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**EMISSIONS OF METALS, CHROMIUM AND NICKEL SPECIES, AND ORGANICS
FROM MUNICIPAL WASTEWATER SLUDGE INCINERATORS**

VOLUME III: SITE 6 EMISSION TEST REPORT

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DISCLAIMER

This material has been funded wholly or in part by the United States Environmental Protection Agency's Risk Reduction Engineering Laboratory and Office of Water under contract numbers 68-02-4442, Work Assignment No. 81, 68-02-4462, Work Assignment No. 90-108, and 68-C0-0027, Work Assignment No. 0-5. It has been subject to the Agency's review and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute an endorsement or recommendation for use.

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 research and the user community.

The problem of disposing of primary and secondary sludge generated at municipal wastewater treatment facilities is one of growing concern. Sludge of this type may contain toxics such as heavy metals and various organic species. Viable sludge disposal options include methods of land disposal or incineration. In determining the environmental hazards associated with incineration, the Risk Reduction Engineering Laboratory and the Office of Water has sponsored a program to monitor the emissions of metals and organics from a series of municipal wastewater sludge incinerators. The following document presents the final results from the Site 6 emissions test program.

E. Timothy Oppelt, Director
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ABSTRACT

The U.S. Environmental Protection Agency (EPA) Office of Water (OW) has drafted risk-based sludge regulations under Section 405d of the Clean Water Act and EPA's Risk Reduction Engineering Laboratory (RREL) has been assisting OW in the collection of supporting data for the proposed regulations. Proposed regulations and a solicitation for public comment was published in the Federal Register on February 6, 1989. Final regulations are scheduled for publication in the Federal Register in January 1992. Because of the associated cancer risk, there is particular concern regarding chromium and nickel species in the emissions from sludge incineration.

An RREL/OW research program was implemented to determine the ratios of hexavalent to total chromium and nickel subsulfide to total nickel in sewage sludge incinerator emissions under varied incinerator operating conditions. Site 6, a multiple hearth furnace was tested under two operating conditions, normal combustion was compared with improved combustion conditions as indicated by reduced CO and THC emissions. This report presents the test results from Site 6, the second of five incinerator test sites. Four incinerators tested under another project with Radian Corporation are included in the site numbering convention used. Thus the second site, covered under the present project, and by this report, is referred to as Site 6.

The effect of lime conditioning on the conversion of total chromium in the sludge to hexavalent chromium emissions was also a primary concern at Site 6. Secondary objectives included comparing the results for chromium and nickel subspecies determined by different analytical procedures, gathering data on other metals and inorganic/organic gases in incinerator emissions, and assessing pollutant removal efficiencies by measuring emissions at both the inlet and outlet to the control system.

The Site 6 plant treats 30 million gallons a day of municipal and industrial wastewater. The blended primary/secondary sludge is dewatered to approximately 28% solids using recessed plate cloth filters. The dried filter cakes are incinerated in an eight-hearth unit and emissions are controlled with a venturi scrubber and impingement tray scrubber.

The flue gas volumetric flow rates at the inlet sampling location were fairly consistent averaging 475 dry standard cubic meters per minute (dscm/min) for normal operating conditions and 430 dscm/min for low CO conditions. Averaged temperatures of the flue gas were 876°F (469°C) for the normal operating conditions and 1027°F (553°C) for the low CO conditions with a moisture content of 33.1% and 33.2%, respectively. The percent dry of oxygen, carbon dioxide were 13.0 and 7.4, respectively for normal conditions and 11.5 and 7.7 respectively for low CO conditions. The carbon monoxide emissions corrected to 7% O₂ for the normal and low CO operating conditions were 1290 and 620 ppm, respectively.

The average flue gas volumetric flow rates at the outlet sampling site ranged from 522.4 dscm/min for normal operating conditions to 531.1 dscm for low CO conditions. Averaged temperatures of the flue gas were 144°F (62.4°C) for normal operating conditions and 147°F (64.1°C) with a moisture content of 6.7% and 7.2% respectively. The percentage of dry oxygen and carbon dioxide were 14.1 and 5.6 respectively for the normal operating conditions and 13.3 and 6.3, respectively for the low CO condition. The carbon monoxide levels corrected to 7% oxygen for both the normal and low CO conditions were 1318 and 592 ppm, respectively. The THC emissions for the normal and low CO were 24 and 8 ppm, respectively.

The metal mass emissions rate for the inlet runs averaged: Arsenic (As) - not detected (< 862 mg/hr), Beryllium (Be) - 15.9 mg/hr, Cadmium (Cd) - 5,840 mg/hr, Chromium (Cr) - 12,400 mg/hr, Lead (Pb) - 86,600 mg/hr, and Nickel (Ni) - 1,220 mg/hr. The metal mass emissions rate for the outlet runs averaged: As - not detected (< 508 mg/hr), Be - not detected (< 2.2 mg/hr), Cd - 1,450 mg/hr, Cr - 83.3 mg/hr, Pb - 21,100 mg/hr, and Ni - 73.9 mg/hr. The particulate mass emission rates averaged 42 kg/hr and 0.7 kg/hr, respectively for the inlet and outlet.

A positive correlation between the CO/CO₂ ratios (an indication of combustion conditions) and the hexavalent to total chromium ratio was demonstrated for the outlet location. At low CO levels (good combustion) the ratio of hexavalent chromium to trivalent chromium was highest, with approximately 10% of the total chromium in the form of hexavalent chromium. At high CO levels (poor combustion), the ratio of hexavalent chromium to total chromium was significantly reduced to less than approximately 1%.

It was anticipated that the nickel subsulfide emissions from multiple hearth incinerators would constitute less than 1% of the total nickel emissions. A wet chemical analysis indicated that within the detection limit (< 10%), no nickel subsulfide was present in the samples. Samples were also analyzed by X-ray absorption near-edge structure (XANES) and extended X-ray absorption fine structure (EXAFS); no nickel subsulfide was detected within the instrumental detection limit of 10% of the total nickel.

EPA is evaluating CO and THC monitoring as a surrogate indicator of organic emissions. With the exclusion of one run, the correlation coefficient between CO and THC emissions under the tested conditions was 0.97 for the 2- and 4-hr runs.

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1.0 INTRODUCTION

The U.S. Environmental Protection Agency (EPA) Office of Water (OW) has been developing new regulations for sewage sludge incinerators and EPA's Risk Reduction Engineering Laboratory (RREL) has been assisting OW in the collection of supporting data. There is particular concern regarding chromium and nickel species in the emissions from incineration of municipal wastewater sludge because of the associated cancer risk. OW has drafted risk-based sludge regulations under Section 405d of the Clean Water Act which have been published for comment in the Federal Register, Volume 54, No. 23, February 6, 1989. Final regulations are scheduled for publication in the Federal Register in January 1992.

The draft regulations are based on the risk incurred by the "most exposed individual" (MEI). The MEI approach involves calculating the risk associated with an individual residing for seventy years at the point of maximum ground level concentration of the emissions just outside the incinerator facility property line. EPA's proposal for regulating sewage sludge incinerators is based on ensuring that the increase in ambient air concentrations of metal pollutants emitted from sludge incinerators is below the ambient air human health criteria. The increase in ambient air concentrations for four carcinogenic metals, arsenic, chromium, cadmium, and nickel, are expressed as annual averages. The concentrations are identified in the proposed regulations as Risk Specific Concentrations (RSC). Both nickel and chromium emissions from sludge incinerators presented a specific problem in establishing RSCs, because unknown portions of the emissions of these metals are in forms which are harmful to human health. In performing the risk calculations, EPA assumed that 1% of the emissions of chromium from the sludge incinerators is in the most toxic form, hexavalent chromium. For nickel,

EPA assumed that 100% of the nickel emissions are in the most toxic form, nickel subsulfide.

Chromium is likely to be emitted in either the highly carcinogenic hexavalent state (Cr^{+6}) or in the noncarcinogenic trivalent state (Cr^{+3}). Trivalent chromium has not been shown to be carcinogenic and is toxic only at levels higher than those normally found in sewage sludge incinerator emissions. Although hexavalent chromium (as the most oxidized form) could be reasonably expected to result from combustion processes, investigators speculate that most of the chromium is likely to be emitted in the trivalent state.¹ This is because hexavalent chromium is highly reactive, and thus likely to react with reducing agents to form trivalent chromium.

Studies have been conducted to determine the potential for chromium in sewage sludge to be converted to the hexavalent form. Analysis of laboratory combusted sludges dosed with various levels of lime and ferric chloride revealed that the hexavalent to total chromium ratio increased with lime dosage.¹ One-hundred percent conversion of chromium to hexavalent chromium was observed in several of the tests.¹ These tests indicate that when lime and ferric chloride are used as sludge conditioners, high ratios of hexavalent to total chromium may be formed under certain incinerator operating conditions.

EPA has previously sponsored emission testing studies for measurement of hexavalent chromium at two sludge incinerators.^{2,3} For one site, the hexavalent chromium concentrations were below the analytical detection limit; for the other site, a hexavalent-to-total chromium ratio of 13% was calculated. The 1% value chosen for the draft regulations may seem low. This is the result, however, of weighting various values to give the most credible ones more influence. With this approach, lower values were assigned a stronger contribution. The lack of a substantial data base on hexavalent chromium emissions prompted the following statement in the EPA's Technical Support Document for the Incineration of Sewage Sludge: "EPA plans to perform additional tests of sewage sludge incinerator emissions for hexavalent chromium before this proposed rule is finalized. The additional data should allow the Agency to better understand how hexavalent chromium is generated in sewage sludge incinerators." As previously stated,

EPA assumed that 100% of the nickel emissions are in the subsulfide form to calculate an RSC. Since the Agency had no nickel subsulfide emission data from sewage sludge incinerators, it took the most conservative approach in conducting the nickel risk analysis and assumed that all emitted nickel compounds cause the same health effects as nickel subsulfide. Again, the Technical Support Document stated: "As additional data become available on the form of chromium and nickel emissions from combustion sources, the Agency will consider what changes, if any, would be appropriate for these proposed regulations."

There were no published EPA emission measurement test methods for the sampling and analysis of hexavalent chromium or nickel subsulfide. In addition, very little data exist on the conditions that may cause their formation. The primary objectives of the RREL/OW research described in this report were to implement sampling and analysis procedures to determine the ratio of hexavalent to total chromium and the ratio of nickel subsulfide to total nickel in sewage sludge incinerator emissions under varied excess air incinerator operating conditions. High excess air in the furnace presents conditions favorable for the formation of hexavalent chromium; low excess air presents conditions favorable for the formation of nickel subsulfide. The effect of lime conditioning on the conversion of total chromium in the sludge to hexavalent chromium emissions was also a primary concern at this test site. Based on the results under normal to high excess air, the test program was modified on-site to reflect normal and improved combustion conditions (reduced CO and THC levels). Secondary objectives include comparing the results for emissions of chromium and nickel subspecies determined by different analytical procedures, as well as gathering data on other metals and inorganic and organic gaseous components in uncontrolled and controlled incinerator emissions. Continuous emissions monitoring of oxygen (O_2), carbon dioxide (CO_2), carbon monoxide (CO), sulfur dioxide (SO_2), and oxides of nitrogen (NO_x) at the control system inlet and CO and total hydrocarbons (THC) at the control system outlet stack were used to establish process and control equipment operation during the manual testing and to provide additional emissions data.

This report presents the results for the Site 9 test program, the second in a series of five emission tests (Sites 5, 6, 7, 8 and 9) completed for this portion of RREL's research program. This report is organized in two volumes. The Emission Report is contained in Volume III, while the Appendices are included in Volume IV.

The following sections present detailed descriptions of the testing and results from the Site 6 program. Section 2.0 presents a summary of the results. Section 3.0 presents the process description and process operating conditions. Section 4.0 provides a detailed discussion of the sampling and analytical results. Section 5.0 describes the sampling location and procedures and Section 6.0 describes the analytical procedures. The quality assurance/quality control activities and results are presented in Section 7.0.

2.0 SITE 6 TEST SUMMARY

2.1 TESTING PROGRAM DESIGN

The main emphasis for the testing at Site 6 was to determine the effect of lime conditioning and excess air on the conversion of total chromium in the sludge to hexavalent chromium emissions. The following criteria were considered in selecting this test site: adequate levels of chromium and nickel in the sludge, suitable sampling locations, use of lime in conditioning sludge, adequate latitude in control of excess air, multiple hearth furnace, venturi scrubber capable of achieving medium to high pressure drops, and not previously tested for RREL.

The incinerator emissions were tested under two operating conditions, high and low levels of excess air in the furnace. High excess air in the furnace presents conditions favorable for the formation of hexavalent chromium, and low excess air presents conditions favorable for the formation of nickel subsulfide. In addition to speciation of chromium and nickel emissions, sampling was also conducted for trace metals and continuous emissions monitoring (CEM) techniques were used to measure O₂, CO₂, CO, SO₂, and NO_x at the control system inlet and CO and THC at the control system outlet stack. The monitoring data were used principally to determine process and control equipment operating conditions during the chromium and nickel speciation tests.

The emission testing at Site 6 was conducted from October 9 to October 13, 1989. The test program sampling matrix is shown in Table 2-1. Sampling was conducted at the inlet and outlet of the venturi/impingement tray scrubber used to control the incinerator emissions. Certain inlet and outlet flue gas conditions (see Table 2-1) were monitored continuously while establishing incinerator operating conditions.

TABLE 2-1. TEST PROGRAM SAMPLING MATRIX

Incinerator Operating Conditions	Flue Gas Samples*		Solid/Liquid Samples		
	Control Device Inlet*	Control Device Outlet	Sludge†	Scrubber† Inlet Outlet	Ash†
Normal Combustion	<u>Hexavalent Chromium Sampling</u>				
	Cr+6 (F) (4x3 runs)	Cr+6 (RC) (4x3 runs)	3 runs	3 runs 3 runs	3 runs
	CEMS (3 runs)	CEMT (3 runs)			
	<u>Nickel/Metals Sampling</u>				
	Ni (2x3 runs)	Ni (2x3 runs)	3 runs	3 runs 3 runs	3 runs
	Mtl (1x3 runs)	Mtl (1x3 runs)			
Low CO and THC	CEMS (3 runs)	CEMT (3 runs)			
	<u>Hexavalent Chromium Sampling</u>				
	Cr+6 (F) (4x2 runs)	Cr+6 (RC) (4x2 runs)	3 runs	3 runs 3 runs	3 runs
	CEMS (2 runs)	CEMT (2 runs)			
	<u>Nickel/Metals Sampling</u>				
	Ni (2x3 runs)	Ni (2x3 runs)	3 runs	3 runs 3 runs	3 runs
	Mtl (1x3 runs)	Mtl (1x3 runs)			
	CEMS (3 runs)	CEMT (3 runs)			

*Cr+6 (F) = Filter sampling train for hexavalent chromium.

Cr+6 (RC) = Recirculating sampling train for hexavalent chromium.

CEMS = Continuous emissions monitoring system (O₂, CO₂, CO, SO₂, NO_x).

CEMT = Continuous emissions monitoring system (O₂, CO₂, CO, SO₂, NO_x, and THC).

Mtl = Multiple metals sampling train, included in the nickel train

Ni = Nickel sampling train

M3 = Method 3 sampling train for CO₂, O₂

† Sludge was grab sampled and analyzed for metals, proximate and ultimate analysis; scrubber water and ash were grab sampled and analyzed for metals.

The specific elements and compounds of interest are presented in Table 2-2. A summary of the sampling and analytical methods used to conduct the testing are presented in Table 2-3. Two approaches were used for measuring the outlet emissions of hexavalent chromium: (1) method 5-type sampling system and (2) a recirculating impinger reagent sampling system. Only the Method 5-type of sampling system was used at the inlet site for measurement of hexavalent chromium because a high temperature recirculating train had not been built. Five runs were conducted at the inlet and outlet locations at Site 6 for arsenic, beryllium, cadmium, chromium, lead, and nickel. The particulate emissions were also determined using the multiple metals sampling system. Composite sludge feed samples, bottom ash samples, and scrubber water inlet and outlet samples were taken during each test series. The sampling and analytical methods are described in detail in Sections 5.0 and 6.0, respectively.

2.2 TEST PROGRAM RESULTS

This section summarizes the test results for the Site 6 test program. Site 6 was originally scheduled for testing at two process operating conditions: (1) normal to high excess air and (2) low excess air. Initial testing under normal combustion conditions resulted in high emissions of CO and THC, which are associated with poor combustion conditions. In response, the desired process operating conditions for the second series of runs were modified from low excess air to setting incinerator conditions that would reduce the levels of CO and THC. This modification was achieved by changing sludge feed rate and the combustion air distribution and burning more auxiliary fuel oil. The emission results and associated emission factors are highlighted in this section; the run by run data, as well as the process sample results, are detailed in Section 4.0. The test program schedule is presented in Section 2.2.1. Particulate and metal results are summarized in Section 2.2.2, hexavalent chromium results are summarized in Section 2.2.3, nickel speciation results are summarized in Section 2.2.4, total hydrocarbon (THC) and carbon monoxide (CO) results are summarized in Section 2.2.5, and conclusions are presented in Section 2.2.6.

TABLE 2-2. SPECIFIC ELEMENTS AND COMPOUNDS OF INTEREST

Metals*	Chromium Species†	Nickel Species‡	Combustion Gas and Criteria Pollutants
Arsenic	Hexavalent chromium	Nickel sulfate	O ₂
Beryllium	Trivalent chromium	Nickel sulfide	CO ₂
Cadmium		Nickel subsulfide	CO
Chromium		Metallic nickel	SO ₂
Lead		Nickel oxide	NO _x
Nickel			THC

* These metals are of specific interest to OW. Analysis by ICAP; chromium and nickel analysis also by XANES.

† The chromium subspecies are of interest to OW since their contribution to total sludge incinerator chromium emissions has been variable, thus giving unreliable estimates of total chromium emissions.

‡ Nickel subspecies are of interest to OW because no data are available on nickel subsulfide.

TABLE 2-3. SUMMARY OF SAMPLING AND ANALYTICAL METHODS

Sampling Location	Parameter	Sampling Method	Analysis Method
Inlet to the Control Device	• Total chromium, Cr ⁺⁶	EPA Draft ^{a,b} Cr ⁺⁶ Methods	IC/PCR, gamma counter, Xanes, ICAP/AA
	• Total nickel, nickel subsulfide	EPA Draft ^c Ni Method	EPA Draft Method, Xanes, ICAP/AA
	• Particulates metals ^d	EPA Draft Ni Method	ICAP/AA
	• O ₂ , CO ₂ , CO NO _x , SO ₂ ,	3A, 10, 7E and 6C	3A, 10, 7E and 6C
	• Fixed gases (O ₂ , CO ₂)	Method 3	Orsat
	• Moisture	Method 4	Gravimetric
Outlet to the Control Device	• Total chromium, Cr ⁺⁶	EPA Draft ^{a,b} Cr ⁺⁶ Methods	IC/PCR, gamma counter Xanes, ICAP/AA
	• Total nickel, nickel subsulfide	EPA Draft ^c Ni Method	EPA Draft Method Xanes, ICAP/AA
	• Particulates, metals ^d	EPA Draft Ni Method	ICAP/AA
	• O ₂ , CO ₂ , CO NO _x , SO ₂ ,	3A, 10, 7E and 6C	3A, 10, 7E and 6C
	• THC	Method 25A	FID
	• Fixed gases (O ₂ , CO ₂)	Method 3	Orsat
	• Moisture	Method 4	Gravimetric
Solid Samples	Sludge feed	Grab samples	- ^e
	Scrubber water:		
	inlet	Grab samples	- ^f
	outlet	Grab samples	- ^f
	Bottom ash	Grab samples	- ^f

^a Recirculating train for labelled hexavalent chromium, with 0.1 N NaOH impinger solution.

^b Method 5 type sampling train for chromium, with 0.1 N NaOH impinger solution.

^c Method 5 type sampling train for nickel.

^d Metals analysis will include chromium, nickel, arsenic, lead, cadmium, and beryllium.

^e Analysis for metals, moisture, proximate and ultimate analysis, and heating value by methods described in Section 5.0.

^f Analysis for total metals by ICAP/AA.

2.2.1 Test Program

The Site 6 test program schedule is summarized in Table 2-4. Included in the table are the sampling locations, run numbers, sample times, incinerator operating conditions, and comments on the sampling and incinerator operating conditions. Testing was conducted with high THC and CO emissions (normal combustion) and with low THC and CO emissions (improved combustion). Due to time limitations, one run was conducted during the transition from normal combustion to improved combustion conditions.

2.2.2 Particulate/Metals Results

The particulate/metals train runs were conducted simultaneously with the nickel speciation runs. Two runs (Runs 5 and 6) were conducted during normal incinerator operations (high THC and CO), one run (Run 8) during transition from normal combustion conditions to improved combustion conditions (low THC and CO), and two runs (Runs 10 and 12) during improved combustion conditions (low THC and CO). Run 8 is presented with the low THC/CO runs. The results for all the metals except lead (see Table 2-5) were similar. The lead emissions from the incinerator increased with the improved combustion (higher furnace temperatures). The particulate and metal removal (collection) efficiency of the venturi/tray scrubber control system is also presented in Table 2-5. The removal efficiency for the particulate emissions averaged 98.1%. The removal efficiency for all the metals was less than the particulate with the exception of chromium which was 99.3%. As discussed in Section 4.3.2, the measured chromium emissions of the multiple metals train are about one half of the measured chromium emissions using the hexavalent chromium sampling train.

2.2.3 Hexavalent Chromium Results

The hexavalent chromium samples from the Method 5-type trains used to conduct the inlet sampling and some of the outlet sampling were analyzed by EPA's Environmental

TABLE 2-4. SUMMARY OF SCRUBBER INLET AND OUTLET FLUE GAS CONDITIONS

Run No. & Condition	Sampling Location	Test Date	Run Time	Flue Gas Conditions				Comments on sampling and Incinerator Operating Conditions
				Temperature (°F)	Moisture (% H ₂ O)	Oxygen (% dry)	Air Flow Rate (dscm/min)	
Run 2 Normal	Inlet Outlet	10/09/89	10:10-11:10	1340	6.6	NA*	450.9	Outlet Cr+6 tests for EMSL, data not released Process data was not taken
Run 3 Normal	Inlet Outlet	10/09/89	12:45-14:45 12:45-14:45	861 138	30.3 6.1	13.4 14.0	538.4 585.2	Clinkers were removed from furnace Inlet Cr+6 data not released
Run 4 Normal	Inlet Outlet	10/09/89	17:00-19:00 17:00-19:00	963 138	29.2 6.9	14.8 14.1	620.8 556.9	With 2 min left on test sludge was interrupted and burnout occurred, should not effect results
Run 5 Normal	Inlet Outlet	10/10/89	09:00-11:00 09:10-11:10	903 146	40.1 6.3	10.3 13.2	378.1 520.5	Sludge cake which was high in moisture, lime added. Feed stopped at end of run.
Run 6 Normal	Inlet Outlet	10/10/89	14:15-16:15 14:30-17:00	928 144	34.9 6.5	12.7 14.4	369.8 466.5	Lost scrubber water at 14:45. Outlet tests stopped and restarted when water flow resumed.
Run 7 Normal	Inlet Outlet	10/10/89	18:50-20:50 18:37-21:37	820 135	32.3 7.3	13.5 14.2	451.1 498.3	Sludge cake wet and low in volatiles, more burners were added to maintain temperatures
Run 8 Transition	Inlet Outlet	10/11/89	09:45-11:45 09:45-11:45	1045 155	40.9 7.4	9.1 11.9	353.6 463.6	Low O ₂ caused a lot of smoking during first part of the sample run.
Run 9 Low CO	Inlet Outlet	10/11/89	16:22-18:22 15:43-18:43	1084 149	34.5 6.9	11.5 13.4	505.9 634.1	Low CO was achieved with additional burners to obtain a high Hearth #1 temperature.
Run 10 Low CO	Inlet Outlet	10/12/89	09:00-11:00 09:00-11:00	1014 143	32.7 7.3	11.5 13.2	372.1 454.2	Fuel pump failure for very short period.
Run 11 Low CO	Inlet Outlet	10/12/89	13:15-15:15 12:31-15:31	1050 148	33.1 8.1	11.5 13.1	522.0 629.0	No problems
Run 12 Low CO	Inlet Outlet	10/12/89	17:45-19:45 17:32-19:32	962 150	32.5 6.6	11.6 13.6	319.6 407.2	No problems
Run 13 Normal	Inlet Outlet	10/13/89	09:00-11:00 09:00-13:00	783 171	31.9 7.4	13.4 14.5	493.9 578.8	No problems

* Not available

TABLE 2-5. SUMMARY OF INLET AND OUTLET PARTICULATE AND METALS MASS EMISSION RATES

Run No. and Location	Particulate Mass Rate (kg/hour)	Flue Gas Metal Mass Emission Rate (mg/hour)					
		Arsenic	Beryllium	Cadmium	Chromium	Lead	Nickel
Run 5 Inlet	50.8	< 969	16.9	5650	14600	83000	1470
Run 5 Outlet	0.7	< 521	< 2.3	1260	65.8	16700	90.7
Efficiency*, %	98.6	NA†	> 86.6	77.7	99.5	79.9	93.8
Run 6 Inlet	37.0	< 761	16.6	3710	11200	42400	1110
Run 6 Outlet	0.6	< 591	< 2.6	1380	113	19300	79.7
Efficiency, %	98.4	NA	> 84.5	62.9	99.0	54.5	92.8
Run 8 Inlet	19.8	< 860	11.2	11100	8990	148000	1010
Run 8 Outlet	1.1	< 531	2.3	2590	164	30500	109
Efficiency, %	94.6	NA	79.4	76.8	98.2	79.4	89.2
Run 10 Inlet	34.4	< 787	13.7	5130	11900	94700	1140
Run 10 Outlet	0.7	< 573	< 2.5	1200	47.3	22500	34.9
Efficiency, %	97.8	NA	> 81.8	76.7	99.6	76.2	96.9
Run 12 Inlet	47.1	< 926	20.1	4750	15100	89000	1300
Run 12 Outlet	0.6	< 278	1.2	987	26.5	19400	44.7
Efficiency, %	98.8	NA	94.0	79.2	99.9	78.2	96.6
Runs 5 and 6 Inlet	43.9	< 865	16.7	4680	12900	62700	1290
Runs 5 and 6 Outlet	0.6	< 556	< 2.4	1320	89.5	18000	85.2
Efficiency, %	98.5	NA	> 85.5	71.9	99.3	71.3	93.4
Runs 8, 10, & 12 Inlet	33.8	< 858	15.0	7000	12000	110000	1150
Runs 8, 10, & 12 Outlet	0.8	< 461	< 2.0	1590	77.3	24100	62.7
Efficiency, %	97.6	NA	> 86.7	77.3	99.4	78.1	94.5
Total Average Inlet	38.8	< 862	15.9	5840	12400	86600	1220
Total Average Outlet	0.7	< 508	< 2.2	1450	83.3	21100	73.9
Efficiency, %	98.1	NA	> 86.1	75.1	99.3	75.7	93.9

* Collection efficiency of air pollution control system.

