metal being present in kiln ash. Barium, copper, strontium, arsenic, chromium, and magnesium were relatively nonvolatile with an average of greater than 75 percent of the discharged metal being present in the kiln ash.
- Observed metal volatilities from most to least volatile were cadmium, lead, bismuth, barium, copper, strontium, arsenic, chromium, and magnesium. This order agrees with the order predicted by metal volatility temperature (temperature at which the vapor pressure of a principal vapor species is 10^{-6} atm) with the notable exception of arsenic. Arsenic has the lowest volatility temperature of the metals tested, but was observed to be one of the least volatile of the metals. This suggests that As_2O_3 was not a predominant arsenic species in the incinerator, or that the arsenic is adsorbed by the clay/ash matrix.
- The average apparent scrubber collection efficiency was lower for the volatile metals (36 to 45 percent) than for the nonvolatile metals (49 to 88 percent) with the notable exception of copper. Copper's average scrubber removal efficiency, at about 30 percent, was significantly lower than the efficiencies seen for the other relatively nonvolatile metals.
- Feed chlorine content had a major effect on the partitioning of the volatile metals (cadmium, lead, and bismuth), and of copper. The fraction of metal discharged in the kiln ash for these metals measurably decreased with increasing feed chlorine content.
- Kiln temperature had a minor effect on the relative volatility of the observed most volatile metals (cadmium and lead) and of arsenic. The fraction of metal discharged in the kiln ash decreased slightly with increasing kiln temperature.
- No test variable measurably affected the discharge distributions of barium, strontium, chromium, and magnesium, among the least volatile metals.

- Total afterburner exit flue gas particulate size distributions were roughly log-normal for all tests in which they were measured. The size distributions for all tests with chlorine-containing feed were roughly comparable; the size distribution was shifted to larger particle size with no chlorine in the feed.
- The afterburner exit flue gas average size distributions of the less volatile metals (chromium, copper, magnesium, and strontium) were coarser than the corresponding overall particulate average size distribution. The size distribution for the relatively more volatile metals (barium and bismuth) were finer than the overall particulate average size distribution.

Test waste POHC destruction and removal efficiencies (DREs) were greater than 99.99 percent as measured in the scrubber discharge flue gas and in the stack downstream of the system's carbon bed/HEPA filter for all of the parametric trace metal tests. POHC DREs were similarly greater than 99.99 percent as measured in the afterburner exit flue gas for all tests except the test with the highest (nominally 8 percent) feed chlorine content. In fact, POHC DREs at all locations were uniformly lower for this high-chlorine-content feed than for other tests with lower feed chlorine contents.

Chloroform, carbon tetrachloride, and benzene were the volatile products of incomplete combustion (PICs) present in flue gas at the highest levels, up to the order of 10 $\mu\text{g}/\text{dscm}$.

All test program data quality objectives (DQOs) were met, with the exception of field matrix spike recovery and the precision of feed clay matrix spike duplicate analyses for Cr(+6). Precision and accuracy DQOs for laboratory matrix spikes and field spikes for other matrices were met. The most important Cr(+6) analyses needed to support the test conclusions noted above were for matrices other than the feed clay. Thus, the failure to achieve the recovery and precision DQOs for a single analyte in a single field matrix has very little impact on the test conclusions stated above.

SECTION 6

QUALITY ASSURANCE

A number of quality assurance (QA) procedures were followed to assess the data quality of the laboratory analytical measurements performed in these tests. In addition, QA efforts performed to ensure that data quality is known for the particulate and CEM measurements involved adherence to Reference Method procedures and CEM manufacturers' specifications. No deviations from the quality assurance project plan (QAPP) occurred for these measurements with the exception that the CO/CO₂ monitor at the stack did not function properly. The major laboratory QA efforts focused on the following measurements:

- Volatile organic compounds in the feed mixtures
- Volatile organic compounds in the VOST traps
- Metals in the feed and incinerator discharges

The data quality objectives (DQOs) for these measurements are listed in Table 43. The QA procedures performed included:

- Analyzing replicate feed mixture samples for toluene, chlorobenzene, and tetrachloroethylene
- Preparing a clay/organic liquid feed mixture matrix spike and analyzing replicate samples for spike recovery
- Spiking all VOST train resin traps with two method surrogates and measuring surrogate recovery
- Preparing eight matrix spike VOST traps and analyzing these for spike recovery (seven analyzed at the IRF using the method described in Appendix D and one shipped to the Acurex analytical chemistry laboratory in Mountain View, California and analyzed via EPA Method 5040)
- Obtaining duplicate VOST samples for analysis at both the IRF and the Acurex Mountain View, California laboratories
- Preparing laboratory matrix spike solutions with the trace metals and analyzing these for spike recovery
- Preparing four hexavalent chromium field matrix spike samples at the IRF and analyzing these for spike recovery

TABLE 43. PRECISION, ACCURACY, AND COMPLETENESS OBJECTIVES

| Measurement parameter | Measurement/ analytical method | Reference | Conditions | Precision (% RSD* or RPD**) | Accuracy (%) | Completeness (%) |
|---|---|--|--|-----------------------------|-----------------------|------------------|
| Volatile organic compounds in clay/ liquid feed | Packed column GC/FID by the method in Appendix D | Appendix D | Direct injection | 50 | 50-140 | 80 |
| Volatile organic compounds on Tenax | Thermal desorption purge and trap by Method 5040, GC/FID analysis by the method in Appendix D | SW-846-3 ^{a,b} Appendix D | Thermal desorption | 100 | 50-150 ^{b,c} | 80 |
| Cr(+ 6) | Methods 3060 ^d and 7197 ^e | SW-846-3 ^a , SW-846-2 ^d | Alkaline digestion and AA analysis | 25 | 75-12 | 80 |
| Metals | Method 6010, 7000 series, ^g or 300 series ^f methods | SW-846-3 ^a , AWPCF ^e | Acid digestion and ICAP or AA analysis | 25 | 75-125 ^f | 80 |

^a(1)

^bRecovery of surrogate spikes of resin samples greater than 20 percent was also an accuracy objective.