† Not applicable.

Monitoring Systems Laboratory (EMSL) in Cincinnati, Ohio. The data from these trains have not been released for publication by EMSL and will not be presented in this report.

The hexavalent chromium results for the recirculating reagent sampling train testing conducted at the outlet are shown in Figure 2-1 where the ratio of hexavalent chromium to total chromium is plotted against the combustion efficiency factor (CO divided by CO_2). Three runs (Runs 3, 7 and 13) were conducted during normal combustion and two runs (Runs 9 and 11) were conducted during improved combustion. Run 7 has been excluded from the plot which shows a direct relationship between good combustion and higher concentrations of hexavalent chromium. During normal combustion (higher THC and CO), the hexavalent chromium constituted only 1% of the total chromium emissions; during good combustion, the hexavalent chromium constituted about 8% of the total chromium emissions. A primary concern with draft sampling methods for hexavalent chromium is the conversion of the collected hexavalent chromium to trivalent chromium in the sampling system. During the Site 6 testing program an average of about 10% of the internal isotopically labeled hexavalent chromium was converted to trivalent chromium. The new procedure instituted for this test program resulted in the 10% loss of hexavalent chromium. These results were the best performance of many draft sampling procedure to this point.

2.2.4 Nickel Speciation

The nickel speciation runs were conducted simultaneously with the particulate/metals train runs. Two runs (Runs 5 and 6) during normal incinerator combustion conditions, one run (Run 8) during the transition from normal to improved combustion, and two runs (Runs 10 and 12) during improved combustion (Low CO and THC) were conducted. Nickel subsulfide cannot be measured directly at levels encountered in these emissions. A wet chemical technique was used to measure sulfidic nickel, the combination of both nickel sulfide and nickel subsulfide. No sulfidic nickel was detected for any of the outlet runs. It was detected in only one of the inlet samples and only at

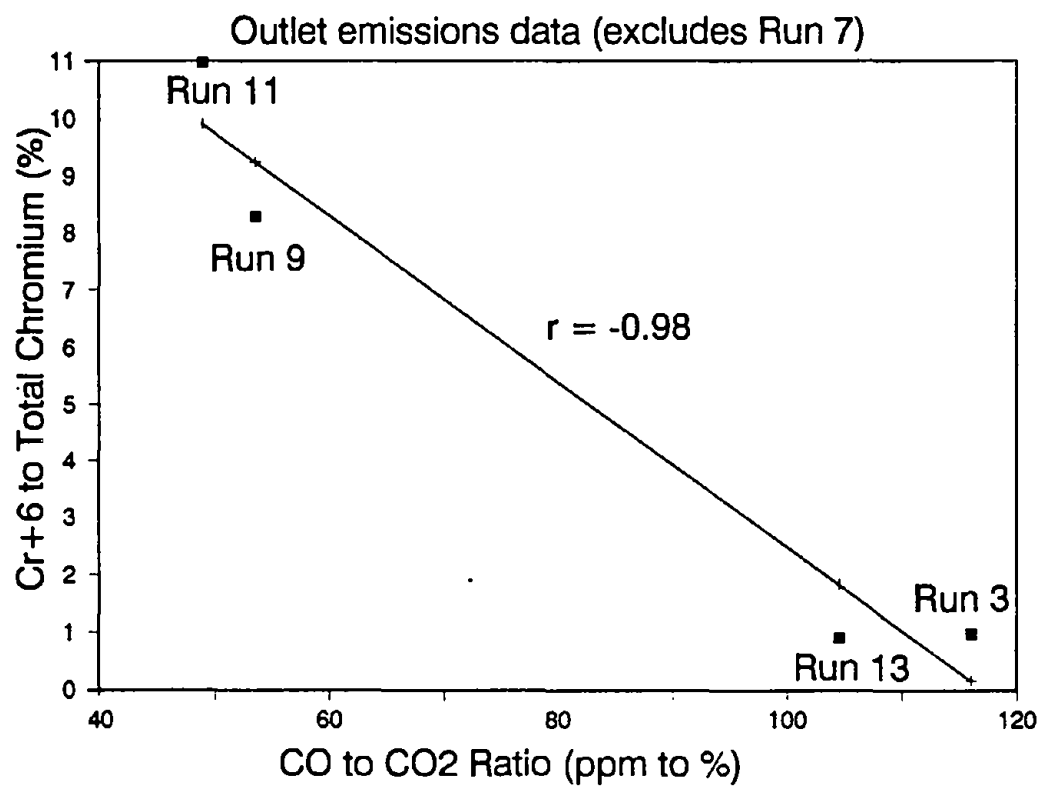


Figure 2-1. Cr⁺⁶ to total chromium versus CO to CO₂ ratios.

the limit of detection which may not be a reliable number. Considering the limit of detection at the outlet, the nickel subsulfide constitutes less than 10% of the total nickel emissions. Considering the limit of detection for the inlet, the nickel subsulfide constitutes less than 12% of the total nickel emissions at this location.

2.2.5 Carbon Monoxide and Total Hydrocarbons

EPA is evaluating monitoring CO and THC emissions as a surrogate indicator of organic emissions. Since no organic compound specific measurements were made at this site, the relationship between CO and THC emissions under the tested conditions is shown in Figure 2-2. When data from Run 5 are excluded, the correlation coefficient is 0.97 for the data from the 2- and 4-hr runs.

2.2.6 Conclusions

From a methods development and data quality perspective, the test program conclusions are as follows:

1. The ratio of hexavalent chromium to total chromium is relatively high (greater than 10%) when lime is used for sludge conditioning, during good combustion conditions, and under the long residence times required for combustion of sludge in a multiple hearth incinerator.
2. The ratio of nickel subsulfide to total nickel was less than detectable (less than 12%) under both furnace operating conditions.
3. There was good correlation between CO emissions and THC emissions.
4. The recirculating impinger reagent train with certain post-sampling procedural modifications yielded acceptable results for the measurement of hexavalent chromium at the outlet.
5. The process operating conditions used for the final series of test runs at Site 6 greatly reduced the level of CO and THC emissions by about 60%.

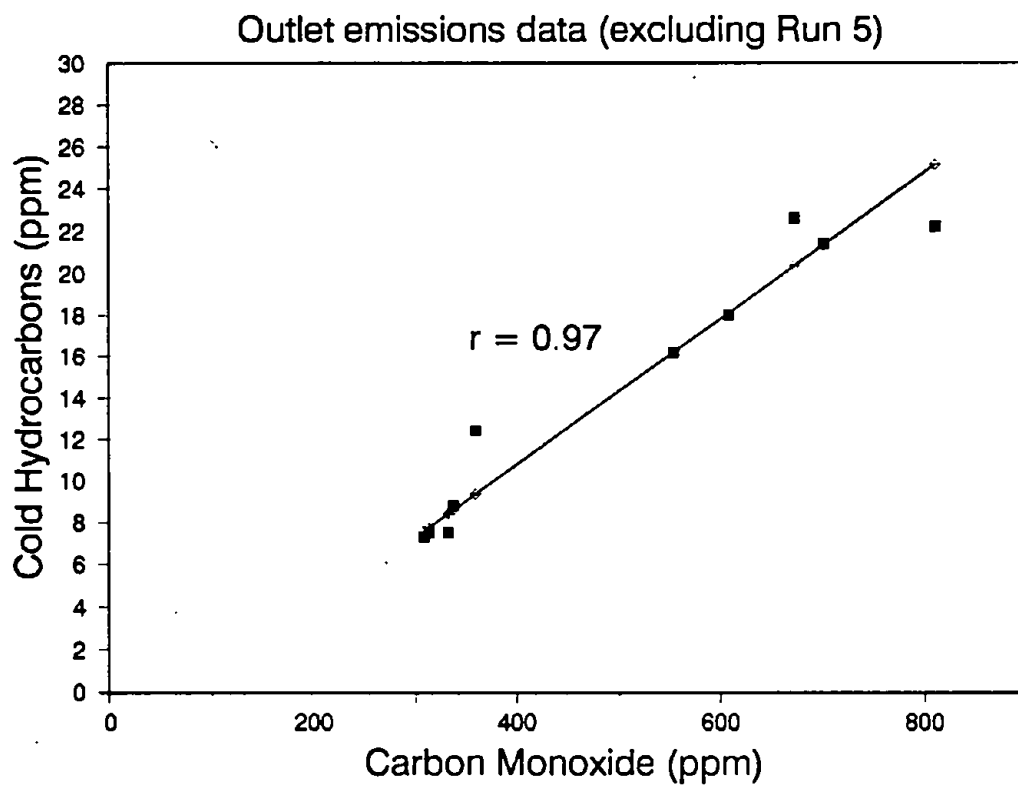


Figure 2-2. Hydrocarbon emissions versus carbon monoxide emissions.

3.0 PROCESS DESCRIPTION AND OPERATION

3.1 FACILITY DESCRIPTION

The Site 6 facility processes an average of 30 million gallons of wastewater per day. The furnace operates 24 hours per day, 5 and three-fourth days a week. The influent to the wastewater treatment facility comes from predominantly (98%) domestic sources. The treatment facility serves a population of approximately 150,000.

Incoming wastewater is screened at four facilities at the plant and degritted at two locations. Screenings and grit are hauled directly to the landfill. The primary treatment consists of four side by side rectangular tanks which receive the degritted and screened wastewater. A chain and flight collector mechanism moves the settled material (primary sludge) to the influent end of the tank and the floating material (grease) to the effluent end of the tank. The primary sludge is pumped to the gravity thickener; the grease is hauled directly to the landfill.

The secondary treatment system consists of three side by side four-pass aeration basins configured to operate in either a step feed or conventional plug flow mode. Diffused air is used. Six circular clarifiers follow this treatment. The waste sludge from this process is concentrated in a dissolved air floatation thickener. Only the three most recent clarifiers have scum removal mechanisms. The secondary scum is pumped to the primary clarifiers. Site 6 advanced treatment includes phosphorus removal. Sludge generated by this process goes to the primary clarifiers.

All sludge is dewatered prior to incineration to reduce the water content of the sludge cake to between 70 to 75% by weight. Dewatering is a critical step in the process of sludge incineration, because it reduces the thermal demand on the incinerators. A gravity thickener is used to increase the percentage of solids in the primary sludge. A

floatation thickener processes the secondary sludge. The combined thickened sludge for those thickeners is then pumped into a storage tank. Lime slurry and ferric chloride solution are used to condition the sludge drawn from the storage tank. Four recessed plate filter presses are available to dewater the conditioned sludge.

The incinerator tested at Site 6 was one of the two identical Nichols eight hearth incinerator which are 22 ft 3 in outside diameter and operates in an excess air mode. The air pollution control system associated with this incinerator consists of an afterburner (which was not used during the test program), a water injection venturi, and an impingement tray scrubber. The incinerator and flue gas treatment system are discussed in greater detail in Section 3.2.

A heat recovery system was installed at the facility but was not functional during the testing program.

3.2 INCINERATOR AND POLLUTION CONTROL SYSTEM

Site 6 has two identical 22 ft 3 in Nichols eight-hearth, multiple hearth furnaces (MHF). Only one of the furnaces is operated at a time. A schematic diagram of the MHF and its pollution control system are presented in Figure 3-1. On most MHFs, the sludge is dropped in through the top, but on this furnace, originally designed for pyrolysis operation, the sludge is screwed into side of Hearth #1. Many MHFs use recycled shaft cooling air to reduce auxiliary fuel consumption. However, Site 6 does not use recycled shaft cooling air due to problems associated with the original design. Air for combustion is admitted through atmospheric ports located in Hearth #7 and Hearth #8. The position of the atmospheric port dampers is controlled with manual loading stations located in the control room. The auxiliary fuel system is oil fired and two (2) burners are located on each of Hearths #2, #4, #5, and #7. Incinerator design data are presented in Table 3-1.

The air pollution control system consists of an adjustable throat venturi scrubber followed by a two (2) plate, impingement tray scrubber. The tray scrubber flue gas exit temperature is nominally 100°F. The position of the venturi adjustable throat is

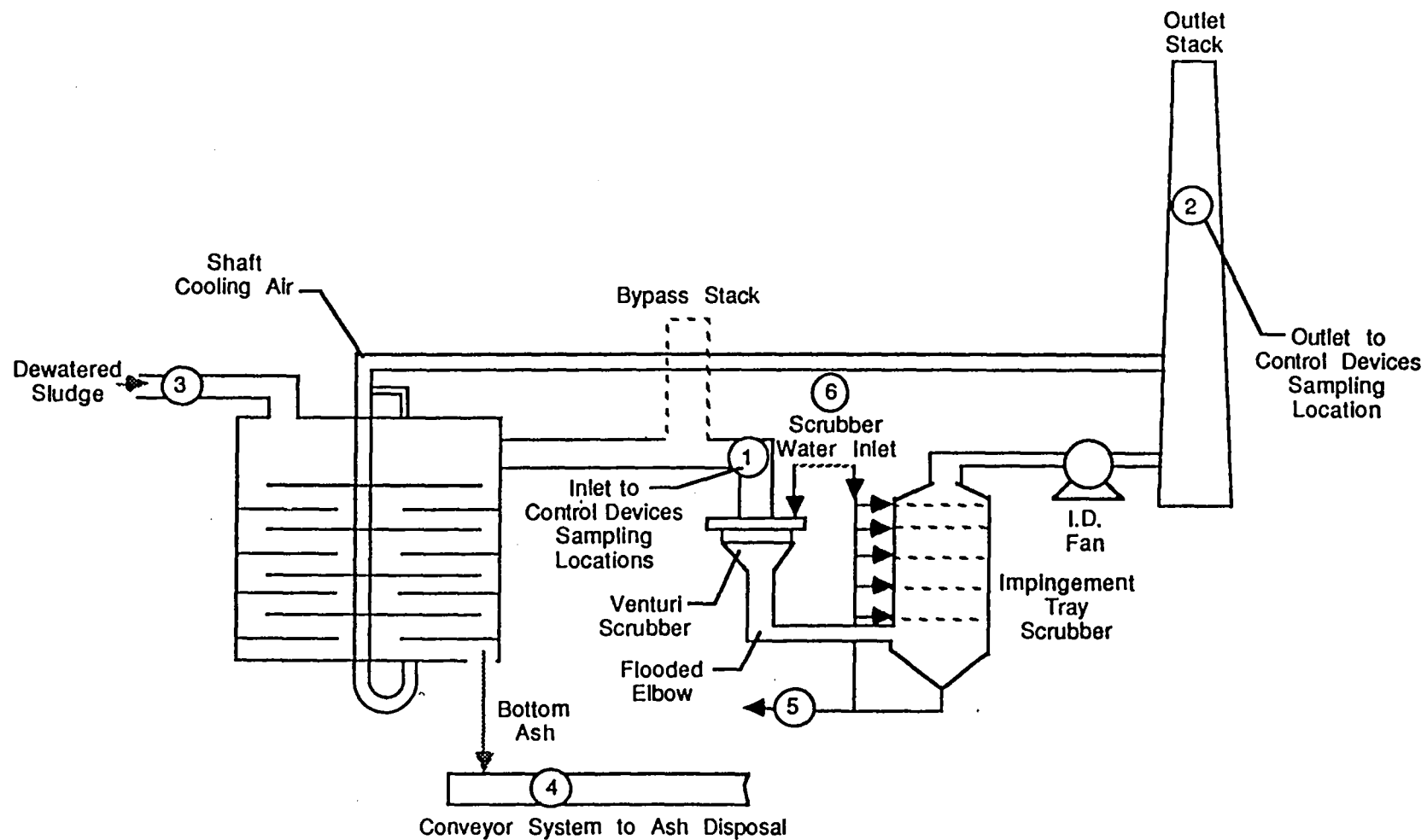


Figure 3-1. Process diagram with sampling locations.

TABLE 3-1. INCINERATOR DESIGN INFORMATION

Design Parameter	Value
<u>Incinerator</u>	
Manufacturer	Nichols
Outside Diameter	22 ft - 3 in
Number of Hearths	8
Recommended Sludge Feed Rate	13,500 lb/hr (wet)
Exhaust Gas Volume (fan rating)	11,518 acfm @ 120 °F
Excess Air	50 - 125%
Oxygen: Furnace Exhaust	7 - 13% Auxiliary
Fuel	Oil
Operating Period	24 hr/day
<u>Pollution Control System</u>	
	<u>Normal</u>
Venturi	233 gpm
Tray Scrubber	776 gpm
<u>Sludge Feed</u>	
Moisture	72% by wt
Solids	28% by wt (wet basis)
Combustible solids	60% by wt (dry basis)
Ash	35% by wt (dry basis)
Heating Value	6,000 Btu/lb

controlled with a manual loading station located in the control room.

The sludge feed to the system is very erratic due to the nature of the dewatering system. The plate and frame filter presses drop sludge into a bunker where the sludge is removed by drag conveyors and deposited onto a belt conveyor system and transported to the furnace. The feed rate is not measured directly and the speed of the drag conveyor is the only indication of the sludge feed rate. Using plant historical data, the sludge feed rate was estimated to be 1.6 dry tons per hour during the testing period. At this site, sludge consistency is also a problem. During the testing period, wide swings in sludge moisture sometimes occurred with each drop of sludge from the filter presses.

3.3 INCINERATOR OPERATING CONDITIONS DURING TESTING

The emission testing at Site 6 was conducted under two separate modes of incinerator operation. The first mode is called "normal operation" which generally results in high levels of carbon monoxide and total hydrocarbons and also a noticeably visible yellow/brown exhaust stack gas. The second mode was an improved operation and resulted in significantly lower emissions of carbon monoxide and total hydrocarbons as well as reduced stack opacity. This test condition is referred to as "low CO".

The furnaces were originally designed for pyrolysis operation; therefore, the auxiliary fuel burner capacity is extremely limited as indicated by the fact that there are only two (2) burners per hearth instead of the more typical four (4) per hearth. To achieve good combustion under the improved conditions (low CO) mode, which requires higher Hearth #1 temperatures, it was necessary to fire burners in Hearths #2, #4, and #5 to raise the temperature in Hearth #1. The very high temperatures in Hearth #3 and Hearth #5 during the improved combustion runs probably contributed to raising the level of heavy metals (particularly lead) emissions.

Figures 3-2 through 3-6 are graphs of the furnace temperature profile during Runs 3, 4, 5, 9, and 13. Runs 3 through 5 are typical of normal mode of operation. Run 9 is typical of improved combustion and Run 13 is an exceptionally steady run in the normal mode.

3-6

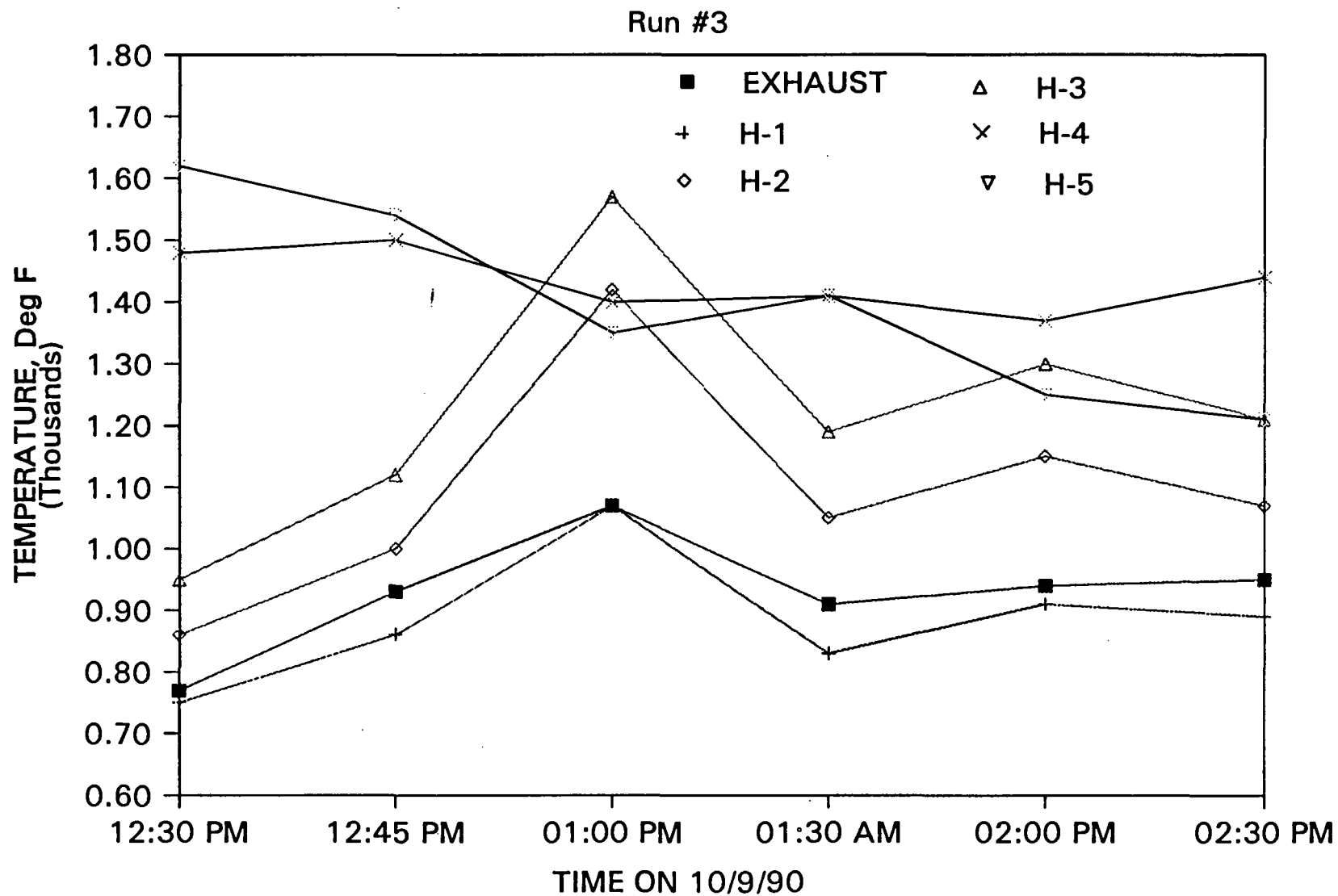


Figure 3-2. Hearth temperature profile during Run 3 - normal mode of operation.