^cPercent recovery from a spiked Tenax cartridge.

^d(2)

^e(7)

^fRecovery of a spiked water solution.

*Relative Standard Deviation.

**Relative Percent Difference.

Results of these QA procedures are discussed in the following sections.

6.1 ORGANIC ANALYSIS OF CLAY/ORGANIC LIQUID FEED SAMPLES

As discussed in Section 3, a composite sample of the feed mixture from each drum prepared was collected and analyzed for the volatile organic hazardous constituents toluene, chlorobenzene, and tetrachloroethylene. Each drum of a given test mixture was prepared by mixing together weighed amounts of the individual liquid organic constituents, then mixing together weighed amounts of the liquid mixture and the clay sorbent. Waste feed for a given test was taken from one, two, or three drums depending on test conditions and durations. Thus, comparing analysis results for each drum of a target test mixture preparation gives a measure of the precision and accuracy of the entire feed preparation, sampling, and analysis procedure.

Table 44 provides such a comparison. For each target test mixture preparation, analysis results for a sample of each drum prepared are compared to the target composition and to each other. The accuracy of the feed preparation, sampling, and analysis operation is measured by the ratio of the analyzed concentration of a mixture component to the target value (termed recovery in Table 44). The precision of the procedure is measured by the relative standard deviation (RSD) of all samples analyzed.

The data in Table 44 show that analyzed concentrations ranged from 26 to 135 percent of target concentrations over 24 samples analyzed. Only 1 measurement out of 63 was out of the 50 to 140 percent recovery range, the accuracy DQO for the measurement. Thus, 98 percent of the measurements were acceptable. This DQO was met. The precision of the procedure as measured by the RSD of the analysis of different drum samples of a given target formulation ranged from 5 to 26 percent. All were within the precision DQO of 50 percent for this measurement. Thus, this DQO was met.

As a further measure of analytical precision, two clay/organic liquid feed samples were subjected to duplicate analysis. The relative percent difference (RPD) for these ranged from 1.3 to 10.1 percent for each of the constituents analyzed, as shown in Table 45. The precision DQO for the volatile organic compounds in the feed mixture was 50 percent RPD. This DQO was therefore met.

Three samples of a clay/organic liquid feed mixture matrix spike were prepared under controlled laboratory conditions (as opposed to bulk feed sample preparation) and analyzed to determine volatile organic constituent matrix spike recovery. The data in Table 46 show that the recoveries ranged from 75 to 104 percent. All were within the accuracy DQO range for this measurement of 50 to 140 percent recovery. Thus, this DQO was also achieved. Further, the RSD of the replicate spike analyses ranged from 8.9 to 11.3 percent, all within the DQO for precision of 50 percent.

It bears noting that the analytical data in Tables 44 and 45 are different from the feed mixture compositions used for DRE determinations in Section 4 (i.e., in Table 17). The data in Table 17 represent analyzed compositions (Tables 44 and 45) corrected for the matrix spike recoveries experienced (Table 46).

6.2 VOLATILE ORGANIC COMPOUNDS IN THE VOST TRAPS

To assess measurement accuracy of the VOST samples analyses, each resin trap was spiked with the surrogates octane and 4-bromofluorobenzene prior to use in sampling. Surrogate recovery was then measured.

TABLE 44. CLAY/ORGANIC LIQUID FEED SAMPLE POHC ANALYSIS RESULTS

| Test | Test date | Drum | Toluene | | Tetrachloroethylene | | Chlorobenzene | |
|----------------------|-----------------|------|-------------------------------|---------------------------|-------------------------------|---------------------------|-------------------------------|---------------------------|
| | | | Measured concentration (wt %) | Recovery ^a (%) | Measured concentration (wt %) | Recovery ^a (%) | Measured concentration (wt %) | Recovery ^a (%) |
| Mixture 1 | | | | | | | | |
| Target concentration | | | 28.6 | | 0 | | 0 | |
| 1,2 | 9/28/88-9/29/88 | 1 | 26.4 | 92 | — | — | — | — |
| | | 2 | 25.5 | 89 | — | — | — | — |
| | | 3 | 24.4 | 85 | — | — | — | — |
| 4 | 9/14/88 | 1 | 22.5 | 79 | — | — | — | — |
| | | 2 | 15.3 | 53 | — | — | — | — |
| | | 3 | 26.6 | 93 | — | — | — | — |
| RSD (%) | | | 18 | | | | | |
| Mixture 2 | | | | | | | | |
| Target concentration | | | 21.7 | | 3.4 | | 3.4 | |
| 5 | 8/25/88 | 1 | 15.1 | 70 | 2.7 | 79 | 3.2 | 94 |
| | | 2 | 15.7 | 72 | 2.3 | 68 | 3.0 | 88 |
| 6 | 9/16/88 | 1 | 19.1 | 88 | 3.1 | 91 | 3.0 | 88 |
| | | 2 | 19.0 | 88 | 2.9 | 85 | 2.9 | 85 |
| | | 3 | 18.9 | 87 | 3.1 | 91 | 3.2 | 94 |
| 7 | 8/30/88 | 1 | 16.0 | 74 | 2.3 | 68 | 2.5 | 74 |
| | | 2 | 19.2 | 88 | 2.9 | 85 | 3.0 | 88 |
| | | 3 | 19.4 | 89 | 2.7 | 79 | 3.0 | 88 |
| 8 | 9/7/88 | 1 | 16.3 | 75 | 2.8 | 82 | 2.7 | 79 |
| | | 2 | 15.4 | 71 | 2.3 | 68 | 2.5 | 74 |
| 9 | 9/9/88 | 1 | 15.2 | 70 | 2.4 | 71 | 2.5 | 74 |
| 10 | 9/20/88 | 1 | 21.1 | 97 | 3.2 | 94 | 3.6 | 106 |
| | | 2 | 20.1 | 93 | 0.9 | 26 | 4.6 | 135 |
| | | 3 | 21.2 | 98 | 3.2 | 94 | 3.1 | 91 |
| RSD (%) | | | 15 | | 26 | | 18 | |
| Mixture 3 | | | | | | | | |
| Target concentration | | | 14.9 | | 6.9 | | 6.9 | |
| 3 | 9/26/88 | 1 | 13.8 | 93 | 6.0 | 87 | 5.4 | 78 |
| | | 2 | 15.3 | 103 | 6.4 | 93 | 6.0 | 87 |
| 11 | 9/22/88 | 1 | 12.8 | 86 | 5.6 | 81 | 5.7 | 83 |
| | | 2 | 14.0 | 94 | 6.1 | 88 | 6.2 | 90 |
| | | 3 | 13.6 | 91 | 6.0 | 87 | 6.0 | 87 |
| RSD (%) | | | 7 | | 5 | | 5 | |

^aRatio of measured concentration to target concentration.