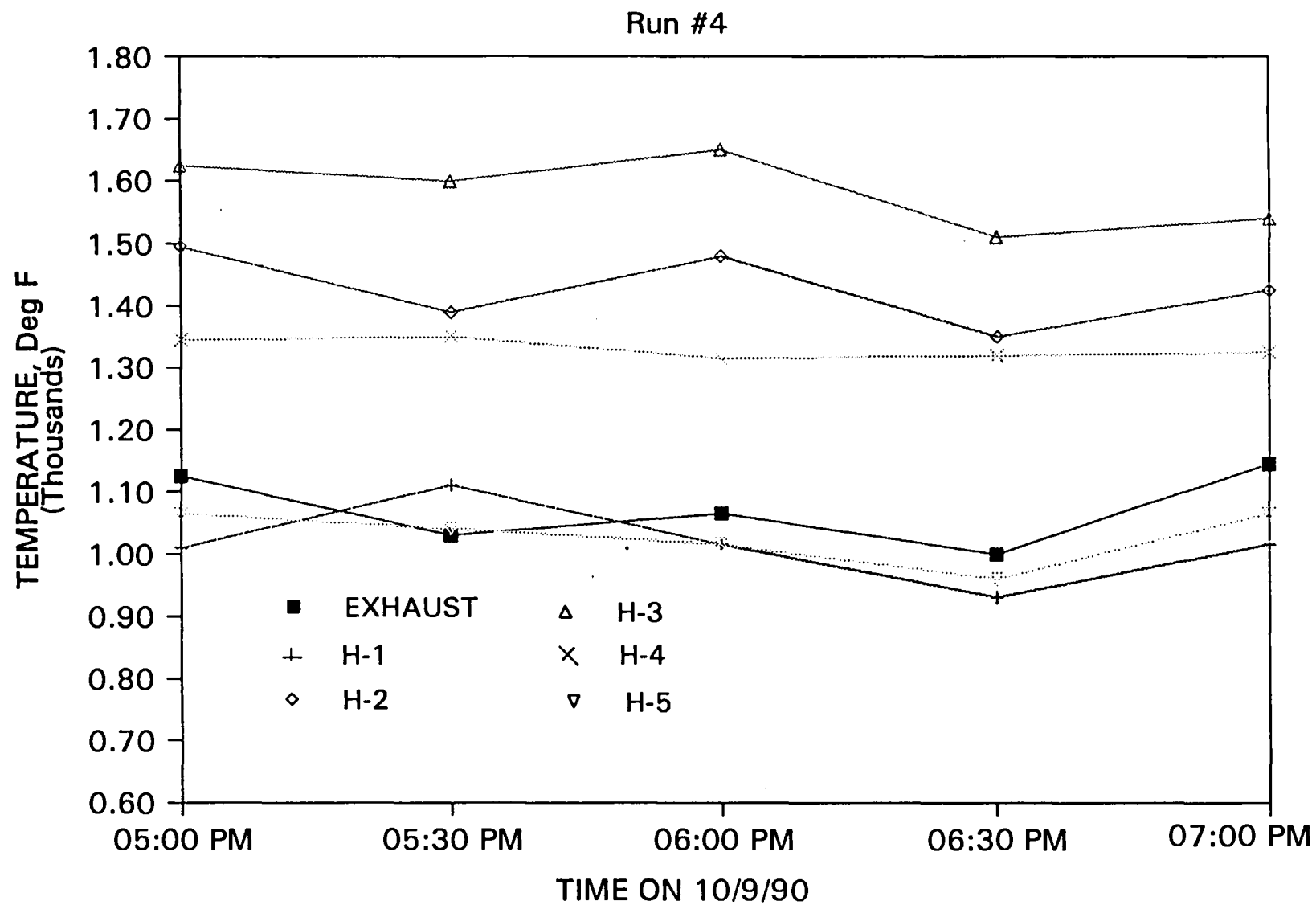


Figure 3-3. Hearth temperature profile during Run 4 - normal mode of operation.

8-8

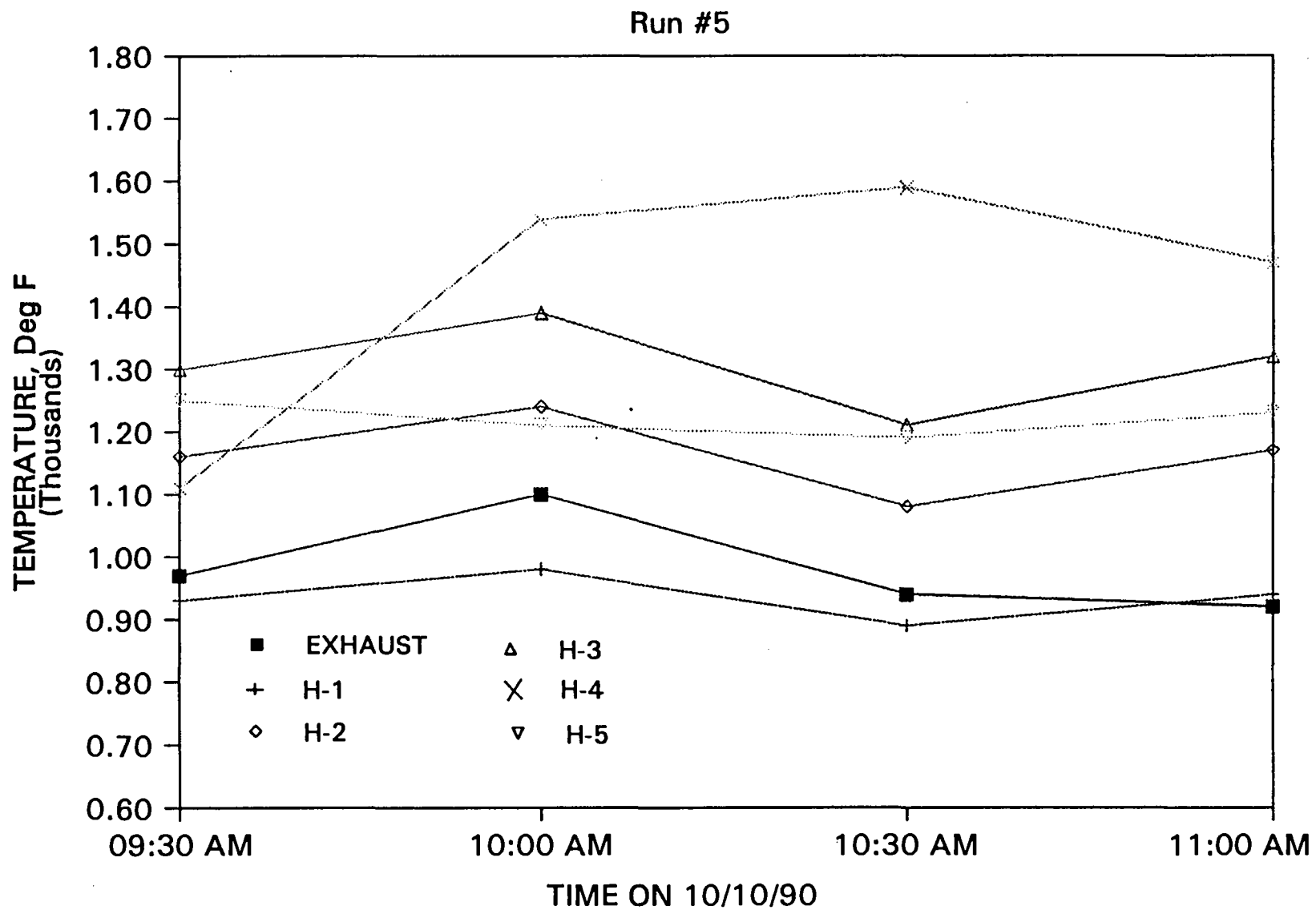


Figure 3-4. Hearth temperature profile during Run 5 - normal mode of operation.

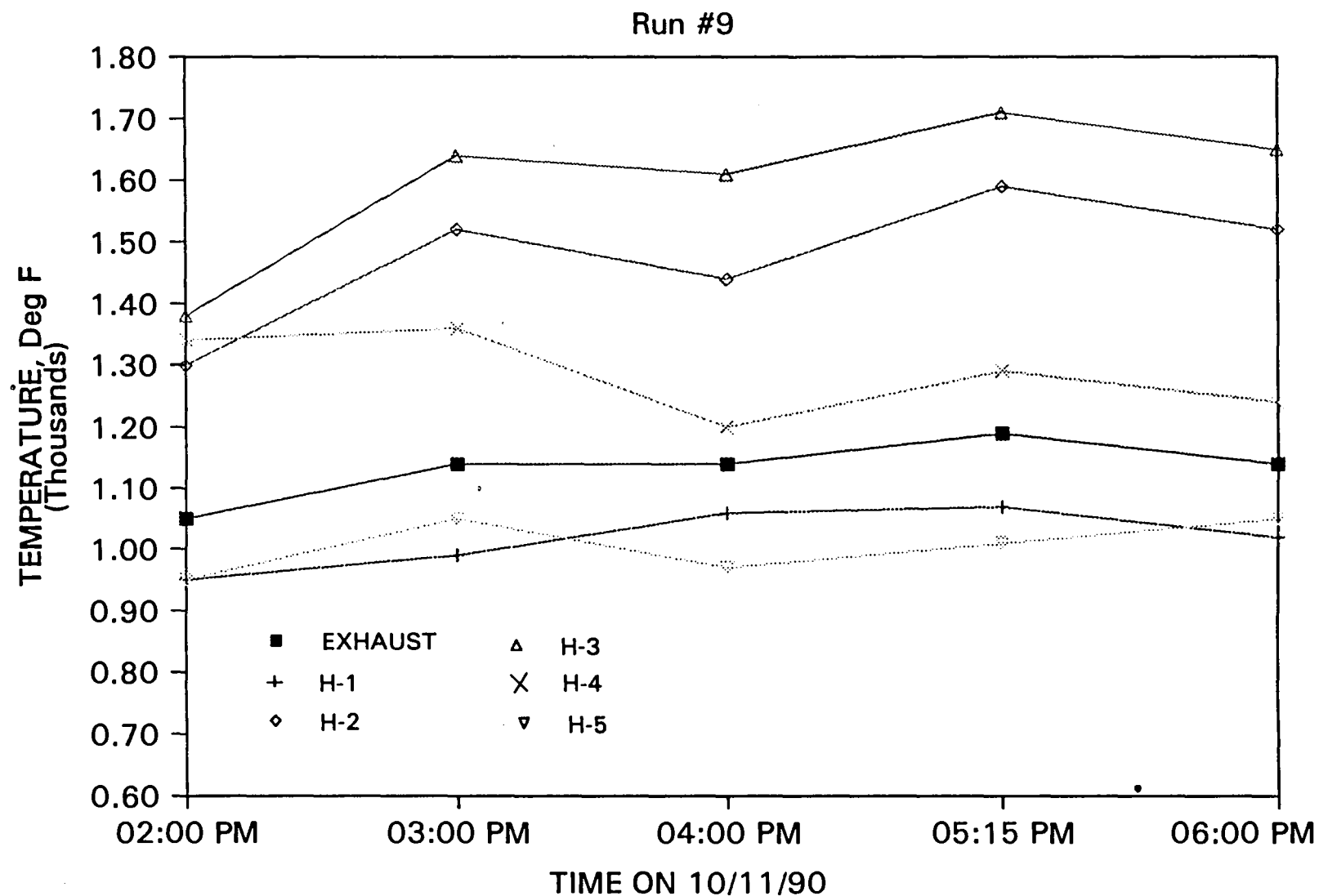


Figure 3-5. Hearth temperature profile during Run 9 - improved combustion mode of operation.

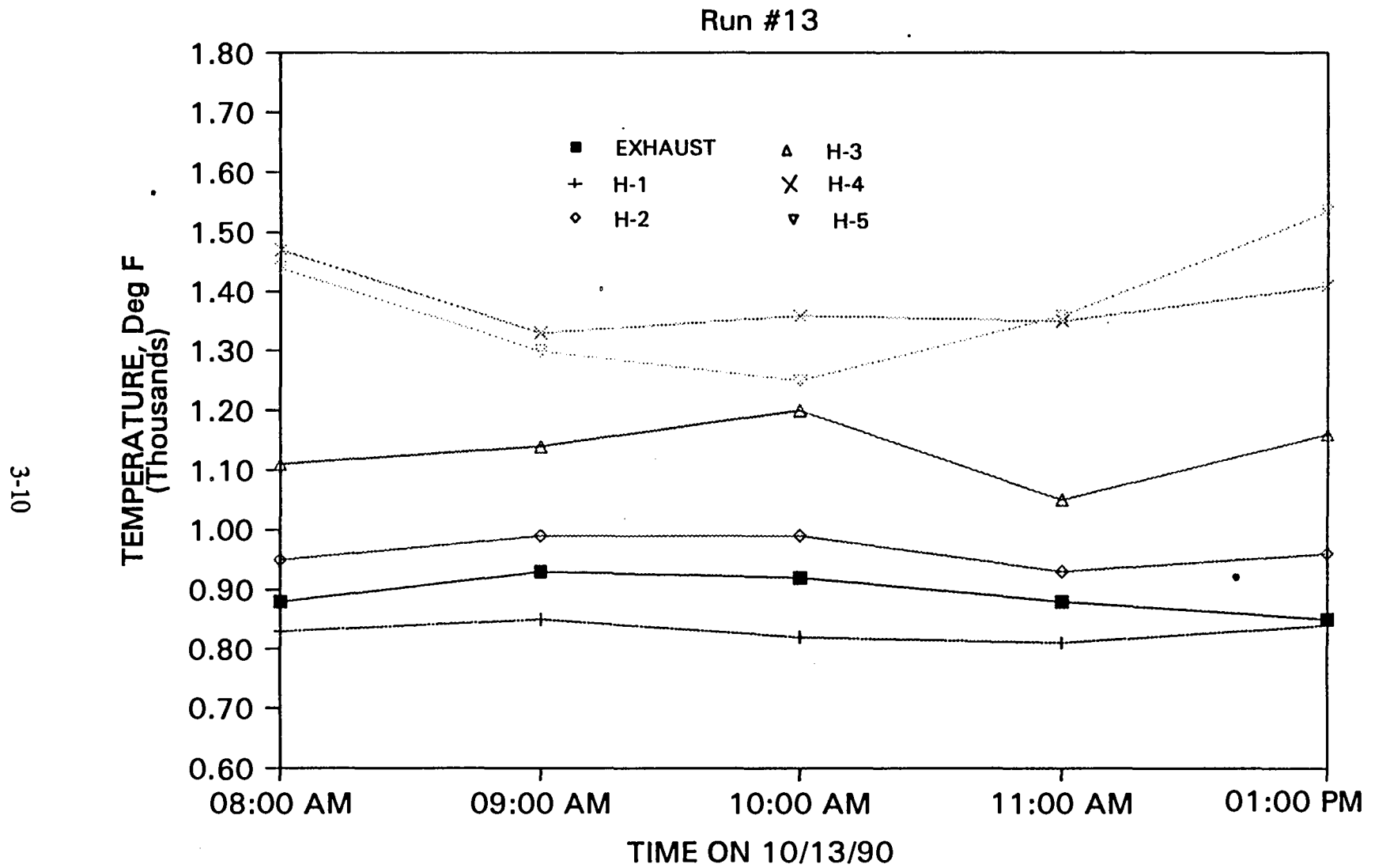


Figure 3-6. Hearth temperature profile during Run 13 - normal mode of operation.

3.4 PROCESS DATA RESULTS

To ensure that the incinerator was operated in a manner to meet program goals, F. Michael Lewis was contracted to monitor and record operations and operate the furnace during special conditions (i.e., low CO condition). Each of the test runs conducted at Site 6 are listed in Table 3-2 along with the operating conditions and the average run values for all hearth temperatures. The auxiliary fuel usage, flue gas temperatures, pressure drop across the venturi scrubber and venturi scrubber/impingement tray scrubber, and THC and CO concentrations are presented in Table 3-3. The sludge feed varied from 3,300 lb/hr to 4,100 lb/hr over the testing period. The differential pressure drop across the venturi scrubber ranged from 24 inches water column (in w.c.) to 30 in w.c. and across the venturi scrubber/impingement tray scrubber ranged from 31 in w.c. to 36 in w.c. During the "normal" furnace conditions, the THC and CO emissions averaged 22.8 ppm and 652 ppm, respectively. During the "low CO" furnace conditions, the THC and CO emissions averaged 7.8 ppm and 322 ppm, respectively.

TABLE 3-2. SUMMARY OF HEARTH TEMPERATURES FOR SITE 6

Run No. / Condition	Hearth#1 Temp (°F)	Hearth#2 Temp (°F)	Hearth#3 Temp (°F)	Hearth#4 Temp (°F)	Hearth#5 Temp (°F)	Hearth#6 Temp (°F)	Hearth#7 Temp (°F)	Hearth#8 Temp (°F)	Comment(s) on Furnace Operations
Run 3 Normal	881 (160)*	1091 (280)	1223 (310)	1433 (65)	1396 (205)	585 (40)	190 (30)	96 (5)	Clinkers were removed from furnace
Run 4 Normal	996 (43)	1428 (73)	1586 (68)	1331 (18)	1029 (53)	378 (53)	136 (13)	89 (3)	With 5 min left in run feed was interrupted & burn out occurred
Run 5 Normal	935 (45)	1162 (80)	1305 (90)	1565 (95)	1220 (30)	490 (60)	135 (5)	90 (0)	Problem with sludge cake, high lime added; feed stopped at 11:00
Run 6 Normal	961 (65)	1176 (153)	1277 (175)	1470 (168)	1246 (200)	577 (73)	170 (25)	90 (5)	Lost scrubber water at 14:45 Outlet tests stopped
Run 7 Normal	848 (65)	946 (138)	1037 (148)	1307 (183)	1286 (368)	678 (183)	320 (200)	133 (53)	Sludge cake was wet, more burners added
Run 8 Transition	993 (144)	1366 (258)	1470 (270)	1527 (90)	1300 (210)	550 (60)	163 (8)	97 (3)	Low O ₂ caused a lot of smoking
Run 9 Low CO	1018 (60)	1474 (145)	1598 (165)	1286 (80)	1006 (50)	316 (40)	127 (8)	90 (0)	Low CO was achieved with a high Hearth #1 temperature
Run 10 Low CO	931 (93)	1225 (225)	1335 (230)	1472 (120)	1505 (255)	602 (160)	200 (35)	97 (5)	Fuel pump failure for very short period
Run 11 Low CO	1025 (70)	1515 (60)	1660 (40)	1307 (70)	1082 (55)	365 (75)	147 (10)	96 (5)	No problems
Run 12 Low CO	1096 (50)	1413 (20)	1600 (10)	1146 (30)	960 (20)	270 (10)	126 (5)	96 (5)	No problems
Run 13 Normal	827 (20)	965 (30)	1125 (75)	1362 (40)	1345 (80)	537 (30)	155 (20)	102 (5)	No problems

* () - Indicates the temperature range above and below average for the test run.

TABLE 3-3. FURNACE AND CONTROL EQUIPMENT OPERATIONS

Run No. Condition	Auxiliary Fuel Usage (% output) Oil Burned Numbers								Venturi ΔP (in WC)	Scrubber ΔP (in WC)	Sludge Feed Rate lb/hr	Outlet Conc. CO (ppm)	Conc. THC (ppm)
	2-1	2-2	4-1	4-2	5-1	5-2	7-1	7-2					
Run 3 Normal	10	20	20	0	0	0	0	0	26	31	4100	673	22.6
Run 4 Normal	0	35	40	40	0	0	0	0	24	31	4100	359	12.4
Run 5 Normal	40	25	0	90	90	0	0	0	28	34	3600	903	45.6
Run 6 Normal	5	5	60	60	50	0	0	0	28	34	3600	608	18.0
Run 7 Normal*	80	100	100	100	15	0	0	0	26	35	3600	810	22.2
Run 8 Transition	100	100	85	85	45	5	0	0	29	36	3700	701	21.4
Run 9 Low CO	100	100	100	100	60	40	0	0	29	36	3700	332	7.5
Run 10 Low CO	100	100	80	80	80	80	0	0	29	35	3300	337	8.8
Run 11 Low CO	100	100	100	100	100	100	0	0	29	36	3300	313	7.5
Run 12 Low CO	100	100	100	100	100	100	0	0	31	36	3300	308	7.3
Run 13 Normal	0	0	20	20	20	20	0	0	30	36	3400	554	16.2

* Sludge was wet and low in volatiles, so burners were used to maintain temperature.

4.0 TEST RESULTS

The results of the emission tests performed at Site 6 from October 9 to October 13, 1989 are presented in this section. Site 6 is a typical multiple hearth incinerator equipped with a venturi scrubber/impingement tray scrubber combination to control emissions. This site was selected for testing principally because (1) the sludge is conditioned with lime and (2) a measurable amount of total chromium is introduced into the process as a contaminant in the ferric chloride used to improve sludge filtration characteristics. The primary objective of this test program was to determine the effect of lime conditioning and excess air on the conversion of total chromium in the sludge to hexavalent chromium emissions. Site 6 was originally scheduled for testing under two process operating conditions: (1) normal to high excess air and (2) low excess air. The emissions testing under the first condition showed high emissions of CO and THC, which are associated with poor combustion. It was decided that the second series of tests should be conducted under incinerator conditions that would reduce the levels of CO and THC indicating improved combustion rather than low excess air. The CO and THC emissions were reduced by changing the sludge feed rate, combustion, air distribution, and using more fuel oil. The two test conditions are referred to in the text and tables as "Normal" and "Low CO." One run (Run 8) was conducted during the transition from normal to improved combustion conditions and is referred to as "Transition".

In addition to the presentation of the results, variability and outliers in the data are discussed. The incinerator operating conditions and other process parameters are discussed in relation to the results; however, a complete statistical analysis of the emission results relating to the operating conditions is not included in this report.

Results are presented in metric units. Flue gas results are presented as measured and as normalized to an equivalent 7% O₂ concentration. Mass emission rates are also

presented for each of the parameters. In addition, emission factors relating stack emissions to sludge feed composition and rates are presented where appropriate. Supporting data for the results presented in this section are included in the appendices in Volume IV.

4.1 FLUE GAS CONDITIONS

A summary of the Site 6 inlet and outlet flue gas conditions is presented in Table 4-1, along with the run numbers, test dates, and run times. A summary of inlet and outlet continuous monitoring measurements is present in Table 4-2.

4.1.1 Inlet Flue Gas Conditions

The flue gas volumetric flow rates at the inlet location were fairly consistent averaging 475 dry standard cubic meters per minute (dscm/min) for normal operating conditions and 430 dscm/min for low CO conditions. The inlet flue gas flow rate was calculated by correcting the measured outlet flue gas flow rate with the difference between the outlet and inlet oxygen concentrations. The average flue gas temperature was 876°F (469°C) under normal operating conditions and 1027°F (553°C) under low CO conditions with moisture contents of 33.1% and 33.2%, respectively. The average percentages of dry oxygen and carbon dioxide were 13.0 and 7.4, respectively under normal conditions and 11.5 and 7.7, respectively under low CO conditions. The average CO concentrations were 1290 and 620 ppm corrected to 7% O₂ under normal and low CO operating conditions, respectively.

4.1.2 Outlet Flue Gas Conditions

The average flue gas volumetric flow rates at the outlet sampling site ranged from 522 dscm/min under normal operating conditions to 531 dscm/min under low CO conditions. Flue gas average temperatures were 144°F (62.4°C) under normal operating

TABLE 4-1. SUMMARY OF INLET AND OUTLET FLUE GAS CONDITIONS

Run No. & Condition	Sampling Location	Test Date	Run Time	Flue Gas Conditions			
				Temperature (F)	Moisture (% H2O)	Oxygen (% dry)	Flow Rate (dscm/min)
Run 2 Normal	Inlet* Outlet	10/09/89	10:10-11:10	139.4	6.6	NA*	450.9
Run 3 Normal	Inlet Outlet	10/09/89	12:45-14:45 12:45-14:45	861.1 138.1	30.3 6.1	13.4 14.0	538.4 585.2
Run 4 Normal	Inlet Outlet	10/09/89	17:00-19:00 17:00-19:00	962.5 137.7	29.2 6.9	14.8 14.1	620.8 556.9
Run 5 Normal	Inlet Outlet	10/10/89	09:00-11:00 09:10-11:10	902.7 146.2	40.1 6.3	10.3 13.2	378.1 520.5
Run 6 Normal	Inlet Outlet	10/10/89	14:15-16:15 14:30-17:00	928.0 143.8	34.9 6.5	12.7 14.4	369.8 466.5
Run 7 Normal	Inlet Outlet	10/10/89	18:50-20:50 18:37-21:37	819.8 135.0	32.3 7.3	13.5 14.2	451.1 498.3
Run 8 Transition	Inlet Outlet	10/11/89	09:45-11:45 09:45-11:45	1045.2 155.0	40.9 7.4	9.1 11.9	353.6 463.6
Run 9 Low CO	Inlet Outlet	10/11/89	16:22-18:22 15:43-18:43	1084.1 148.7	34.5 6.9	11.5 13.4	505.9 634.1
Run 10 Low CO	Inlet Outlet	10/12/89	09:00-11:00 09:00-11:00	1013.7 143.2	32.7 7.3	11.5 13.2	372.1 454.2
Run 11 Low CO	Inlet Outlet	10/12/89	13:15-15:15 12:31-15:31	1049.3 147.6	33.1 8.1	11.5 13.1	522.0 629.0
Run 12 Low CO	Inlet Outlet	10/12/89	17:45-19:45 17:32-19:32	962.1 149.7	32.5 6.6	11.6 13.6	319.6 407.2
Run 13 Normal	Inlet Outlet	10/13/89	09:00-11:00 09:00-13:00	783.1 170.8	31.9 7.4	13.4 14.5	493.9 578.8

* Not available.