TABLE 45. CLAY/ORGANIC LIQUID FEED REPLICATE VOLATILE ORGANIC ANALYSIS RESULTS

| Test | Test date | Drum | Toluene | | | Tetrachloroethylene | | | Chlorobenzene | | |
|------|---------------------|------|---------------------|---------------------|---------|---------------------|---------------------|---------|---------------------|---------------------|---------|
| | | | 1st Analysis (wt %) | 2nd Analysis (wt %) | RPD (%) | 1st Analysis (wt %) | 2nd Analysis (wt %) | RPD (%) | 1st Analysis (wt %) | 2nd Analysis (wt %) | RPD (%) |
| 3 | 9/26/88 | 2 | 15.3 | 15.1 | 1.3 | 6.4 | 6.3 | 1.4 | 6.0 | 5.9 | 1.9 |
| 1,2 | 9/28/88- 9/29/88 | 3 | 24.4 | 27.0 | 10.1 | | | | | | |

TABLE 46. VOLATILE ORGANIC CONSTITUENT RECOVERY FROM CLAY/ORGANIC LIQUID FEED MATRIX SPIKE SAMPLES

| Compound | Spike | Percent recovery | | Relative Standard Deviation (RSD) |
|---------------------|-------|------------------|-----------------|-----------------------------------|
| | | Replicate spike | Replicate spike | |
| Toluene | 86 | 88 | 104 | 8.9 |
| Chlorobenzene | 78 | 81 | 100 | 11.3 |
| Tetrachloroethylene | 75 | 79 | 95 | 10.4 |

Table 47 summarizes octane recovery from the VOST trap pairs. The data in the table show that octane recovery ranged from 0 to 217 percent, with 7 of 75 recoveries not within the DQO range of 50 to 150 percent. Measurement completeness was 91 percent, and met the DQO for recovery completeness of 80 percent.

Table 48 similarly summarizes 4-bromofluorobenzene recovery from the VOST trap pairs. The data show that 4-bromofluorobenzene recovery ranged from 0 to 120 percent, with 3 of 75 recoveries not within the DQO range of 50 to 150 percent. Measurement completeness was 96 percent, and met the DQO for recovery completeness of 80 percent.

As a further measure of VOST analysis accuracy, and as a measure of analysis precision, seven matrix spike VOST trap pairs were prepared and analyzed at the IRF. Results of these analyses are summarized in Table 49. The data in the table show that matrix spike recoveries ranged from 73 to 194 percent, with 2 of 35 recoveries not within the DQO range of 50 to 150 percent. Measurement completeness was 94 percent, and met the DQO for recovery completeness of 80 percent. Further, the RSD of the matrix spike analyses ranged from 3.6 to 29 percent, all within the DQO for precision of 100 percent.

In addition to the seven traps analyzed at the IRF, an eighth matrix spike VOST trap pair was prepared and shipped to Acurex's Mountain View, California, laboratory for analysis by EPA Method 5040. This spike sample was prepared identically to one analyzed at the IRF. Table 50 summarizes the analysis results for this VOST trap pair and compares results of the Method 5040 GC/MS analysis with the GC/FID analysis of an identically prepared trap pair. Matrix spike and surrogate recoveries for the GC/MS-analyzed sample ranged from 46 to 137 percent, with one out of eight compounds not within the DQO recovery range of 50 to 150 percent. Measurement completeness was 87 percent, and met the DQO for recovery completeness of 80 percent. In addition, the RPD between the GC/MS-analyzed traps and the GC/FID-analyzed traps ranged from 28 to 80 percent, all within the precision DQO for this measurement of 100 percent RPD.

To further compare VOST trap analysis results between the GC/FID analysis method routinely performed at the IRF (see Appendix D) and the GC/MS Method 5040 analysis method, two sampling trains were operated simultaneously at the scrubber exit during Test 10 to obtain two sets of VOST traps from the same flue gas. One set was analyzed at the IRF by GC/FID (Appendix D), and one set was analyzed by Method 5040 at the Acurex Mountain View laboratory. Unfortunately, the GC/MS analysis of one of the trap pairs (the third pair) was invalidated due to a leak during analysis. Table 51 summarizes the VOST results reported for each method.

The precision DQO for the measurement of volatile organic compounds in VOST traps is 100 percent RPD. The data in Table 51 show that reasonably good agreement between the two analysis methods, within about 100 percent RPD, was achieved for chloroform, bromodichloromethane, dibromochloromethane, bromoform, toluene, chlorobenzene, and tetrachloroethylene (the last three compounds being the POHCs for these tests). However, agreement between the two analysis methods was not as good for carbon tetrachloride, benzene, dichlorobenzenes, and 1,2-dichloroethane. Surrogate recoveries were acceptable by both methods. Given the mixed nature of these results, further method comparisons are recommended and planned in future tests.

TABLE 47. OCTANE SURROGATE RECOVERY FROM VOST TRAPS

| Afterburner exit traps | | Scrubber system exit traps | | Other traps | |
|--------------------------|---------------------|----------------------------|---------------------|---------------------------|---------------------|
| Sample | Octane recovery (%) | Sample | Octane recovery (%) | Sample | Octane recovery (%) |
| <u>Test 4 (9/14/88)</u> | | <u>Test 4 (9/14/88)</u> | | Stack Samples | |
| 1st trap pair | 121 | 1st trap pair | 118 | <u>Test 8 (9/07/88)</u> | |
| 2nd trap pair | 101 | 2nd trap pair | 107 | 1st trap pair | 123 |
| 3rd trap pair | 76 | 3rd trap pair | 109 | 2nd trap pair | 109 |
| <u>Test 5 (8/25/88)</u> | | <u>Test 5 (8/25/88)</u> | | 3rd trap pair | 134 |
| 1st trap pair | 137 | 1st trap pair | 217 | <u>Test 9 (9/09/88)</u> | |
| 2nd trap pair | 151 | 2nd trap pair | 112 | 1st trap pair | 102 |
| 3rd trap pair | 117 | 3rd trap pair | 153 | 2nd trap pair | 34 |
| <u>Test 6 (9/16/88)</u> | | <u>Test 6 (9/16/88)</u> | | 3rd trap pair | 119 |
| 1st trap pair | 107 | 1st trap pair | 109 | <u>Test 11 (9/22/88)</u> | |
| 2nd trap pair | 114 | 2nd trap pair | 111 | 1st trap pair | 91 |
| 3rd trap pair | 115 | 3rd trap pair | 0 | 2nd trap pair | 122 |
| <u>Test 7 (8/30/88)</u> | | <u>Test 7 (8/30/88)</u> | | 3rd trap pair | 123 |
| 1st trap pair | 114 | 1st trap pair | 111 | Field blank traps | |
| 2nd trap pair | 110 | 2nd trap pair | 111 | 8/25/88-1 | 36 |
| 3rd trap pair | 76 | 3rd trap pair | 98 | 8/25/88-2 | 94 |
| <u>Test 8 (9/07/88)</u> | | <u>Test 8 (9/07/88)</u> | | 8/30/88 | 80 |
| 1st trap pair | 117 | 1st trap pair | 96 | 9/07/88 | 80 |
| 2nd trap pair | 136 | 2nd trap pair | 100 | 9/09/88 | 120 |
| 3rd trap pair | 108 | 3rd trap pair | 114 | 9/14/88 | 101 |
| <u>Test 9 (9/09/88)</u> | | <u>Test 9 (9/09/88)</u> | | 9/16/88 | 105 |
| 1st trap pair | 79 | 1st trap pair | 133 | 9/20/88 | 106 |
| 2nd trap pair | 116 | 2nd trap pair | 135 | 9/22/88 | 100 |
| 3rd trap pair | 116 | 3rd trap pair | 118 | Laboratory method | |
| <u>Test 10 (9/20/88)</u> | | <u>Test 10 (9/20/88)</u> | | blank traps | |
| 1st trap pair | 122 | 1st trap pair | 81 | 8/25/88 | 72 |
| 2nd trap pair | 117 | 2nd trap pair | 117 | Matrix spike traps | |
| 3rd trap pair | 115 | 3rd trap pair | 178 | 8/25/88 | 98 |
| <u>Test 11 (9/22/88)</u> | | <u>Test 11 (9/22/88)</u> | | 8/30/88 | 103 |
| 1st trap pair | 115 | 1st trap pair | 111 | 9/07/88 | 108 |
| 2nd trap pair | 0 | 2nd trap pair | 117 | 9/09/88 | 104 |
| 3rd trap pair | 111 | 3rd trap pair | 112 | 9/14/88 | 107 |
| | | | | 9/16/88 | 109 |
| | | | | 9/20/88 | 112 |
| | | | | 9/22/88 | 105 |

TABLE 48. 4-BROMOFLUOROBENZENE SURROGATE RECOVERY FROM VOST TRAPS

| Afterburner exit traps | | Scrubber system exit traps | | Other traps | |
|--------------------------|-------------------------------------|----------------------------|-------------------------------------|--------------------------------------|-------------------------------------|
| Sample | 4-Bromo-fluoro-benzene recovery (%) | Sample | 4-Bromo-fluoro-benzene recovery (%) | Sample | 4-Bromo-fluoro-benzene recovery (%) |
| <u>Test 4 (9/14/88)</u> | | <u>Test 4 (9/14/88)</u> | | Stack Samples | |
| 1st trap pair | 107 | 1st trap pair | 100 | <u>Test 8 (9/07/88)</u> | |
| 2nd trap pair | 86 | 2nd trap pair | 104 | 1st trap pair | 96 |
| 3rd trap pair | 81 | 3rd trap pair | 98 | 2nd trap pair | 98 |
| <u>Test 5 (8/25/88)</u> | | <u>Test 5 (8/25/88)</u> | | 3rd trap pair | 111 |
| 1st trap pair | 105 | 1st trap pair | 91 | <u>Test 9 (9/09/88)</u> | |
| 2nd trap pair | 115 | 2nd trap pair | 105 | 1st trap pair | 120 |
| 3rd trap pair | 101 | 3rd trap pair | 112 | 2nd trap pair | 108 |
| <u>Test 6 (9/16/88)</u> | | <u>Test 6 (9/16/88)</u> | | 3rd trap pair | 120 |
| 1st trap pair | 83 | 1st trap pair | 104 | <u>Test 11 (9/22/88)</u> | |
| 2nd trap pair | 101 | 2nd trap pair | 106 | 1st trap pair | 79 |
| 3rd trap pair | 105 | 3rd trap pair | 0 | 2nd trap pair | 108 |
| <u>Test 7 (8/30/88)</u> | | <u>Test 7 (8/30/88)</u> | | 3rd trap pair | 107 |
| 1st trap pair | 94 | 1st trap pair | 99 | Field blank traps | |
| 2nd trap pair | 99 | 2nd trap pair | 95 | 8/25/88-1 | 27 |
| 3rd trap pair | 80 | 3rd trap pair | 37 | 8/25/88-2 | 94 |
| <u>Test 8 (9/07/88)</u> | | <u>Test 8 (9/07/88)</u> | | 8/30/88 | 81 |
| 1st trap pair | 104 | 1st trap pair | 104 | 9/07/88 | 88 |
| 2nd trap pair | 115 | 2nd trap pair | 81 | 9/09/88 | 96 |
| 3rd trap pair | 95 | 3rd trap pair | 103 | 9/14/88 | 97 |
| <u>Test 9 (9/09/88)</u> | | <u>Test 9 (9/09/88)</u> | | 9/16/88 | 102 |
| 1st trap pair | 89 | 1st trap pair | 102 | 9/20/88 | 94 |
| 2nd trap pair | 92 | 2nd trap pair | 101 | 9/22/88 | 88 |
| 3rd trap pair | 106 | 3rd trap pair | 102 | Laboratory method blank traps | |
| <u>Test 10 (9/20/88)</u> | | <u>Test 10 (9/20/88)</u> | | 8/25/88 | 92 |
| 1st trap pair | 107 | 1st trap pair | 101 | Matrix spike traps | |
| 2nd trap pair | 104 | 2nd trap pair | 91 | 8/25/88 | 97 |
| 3rd trap pair | 92 | 3rd trap pair | 110 | 8/30/88 | 96 |
| <u>Test 11 (9/22/88)</u> | | <u>Test 11 (9/22/88)</u> | | 9/07/88 | 93 |
| 1st trap pair | 96 | 1st trap pair | 106 | 9/09/88 | 110 |
| 2nd trap pair | 110 | 2nd trap pair | 97 | 9/14/88 | 105 |
| 3rd trap pair | 105 | 3rd trap pair | 105 | 9/16/88 | 106 |
| | | | | 9/20/88 | 105 |
| | | | | 9/22/88 | 98 |