TABLE 4-2. SUMMARY OF INLET AND OUTLET CONTINUOUS EMISSION MEASUREMENTS

Run No. & Condition	Sampling Location	Test Date	Diluent (% dry)		Pollutant Gases (actual ppm and/or corrected to 7% O ₂)						
			Oxygen	Carbon Dioxide	Sulfur Dioxide Actual @7% O ₂		Nitrogen Oxides Actual @7% O ₂		Carbon Monoxide Actual @7% O ₂		Cold THC Actual
Run 3 Normal	Inlet Outlet	10/09/89	13.4 14.0	6.3 5.8	41.5 21.4	76.9 43.1	153 142	284 286	702 673	1301 1357	22.6
Run 4 Normal	Inlet Outlet	10/09/89	14.8 14.1	5.0 5.7	38.4 18.2	87.5 37.2	115 142	261 290	406 359	925 735	12.4
Run 5 Normal	Inlet Outlet	10/10/89	10.3 13.2	8.7 6.3	32.1 25.3	42.1 45.7	151 124	198 226	1023 903	1341 1631	45.6
Run 6 Normal	Inlet Outlet	10/10/89	12.7 14.4	6.7 5.3	35.7 18.4	60.5 39.3	207 166	351 355	752 608	1275 1300	18.0
Run 7 Normal	Inlet Outlet	10/10/89	13.5 14.2	6.0 5.3	29.9 21.8	56.2 45.2	214 165	401 342	894 810	1679 1681	22.2
Run 8 Transition	Inlet Outlet	10/11/89	9.1 11.9	9.9 7.4	110 15.9	130 24.6	180 102	212 157	848 701	999 1083	21.4
Run 9 Low CO	Inlet Outlet	10/11/89	11.5 13.4	7.6 6.2	39.7 16.0	58.7 29.7	192 133	284 247	445 332	659 615	7.5
Run 10 Low CO	Inlet Outlet	10/12/89	11.5 13.2	7.8 6.3	58.0 18.6	85.8 33.6	177 141	262 255	424 337	627 609	8.8
Run 11 Low CO	Inlet Outlet	10/12/89	11.5 13.1	7.8 6.4	65.0 5.4	97.2 9.6	159 127	238 225	395 313	590 557	7.5
Run 12 Low CO	Inlet Outlet	10/12/89	11.6 13.6	7.7 6.1	45.9 8.6	68.6 16.4	166 114	249 217	403 308	603 587	7.3
Run 13 Normal	Inlet Outlet	10/13/89	13.4 14.5	6.1 5.3	26.2 22.7	48.6 49.3	191 147	353 320	659 554	1220 1203	16.2

conditions and 147°F (64.1°C) under low CO conditions with moisture contents of 6.7% and 7.2%, respectively. The average percentages of dry oxygen and carbon dioxide were 14.1 and 5.6, respectively, under normal operating conditions and 13.3 and 6.3, respectively, under low CO condition, while the carbon monoxide levels corrected to 7% oxygen for the normal and low CO conditions were 1318 and 592 ppm, respectively. The THC emissions measured under the normal and low CO conditions were 24 and 8 ppm, respectively.

4.2 PARTICULATES/METAL RESULTS

Particulates/metal emissions were determined using the draft EPA method procedure for "Methodology for the Determination of Trace Metals Emissions in Exhaust Gases for Stationary Source Combustion Processes" (reproduced in Volume IV: Site 6 Draft Test Report, Appendices)

Five runs (Runs 5, 6, 8, 10, and 12) were conducted at the inlet and outlet of Site 6 for arsenic (As), beryllium (Be), cadmium (Cd), chromium (Cr), lead (Pb), and nickel (Ni). The particulate emissions were determined using the multiple metals sampling system. The emission results for metals and particulates are shown in Table 4-3 on a concentration basis and in Table 4-4 on a mass emission rate basis. The removal efficiency for particulates and metals are also shown in Table 4-4.

For Condition 1 "Normal Operation", test runs were conducted over a 2-hr period with no special effort taken to control the incinerator operating conditions. For Condition 2, the test runs were also conducted over a 2-hr period, with the incinerator operated to minimize CO and THC emissions as indicated by the continuous emission monitoring results. For the inlet sampling, high moisture content required use of a large first impinger in the particulates/metals train. Results from the test program provide data on average emissions from sludge incinerators during typical operations (steady-state and transient operating conditions) and under optimum combustion conditions.

Research Triangle Institute (RTI) analyzed all the total metals samples. Special sample handling procedures were not required for these samples because mercury was

TABLE 4-3. SUMMARY OF INLET AND OUTLET PARTICULATE MASS AND TARGET METALS

Run No. / Location	Particulate Mass (mg/dscm)	Flue Gas Metals Concentration ($\mu\text{g/dscm}$)					
		Arsenic	Beryllium	Cadmium	Chromium	Lead	Nickel
Run 5 Inlet	2239	< 42.7*	0.74	249	642	3660	64.8
Outlet	22.3	< 16.7	< 0.07	40.3	2.1	535	2.9
Run 6 Inlet	1666	< 34.3	0.75	167	504	1910	50.1
Outlet	21.3	< 21.1	< 0.09	49.1	4.0	689	2.8
Run 8 Inlet	934	< 40.5	0.53	525	424	6960	47.4
Outlet	38.7	< 19.1	0.08	93.0	5.9	1096	3.9
Run 10 Inlet	1542	< 35.2	0.61	230	531	4244	50.9
Outlet	27.2	< 21.0	< 0.09	43.9	1.7	826	1.3
Run 12 Inlet	2457	< 48.3	1.05	248	786	4641	67.8
Outlet	24.1	< 11.4	0.05	40.4	0.8	795	1.8
Avg. Normal Inlet	1953	< 38.5	0.7	208	573	2785	57.5
Outlet	21.8	< 18.9	< 0.1	44.7	3.1	612	2.9
Avg. Low CO Inlet	1644	< 41.4	0.7	334	580	5282	55.4
Outlet	30.0	< 17.2	< 0.1	59.1	2.8	906	2.3
Test Average Inlet	1799	< 39.9	0.7	271	577	4033	56.4
Outlet	25.9	< 18.0	< 0.1	51.9	2.9	759	2.6

* < - Below the level of detection, less than indicated detection limit.

TABLE 4-4. SUMMARY OF INLET AND OUTLET PARTICULATE AND METALS MASS EMISSION RATES

Run No. and Locations	Particulate Mass Rate (kg/hour)	Flue Gas Metal Mass Emission Rate (mg/hour)					
		Arsenic	Beryllium	Cadmium	Chromium	Lead	Nickel
Run 5							
Inlet	50.8	< 969	16.9	5648	14564	83037	1471
Outlet	0.7	< 521	< 2.3	1258	65.8	16710	90.7
Efficiency*, %	98.6	NA†	> 86.6	77.7	99.5	79.9	93.8
Run 6							
Inlet	37.0	< 761	16.6	3708	11173	42374	1112
Outlet	0.6	< 591	< 2.6	1375	113	19297	79.7
Efficiency, %	98.4	NA	> 84.5	62.9	99.0	54.5	92.8
Run 8							
Inlet	19.8	< 860	11.2	11140	8991	147661	1006
Outlet	1.1	< 531	2.3	2586	164	30475	109
Efficiency, %	94.6	NA	79.4	76.8	98.2	79.4	89.2
Run 10							
Inlet	34.4	< 787	13.7	5131	11851	94744	1136
Outlet	0.7	< 573	< 2.5	1196	47.3	22517	34.9
Efficiency, %	97.8	NA	> 81.8	76.7	99.6	76.2	96.9
Run 12							
Inlet	47.1	< 926	20.1	4752	15081	88996	1301
Outlet	0.6	< 278	1.2	987	26.5	19432	44.7
Efficiency, %	98.8	NA	94.0	79.2	99.9	78.2	96.6
Ave. Normal							
Inlet	43.9	< 865	16.7	4678	12868	62705	1292
Outlet	0.6	< 556	< 2.4	1317	89.5	18003	85.2
Efficiency, %	98.5	NA	> 85.5	71.9	99.3	71.3	93.4
Avg. Low CO							
Inlet	33.8	< 858	15.0	7007	11974	110467	1147
Outlet	0.8	< 461	< 2.0	1590	77.3	24141	62.7
Efficiency, %	97.6	NA	> 86.7	77.3	99.4	78.1	94.5
Test Average							
Inlet	38.8	< 862	15.9	5843	12421	86586	1219
Outlet	0.7	< 508	< 2.2	1453	83.3	21072	73.9
Efficiency, %	98.1	NA	> 86.1	75.1	99.3	75.7	93.9

* Control device removal efficiency.

† Not applicable.

not measured, and the other target metals remain stable until analyzed.

A reagent blank was collected to assess background contamination levels of each target metal. A complete discussion of Quality Assurance/Quality Control procedures and results is presented in Section 7.0.

4.2.1 Control Device Inlet Results

The flue gas metals and particulate concentrations at the control device inlet are shown in Table 4-5. The average values for "normal" (Runs 5 and 6) represent metals emissions during normal furnace operations. The average values for "Low CO" (Runs 8, 10, and 12) represent metal emissions during improved combustion conditions. Arsenic was below the level of detection, which was about 40 $\mu\text{g}/\text{dscm}$ for all inlet sample runs. Since arsenic was below the level of detection for all samples it was decided not to have them reanalyzed by GFAAS. The average beryllium concentration of 0.7 $\mu\text{g}/\text{dscm}$ was the same for both operating conditions. The cadmium emission concentrations were approximately the same for both conditions if the transition run (Run 8) is not considered in the averages of 208 and 239 $\mu\text{g}/\text{dscm}$ for the normal and Low CO conditions, respectively. The chromium emission concentrations were also similar for both runs, averaging 573 and 580 $\mu\text{g}/\text{dscm}$ for the normal and low CO conditions, respectively. The low CO conditions yielded a significant increase in the lead emission concentrations with averages of 2785 and 5282 $\mu\text{g}/\text{dscm}$ for the normal and low CO conditions, respectively. The nickel emissions were similar for both operating conditions averaging 57.5 and 55.4 $\mu\text{g}/\text{dscm}$ for the normal and low CO conditions, respectively. The particulate concentration were similar for both conditions and averaged 1950 and 2000 mg/dscm , respectively for the normal and low CO conditions.

The flue gas metal and particulate mass emission rates at the control device inlet are shown in Table 4-6. Because the flue gas flow rate was consistent from run to run, the mass emission rates showed the same correlations as the metal concentrations. The metal mass emissions rates for the inlet runs averaged: arsenic - not detected (< 862 mg/hr), beryllium - 15.9 mg/hr , cadmium - 5,840 mg/hr , chromium - 12,400 mg/hr , lead

TABLE 4-5. SUMMARY OF INLET PARTICULATE MASS AND TARGET METALS

Run No. and Condition	Particulate Mass (mg/dscm)	Flue Gas Metals Concentration (g/dscm)					
		Arsenic	Beryllium	Cadmium	Chromium	Lead	Nickel
Run 5 Normal	2239	< 42.7	0.74	249	642	3660	64.8
Run 6 Normal	1666	< 34.3	0.75	167	504	1910	50.1
Run 8 Transition	934	< 40.5	0.53	525	424	6960	47.4
Run 10 Low CO	1542	< 35.2	0.61	230	531	4244	50.9
Run 12 Low CO	2457	< 48.3	1.05	248	786	4641	67.8
Runs 5 & 6 Normal	1953	< 38.5	0.7	208	573	2785	57.5
Runs 8,10,12 Low CO	1644	< 41.4	0.7	334	580	5282	55.4
Test Average Inlet	1799	< 39.9	0.7	271	577	4033	56.4

TABLE 4-6. SUMMARY OF INLET PARTICULATE AND METALS MASS EMISSION RATES

Run No. and Locations	Particulate Mass Rate (kg/hour)	Flue Gas Metals Mass Emission Rate (mg/hour)					
		Arsenic	Beryllium	Cadmium	Chromium	Lead	Nickel
Run 5 Normal	50.8	< 969	16.9	5648	14564	83037	1471
Run 6 Normal	37.0	< 761	16.6	3708	11173	42374	1112
Run 8 Transition	19.8	< 860	11.2	11140	8991	147661	1006
Run 10 Low CO	34.4	< 787	13.7	5131	11851	94744	1136
Run 12 Low CO	47.1	< 926	20.1	4752	15081	88996	1301
Runs 5 and 6 Normal	43.9	< 865	16.7	4678	12868	62705	1292
Runs 8,10,&12 Low CO	33.8	< 858	15.0	7007	11974	110467	1147
Total Average Inlet	38.8	< 862	15.9	5843	12421	86586	1219

- 86,600 mg/hr, and nickel - 1,220 mg/hr. The particulate mass emission rates were similar under normal and low CO conditions and averaged 42.9 and 40.8 kg/hr, respectively.

For each inlet sampling run, the micrograms of each target metal collected was divided by the grams of particulate collected (see Table 4-7) to yield the concentration of metal in the fly ash. All metal concentrations in the inlet fly ash samples were similar under both combustion conditions with the exception of the lead concentrations which were almost twice as much for the Low CO conditions because of the higher furnace temperatures. The metals concentrations in terms of microgram of metal per gram of particulate for the inlet sampling runs averaged: arsenic - not detected ($< 18 \mu\text{g/g}$), beryllium - $0.43 \mu\text{g/g}$, cadmium - $205 \mu\text{g/g}$, chromium - $342 \mu\text{g/g}$, lead - $2980 \mu\text{g/g}$, and nickel - $34 \mu\text{g/g}$.

4.2.2 Control Device Outlet Results

The flue gas metals and particulate concentrations at the control device outlet are shown in Table 4-8. The average values for Runs 5 and 6 represent metals emissions during normal furnace operations. The average values for Runs 8, 10, and 12 represent metal emissions during the improved combustion conditions. Arsenic emissions were below the level of detection, which was about $18 \mu\text{g/dscm}$ for all outlet sampling runs. The beryllium emissions were at or below the level of detection, which was about $0.1 \mu\text{g/dscm}$ for all outlet runs. The cadmium emission concentrations were approximately the same under both conditions when Run 8 (the transition run) is not included in the averages of 44.7 and $42.2 \mu\text{g/dscm}$ for the normal and low CO conditions, respectively. The chromium emission concentrations were also similar under both normal and low CO conditions averaging 3.1 and $2.9 \mu\text{g/dscm}$, respectively. A greater control device removal efficiency average of $906 \mu\text{g/dscm}$ was obtained for the higher emission concentrations of lead encountered during the low CO conditions versus $612 \mu\text{g/dscm}$ under normal operating conditions. The nickel emissions were similar under both normal and low CO conditions and averaged $2.9 \mu\text{g/dscm}$ and $2.3 \mu\text{g/dscm}$, respectively. The

TABLE 4-7. SUMMARY OF METAL CONCENTRATIONS IN FLY ASH

Run No./ Location	Metals Concentration in Fly Ash (μg metal/g particulate)				
	Beryllium	Cadmium	Chromium	Lead	Nickel
Run 5					
Inlet	0.33	111	287	1635	29
Outlet	< 3.3*	1811	111	24046	131
Run 6					
Inlet	0.45	100	303	1146	30
Outlet	< 4.3	2301	211	32301	133
Run 8					
Inlet	0.57	562	455	7455	51
Outlet	2.1	2401	163	28296	101
Run 10					
Inlet	0.40	149	345	2752	33
Outlet	< 3.4	1613	81	30387	47
Run 12					
Inlet	0.43	101	320	1889	28
Outlet	2.1	1676	55	32992	76
Runs 5 & 6					
Inlet	0.39	106	295	1390	30
Outlet	< 3.8	2056	161	28173	132
Runs 10 & 12					
Inlet	0.41	125	333	2320	30
Outlet	< 2.7	1645	68	31689	61
Average					
Inlet	0.43	205	342	2975	34
Outlet	< 3.0	1960	124	29604	97

* < - Below the level of detection, less than indicated detection limit.

Note: Arsenic was below the limit of detection in all runs, inlet values were less than 25 μg and outlet less than 695 μg .

TABLE 4-8. SUMMARY OF OUTLET PARTICULATE MASS AND TARGET METALS

Run No. and Location	Particulate Mass (mg/dscm)	Flue Gas Metals Concentration(μ g/dscm)					
		Arsenic	Beryllium	Cadmium	Chromium	Lead	Nickel
Run 5 Normal	22.3	< 16.7	< 0.07	40.3	2.1	535	2.9
Run 6 Normal	21.3	< 21.1	< 0.09	49.1	4.0	689	2.8
Run 8 Transition	38.7	< 19.1	0.08	93.0	5.9	1096	3.9
Run 10 Low CO	27.2	< 21.0	< 0.09	43.9	1.7	826	1.3
Run 12 Low CO	24.1	< 11.4	0.05	40.4	0.8	795	1.8
Runs 5 & 6 Normal	21.8	< 18.9	< 0.1	44.7	3.1	612	2.9
Runs 8,10,12 Low CO	30.0	< 17.2	< 0.1	59.1	2.8	906	2.3
Test Average Outlet	25.9	< 18.0	< 0.1	51.9	2.9	759	2.6

particulate concentrations were slightly increased under the low CO conditions averaging 25.7 mg/dscm, as opposed to 21.8 μ g/dscm under normal conditions.

The flue gas metal and particulates mass emission rates at the control device inlet are shown in Table 4-9. Because the flue gas flow rate was consistent from run to run, the mass emission rates show the same correlations as the metals concentrations. The metal mass emissions rates for all outlet runs averaged: arsenic - not detected (< 508 mg/hr), beryllium - not detected (< 2.2 mg/hr), cadmium - 1,450 mg/hr, chromium - 83.3 mg/hr, lead - 21,100 mg/hr, and nickel - 73.9 mg/hr. The particulate mass emission rates were similar under both normal and low CO conditions averaging 0.7 and 0.7 kg/hr, respectively.

For each outlet sampling run, the micrograms of each target metal collected was divided by the grams of particulate collected (see Table 4-7) to provide the concentration of metal in the fly ash. All metal concentrations in the outlet fly ash samples were higher under the low CO conditions with the exception of the lead concentration which was slightly higher under the low CO₂ conditions. The metal concentrations in terms of microgram of metal per gram of particulate for the outlet sample runs averaged: arsenic - not detected, beryllium - not detected, cadmium - 1960 ug/g, chromium - 124 ug/g, lead - 29,600 ug/g, and nickel - 97 ug/g.

4.2.3 Removal Efficiency of Control Device for Metals and Particulate

The pollutant removal (collection) efficiencies reported for the venturi scrubber/impingement tray scrubber were based on the mass emission rates, see Table 4-4. The efficiencies cannot be based on the emission concentrations, unless corrected for dilution air, since the cooling shaft air enters the ducting between the inlet and outlet locations. The pollutant removal efficiencies measured for the particulate and target metal runs were similar for both operating conditions and averaged: particulate - 98.1%, arsenic - inlet and outlet samples were below the level of detection, beryllium - > 86.1% (all outlet sample values were at or below the limit of detection), cadmium - 75.1%, chromium - 99.3%, lead - 75.7%, and nickel - 93.9%. The lower removal efficiencies for

TABLE 4-9. SUMMARY OF OUTLET PARTICULATE AND METALS MASS EMISSION RATES

Run No. and Locations	Particulate Mass Rate (kg/hour)	Flue Gas Metal Mass Emission Rate (mg/hour)					
		Arsenic	Beryllium	Cadmium	Chromium	Lead	Nickel
Run 5 Normal	0.7	< 521	< 2.3	1258	65.8	16710	90.7
Run 6 Normal	0.6	< 591	< 2.6	1375	113	19297	79.7
Run 8 Transition	1.1	< 531	2.3	2586	164	30475	109
Run 10 Low CO	0.7	< 573	< 2.5	1196	47.3	22517	34.9
Run 12 Low CO	0.6	< 278	1.2	987	26.5	19432	44.7
Runs 5 and 6 Normal	0.6	< 556	< 2.4	1317	89.5	18003	85.2
Runs 8,10,&12 Low CO	0.8	< 461	< 2.0	1590	77.3	24141	62.7
Total Average Outlet	0.7	< 508	< 2.2	1453	83.3	21072	73.9

many of the metals compared to the total particulate are expected because the metals are typically concentrated in the smaller diameter particles which are in turn more difficult to collect. It is not known why the collection efficiency of chromium was greater than the particulate removal efficiency.

4.2.4 Sludge Feed Results

A composite sludge feed sample was collected over the duration of each of the test runs. The sludge feed metal concentrations on a wet basis are presented in Table 4-10. The concentrations of metals in the sludge were fairly consistent and averaged: arsenic - not detected ($< 4.6 \mu\text{g/g}$), beryllium - $0.088 \mu\text{g/g}$, cadmium - $1.28 \mu\text{g/g}$, chromium - $18.5 \mu\text{g/g}$, lead - $81 \mu\text{g/g}$, and nickel - $6.1 \mu\text{g/g}$.

The metal feed rates based on the concentration of metals in the sludge and the sludge feed rates are presented in Table 4-11. The mass feed rates of metals in the sludge were fairly consistent and averaged: arsenic - not detected ($< 8 \text{ g/hr}$), beryllium - 0.088 g/hr , cadmium - 1.28 g/hr , chromium - 18.5 g/hr , lead - 81 g/hr , and nickel - 6.1 g/hr .

The results of the sludge proximate and ultimate analyses are presented in Table 4-12. All of these results were fairly consistent from run-to-run with the exception of the moisture content on Run 4. The results for the metals runs (Runs 5, 6, 8, 10, and 12) averaged: moisture - 73.25%, volatile matter - 57.96% (dry basis), fixed carbon - 4.23% (dry basis), ash - 37.85% (dry basis), sulfur - 0.53% (dry basis), carbon - 32.24% (dry basis), hydrogen - 4.6% (dry basis), nitrogen - 3.44% (dry basis), oxygen - 21.34% (dry basis), and BTU per pound - 5953 (dry basis).

4.2.5 Scrubber Water Results

Scrubber water influent and effluent samples were collected for all sampling runs. The scrubber water effluent samples had to be collected from the bottom of the discharge pipe. The water effluent samples therefore may not be representative of the

TABLE 4-10. METALS CONCENTRATION ($\mu\text{g/g}$) IN SLUDGE ON WET BASIS

Run Number	Metal Concentration ($\mu\text{g/g}$, wet basis)					
	As	Be	Cd	Cr	Pb	Ni
3-Sludge	ND*	0.06	0.675	53.0	8.88	4.86
4-Sludge	ND	0.039	0.808	57.9	10.5	4.65
5-Sludge	ND	0.059	0.867	60.7	9.6	4.91
6-Sludge	ND	0.059	0.846	52.5	11.9	4.46
7-Sludge	ND	0.059	0.825	62.1	16.8	4.36
8-Sludge	ND	0.059	0.691	52.1	12.8	3.73
9-Sludge	ND	0.040	0.818	52.7	9.24	4.63
10-Sludge	ND	0.08	1.00	50.6	13.3	4.06
11-Sludge	ND	0.040	0.774	52.0	13.4	2.72
12-Sludge	ND	0.02	0.62	36.6	10.5	1.98
13-Sludge	ND	0.020	0.909	61.1	11.0	3.20
Detection Limit†	4.6	0.02	0.06	0.02	1.68	0.24

* ND - Below the analytical limit of detection.

† Detection Limit - Values shown represent limit of detection in μg of metal divided by g of wet sludge.

TABLE 4-11. FEED RATE OF METALS IN SLUDGE (g/hr)

Run No.	Feed Rate of Specified Metal (g/hr)					
	As*	Be	Cd	Cr	Pb	Ni
3	< 9	0.112	1.26	16.5	99	9.05
4	< 9	0.073	1.50	19.5	108	8.66
5	< 8	0.097	1.42	15.7	99	8.02
6	< 8	0.096	1.38	19.4	86	7.29
7	< 8	0.096	1.35	27.5	101	7.13
8	< 8	0.100	1.16	21.5	88	6.27
9	< 8	0.067	1.37	15.5	89	7.78
10	< 7	0.120	1.50	19.9	76	6.08
11	< 7	0.059	1.16	20.1	78	4.08
12	< 7	0.030	0.93	15.7	55	2.97
13	< 7	0.030	1.36	16.5	92	4.79
Average Metals Runs†	< 8	0.088	1.28	18.5	81	6.1

* < - not detected, value represents detection limit.

† Runs 5, 6, 8, 10, and 12.

TABLE 4-12. RESULTS FOR PROXIMATE AND ULTIMATE ANALYSES OF SLUDGE SAMPLES

Run No	Moisture (percent)	Dry Basis Analysis (Percent)*								
		Volatile matter	Fixed Carbon	Ash	S	C	H	N	O	Btu/lb
3	70.25	58.93	6.68	34.39	0.52	34.11	5.00	3.17	22.82	6272
4	82.47	57.32	7.73	34.95	0.56	34.74	4.78	3.26	21.72	6361
5	72.40	58.40	4.84	36.76	0.53	32.98	4.90	3.06	21.77	5998
6	72.74	58.97	5.60	35.43	0.52	33.28	4.77	3.48	22.52	6368
7	75.98	59.56	5.64	34.80	0.53	34.52	4.56	3.59	22.01	6086
8	74.13	59.33	4.99	35.68	0.54	33.54	4.51	3.78	21.96	6304
9	72.37	55.85	2.76	41.39	0.44	29.25	4.37	1.62	22.92	5280
10	74.43	57.36	3.31	39.33	0.54	32.66	4.51	3.61	19.36	5897
11	73.45	56.61	3.87	39.52	0.51	31.00	4.64	3.57	20.76	5548
12	72.55	55.51	2.43	42.06	0.51	28.74	4.31	3.28	21.10	5199
13	72.08	61.24	0.04	38.72	0.48	30.08	4.50	3.18	23.05	5477
Avg	73.25	57.96	4.23	37.85	0.53	32.24	4.60	3.44	21.34	5953
Metals										
Runs†										

* Elemental analysis - S (Sulfur), C (Carbon), H (Hydrogen), N (Nitrogen), and O (Oxygen).

† Runs 5, 6, 8, 10, and 12.

discharge effluent emissions. Due to the nature of the scrubber water effluent sampling location, the results should be treated as an approximation. The scrubber water flow rate is not routinely measured by the facility, but was estimated to be 800 gal/min.

The concentrations of metals in the scrubber influent and effluent is presented in Table 4-13. The mass discharge rates of the metals collected in the scrubber are presented in Table 4-14. These values represent the effluent concentration minus the influent concentration times the scrubber water flow rate of 800 gal/min. The average value for the metals runs were: arsenic - not detected, beryllium - at the level of detection (0.02 g/hr), cadmium - 1.44 g/hr, chromium - 15.0 g/hr, lead - 29.8 g/hr, and nickel - 1.0 g/hr.

4.2.6 Bottom Ash Results

Incinerator bottom ash samples were collected from the hopper once per test at the conclusion of the sample run. The bottom ash metal concentration results are shown in Table 4-15.