TABLE 49. VOLATILE ORGANIC CONSTITUENT RECOVERY FROM VOST MATRIX SPIKE SAMPLES

| Compound | Percent recovery | | | | | | | Relative Standard Deviation (% RSD) |
|-----------------------|---------------------|---------------------|---------------------|---------------------|---------------------|----------------------|----------------------|--|
| | Test 4 (9/14/88) | Test 5 (8/25/88) | Test 7 (8/30/88) | Test 8 (9/07/88) | Test 9 (9/09/88) | Test 10 (9/20/88) | Test 11 (9/22/88) | |
| 1,1,1-Trichloroethane | 111 | 93 | 107 | 113 | 98 | 103 | 126 | 9.4 |
| Benzene | 73 | 116 | 130 | 127 | 194 | 92 | 153 | 29.0 |
| Toluene | 123 | 124 | 115 | 147 | 124 | 131 | 121 | 10.0 |
| Tetrachloroethylene | 104 | 103 | 101 | 100 | 107 | 111 | 100 | 3.6 |
| Chlorobenzene | 102 | 96 | 100 | 100 | 108 | 107 | 97 | 4.2 |

**TABLE 50. VOLATILE ORGANIC CONSTITUENT RECOVERY FROM THE
VOST MATRIX SPIKE SAMPLE FOR TEST 10 (9/20/88)
BY METHOD 5040 AND GC/FID ANALYSIS**

| Compound | Percent recovery | | RPD (%) |
|-----------------------------------|------------------|-------|---------|
| | GC/FID | GC/MS | |
| 1,1,1-Trichloroethane | 103 | 64 | 47 |
| Benzene | 92 | 65 | 34 |
| Toluene | 131 | 71 | 59 |
| Tetrachloroethylene | 111 | 84 | 28 |
| Chlorobenzene | 107 | 46 | 80 |
| 1,1-Dichloroethane | — ^a | 137 | — |
| 4-Bromofluorobenzene ^b | 105 | 57 | 59 |
| Toluene-d8 ^b | — ^a | 87 | — |

^aNot reported.

^bMethod surrogates.

TABLE 51. VOLATILE ORGANIC CONSTITUENT CONCENTRATIONS FOR DUPLICATE VOST SAMPLES TAKEN AT THE SCRUBBER EXIT DURING TEST 10 (9/20/88) AND ANALYZED BY GC/FID AND GC/MS (METHOD 5040)

| Compound | Flue gas concentration ($\mu\text{g}/\text{dscm}$) | | | | | |
|-------------------------------------|---|----------------|-----------------------------------|---------------|----------------|-----------------------------------|
| | 1st Trap pair | | Relative Percent Difference | 2nd Trap pair | | Relative Percent Difference |
| | GC/FID | GC/MS | | GC/FID | GC/MS | |
| Chloroform | 22.0 | 14.7 | 40 | 12.2 | 2.7 | 128 |
| Carbon Tetrachloride | 3.9 | 0.7 | 139 | 1.5 | <0.25 | — |
| Benzene | 19.2 | 0.7 | 186 | 8.4 | <0.25 | — |
| Bromodichloromethane | 12.7 | 16.9 | 28 | 3.8 | 4.8 | 23 |
| Dibromochloromethane | 27.3 | 11.6 | 81 | 77.6 | 7.0 | 167 |
| Bromoform | 11.6 | 10.2 | 13 | 15.7 | 15.0 | 4.6 |
| 1,2-Dichloroethane | 3.2 | <0.2 | — | 2.2 | <0.25 | — |
| 1,4-Dichlorobenzene | 4.4 | <0.2 | — | <1.5 | <0.25 | 0 |
| 1,2-Dichlorobenzene | 6.5 | <0.2 | — | 4.6 | <0.25 | — |
| Toluene | 19.1 | 13.4 | 35 | 15.5 | 10.0 | 43 |
| Chlorobenzene | 1.7 | 1.1 | 43 | <1.6 | 0.9 | 0 |
| Tetrachloroethylene | 2.7 | 2.5 | 7.7 | 4.8 | 1.6 | 102 |
| Surrogate Recoveries (%) | | | | | | |
| Octane | 117 | — ^a | — | 81 | — ^a | — |
| 4-bromofluorobenzene | 91 | 65 | 33 | 101 | 79 | 24 |
| Toluene-d ₈ ^b | — | 99 | — | — | 80 | — |

^aGC/FID surrogate only.

^bGC/MS surrogate only.

6.3 METALS ANALYSIS

For metals analysis in the laboratory, matrix spike and matrix spike duplicate samples were prepared in the laboratory and analyzed to assess analytical precision and accuracy. In addition, field matrix spike and spike duplicate samples were prepared at the IRF and shipped to the Mountain View laboratory for analysis for the chromium valence state tests. Analysis results are discussed in the following subsections.

6.3.1 Chromium Valence State Tests

Table 52 summarizes the results of the laboratory matrix spike/matrix spike duplicate analyses for the chromium valence state test series. Indicated in the table are the group of samples to which the matrix spike samples apply. Matrix spike recovery for laboratory-prepared total chromium and Cr(+6) samples ranged from 89 to 115 percent, all within the DQO range of 75 to 125 percent. The RPD between the values obtained for the matrix spikes and the matrix spike duplicates ranged from 0 to 24 percent. All values were within the DQO for measurement precision of 25 percent.

Table 53 summarizes the results of the Cr(+6) spike analyses of field matrix spike samples prepared at the IRF. Two sets of spike recovery values are noted in the table, one set obtained from analyses performed 12 days after sample preparation, and one set obtained 69 days after sample preparation. The QAPP for these tests specified an analytical hold time for Cr(+6) samples of 7 days between sample collection and analysis. However, between 11 and 12 days actually elapsed between collection and analysis. As a consequence, it was decided to reanalyze the matrix spike samples after a second period of hold time to evaluate the possible effects of exceeding hold time on test conclusions.

The data in Table 53 show that Cr(+6) matrix spike recovery ranged from 32 percent for the feed clay matrix to 90 percent for the kiln ash matrix for the original analysis. A range of recoveries is noted for the scrubber blowdown matrix. This is because a blank sample of the actual matrix spiked was not analyzed. Instead, blank samples with background Cr(+6) concentrations which were known to bracket the actual matrix spiked were analyzed. Thus, actual matrix spike recovery would be in the range noted in Table 53.

The accuracy DQO for this measurement was 75 to 125 percent recovery. Kiln ash, aqueous feed spike solution, and possibly scrubber blowdown matrix spike recovery for the original analyses met this objective. Cr(+6) recoveries from the feed clay spike recoveries were low. Furthermore, the precision of the measurement was 48 percent RPD, greater than the 25 percent DQO for the measurement.

Matrix spike recoveries were uniformly increased for the second analyses series, performed 8 weeks after the original analyses. For these analyses spike recoveries were within the DQO range for all samples except the scrubber blowdown. The precision of the feed clay matrix spike duplicate analyses, at 14 percent RSD, was also within the DQO.

The fact that better QA measurement parameter performance was achieved after samples had aged considerably suggests that exceeding sample hold time did not affect the quality of the data reported. With respect to the inability to uniformly achieve the DQOs in the original sample analyses, it must be noted that the sample matrix failing the spike recovery and precision objective was the feed clay matrix. Acceptable spike recovery was achieved for the matrices of more importance in the tests—the matrices whose analyses were used to formulate

TABLE 52. SUMMARY OF LABORATORY MATRIX SPIKE SAMPLE ANALYSIS RESULTS FOR THE CHROMIUM VALENCE STATE TEST SERIES

| Test | Analyte | Corresponding sample | Spike recovery (%) | Duplicate spike Relative Percent Difference (%) |
|---------|----------|-------------------------------|--------------------|---|
| 1, 2, 3 | Total Cr | Ash | 115 | 7.9 |
| 1 | Total Cr | Water ^a | 105 | 1.9 |
| 2 | Total Cr | Water ^a | 101 | 0.2 |
| 3 | Total Cr | Water ^a | 98 | 1.6 |
| 1, 2, 3 | Total Cr | Afterburner exit train filter | 89 | 0.9 |
| 1 | Total Cr | Scrubber exit train filter | 89 | 0.9 |
| 2 | Total Cr | Scrubber exit train filter | 89 | 0.9 |
| 3 | Total Cr | Scrubber exit train filter | 89 | 0.9 |
| 1 | Cr(+6) | All | 94 | 24 |
| 2 | Cr(+6) | All | 94 | 6.2 |
| 3 | Cr(+6) | All | 102 | 0 |

^aApplies to feed, impinger, and probe wash samples.

TABLE 53. CR(+6) MATRIX SPIKE RECOVERIES

| Matrix | Cr(+6) Concentration (ppm) | | Spike recovery (%) | | | |
|--------------------------------|----------------------------|--------------|-----------------------|---------------------|-----------------------|---------------------|
| | Blank level | Spiked level | Analyzed | | Original ^a | Repeat ^b |
| | | | Original ^a | Repeat ^b | | |
| Feed clay | 2.3 | 10 | 3.9 | 10.8 | 32 | 88 |
| Feed clay duplicate | 2.3 | 10 | 6.4 | 12.4 | 52 | 101 |
| RPD (percent) | | | | | 48 | 14 |
| Scrubber blowdown ^c | 0.10 to 0.28 | 0.10 | 0.19 | 0.49 | 50 to 95 | 129 to 245 |
| Kiln ash | 0.27 | 10 | 9.2 | 10.8 | 90 | 105 |
| Aqueous feed spike | — | 3,400 | 2,700 | 3,100 | 79 | 91 |

^aAnalyzed 12 days after sample preparation.

^bAnalyzed 69 days after sample preparation (8 weeks after original analysis).

^cMatrix blank samples which bracket the matrix spike sample were analyzed.

test conclusions. These were the kiln ash, aqueous feed spike solution, and possibly the scrubber blowdown. Given this, it is felt that test conclusions are valid as stated in Section 5.

6.3.2 Parametric Trace Metal Tests

Table 54 summarizes the trace metal recoveries for the laboratory matrix spikes corresponding to analysis of water-related samples for the parametric test series. These spike and duplicate spike samples apply to aqueous feed spike solutions, impinger, blowdown and probe wash samples obtained during the trace metals parametric test series. Matrix spike recovery for the trace metal constituents ranged from 81 to 123 percent, all within the DQO for recovery of 75 to 125 percent. The RPD between the values obtained for the matrix spike and the matrix spike duplicates ranged from 0 to 37 percent. One out of 72 duplicate spike pairs did not fall within the DQO limit for precision of 25 percent. Measurement completeness was 98 percent and met the DQO for precision completeness of 80 percent.