The bottom ash metal concentrations were converted to metal mass flow rates. The bottom ash flow rate was determined using the percent ash values from the proximate analyses (in Table 4-12). The percent ash values were then multiplied by the appropriate sludge feed rates to yield the total ash production rate. The average particulate emissions measured at the incinerator outlet (scrubber inlet) were subtracted from the total ash rates to give a bottom ash flow rate for each run. The average ash flow rate was 11 tons/hr. These results, at best, represent a rough estimate of the ash flow rate. The metals mass flow rates for the bottom ash are presented in Table 4-16 and for the metals runs (5, 6, 8, 10, and 12) averaged: arsenic - not detected (0.6 g/hr), beryllium - at the level of detection (0.03 g/hr), cadmium - 0.13 g/hr, chromium 10.4 g/hr, lead 38.4 g/hr, and nickel - 2.7 g/hr.

TABLE 4-13. SCRUBBER WATER METAL CONCENTRATIONS ($\mu\text{g/ml}$)

Run No.\ Location	Sample Analysis Results ($\mu\text{g/ml}$)					
	As	Be	Cd	Cr	Pb	Ni
SWI-3*	ND‡	ND	0.003	0.019	0.177	0.016
SWE-3*	ND	0.001	0.053	0.771	1.09	0.064
SWI-4	ND	ND	ND	0.01	ND	0.021
SWE-4	ND	ND	0.083	0.967	2.25	0.091
SWI-5	ND	ND	ND	0.019	0.295	0.016
SWE-5	ND	ND	0.115	0.847	1.66	0.09
SWI-6	ND	ND	ND	0.026	ND	0.014
SWE-6	ND	ND	0.052	0.444	0.671	0.043
SWI-7	ND	ND	ND	0.01	0.117	0.067
SWE-7	ND	0.001	0.139	1.26	1.31	0.125
SWI-8	ND	ND	ND	0.015	ND	0.037
SWE-8	ND	ND	0.056	0.262	2.04	0.054
SWI-9	ND	ND	ND	0.009	ND	0.031
SWE-9	ND	0.002	0.164	1.56	3.27	0.162
SWI-10	ND	ND	ND	0.013	ND	0.035
SWE-10	ND	0.001	0.098	1.27	1.82	0.12
SWI-11	ND	ND	ND	0.02	ND	0.034
SWE-11	ND	0.001	0.083	1.53	2.51	0.129
SWI-12	ND	ND	0.021	0.004	ND	0.049
SWE-12	ND	0.002	0.0959	1.39	2.3	0.121
SWI-13	ND	ND	ND	0.011	ND	0.048
SWE-13	ND	0.002	0.098	1.96	1.23	0.406
Detection Limit	0.23	0.001	0.003	0.005	0.084	0.012

* SWI - Scrubber water influent, SWE - scrubber water effluent.
‡ ND - Not detected, below the limit of detection.

TABLE 4-14. DISCHARGE RATE OF METALS IN SCRUBBER WATER

Run No.	Metal Discharge Emissions in Scrubber Water (g/hr)					
	As	Be	Cd	Cr	Pb	Ni
3	ND*	0.02	0.91	13.66	16.59	0.87
4	ND	ND	1.51	17.39	40.88	1.27
5	ND	ND	2.09	15.04	24.80	1.34
6	ND	ND	0.94	7.59	12.19	0.53
7	ND	0.02	2.53	22.71	21.67	1.05
8	ND	ND	1.02	4.49	37.06	0.31
9	ND	0.04	2.98	28.18	59.41	2.38
10	ND	0.02	1.78	22.84	33.07	1.54
11	ND	0.02	1.51	27.43	45.60	1.73
12	ND	0.04	1.36	25.18	41.79	1.31
13	ND	0.04	1.78	35.41	22.35	6.50
Average Metals Runs†	ND	0.02	1.44	15.03	29.78	1.01
Detection Limit‡	4	0.02	0.05	0.1	1.5	0.2

* ND - Not detected.

† Runs 5, 6, 8, 10, and 12.

‡ Detection Limit - Values represent the detection limit expressed in g/hr.

TABLE 4-15. METALS CONCENTRATION IN BOTTOM ASH

Run No.	Metal Concentration in Bottom Ash ($\mu\text{g/g}$)					
	As	Be	Cd	Cr	Pb	Ni
3-Ash	ND*	0.278	0.892	329	76.3	26.2
4-Ash	ND	0.419	0.698	349	56.5	27.5
5-Ash	ND	0.22	0.619	276	50.9	22
6-Ash	ND	0.377	1.41	413	89.3	27.2
7-Ash	ND	0.336	1.68	468	113	30.8
8-Ash	ND	0.217	0.886	329	54.7	25.8
9-Ash	ND	0.359	0.579	369	50.1	28.4
10-Ash	ND	0.279	0.796	334	61.5	24.5
11-Ash	ND	0.3	0.519	163	35.6	19.3
12-Ash	ND	0.139	0.775	270	36.6	19.3
13-Ash	ND	0.118	0.928	271	73.7	19
Detection† Limit	4.6	0.02	0.06	0.02	1.68	0.24

* ND - Not detected, below the level of detection.

† Detection Limit - Values shown represent limit of detection in μg divided by g of bottom ash.

TABLE 4-16. MASS FLOW RATE OF METALS IN BOTTOM ASH

Run No.	Mass Flow Rate of Specified Metal in Bottom Ash (g/hr)					
	As	Be	Cd	Cr	Pb	Ni
3	< 0.7*	0.042	0.134	11.48	49.5	3.94
4	< 0.4	0.031	0.052	4.19	25.9	2.04
5	< 0.5	0.024	0.067	5.51	29.9	2.38
6	< 0.6	0.048	0.179	11.33	52.4	3.45
7	< 0.5	0.032	0.162	10.92	45.2	2.98
8	< 0.5	0.024	0.099	6.09	36.6	2.87
9	< 0.7	0.055	0.088	7.62	56.1	4.32
10	< 0.6	0.038	0.107	8.27	44.9	3.29
11	< 0.7	0.041	0.071	4.85	22.2	2.63
12	< 0.7	0.020	0.114	5.37	39.6	2.83
13	< 0.7	0.017	0.131	10.44	38.4	2.69
Average Metals runs†	< 0.6	0.031	0.113	7.31	40.7	2.97

* < - Not detected, value represents detection limit.

† Runs 5, 6, 8, 10, and 12.

4.2.7 Metal Emission Factors

One of the objectives of the overall sewage sludge incinerator test program was to develop emission factors relating the stack emissions of the target compounds to the sludge feed rate of these compounds. At Site 6, metal emissions testing was performed at both the inlet and outlet of the control device; therefore, uncontrolled and controlled emissions can be related to the sludge feed composition. The ratios of uncontrolled metal emissions (outlet of the incinerator or inlet to the control device) to the metal feed rates in the sludge are presented in Table 4-17. The metals feed rate to the incinerator was to be calculated based on the sludge feed rate and the metals analyses of the sludge feed. These ratios were calculated based on the metals at each location compared to the metals in the ash and the inlet sampling location. The metals in the sludge feed was not used because they did not seem to be reliable based on the material balance. The metal feed rate to the incinerator was based on the sludge feed rate, the percent ash in the sludge, and the metals in ash discharge plus the metals emissions at the inlet location. The metals at the inlet location in terms of metals emitted from the incinerator compared to metals feed to the incinerator averaged: arsenic - not detected, beryllium - 35.1%, cadmium - 97.8%, chromium - 62.9%, lead - 39.7%, and nickel - 29.1%. The corresponding control device outlet (exhaust stack) emission factors are also presented in Table 4-17. The metals at the outlet location in terms of metals emitted to the atmosphere compared to metals feed to the incinerator averaged: arsenic - not detected, beryllium - at or below detection ($< 6.9\%$), cadmium - 92.2%, chromium - 1.1%, lead - 14.1%, and nickel - 2.4%. The controlled metal emission factors decrease in the same proportion as the control device removal efficiency.

Another factor used is the ratios of metals in terms of μg of metal measured in the emissions to g of particulate (see Table 4-18). The inlet metal emissions factors in terms of μg of metal measured in the emissions to g of particulate averaged: arsenic - not detected for all samples ($< 19 \mu\text{g/g}$), beryllium - $0.43 \mu\text{g/g}$, cadmium - $205 \mu\text{g/g}$, chromium - $342 \mu\text{g/g}$, lead - $2980 \mu\text{g/g}$, and nickel - $34 \mu\text{g/g}$.

TABLE 4-17. INLET AND OUTLET METAL EMISSION FACTORS

Run No. Location	g metal at location/g metal in sludge					
	As	Be	Cd	Cr	Pb	Ni
Run 5 Inlet	ND*	0.415	0.988	0.726	0.393	0.382
Run 5 Outlet	ND	< 0.087†	0.949	0.012	0.115	0.037
Run 6 Inlet	ND	0.257	0.954	0.497	0.247	0.244
Run 6 Outlet	ND	< 0.051	0.885	0.010	0.130	0.023
Run 8 Inlet	ND	0.317	0.991	0.596	0.530	0.259
Run 8 Outlet	ND	< 0.087	0.963	0.026	0.189	0.036
Run 10 Inlet	ND	0.267	0.980	0.589	0.416	0.256
Run 10 Outlet	ND	0.062	0.918	0.006	0.145	0.010
Run 12 Inlet	ND	0.497	0.977	0.737	0.397	0.315
Run 12 Outlet	ND	0.056	0.897	0.004	0.126	0.016
Average Inlet	ND	0.351	0.978	0.629	0.397	0.291
Average Outlet	ND	< 0.069	0.922	0.011	0.141	0.024

* ND - Not detected, all sample measurements were below the analytical detection limit.

† < - Outlet samples were below analytical detection limit, calculated ratio is less than value shown.

TABLE 4-18. RATIO OF METAL TO PARTICULATE

Run No./ Location	Ratio of Metal to Particulate (μg metal/g particulate)					
	As	Be	Cd	Cr	Pb	Ni
Run 5						
Inlet	< 19*	0.33	111	287	1635	29
Outlet	< 750	< 3.26	1811	111	24046	131
Run 6						
Inlet	< 21	0.45	100	303	1146	30
Outlet	< 989	< 4.30	2301	211	32301	133
Run 8						
Inlet	< 43	0.57	562	455	7455	51
Outlet	< 493	2.14	2401	163	28296	101
Run 10						
Inlet	< 23	0.40	149	345	2752	33
Outlet	< 773	< 3.36	1613	81	30387	47
Run 12						
Inlet	< 20	0.43	101	320	1889	28
Outlet	< 471	2.05	1676	55	32992	76
Run 5&6						
Inlet	< 20	0.39	106	295	1390	30
Outlet	< 870	< 3.78	2056	161	28173	132
Run 10&12						
Inlet	< 21	0.41	125	333	2320	30
Outlet	< 622	< 2.71	1645	68	31689	61
Average						
Inlet	< 25	0.43	205	342	2975	34
Outlet	< 695	< 3.02	1960	124	29604	97

* < - Below the level of detection, value indicates detection limit.

The ratio of metals for the controlled flue gas metals measured in the emissions to g of particulate in the controlled emissions averaged: arsenic - not detected in any sample ($< 695 \mu\text{g/g}$), beryllium - at or below the detection limit (less than $3 \mu\text{g/g}$), cadmium - $1960 \mu\text{g/g}$, chromium - $124 \mu\text{g/g}$, lead - $29,600 \mu\text{g/g}$, and nickel - $97 \mu\text{g/g}$.

4.3 HEXAVALENT CHROMIUM RESULTS

4.3.1 Control Device Inlet Results

The inlet location samples collected for hexavalent chromium were analyzed by EPA/EMSL in Cincinnati but the data were not released by EMSL for publication.

4.3.2 Control Device Outlet Results

The testing for hexavalent and total chromium at Site 6 had three major objectives: (1) evaluate the emission testing methodology, (2) determine the effect of lime on the conversion of total chromium in the sludge to hexavalent chromium in the emissions, and (3) determine the effect of excess air and other combustion conditions on the conversion of other forms of chromium to the hexavalent state. Testing for hexavalent chromium was conducted using a recirculating impinger reagent (RC) train spiked with the hexavalent chromium isotope (^{51}Cr) in all four chromium trains for Runs 3 and 7 and in two of the four trains for Runs 9, 11, and 13. Two of the four trains were spiked with a second hexavalent chromium isotope (^{53}Cr) for Runs 9, 11, and 13. The second hexavalent chromium isotope (^{53}Cr) samples were analyzed by the EPA/EMSL but the results were not released by EMSL for publication.

The inlet and outlet flue gas conditions shown in Tables 4-1 and 4-2 were monitored continuously while establishing incinerator operating conditions thought to favor conversion of trivalent to hexavalent chromium. It was also thought that the concentration of SO_2 and THC would provide information regarding the conversion of hexavalent chromium to trivalent chromium during sample collection.

The outlet emission concentrations of hexavalent chromium are summarized in Table 4-19. The recirculating impinger reagent train or RC train was used to collect the hexavalent and total chromium emissions. This train is designed to provide immediate contact of the incoming hexavalent chromium with a basic solution coupled with continuous rinsing of the sample probe to reduce conversion of hexavalent chromium to trivalent chromium during sample collection. The "⁵¹Cr RC" train (see Table 4-19) was spiked with ⁵¹Cr to assess conversion of hexavalent chromium to trivalent chromium during sampling and sample recovery. Analysis of this isotope requires use of a scintillation counter. The level of hexavalent ⁵¹Cr spiked is below the detection level of the other analytical techniques for hexavalent chromium and total chromium. As shown in Table 4-19, the conversion of hexavalent to trivalent chromium in the RC train averaged approximately 10% for a sample collection period of two to four hours. Although the results for the RC trains spiked with ⁵³Cr are not shown, the conversion ratio was demonstrated using both the ⁵³Cr and ⁵¹Cr spikes. The "MMt1" designation in Table 4-19 indicates the total chromium results from the multiple metals train sampling runs. Based on the total chromium results of the hexavalent chromium train, it appears that the total chromium results of the multiple metals train is probably low by a factor of about two.

In Figure 4-1, the CO/CO₂ ratios from Table 4-2 are plotted against the hexavalent to total chromium ratios from Table 4-19. A relationship between good combustion and a higher ratio of hexavalent to trivalent chromium is evident. At low CO levels (good combustion), the ratio of hexavalent chromium to trivalent chromium is highest, with approximately 10% of the total chromium in the form of hexavalent chromium. At high CO levels (poor combustion), the ratio of hexavalent chromium to total chromium is significantly reduced to less than approximately 1%.

The recirculating impinger reagent train approach had demonstrated two problems prior to the Site 6 test: (1) the recirculating train did not completely prevent conversion of hexavalent to trivalent chromium in the presence of high levels of sulfur dioxide, and (2) trivalent chromium in the alkaline collection media was found to slowly convert to hexavalent chromium. In an effort to correct these problems, two new

TABLE 4-19. SUMMARY OF OUTLET SAMPLING RESULTS FOR
HEXAVALENT AND TOTAL CHROMIUM

Run No./ Date	Train and Isotopic Spike	Sample Fractions Cr+6 (ug)	Total Cr (ug)	Conversion of Hexavalent Chromium During Sampling, %	Cr+6 (ug/dscm)	Total Cr (ug/dscm)	Ratio of Cr ⁶⁺ to Cr, %
Run 3 10/09	A-51Cr RC*	0.36	13.2	1.2	0.15	5.6	2.7
	B-51Cr RC	0.10	13.7	1.1	0.06	8.6	0.7
	C-51Cr RC	0.05	7.9	24.6	0.03	4.3	0.6
	D-51Cr RC	0.01	12.5	12.7	0.005	6.1	0.1
	Average				0.06	6.2	1.0
Run 5 Run 6 10/10	MMtl-D	----	5.8	----	----	2.1	----
	MMtl-D	----	8.8	----	----	4.0	----
Run 7 10/10	A-51Cr RC	0.62	15.0	18.1	0.17	4.1	4.1
	B-51Cr RC	0.57	14.3	12.6	0.15	3.9	4.0
	C-51Cr RC	0.55	15.6	15.3	0.16	4.4	3.5
	D-51Cr RC	0.34	47.9	9.6	0.10	13.7†	2.3‡
	Average				0.14	4.2	3.5
Run 8 10/11	MMtl-D	----	14.2	----	----	5.9	----
Run 9 10/11	C-51Cr RC	0.74	13.0	6.1	0.18	3.1	5.7
	D-51Cr RC	1.33	16.1	3.3	0.29	3.6	8.2
	Average				0.24	2.9	7.0
Run 10 10/12	MMtl-D	----	3.8	----	----	1.7	----
Run 11 10/12	C-51Cr RC	1.35	16.6	5.4	0.31	3.8	8.1
	D-51Cr RC	1.49	13.4	2.7	0.38	3.4	11.1
	Average				0.34	3.1	9.6
Run 12 10/12	MMtl-D		1.7		----	0.8	---
Run 13 10/13	C-51Cr RC	0.21	15.3	6.0	0.04	2.7	1.4
	D-51Cr RC	0.12	13.7	3.2	0.02	2.3	0.9
	Average				0.03	3.3	1.1

* RC - Recirculating impinger reagent train for hexavalent chromium.

† Outlier, not included in the average.

‡ Average total chromium value used to calculate ratio.

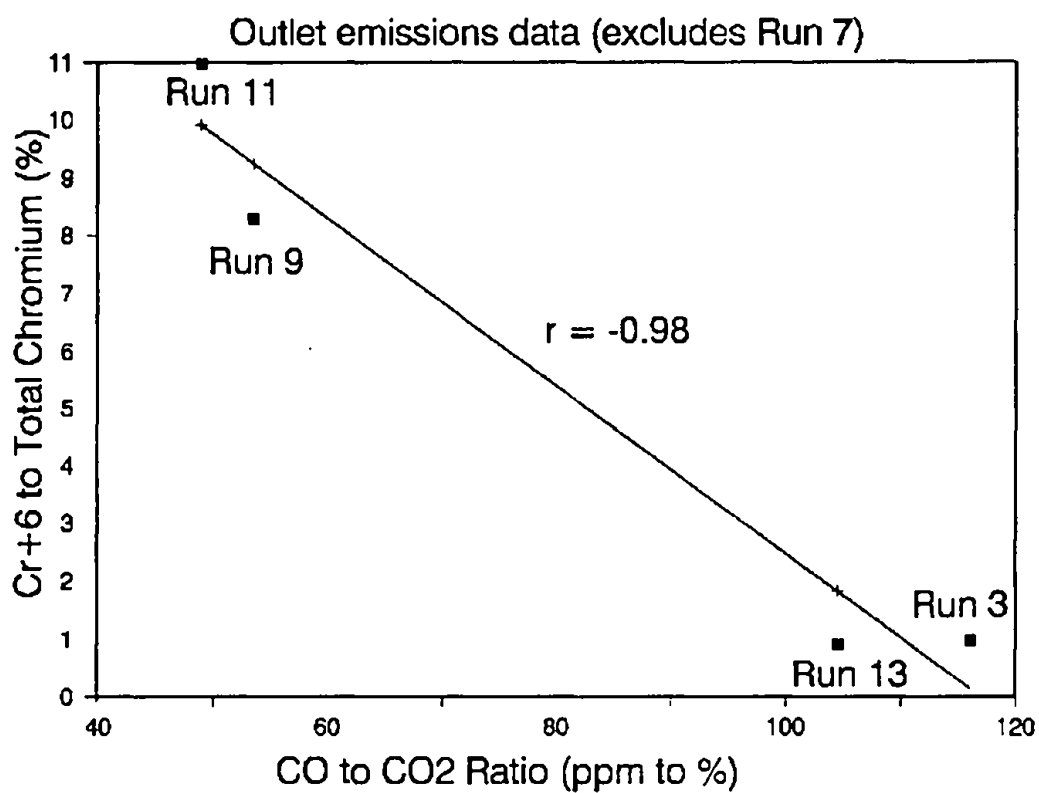


Figure 4-1. Cr⁺⁶ to total chromium versus CO to CO₂ ratios.

procedures were added to the recirculating train sampling protocol for testing at Site 6: (1) a 30-minute nitrogen purge of the sample at a rate of 20 L/min immediately after sample collection, and (2) pressure filtering of the sample through a 0.2 um filter following the nitrogen purge. The net results of these procedural changes were to purge oxygen and sulfur dioxide from the sample and to remove all the insoluble trivalent chromium and other materials from the filtrate solution containing the hexavalent chromium. Conversion was almost completely eliminated and, as shown in Table 4-20, the samples remained stable over a 2-month period.

Samples for the inlet and outlet runs were also analyzed for hexavalent and total chromium using X-ray absorption near-edge structure (XANES) and extended X-ray absorption fine structure (EXAFS) by BYU; hexavalent chromium was below the instrument's detection limit for all samples indicating a hexavalent to total chromium ratio of less than 20%.

4.4 NICKEL SPECIATION RESULTS

The major objective of the nickel speciation testing was to determine the percent of the nickel emissions in the form of nickel subsulfide. It was anticipated that the nickel subsulfide emissions from multiple hearth incinerators would constitute less than 1% of the total nickel emissions, because these incinerators typically operate with high excess air which is not favorable for the formation of nickel subsulfide. The first laboratory to conduct the wet chemical speciation of nickel experienced problems. Dr. Vladimir Zatzka, the developer of the Nickel Producers Environmental Association (NiPERA) nickel speciation method, was then contracted to conduct the sample analysis. This wet chemical analytical method involves sequential leaching. The first leaching step removes all soluble nickel. Peroxide is used to convert the nickel sulfides and subsulfides to soluble nickel sulfate which is then leached in the second step. The third step leaches the metallic nickel compounds and finally total digestion of the remaining sample typically containing nickel oxides. The results of the sequential leaching nickel analysis shown in Table 4-21 indicate that within the detection limit of the wet chemical

TABLE 4-20. STABILITY STUDY OF OUTLET SAMPLING RESULTS FOR HEXAVALENT CHROMIUM

Run No. Date	Train	Actual Cr+6 (ug)	Conversion of Hexavalent Chromium During Sampling, %	Concentration of Cr+6		Difference Cr ⁺⁶ (ppb)
				10/25/89 (ppb)	12/12/89 (ppb)	
Run 9 10/11	C	0.74	6.1	0.78	0.80	0.02
	D	1.33	3.3	0.46	0.46	0.00
	Average		4.7			0.01
Run 11 10/12	C	1.35	5.4	0.57	0.59	0.02
	D	1.49	2.7	0.52	0.57	0.05
	Average		4.1			0.04
Run 13 10/13	C	0.21	6.0	0.25	0.29	0.04

TABLE 4-21. SUMMARY OF NICKEL SPECIES EMISSIONS: SITE 6.

Run No.	Nickel							
	Soluble		Sulfidic*		Oxidic		Total	
	ug/dscm	% Total	ug/dscm	% Total	ug/dscm	% Total	ug/dscm	% Total
Outlet								
Run 5	1.6	58	< 0.15†	< 5	1.2	42	2.8	100
Run 6	0.9	42	< 0.18	< 8	1.3	58	2.2	100
Run 10	1.1	60	< 0.18	< 10	0.7	40	1.8	100
Run 12	0.7	39	< 0.20	< 11	1.1	61	1.8	100
Inlet								
Run 5	65	41	< 18	< 12	92	59	157	100
Run 6	98	41	< 28	< 12	140	59	238	100
Run 8	18	21	< 6	< 7	66	79	84	100
Run 10	65	41	19	12	74	47	158	100
Run 12	64	77	< 13	< 15	19	23	83	100
Run 8AF‡	15	17	< 6	< 7	72	83	87	100

* The sulfidic nickel is a combination of the nickel sulfide and nickel subsulfide.

† < - Below limit of detection, values indicate detection limit.

‡ The effect of quartz fibers on the leach recovery was investigated by spreading one sample portion (inlet Run 8) over one quartz fiber filter (85 mm) and carrying it through the speciation process. The results are shown above as Run 8AF.

method, no nickel subsulfide was present in the samples. Consideration of the detection limit indicates nickel subsulfide to total nickel ratio is less than 12% for the inlet emissions and less than 10% for the outlet emissions. BYU also analyzed samples from the same runs by XANES and EXAFS; no nickel subsulfide was detected within the instrumental detection limit of 10% of the total nickel.

4.5 CONTINUOUS EMISSION MONITORING RESULTS

Continuous emission monitoring (CEM) was performed at the inlet and outlet sampling locations at Site 6. The inlet CEM systems included oxygen (O_2), carbon dioxide (CO_2), carbon monoxide (CO), and oxides of nitrogen (NO_x). The outlet CEMs included oxygen (O_2), carbon dioxide (CO_2), carbon monoxide (CO), oxides of nitrogen (NO_x), and total hydrocarbons (THC). The CEM probes were located upstream of the manual sampling locations. All measurements were made on a dry basis. The averages of the CEM data on a run-by-run basis are presented in Table 4-2. The one-minute averages for each compound for all the runs included in Appendix E of Volume IV: Site 6 Report, Appendices. To provide an indication of how the monitored emissions changed with time, the 15-minute averages are presented in Table 4-22. The indicated time (i.e., 12:06) is the time at the end of the 15-min average. Runs 3, 4, 5, 6, 7 and 13 represent normal furnace operating conditions. Run 8 represents a transition period when the furnace operating conditions were being changed to yield low CO emissions. Runs 9, 10, 11, and 12 represent the low CO conditions. As previously mentioned, several additional auxiliary burners were put into operation to obtain the low CO conditions.