Table 55 summarizes the trace metal recoveries for the laboratory matrix spikes corresponding to analysis of filter and ash samples for the parametric test series. For the kiln ash samples, matrix spike recovery of the trace metal constituents ranged from 72 to 137 percent. Five of the 27 spike samples were not within the DQO range for recovery of 75 to 125 percent. Measurement completeness was 81 percent, and met the DQO for recovery completeness of 80 percent. The RPD between the values obtained for the matrix spike and matrix spike duplicates ranged from 0.3 to 27 percent. One out of 27 duplicate spikes exceeded the DQO for precision

TABLE 54. LABORATORY MATRIX SPIKE AQUEOUS SAMPLE ANALYSIS RESULTS FOR THE PARAMETRIC TEST SERIES

| Metal | Test 4 (9/14/88) | | Test 5 (8/25/88) | | Test 6 (9/16/88) | | Test 7 (8/30/88) | | Test 8 (9/07/88) | | Test 9 (9/09/88) | | Test 10 (9/20/88) | | Test 11 (9/22/88) | |
|-----------|--------------------------|---------------------------------------|--------------------------|---------------------------------------|--------------------------|---------------------------------------|--------------------------|---------------------------------------|--------------------------|---------------------------------------|--------------------------|---------------------------------------|--------------------------|---------------------------------------|--------------------------|---------------------------------------|
| | Spike recovery (%) | Dupli- cate spike RPD (%) | Spike recovery (%) | Dupli- cate spike RPD (%) | Spike recovery (%) | Dupli- cate spike RPD (%) | Spike recovery (%) | Dupli- cate spike RPD (%) | Spike recovery (%) | Dupli- cate spike RPD (%) | Spike recovery (%) | Dupli- cate spike RPD (%) | Spike recovery (%) | Dupli- cate spike RPD (%) | Spike recovery (%) | Dupli- cate spike RPD (%) |
| Arsenic | 86 | 9.2 | 86 | 5.9 | 88 | 3.4 | 95 | 3.3 | 81 | 1.6 | 86 | 4.2 | 89 | 9.3 | 94 | 5.3 |
| Barium | 97 | 7.6 | 97 | 0.6 | 105 | 2.7 | 101 | 3.6 | 105 | 3.0 | 104 | 0.9 | 93 | 4.2 | 96 | 3.9 |
| Bismuth | 123 | 37 | 88 | 17 | 98 | 1.0 | 93 | 14 | 90 | 15 | 83 | 0.8 | 84 | 7.4 | 114 | 13 |
| Cadmium | 103 | 5.2 | 101 | 0.3 | 106 | 2.5 | 106 | 2.8 | 105 | 0.9 | 102 | 1.7 | 101 | 2.9 | 98 | 4.3 |
| Chromium | 97 | 6.5 | 97 | 0.2 | 108 | 2.6 | 102 | 2.8 | 104 | 2.2 | 102 | 0.8 | 97 | 3.9 | 98 | 2.9 |
| Copper | 92 | 7.6 | 92 | 0.5 | 102 | 2.4 | 96 | 3.6 | 99 | 2.3 | 84 | 2.0 | 83 | 2.9 | 95 | 4.0 |
| Lead | 105 | 11 | 104 | 1.9 | 116 | 6.4 | 104 | 11 | 106 | 6.7 | 122 | 2.1 | 109 | 5.1 | 100 | 3.4 |
| Magnesium | 94 | 5.3 | 91 | 0.5 | 103 | 1.0 | 95 | 4.5 | 107 | 2.4 | 98 | 1.4 | 91 | 2.8 | 97 | 2.2 |
| Strontium | 108 | 5.1 | 101 | 0 | 106 | 3.1 | 105 | 2.9 | 109 | 0.9 | 103 | 3.3 | 107 | 2.6 | 117 | 6.3 |

TABLE 55. LABORATORY MATRIX SPIKE FILTER AND ASH SAMPLE ANALYSIS RESULTS FOR THE PARAMETRIC TEST SERIES

| Metal | Kiln Ash | | | | | | Afterburner exit sampling train filter | | Scrubber exit sampling train filter | | | |
|-----------|--------------------------|----------------------------------|--------------------------|----------------------------------|--------------------------|----------------------------------|--|----------------------------------|---|----------------------------------|--------------------------|----------------------------------|
| | Tests 4,5,8,9,10,11 | | Test 6 | | Test 7 | | Tests 4,6,7,8,9,11 | | Tests 4,7,9 | | Tests 6,10,11 | |
| | Spike recovery (%) | Duplicate spike RPD (%) | Spike recovery (%) | Duplicate spike RPD (%) | Spike recovery (%) | Duplicate spike RPD (%) | Spike recovery (%) | Duplicate spike RPD (%) | Spike recovery (%) | Duplicate spike RPD (%) | Spike recovery (%) | Duplicate spike RPD (%) |
| | | | | | | | | | | | | |
| Arsenic | 125 | 6.5 | 117 | 91 | 124 | 18 | 105 | 5.6 | 77 | 19 | 70 | 3.6 |
| Barium | 106 | 1.3 | 80 | 2.1 | 111 | 6.9 | 100 | 3.7 | 104 | 3.7 | 90 | 1.2 |
| Bismuth | 136 | 13 | 101 | 27 | 91 | 3.6 | 92 | 22 | 64 | 16 | 93 | 1.1 |
| Cadmium | 86 | 1.7 | 93 | 0.3 | 98 | 9.1 | 110 | 3.9 | 93 | 0.4 | 91 | 2.5 |
| Chromium | 93 | 5 | 90 | 3.1 | 115 | 11 | 105 | 6.0 | 94 | 1.0 | 89 | 0.9 |
| Copper | 137 | 5.7 | 72 | 1.3 | 91 | 18 | 92 | 4.1 | 94 | 2.2 | 77 | 1.9 |
| Lead | 111 | 18 | 104 | 1.6 | 117 | 11 | 108 | 3.5 | 100 | 0 | 96 | 1.7 |
| Magnesium | 81 | 5.1 | 84 | 7.3 | 100 | 3.4 | 81 | 9.7 | 122 | 13 | 102 | 4.3 |
| Strontium | 129 | 7.6 | 72 | 4.3 | 104 | 20 | 97 | 3.0 | 104 | 23 | 123 | 14 |

of 25 percent. Measurement completeness was 96 percent and met the DQO for precision completeness of 80 percent.

For the filter samples, matrix spike recovery of the trace metal constituents ranged from 64 to 123 percent. Two of 27 spike samples were not within the DQO range for recovery of 75 to 125 percent. Measurement completeness was 92 percent, and met the DQO for recovery completeness of 80 percent. The RPD between the values obtained for the matrix spike and matrix spike duplicates ranged from 0 to 23 percent, all within the DQO for precision of 25 percent.

As a further measure of trace metal analytical accuracy and precision, recall from Section 4.1 that a composite sample of the aqueous metal spike solution for each test was analyzed for the 9 test trace metals. These can be considered field aqueous solution matrix spike samples. Comparing analysis results from test to test will give a measure of the accuracy and precision of the entire aqueous spike solution preparation, sample collection and preservation, and laboratory analysis process.

Table 19 summarized the results of all aqueous metal spike solution analyses. Table 56 presents these results in different form. Specifically, Table 56 tabulates aqueous spike solution matrix spike recoveries where recovery is defined as the analyzed concentration divided by the target preparation concentration. The data in Table 56 show that analysis recoveries ranged from 67 to 122 percent. Seven of 8 analyses were within the recovery DQO for this measurement of 75 to 125 percent for all the metals except arsenic. Thus, measurement completeness was 88 percent for all metals except arsenic, compared to a completeness DQO of 80 percent. Only 4 of 8 arsenic spike recoveries were between 75 and 125 percent, for a completeness of 50 percent. Arsenic measurement accuracy failed its DQO on this basis. However, the failing recoveries were between 71 and 74 percent. These marginally low recoveries suggest that failure to achieve the recovery DQO for arsenic from this matrix would have no impact on test program conclusions regarding arsenic distributions.

The data in Table 56 also show that the precision of individual metal analyses for the aqueous spike solutions ranged from 9 to 18 percent RSD. All precisions were within the DQO for this measurement of 25 percent. Thus, the measurement precision DQO was met.

Finally, recall from Section 4.1 that clay matrix samples for all parametric trace metal tests were analyzed for lead, magnesium, and strontium, and for all tests were analyzed for chromium. Comparing all these analyses gives a measure of the analytical precision of these metal analyses in the clay matrix. Table 57 provides this comparison. As indicated in Table 57, the demonstrated analytical precision for the four metals ranged from 3.6 to 16 percent RSD. Again, all these results were within the precision DQO of 25 percent RSD.

TABLE 56. AQUEOUS SPIKE SOLUTION ANALYSIS ACCURACY AND PRECISION

| Metal | Prepared spike solution concentration (ppm as metal) | Analysis recovery (%) ^a | | | | | | | | RSD (%) |
|-----------|---|------------------------------------|---------------------|---------------------|---------------------|---------------------|---------------------|----------------------|----------------------|------------|
| | | Test 4 (9/14/88) | Test 5 (8/25/88) | Test 6 (9/16/88) | Test 7 (8/30/88) | Test 8 (9/07/88) | Test 9 (9/09/88) | Test 10 (9/20/88) | Test 11 (9/22/88) | |
| Arsenic | 3230 | 87 | 71 | 90 | 74 | 74 | 71 | 111 | 102 | 18 |
| Barium | 2580 | 89 | 70 | 93 | 93 | 97 | 97 | 97 | 89 | 10 |
| Bismuth | 9040 | 111 | 73 | 122 | 82 | 104 | 122 | 122 | 122 | 18 |
| Cadmium | 650 | 93 | 63 | 83 | 82 | 80 | 83 | 98 | 83 | 12 |
| Chromium | 3230 | 96 | 71 | 90 | 90 | 96 | 93 | 102 | 90 | 10 |
| Copper | 32,300 | 90 | 71 | 93 | 93 | 99 | 96 | 93 | 93 | 9 |
| Lead | 3230 | 105 | 74 | 93 | 96 | 99 | 96 | 108 | 93 | 11 |
| Magnesium | 5040 | 91 | 67 | 89 | 85 | 87 | 91 | 97 | 85 | 10 |
| Strontium | 16,100 | 112 | 68 | 93 | 87 | 93 | 99 | 118 | 106 | 16 |

^aAnalyzed amount (see Table 19)/prepared amount.

TABLE 57. FEED CLAY ANALYSIS PRECISION

| Metal | Clay sample metal concentration (ppm) | | | | | | | | | | RSD (%) |
|-----------|---------------------------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|-------------------|-------------------|---------|
| | Test 1/2 composite (9/28,29/88) | Test 3 (9/26/88) | Test 4 (9/14/88) | Test 5 (8/25/88) | Test 6 (9/16/88) | Test 7 (8/30/88) | Test 8 (9/07/88) | Test 9 (9/09/88) | Test 10 (9/20/88) | Test 11 (9/22/88) | |
| Chromium | 54 | 52 | 55 | 55 | 54 | 53 | 54 | 55 | 50 | 50 | 3.6 |
| Lead | NA ^a | NA | 3.2 | 3.7 | 3.5 | 2.9 | 3.1 | 2.3 | 3.0 | 2.5 | 16 |
| Magnesium | NA | NA | 23,000 | 23,000 | 22,000 | 22,000 | 22,000 | 22,000 | 20,000 | 22,000 | 4.2 |
| Strontium | NA | NA | 33 | 37 | 36 | 32 | 33 | 30 | 33 | 35 | 6.7 |

^aNA: not analyzed.

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