EPA is evaluating CO and THC monitoring as a surrogate indicator of organic emissions. Since no organic compound specific measurements were made at this site, the relationship between CO and THC emissions under the tested conditions is shown in Figure 4-2. When Run 5 is excluded, the correlation coefficient between the CO and THC is 0.97 for the data from the 2- and 4-hr runs.

TABLE 4-22. SUMMARY OF INLET AND OUTLET CONTINUOUS EMISSION MONITORING RESULTS
(15-min averages)

Time 24 hr.	Inlet Location					Outlet Location					
	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	THC (ppm)
Run 3 - October 9, 1989											
12:06	17.2	3.3	313.1	3.0	60.7	15.1	4.7	458.5	20.8	125.1	13.3
12:21	12.7	3.2	241.2	11.1	36.0	14.1	5.6	501.9	22.1	120.7	16.3
13:02	8.9	10.2	1115.4	45.4	177.1	11.0	8.3	1124.4	31.3	142.7	47.5
13:17	15.8	4.4	770.8	33.2	155.4	15.8	4.2	729.0	24.2	146.5	34.6
13:32	15.0	4.8	668.6	28.4	134.4	14.7	5.0	688.5	27.0	141.2	20.5
13:47	13.4	6.2	806.7	40.3	161.4	13.9	5.8	734.6	31.0	154.0	22.5
14:02	11.9	7.4	625.6	63.1	165.6	13.4	6.2	497.7	27.0	146.1	15.7
14:17	14.1	5.7	584.0	40.5	146.9	14.3	5.5	553.1	26.1	151.6	15.4
14:32	13.0	6.5	573.4	56.7	152.6	13.8	5.9	474.7	24.2	151.1	13.1
14:47	14.1	5.5	453.1	36.9	148.4	14.6	5.1	393.7	23.1	150.9	11.4
Run 4 - October 9, 1989											
17:43	14.2	5.6	416.2	19.7	106.9	13.2	6.0	448.7	21.2	132.5	12.4
17:58	9.6	4.2	303.4	30.8	101.4	13.8	5.7	310.9	18.4	125.7	9.1
18:13	14.2	4.7	308.3	41.8	97.1	14.0	5.2	330.5	18.7	125.2	9.6
18:28	15.1	4.7	343.3	40.8	109.4	14.3	5.2	350.5	20.4	140.0	11.4
18:43	15.6	4.2	387.4	38.0	112.6	14.8	4.8	434.4	20.6	146.8	13.3
18:58	13.7	5.9	594.2	48.3	145.1	13.4	6.0	610.4	25.3	176.2	18.8
19:13	11.3	7.5	524.8	116.0	129.8	12.9	6.3	598.4	47.1	111.7	57.6

(Continued)

TABLE 4-22. (Continued)

Time 24 hr.	Inlet Location					Outlet Location					
	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	THC (ppm)
Run 5 - October 10, 1989											
09:28	9.6	9.2	1004.7	28.5	124.7	12.7	6.7	754.6	22.3	111.9	31.4
09:43	8.7	9.9	1249.6	62.0	150.9	12.0	7.3	1512.2	47.2	124.9	107.0
09:58	9.3	9.5	1240.9	42.9	169.4	12.5	6.9	1156.5	34.7	132.9	58.6
10:13	10.9	8.2	882.6	31.3	157.1	13.6	6.1	661.8	26.4	129.7	28.3
10:28	11.8	7.5	562.4	26.7	135.1	14.2	5.6	401.8	24.3	125.4	14.2
10:43	9.8	9.2	916.8	31.1	125.8	12.9	6.7	711.5	27.1	116.6	33.2
10:58	11.6	7.7	1171.8	41.4	153.5	14.0	5.7	940.2	35.4	137.3	43.2
11:13	9.4	9.6	1004.3	43.0	146.0	12.7	6.9	890.9	36.7	132.5	49.3
Run 6 - October 10, 1989											
13:58	8.7	9.9	796.1	23.3	160.9	11.1	8.0	618.3	20.6	125.3	25.5
14:30	12.1	7.3	677.5	60.2	163.1	13.7	6.1	559.8	20.5	154.1	18.2
14:45	13.4	6.3	752.8	44.7	169.7	15.1	4.9	593.8	21.1	132.5	20.3
15:40	13.4	6.2	450.4	20.8	142.0	14.9	5.0	353.6	17.1	127.0	11.4
15:55	12.4	6.8	604.3	31.5	191.3	14.8	5.0	437.1	22.1	162.7	12.3
16:10	13.0	6.3	768.9	37.7	191.9	14.6	5.1	605.6	28.9	180.4	15.2
16:25	12.4	6.7	868.1	39.3	198.9	13.9	5.6	687.1	32.3	188.1	17.9
16:40	11.8	7.4	892.1	42.3	212.4	13.2	6.2	702.5	33.6	200.5	20.8
16:55	12.3	7.0	826.1	39.9	204.6	13.3	6.2	686.3	33.5	202.5	21.8

(Continued)

TABLE 4-22. (Continued)

Time 24 hr.	Inlet Location					Outlet Location					
	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	THC (ppm)
Run 7 - October 10, 1989											
18:27	14.8	4.7	296.7	16.4	148.3	15.2	4.4	323.2	18.7	148.4	12.0
18:42	14.2	5.2	666.5	23.9	164.2	15.2	4.3	574.4	25.2	146.2	16.2
18:57	13.8	5.3	1138.6	28.3	205.5	15.3	4.2	907.3	26.5	161.1	20.6
19:12	14.6	4.7	1131.8	27.5	169.2	15.5	4.0	963.5	30.1	146.8	26.8
19:27	12.4	6.6	1246.7	36.8	228.8	13.9	5.4	1061.4	37.0	166.2	28.4
19:42	13.5	5.7	1242.4	31.2	242.1	14.1	5.2	1310.8	35.3	189.1	31.1
19:57	13.6	5.6	1089.6	30.0	204.5	13.8	5.5	1005.7	34.8	168.6	25.6
20:12	12.3	6.8	1012.7	42.8	221.2	12.8	6.4	890.4	35.1	179.5	24.5
20:27	11.3	7.8	769.7	56.5	197.7	12.0	7.2	692.9	31.8	164.8	22.9
20:42	12.8	6.4	732.1	37.8	200.1	13.4	6.0	684.2	31.8	178.9	22.8
20:57	13.4	5.9	733.0	32.9	203.5	14.0	5.5	692.8	30.0	181.8	19.9
21:12	13.1	6.1	639.5	33.7	191.8	14.2	5.3	563.8	29.7	161.3	18.6
21:27	13.0	6.2	657.0	36.2	184.9	14.0	5.5	581.4	33.2	152.9	20.7

(Continued)

TABLE 4-22. (Continued)

Time 24 hr.	Inlet Location					Outlet Location					
	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	THC ppm
Run 8 - October 11, 1989											
09:13	10.3	8.5	570.7	51.2	163.5	12.4	6.9	443.7	22.2	114.7	15.4
09:28	8.3	10.6	632.5	90.3	197.2	11.4	7.8	454.2	17.1	131.0	14.9
09:43	7.9	11.1	788.2	114.8	189.9	11.1	8.2	630.2	21.3	121.6	23.2
10:00	9.5	9.7	735.5	94.8	186.0	12.0	7.3	562.9	20.6	114.1	17.5
10:15	9.4	9.6	971.6	114.6	175.3	11.9	7.4	787.1	21.9	97.1	21.1
10:30	9.4	9.7	911.7	92.1	171.8	12.5	6.9	666.7	20.3	89.6	18.7
10:45	11.0	8.4	711.5	90.3	131.6	13.0	6.5	586.5	20.0	76.6	17.4
11:00	10.1	9.0	760.6	67.4	165.4	13.3	6.1	496.7	18.8	92.7	14.7
11:15	8.0	10.7	698.6	107.1	182.9	11.7	7.5	467.6	19.1	114.7	13.2
11:30	8.2	10.7	624.2	117.3	174.1	10.7	8.4	481.9	21.2	119.8	15.1
11:45	7.5	11.3	1044.9	152.2	192.2	10.1	9.0	962.9	27.0	119.8	27.0
Run 9 - October 11, 1989											
15:41	10.8	8.2	436.2	53.1	178.3	13.2	6.4	326.2	12.3	123.4	6.5
16:00	12.0	7.2	355.5	44.1	181.1	13.9	5.7	261.5	13.2	128.2	5.8
16:15	11.7	7.4	325.1	38.5	178.8	14.2	5.5	223.0	13.0	120.0	4.9
16:30	12.7	6.5	296.6	33.2	173.1	14.6	5.2	224.0	13.9	125.1	5.1
16:45	12.0	7.0	439.4	32.1	198.8	14.5	5.1	321.3	18.0	137.1	8.1
17:00	10.6	8.2	488.5	43.3	217.3	13.2	6.3	364.8	21.4	152.6	9.9
17:15	10.5	8.4	490.9	52.2	202.2	12.5	6.9	398.9	21.1	148.9	9.2
17:30	10.8	8.2	500.4	50.1	201.1	13.0	6.6	383.3	18.6	141.8	8.2
17:45	10.6	8.3	537.1	47.8	206.4	13.2	6.4	388.4	18.3	138.2	8.1
18:00	10.9	8.1	556.6	46.7	198.1	13.2	6.3	423.4	19.2	140.0	8.8
18:15	10.0	8.9	541.3	58.2	206.3	12.4	7.1	417.9	18.9	141.3	8.4
18:30	11.6	7.5	395.8	43.7	169.5	13.0	6.6	327.4	15.7	128.2	7.1
18:45	12.9	6.5	299.5	33.2	137.1	13.3	6.4	277.9	14.9	111.2	6.8

(Continued)

TABLE 4-22. (Continued)

Time 24 hr.	Inlet Location					Outlet Location					
	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	THC (ppm)
Run 10 - October 12, 1989											
09:25	11.5	7.8	318.1	33.0	155.6	13.4	6.4	248.1	16.9	119.2	5.8
09:40	12.3	7.1	331.9	32.5	160.1	13.9	5.9	266.6	18.3	127.6	6.2
09:55	12.2	7.1	431.0	32.8	188.0	14.3	5.6	329.9	18.4	144.0	8.2
10:10	11.6	7.5	521.1	37.5	211.2	13.6	6.1	404.5	21.7	169.1	10.3
10:25	10.3	8.8	417.3	57.9	201.9	12.0	7.7	331.8	23.2	169.5	10.2
10:40	11.5	7.8	362.1	57.8	175.1	12.2	7.5	318.9	24.0	160.7	9.8
10:55	11.4	7.9	432.1	54.2	156.3	12.8	7.0	351.0	23.4	132.4	9.5
11:10	11.6	7.7	477.5	59.4	158.4	12.9	6.7	391.7	24.2	130.4	9.9
Run 11 - October 12, 1989											
12:22	11.7	7.5	260.3	59.2	143.4	13.0	6.7	207.7	9.3	133.6	5.3
12:53	11.9	7.3	400.7	61.1	143.8	13.6	6.0	297.9	9.9	117.9	8.3
13:08	11.5	7.7	438.3	66.4	167.5	12.7	6.8	352.9	9.5	146.3	9.2
13:23	11.7	7.7	345.9	62.6	157.6	12.4	7.2	291.6	7.4	148.7	8.0
13:38	12.1	7.3	385.5	57.6	148.0	12.8	6.8	323.1	5.3	139.1	8.4
13:53	11.2	7.9	459.7	62.7	161.3	12.8	6.8	352.0	5.2	133.2	8.6
14:08	11.0	8.1	514.8	64.6	164.9	12.6	6.9	394.3	6.3	132.4	9.2
14:23	10.6	8.5	418.8	69.7	170.0	12.0	7.6	322.2	16.6	109.0	7.6
14:38	11.7	7.5	370.7	56.6	145.3	13.0	6.6	289.4	10.3	122.8	7.1
14:53	10.7	8.3	394.3	61.8	166.5	12.4	7.2	299.6	11.6	137.5	7.2
15:08	12.1	7.1	315.5	50.3	141.1	13.1	6.5	252.8	10.4	128.5	6.2
15:23	11.4	7.7	314.1	51.9	142.3	13.1	6.5	230.2	10.4	119.6	5.3
15:38	12.0	7.2	319.4	52.7	146.9	13.4	6.2	241.3	10.1	121.9	5.4

(Continued)

TABLE 4-22. (Continued)

Time 24 hr.	Inlet Location					Outlet Location					
	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	O2 (%)	CO2 (%)	CO (ppm)	SO2 (ppm)	NOx (ppm)	THC (ppm)
Run 12 - October 12, 1989											
17:47	11.3	7.8	465.1	38.4	186.9	13.2	6.6	354.4	12.0	133.9	7.5
18:02	13.2	6.2	339.9	37.3	141.5	13.8	6.1	302.8	8.2	115.9	7.1
18:17	12.1	7.1	336.4	41.9	147.8	13.7	6.1	261.9	7.1	107.4	6.0
18:32	11.8	7.4	297.2	42.9	141.5	13.7	6.1	222.1	6.3	101.1	5.3
18:47	11.5	7.6	387.3	40.5	150.6	14.0	5.8	269.8	7.5	98.2	6.5
19:02	9.3	9.2	536.7	51.0	210.0	13.2	6.4	344.3	11.0	126.8	8.4
19:17	10.7	8.2	455.4	49.2	175.9	12.8	6.8	342.0	10.1	128.4	8.6
19:32	11.1	7.9	393.1	49.0	160.1	12.9	6.8	302.2	8.0	120.8	7.5
19:47	12.4	6.8	365.4	44.3	146.9	13.5	6.3	303.1	7.8	116.4	7.8
Run 13 - October 13, 1989											
09:27	12.9	6.3	760.2	32.3	190.3	14.2	5.4	631.5	28.2	157.3	16.9
09:42	12.9	6.4	659.6	26.1	206.8	14.1	5.5	557.2	26.8	164.9	14.6
09:57	13.2	6.2	643.9	22.7	186.9	14.7	5.0	525.0	24.3	142.7	14.2
10:12	13.2	6.1	563.9	22.8	189.5	14.7	5.0	454.0	25.0	144.2	13.3
10:27	14.2	5.5	535.7	20.5	164.5	15.4	4.5	434.3	24.7	127.8	14.2
10:42	13.3	6.1	532.4	23.7	166.6	14.6	5.1	437.9	28.0	134.3	14.8
10:57	13.5	5.8	508.1	24.2	161.7	14.5	5.0	438.5	30.0	140.0	16.1
11:12	13.3	5.9	703.0	30.9	184.0	14.6	5.0	574.0	35.2	149.9	18.6
11:27	13.2	6.0	741.0	31.4	191.9	14.3	5.3	629.2	37.6	154.7	20.2
11:42	12.8	6.4	741.6	31.4	207.0	13.8	5.7	621.0	38.3	161.3	18.7
11:57	13.1	6.1	730.7	28.9	202.9	13.9	5.6	642.5	36.5	162.2	18.6
12:12	13.0	6.3	663.2	25.9	196.6	13.8	5.8	587.2	33.6	156.3	17.1
12:27	13.0	6.3	635.1	25.7	189.7	14.0	5.6	546.8	32.0	144.5	15.5
12:42	12.9	6.4	641.0	27.2	196.3	14.1	5.5	531.1	32.9	146.6	14.5
12:57	13.8	5.7	683.4	24.5	190.5	14.6	5.2	584.5	33.4	149.0	16.8

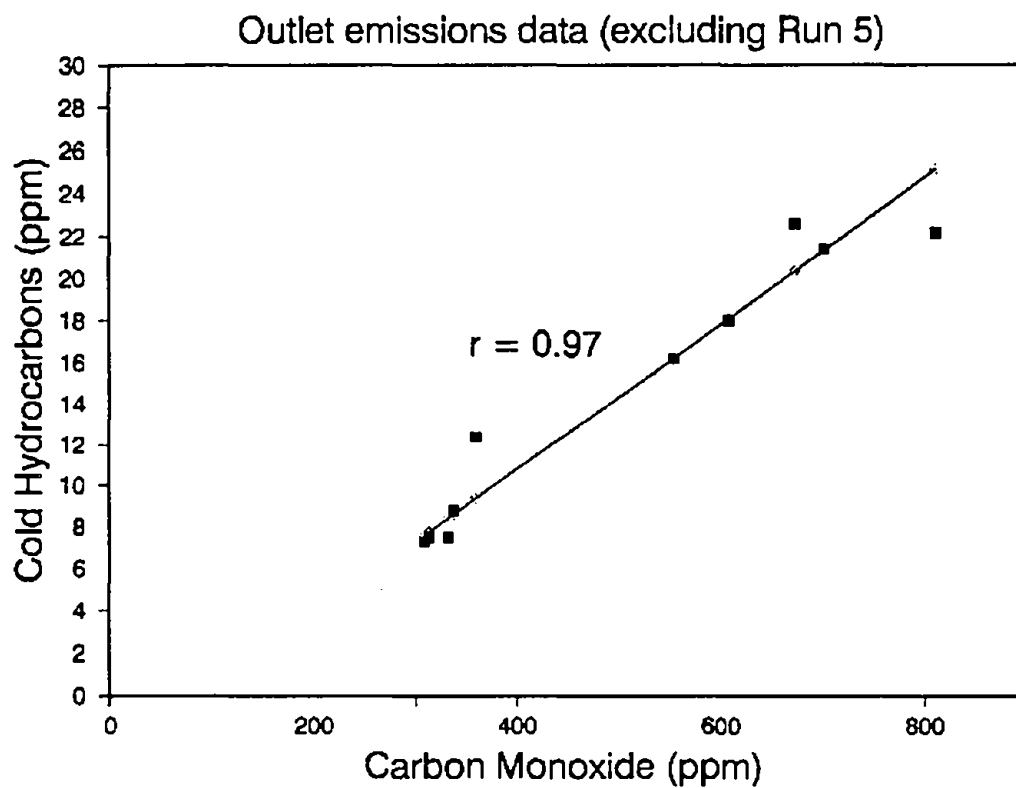


Figure 4-2. Hydrocarbon emissions versus carbon monoxide emissions.

4.6 CONCLUSIONS FROM SITE 6 TEST

From the perspective of methods development and data quality, the conclusions that may be drawn from the Site 6 testing are:

1. The ratio of hexavalent chromium to total chromium is relatively high (greater than 10%) when lime is used for sludge conditioning, during good combustion conditions, and under the long residence times required for combustion of sludge in a multiple hearth incinerator.
2. The ratio of nickel subsulfide to total nickel was less than detectable (less than 12%) under both furnace operating conditions.
3. There was good correlation between CO emissions and THC emissions.
4. The recirculating impinger reagent train with certain post-sampling procedural modifications yielded acceptable results for the measurement of hexavalent chromium at the outlet.
5. The process operating conditions used for the final series of test runs at Site 6 greatly reduced the level of CO and THC emissions by approximately 60%.

5.0 SAMPLING LOCATION SELECTION AND SAMPLING PROCEDURES

Sampling procedures used during the Site 6 program are briefly described in this section. Standard EPA methods or draft EPA procedures were used for all sampling. Emission sampling locations are discussed in Section 5.1, and methods and procedures are discussed in Section 5.2.

5.1 EMISSION SAMPLING LOCATIONS

Emission sampling was conducted at the inlet to the control system (incinerator discharge) and outlet of the control system which consists of venturi scrubber/impingement tray scrubber at the outlet stack. The particulars for each of these sampling locations are described below.

5.1.1 Inlet to the Control System

The sampling location for the inlet to the control system is shown in Figures 5-1 (Point 1) and Figure 5-2. The flue gas at this point was coming directly from the incinerator at a temperature of about 1,000°F (538°C). As shown, the ducting exits the furnace horizontally, turns, and goes down vertically into the venturi scrubber. The exact direction of the flow was determined using a directional probe and the "Alternative Measurement Site Selection Procedure" in EPA Method 1, Section 2.5 (40 CFR 60, July 1, 1988). The sampling train nozzles were then directed into the flow path for testing. As in the majority of incinerators, the duct is not long enough to provide a uniform flow pattern and meet the EPA Method 1 criteria. The objective of the program was, however, to determine the ratios of nickel subsulfide to total nickel and hexavalent

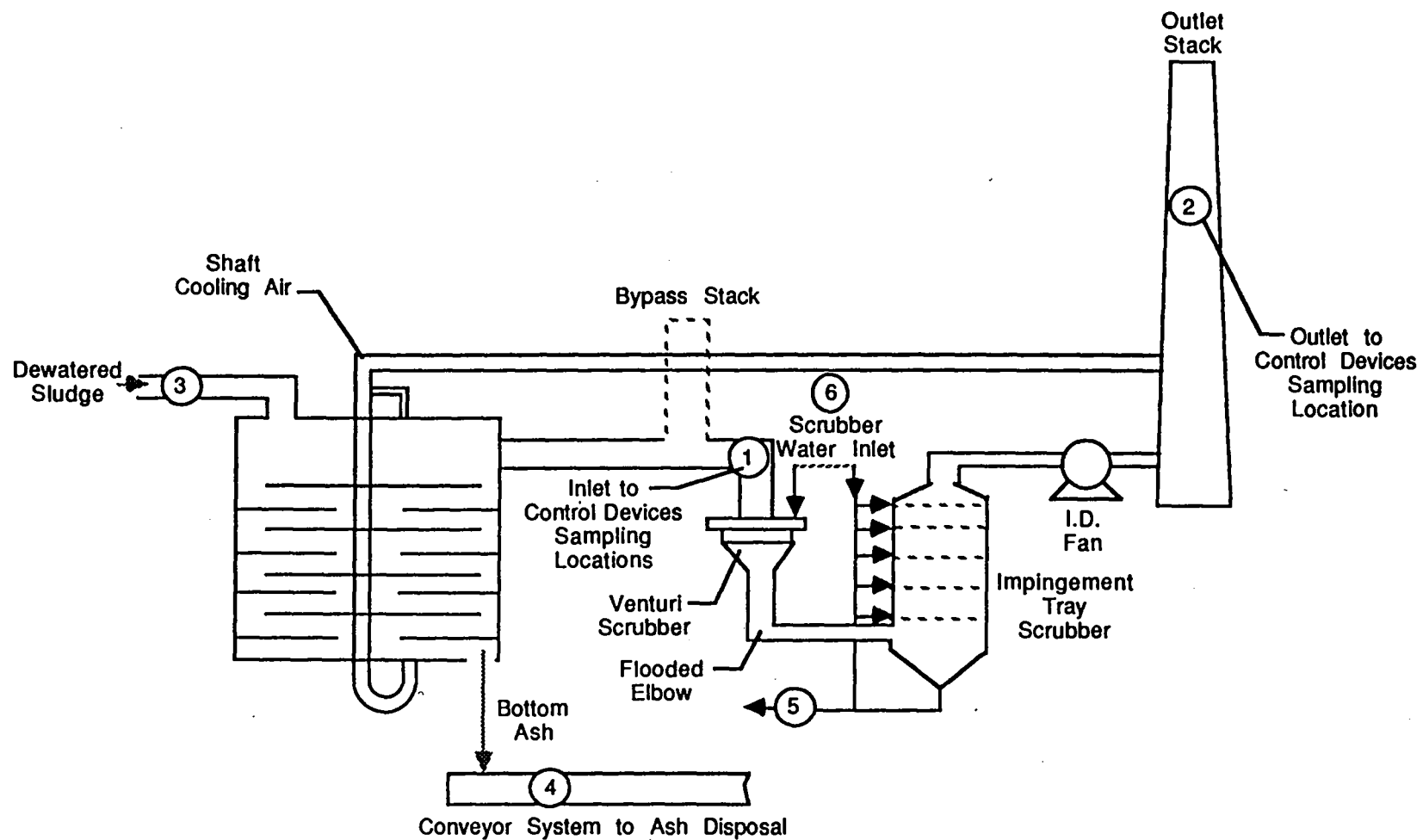


Figure 5-1. Process diagram with sampling locations.

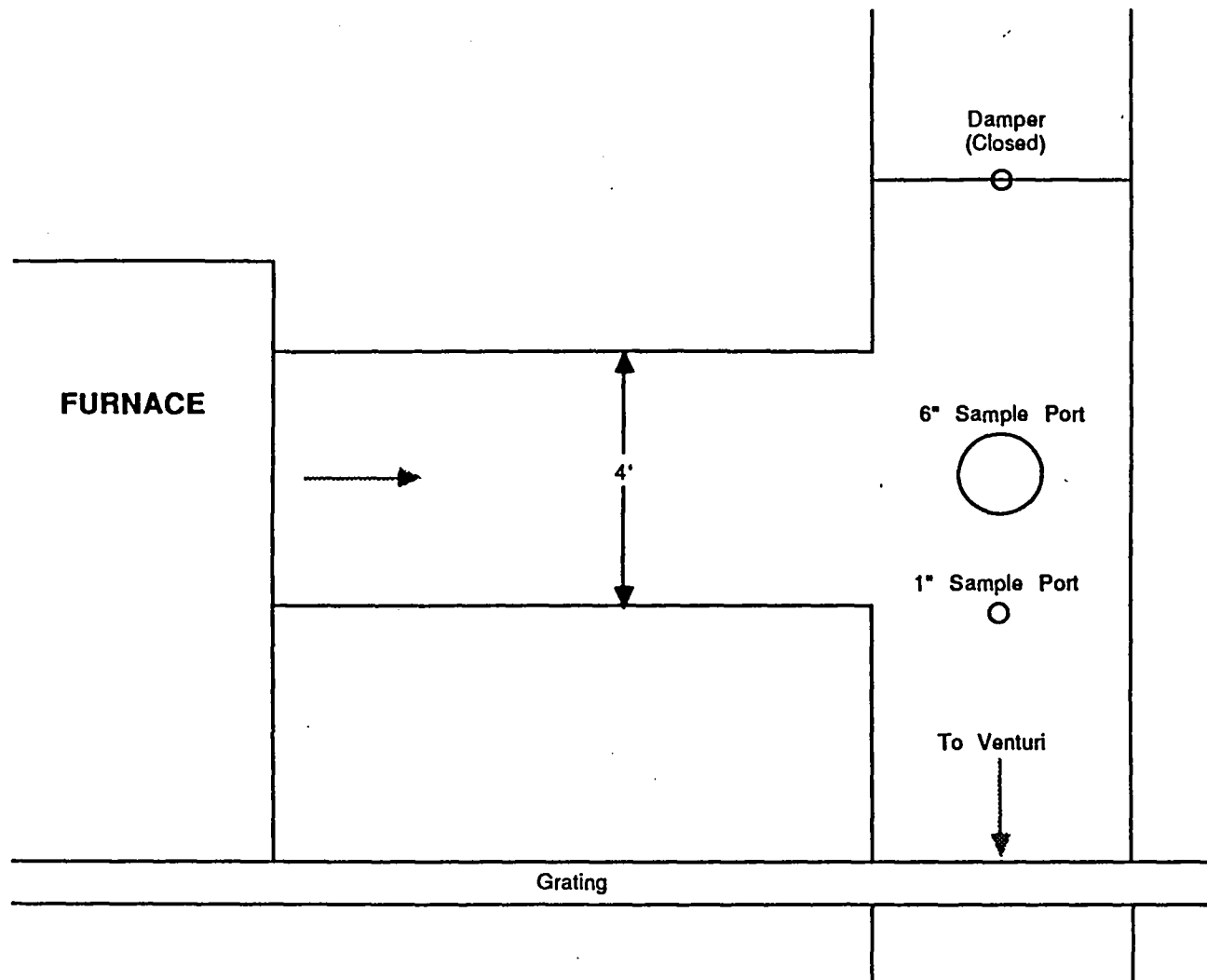


Figure 5-2. Inlet sampling location.

chromium to total chromium, rather than the absolute concentration of these emissions. Considering this and the fact that four sampling systems were operated simultaneously at the same point and the samples collected were analyzed separately to provide four results for each run, the sampling location was adequate for the purposes of this test.

A 6-in sample port was installed at this location for the manual testing and a 1-in sample port was installed for extraction of the continuous emission monitoring samples. Because of the likelihood of poor gas velocity distribution at this location, and the fact that the inlet duct is refractory-lined and should not incorporate numerous ports, the standard flue gas volumetric flow rate at the inlet location was not determined. The outlet standard flue gas volumetric flow rate corrected using the inlet and outlet oxygen concentration was used to calculate flow rates and emission for these locations. This calculation eliminated the contribution from dilution by the shaft cooling air.

5.1.2 Outlet of the Control System

The sampling location at the outlet of the control system (stack) is shown in Figures 5-1 (Point 2) and Figure 5-3. The flue gas at this point has passed through the venturi scrubber and dilution air from the shaft cooling air system has mixed with the flue gas upstream of the sampling location. The flue gas temperature at this point is typically about 100°F (38°C). The sampling point is located in vertical, circular ducting which has two ports at 90° apart. Prior to testing a velocity traverse was conducted to determine the flue gas flow rate. Testing was then conducted at a single point of average velocity in the outlet stack. The single point of average velocity was then used to determine the flue gas flow rate for that test run.

5.2 SAMPLING PROCEDURES

5.2.1 Total Metals

Sampling for total metals at the inlet and outlet (stack) locations followed the

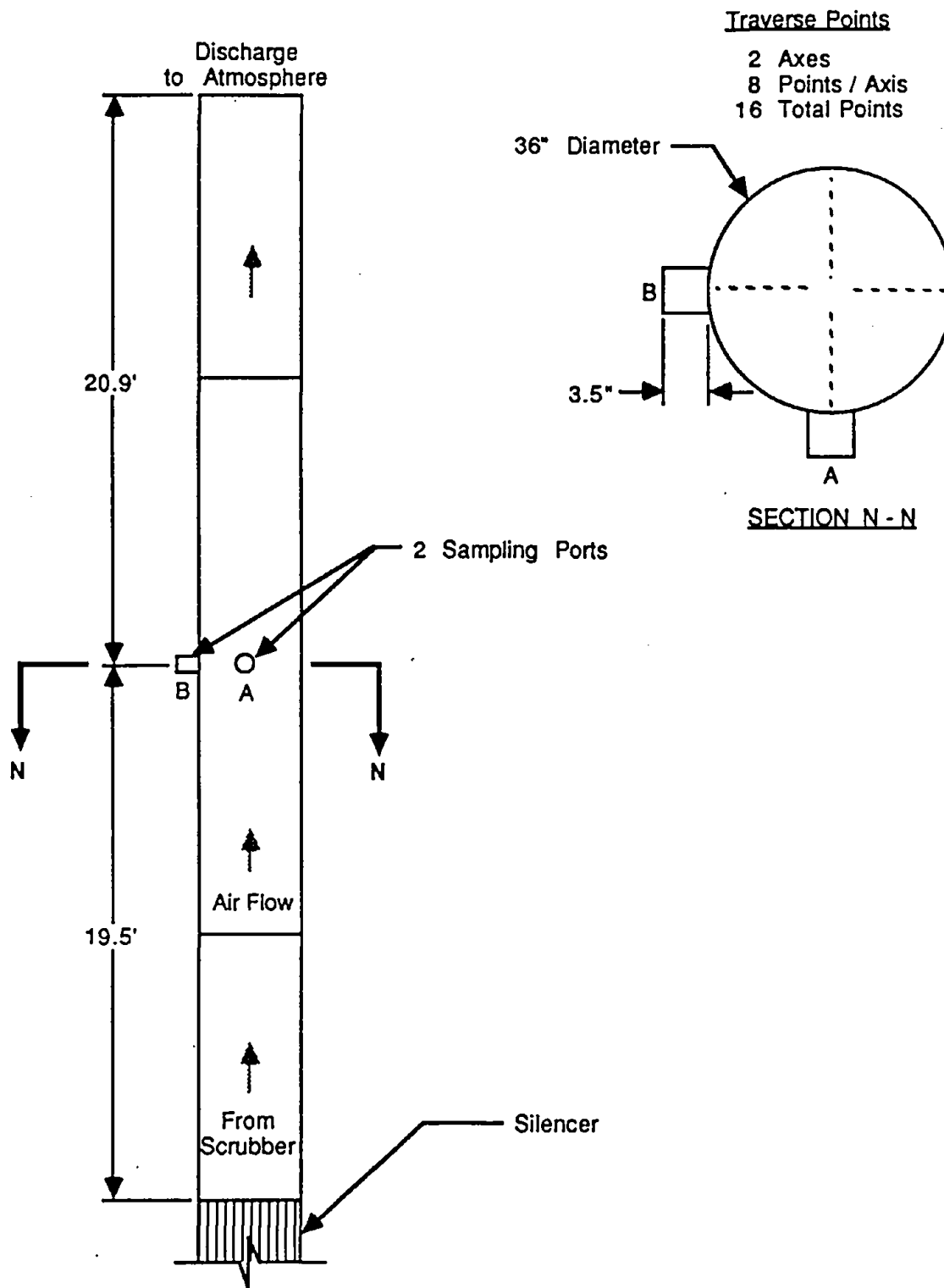


Figure 5-3. Duct dimensions with sampling port and point locations.

procedures in the draft EPA method, "Methodology for the Determination of Trace Metals Emissions in Exhaust Gases from Stationary Source Combustion Processes." A diagram of the multiple metals sampling train used in this test program is shown in Figure 5-4 and a copy of the draft method is reproduced in Appendix B found in Volume IV: Site 6 Draft Test Report, Appendices. The sampling train is similar to the EPA Method 5 train (40 CFR Part 60) with the following exceptions:

- A glass or quartz nozzle and probe liner are used;
- a Teflon filter support is used;
- a low metals background quartz fiber filter is used;
- 5% nitric acid/10% hydrogen peroxide solution replaced water in the impingers;
- the glassware is cleaned according to the procedure in Table 5-1; and
- the sample is recovered as shown in Table 5-2 and Figure 5-5.

For the inlet sampling system, the nozzle and probe liner were quartz glass and the filter holder was borosilicate glass. For the outlet sampling system, the nozzle, probe liner, and filter holder were borosilicate glass. In both cases, Teflon frits were used to support the filters. The probe and filter holder were heated to $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$ to prevent moisture condensation. High purity quartz fiber filters without organic binder and with a 99.95% collection efficiency for 0.3 micron dioctyl phthalate (DOP) smoke particles were used.

For the normal combustion testing, the samples were collected over a 2-hr period at the inlet sampling site and over 2-, 3- or 4-hour period at the outlet location with no special effort to control incinerator operating conditions. For the improved combustion testing, the samples were collected over a 2-hour period at both the inlet and outlet location, and the incinerator process conditions were modified to reduce the CO and THC emissions. For the inlet sampling, the high moisture content at this location required the use of a extra large (2-L) first impinger to allow operation of the train over the 2-hr period. Sampling for total metals were conducted simultaneously with the nickel

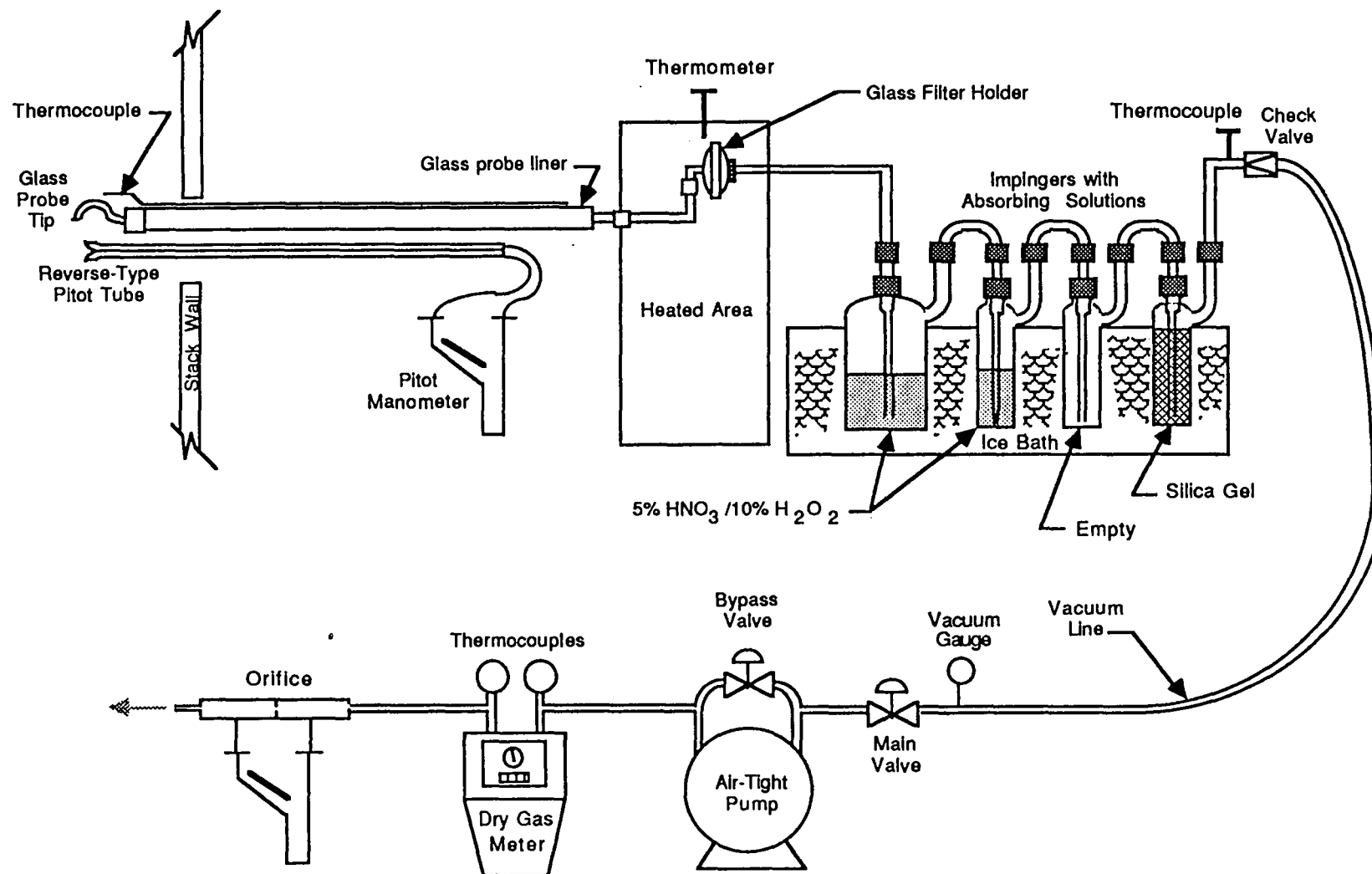


Figure 5-4. Schematic of multiple metals sampling train.

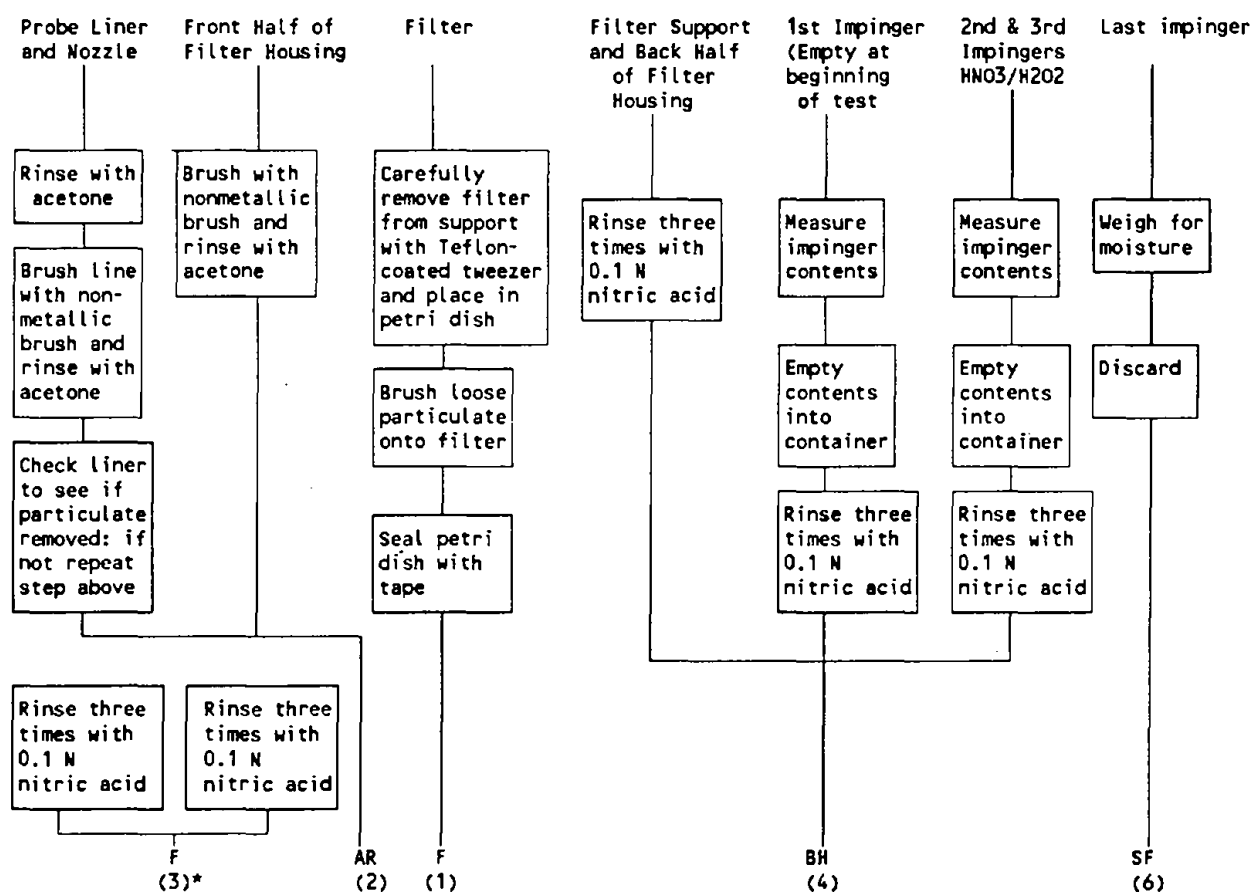
TABLE 5-1. TOTAL METALS GLASSWARE CLEANING PROCEDURES

NOTE: Use disposable gloves and adequate ventilation.

1. Soak all glassware in hot, soapy water (Alconox).
2. Rinse with tap water, three times.
3. Rinse with deionized water, three times.
4. Soak in 10% HNO₃ for 10 hours.
5. Rinse with deionized water, three times.
6. Cap glassware with Teflon tape.
7. Leave cleaned glassware capped until field assembly.

TABLE 5-2. SAMPLE RECOVERY COMPONENTS FOR TOTAL METALS TRAIN

Component	Code	Item
1	AR	Acetone rinses of probe liner, nozzle and front half of filter housing
2	PR-HNO ₃	0.1 N nitric acid rinses of probe liner, nozzle, and front half of filter housing
3	F	Filter
4	BH	0.1 N nitric acid rinses of back half of filter housing, HNO ₃ /H ₂ O ₂ impinger contents, and 0.1 N nitric acid rinses of impingers 1, 2, 3, and connecting glassware



* Number in parentheses indicates container number.

Figure 5-5. Sample recovery procedures for multiple metals train.

speciation testing. Four sampling trains (quadruplicate trains) were used to collect the required number of sampling for the multiple laboratory analyses. Only one sample was intended for multiple metals analysis, two for nickel speciation, and the last was operated in the event of sample system failure during testing.

Samples were analyzed by inductively-coupled argon plasma spectroscopy and atomic absorption spectroscopy for total Cr, Ni, As, Pb, Cd, and Be. Samples were handled and shipped according to the draft method.

5.2.2 Nickel/Nickel Subsulfide

Sampling for nickel/nickel subsulfide at the inlet and outlet locations followed the draft EPA method, "Methodology for the Determination of Nickel Compound Emissions from Stationary Sources." A diagram of the nickel sampling train is shown in Figure 5-6 and the method description is presented in Appendix B found in Volume IV: Site 6 Draft Test Report, Appendices. The sampling train is identical to the EPA Method 5 train (40 CFR Part 60) with the following exceptions:

- A glass or quartz nozzle and probe liner are used;
- a low metals background quartz fiber filter is used;
- the glassware is cleaned according to the procedure in Table 5-3; and
- the sample is recovered as shown in Table 5-4 and Figure 5-7.

For the inlet sampling system, the nozzle and probe liner were quartz glass and the filter holder was borosilicate glass. For the outlet sampling system, the nozzle, probe liner, and filter holder were borosilicate glass. Although not required, a Teflon frit was used to support the filters. The probe and filter holder were heated to $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$ to prevent moisture condensation. High purity quartz fiber filters without organic binder and with a 99.95% collection efficiency for 0.3 micron dioctyl phthalate (DOP) smoke particles were used.

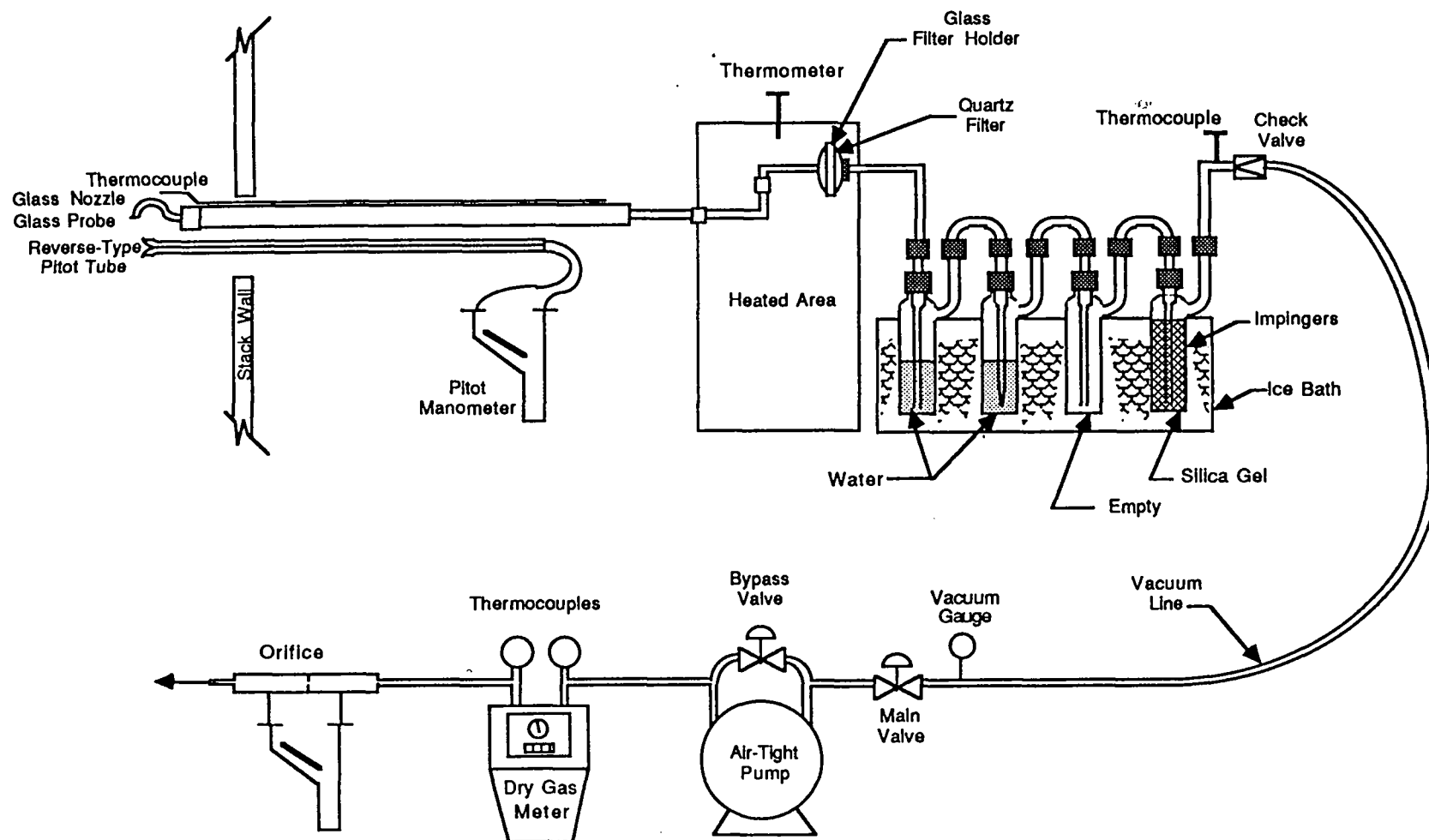


Figure 5-6. Schematic of nickel/nickel subsulfide sampling train.

TABLE 5-3. NICKEL/NICKEL SUBSULFIDE GLASSWARE CLEANING PROCEDURES

NOTE: Use disposable gloves and adequate ventilation.

1. Soak all glassware in hot, soapy water (Alconox).
2. Rinse with tap water, three times.
3. Rinse with deionized water, three times.
4. Soak in 10% HNO₃ for 10 hours.
5. Rinse with deionized water, three times.
6. Cap glassware with Teflon tape.
7. Leave cleaned glassware capped until field assembly.

TABLE 5-4. SAMPLE RECOVERY COMPONENTS FOR THE NICKEL/NICKEL SUBSULFIDE TRAIN

Component	Code	Item
1	AR	Acetone rinses of probe liner, nozzle and front half of filter housing
2	F	Filter*

* The samples sent to BYU were immediately placed on dry ice.
The samples sent to RTI were placed in a desiccator and stored under a dry nitrogen atmosphere.

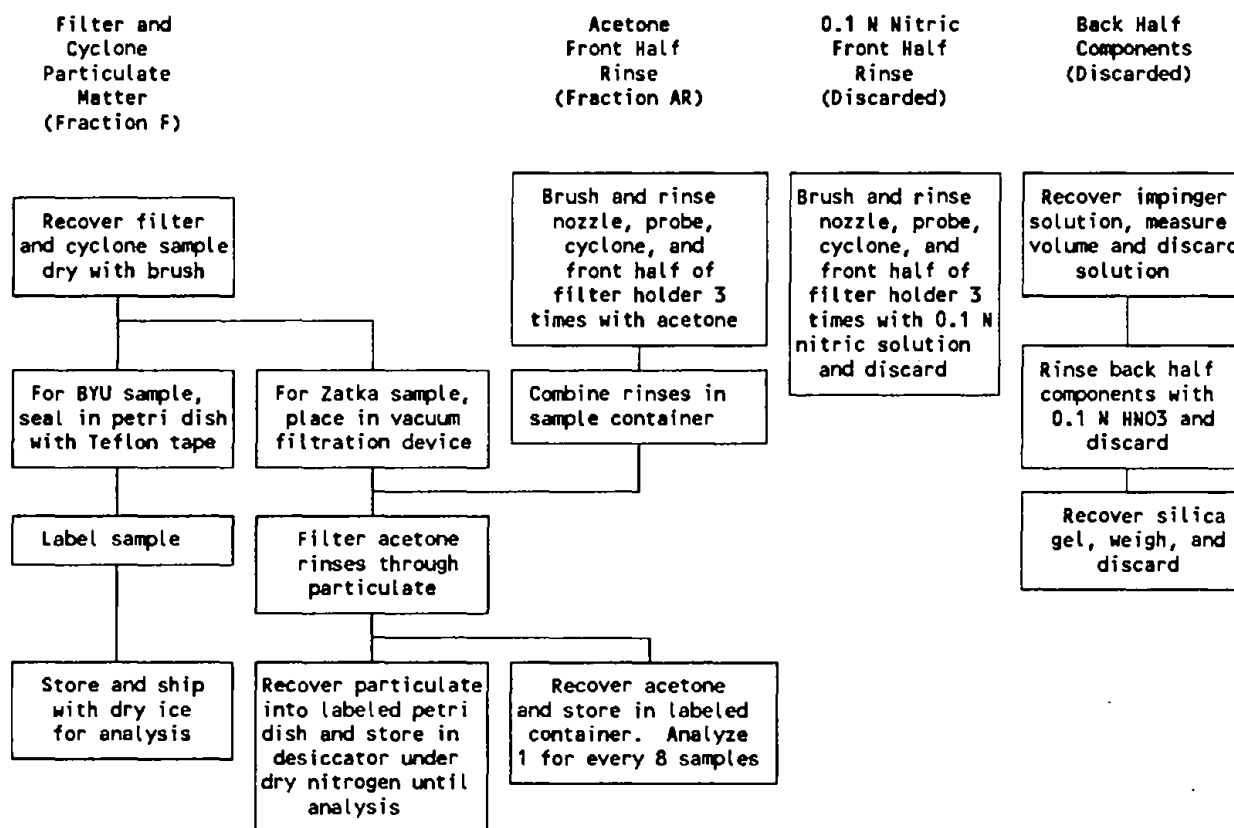


Figure 5-7. Schematic of sample recovery procedures for nickel train.

The nickel speciation sampling trains were operated simultaneously with the multiple metal train under both operating conditions.

A representative filter from each operational condition at the outlet sampling location and bulk samples for the inlet location were sent for analyses by XANES and EXAFS by Brigham Young University (BYU). These filter samples were placed on dry ice immediately after recovery. The remaining inlet and outlet filters were analyzed by Dr. Vladimir Zarka. The inlet location samples were recovered and stored dry because of the large volume of sample. For the outlet samples, the acetone probe rinse was vacuum filtered through the filter. The acetone filtrate was archived with the exception of one sample for the 12 daily samples. That filtrate sample was analyzed for total nickel to reaffirm that the nickel compounds are not soluble in acetone. The filters to be analyzed by Dr. Zarka were stored in a desiccator under a dry nitrogen atmosphere and sent to Dr. Zarka at the conclusion of each run condition; past experience has shown that oxidation of nickel compounds can occur over a several week period.

5.2.3 Chromium and Hexavalent Chromium (Recirculating Train)

Sampling for hexavalent and total chromium (Cr^{+6}/Cr) using the recirculating reagent sampling train was conducted at the outlet location. Sampling and analysis followed the procedures in the draft EPA method, "Determination of Hexavalent Chromium from Stationary Sources." A diagram of the recirculating reagent sampling train is shown in Figure 5-8; the draft method is reproduced in Appendix B of Volume IV: Site 6 Draft Test Report, Appendices. This procedure is based on EPA Method 5 with the following modifications:

- The reagents are continuously recirculated from the first impinger back to the nozzle to provide a flow of reagents through the probe, and thus preventing hexavalent chromium in the probe drying out and possibly converting to another valence state;
- 0.1 N KOH replaces water in the impingers;

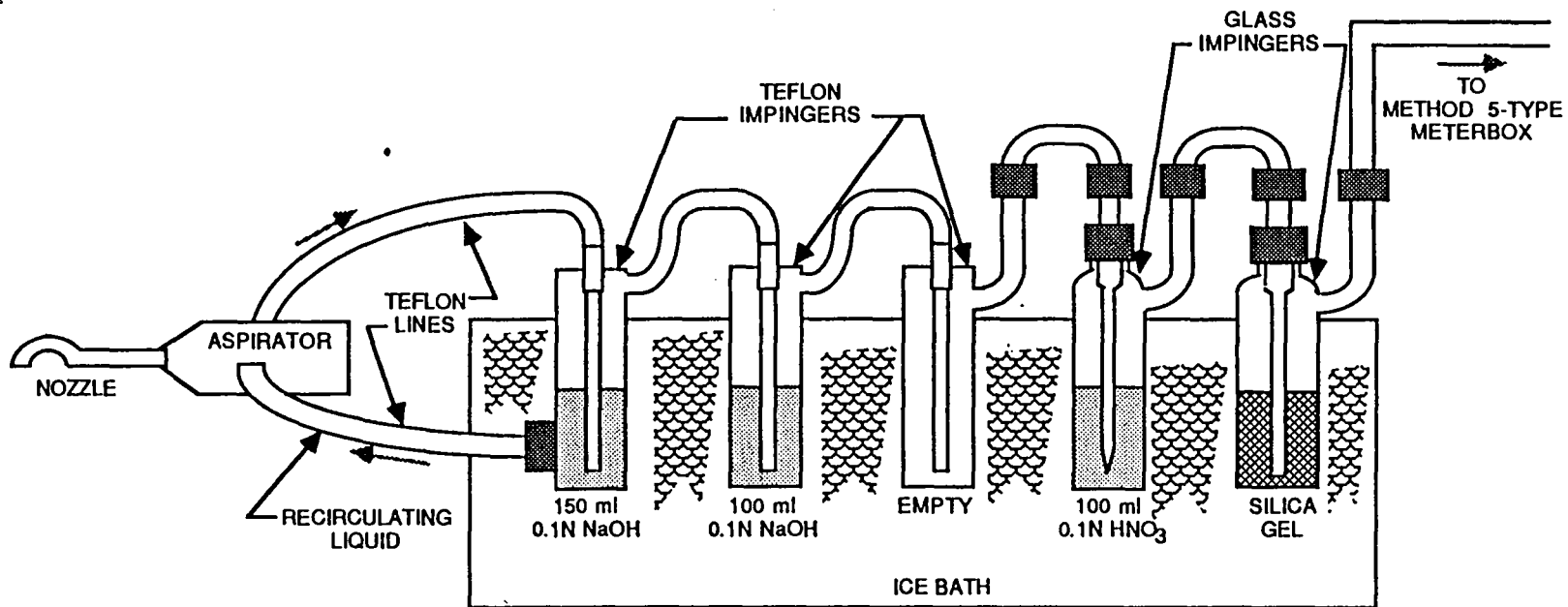


Figure 5-8. Schematic of recirculating reagent sampling train for hexavalent chromium.

- the entire surface exposed to sample is constructed of Teflon and/or glass;
- the Teflon and/or glass components are cleaned according to the procedure in Table 5-5;
- the sample is recovered as shown in Table 5-6 and Figure 5-9; and
- the train does not have a filter section.

The sampling system was expected to be operated isokinetically, however, the pressure drop across the aspirator made it difficult to maintain isokinetic sampling. Two-, three-, and four-hour quad train runs were conducted at the outlet location under each of the two test conditions.

The impinger solutions were taken from a common solution that was spiked with radioactively labelled chromium to serve as a recovery standard for conversion of hexavalent chromium in the samples. To verify the spike concentration, and as a check for contamination, control samples of the solution were set aside and analyzed with the field samples. Immediately after a sample was recovered from the sampling train, the combined impinger solutions were pressure filtered through a 0.45 micron Teflon filter. The filtrate was stored and shipped cold for next day analysis for hexavalent chromium. Problems were encountered with these analyses and the next day analysis could not be conducted.

The Teflon filters and nitric acid rinse samples analyzed for total chromium did not require any special handling procedures. Filters and nitric rinse portions were shipped to the respective laboratories for analysis.

5.2.4 Chromium and Hexavalent Chromium (Filter Train)

The Cr^{+6}/Cr train sampling using the filter was conducted at the inlet for all runs and at the outlet location on two runs (Runs 1 and 2). Sampling followed the procedures of the draft EPA method, "Determination of Hexavalent Chromium from Stationary Sources," dated December 13, 1984. A diagram of the sampling train is shown in Figure 5-10 and the method description is not presented in this report since the

TABLE 5-5. Cr⁺⁶/Cr TEFLON/GLASS COMPONENTS CLEANING PROCEDURES

NOTE: Use disposable gloves and adequate ventilation.

1. Soak all components in hot, soapy water (Alconox).
2. Rinse with tap water, three times.
3. Rinse with deionized water, three times.
4. Soak in 10% HNO₃ for 10 hours.
5. Rinse with deionized water, three times.
6. Cap Teflon/glassware with Teflon tape.
7. Leave cleaned Teflon/glassware capped until field assembly.

TABLE 5-6. SAMPLE RECOVERY COMPONENTS FOR THE Cr⁺⁶/Cr RECIRCULATING TRAIN

Component	Code	Item
1	IMP	0.1 N potassium hydroxide impinger catches and DI water rinses of Teflon impingers (1, 2, 3, and 4) and connecting tubing
2	NR	0.1 N nitric acid rinses of probe liner, nozzle, and connecting tubing
3	F	Filter*

*The impinger catch and KOH rinses will be pressure filtered on-site through a 0.45 micron Teflon filter immediately after sample recovery. The filter to be analyzed by BYU will be placed on dry ice. The impinger catch and rinses will shipped to RREL, General Engineering Laboratories, and Entropy for next day analysis by the respective laboratories.

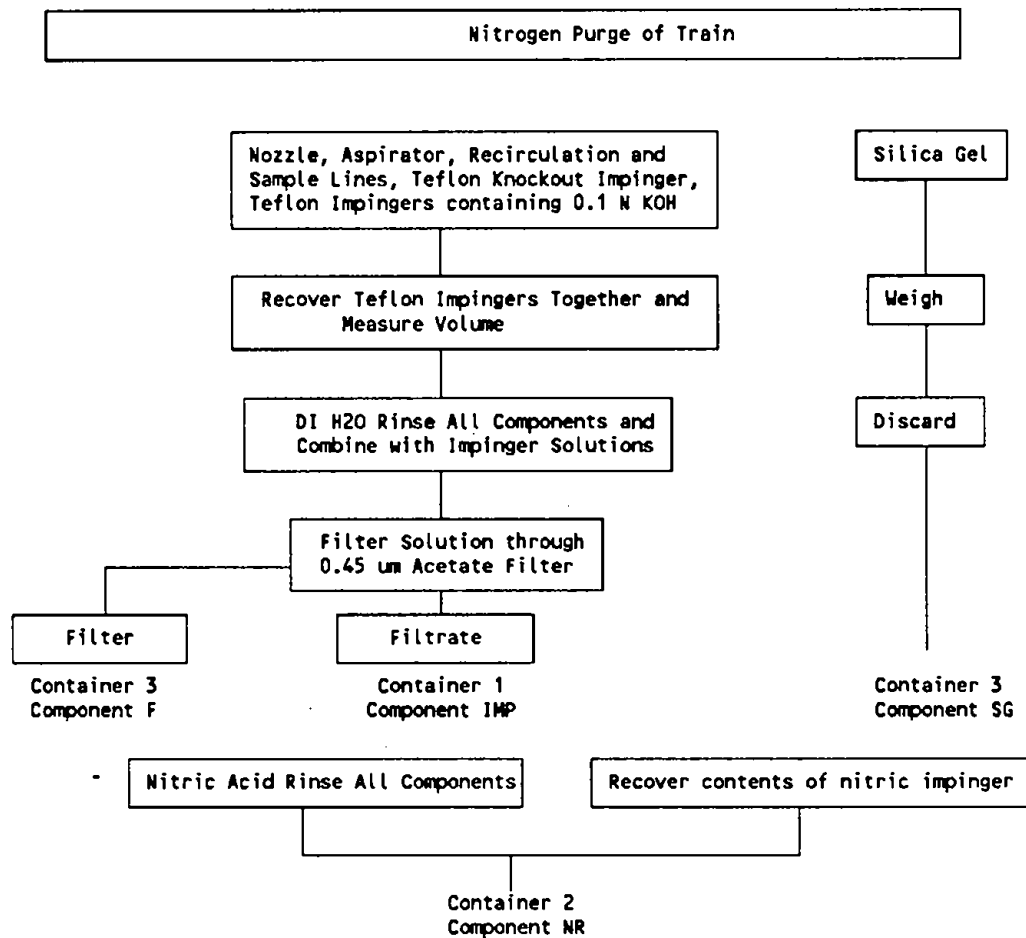


Figure 5-9. Sample recovery scheme for hexavalent chromium recirculating impinger train.

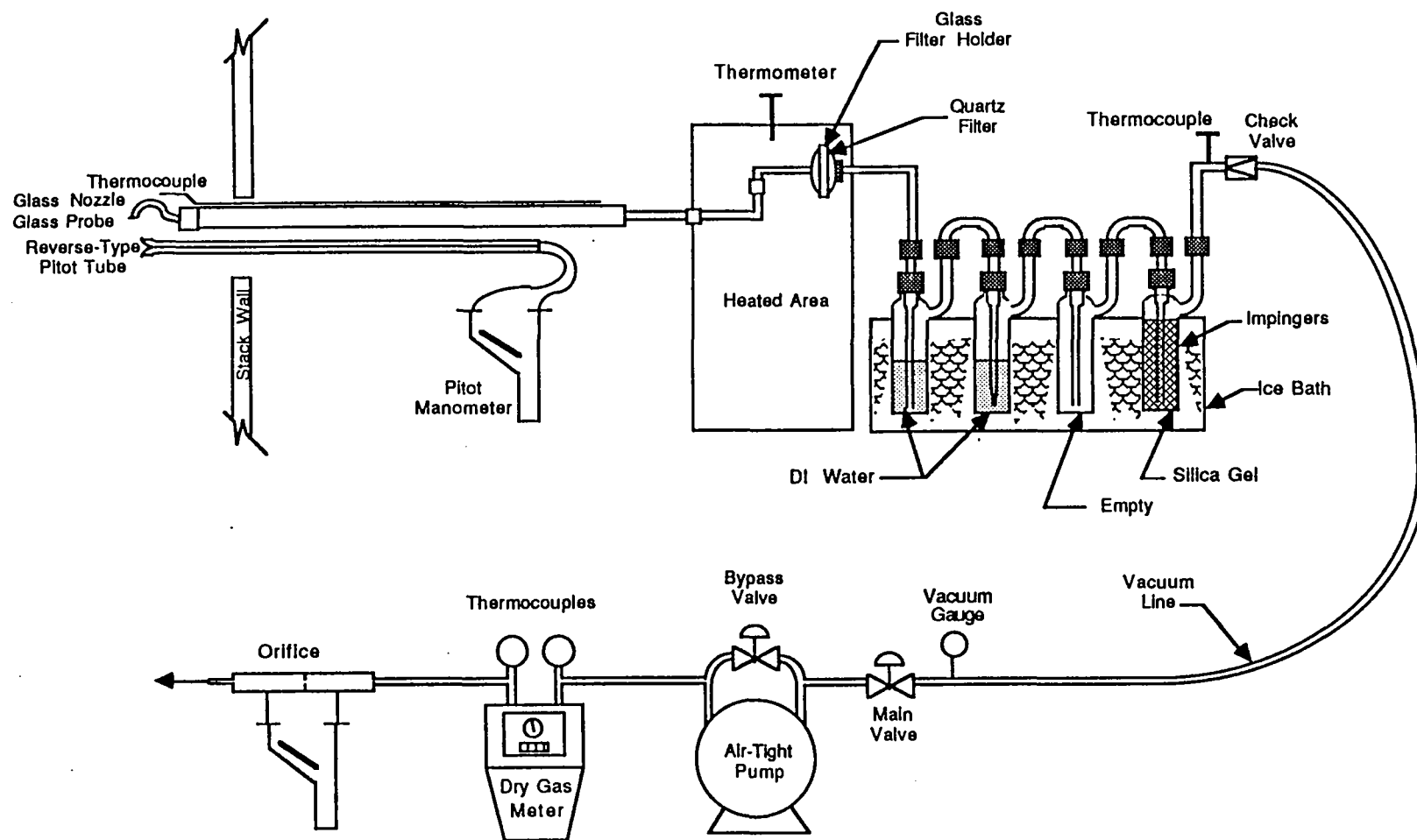


Figure 5-10. Chromium filter sampling train.

data was not released by EPA. This procedure involves the use of the EPA Method 5 sampling train with the following modifications:

- A glass nozzle and probe liner are used;
- the glassware is cleaned according to the procedure in Table 5-7; and
- the sample is recovered as shown in Table 5-8 and Figure 5-11.

The sampling train nozzle, probe liner, and filter holder were made of borosilicate glass. Both the probe and filter holder were heated to $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$ to prevent moisture condensation. High purity quartz fiber filters without organic binder and with a 99.95% collection efficiency for 0.3 micron diocyl phthalate (DOP) smoke particles were used.

Two quad-train runs (Runs 1 and 2) were conducted at the outlet location under normal incinerator operating conditions, each for 2 hr. Six of the eight filters were spiked with isotopically labelled chromium, prior to the tests. To verify the spike concentration and as a check for contamination, two unused spiked filters were set aside as control samples. Upon completion of the sampling, the nozzle, probe liner, cyclone (if applicable) and filter front half were rinsed three times with acetone and the rinses placed in a sample container. The nozzle, probe liner, cyclone (if applicable), and filter front half were then rinsed three times with 0.1 N HNO_3 and the rinses placed in a separate container.

All inlet location hexavalent chromium sampling was conducted using the filter train. The sample train preparation and sample recovery procedures were the same as described above for the outlet hexavalent chromium filter train samples.

The spiked filter samples were analyzed by EPA's Environmental Monitoring Systems Laboratory (EMSL) for hexavalent and total chromium. Immediately after recovery they were placed on dry ice and shipped the same day to EMSL for next day analysis. The unspiked filters and accompanying acetone and nitric rinses were analyzed by EPA for total chromium only and did not require special handling procedures. The used and unused isotopically spiked filters were analyzed for native and isotopically labelled hexavalent chromium. The results of the filter train testing have not been

TABLE 5-7. Cr/Cr⁶ GLASSWARE CLEANING PROCEDURES

NOTE: Use disposable gloves and adequate ventilation.

1. Soak all glassware in hot, soapy water (Alconox).
2. Rinse with tap water, three times.
3. Rinse with deionized water, three times.
4. Soak in 10% HNO₃ for 10 hours.
5. Rinse with deionized water, three times.
6. Cap glassware with Teflon tape.
7. Leave cleaned glassware remained capped until field assembly.

TABLE 5-8. SAMPLE RECOVERY COMPONENTS FOR Cr/Cr⁶ FILTER TRAIN

Component	Code	Item
1	AR	Acetone rinse of probe liner, nozzle, and front half of filter housing
2	PR	0.1 N nitric acid rinses of probe liner, nozzle, and front half of filter housing
3	F	Filter*

* Spiked filters placed on dry ice immediately after recovery and sent that day to EMSL for next day analysis.

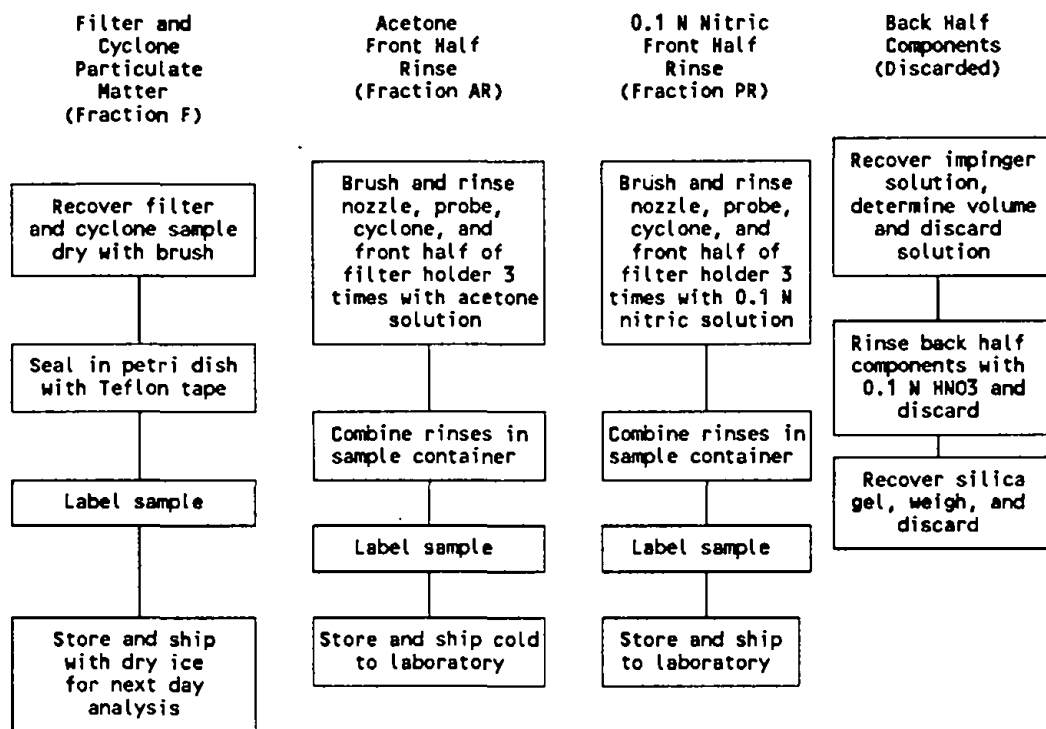


Figure 5-11. Sample recovery scheme for hexavalent chromium filter train.

released by EMSL and are not presented in this report.

5.2.5 Continuous Emissions Monitoring Systems

Continuous emission monitoring systems (CEMSs) were used at the control device inlet and outlet to monitor CO, CO₂, O₂, NO_x, and SO₂; CO and total hydrocarbons (THC as propane) were monitored at the stack outlet location. The primary intent of the continuous monitoring effort was to: (1) determine concentrations of these, and (2) provide a real-time indication of combustion conditions. The continuous monitoring systems were calibrated daily, but no attempt was made to certify the monitors using EPA test methods. The sampling and analytical systems used to determine CO, CO₂, O₂, NO_x, SO₂, and THC are discussed in the following sections.

5.2.5.1 Sample and Data Acquisition - Sample gas was drawn through a sample gas conditioner consisting of an ice bath and knockout trap to remove moisture and thus provide a dry gas stream for analysis. Sample gas from the gas conditioner was pumped through a manifold at a flow rate which exceeded the total sample requirements of the various gas analyzers. The sample manifold was used to provide slip stream sample flows to each analyzer. A separate gas conditioning system and sample line was used for the inlet and outlet sampling locations.

To maximize representativeness of the CEM measurements, all gases for calibration were introduced at the inlet of the sample line. The instruments were calibrated prior to each test run. At the end of each test run, a post-test calibration was performed. If instrument drift exceeded 2% of the span value for any measurement, the data was adjusted linearly to account for the drift. Data from the analyzers was collected and recorded using a microprocessor data acquisition/reduction system. A hard copy of the reduced data (engineering units) was printed at the end of each sample run and the raw data were stored on the computer hard drive and on floppy disks.

5.2.5.2 Carbon Monoxide/Carbon Dioxide Analysis - A TECO Model 48 analyzer was used to measure CO concentrations in the flue gas. The TECO Model 48 is a gas filter correlation (GFC) analyzer. The instrument measures the concentration of CO by infrared adsorption at a characteristic wavelength. A Fuji 3300 analyzer was used to determine CO₂ concentration. The Fuji 3300 is a non-dispersive infrared (NDIR) analyzer.

5.2.5.3 Oxygen Analysis - A Teledyne Model 320P-4 O₂ analyzer was used to continuously measure flue gas oxygen concentrations. The Teledyne analyzer uses an electro-chemical cell to produce a linearized voltage signal that is proportional to the ratio of oxygen concentration of a reference gas (ambient air) and the oxygen concentration of the sample.

5.2.5.4 Nitrogen Oxides (NO_x) Analysis - A TECO Model 10 analyzer was used for NO_x measurement. This instrument determines NO_x concentrations by converting all nitrogen oxides present in the sample to nitric oxides and then reacting the nitric oxide with ozone. The reaction produces a chemiluminescence proportional to the NO_x concentration in the sample. The chemiluminescence is measured using a high-sensitivity photomultiplier. Also, during the time between the manual sampling runs, the ratio of NO to NO₂ was determined. This ratio is of interest because NO₂ can be effectively removed by the venturi scrubber.

5.2.5.5 Sulfur Dioxide (SO₂) Analysis - Sulfur dioxide in the flue gas was measured using a Maihak UNOR 6N analyzer. This instrument measures SO₂ on the basis of infrared adsorption.

5.2.5.6 Total Hydrocarbon Analysis - A Beckman 400A analyzer was used to measure total hydrocarbons (THC) as propane in the flue gas. This instrument is a continuous flame ionization analyzer (FIA). The detector is a burner where a regulated flow of sample gas passes through a flame sustained by regulated amounts of air and hydrogen.

Hydrocarbons passing through the flame undergo a complex ionization that produces electrons that are detected by polarized electrodes. The THC analyzer was calibrated using propane standards, and the THC concentrations were reported in parts per million by volume (ppmv) as propane.

5.2.6 EPA Methods 1, 2, 3, and 4

The methods used to determine the flue gas moisture content, molecular weight, and volumetric flow rate are described in the following sections.

5.2.6.1 Volumetric Gas Flow Rate Determination - The volumetric gas flow rate at the outlet location was determined during this program using procedures described in EPA Method 2. Based on this method, the volumetric gas flow rate is determined by measuring the cross-sectional area of the stack and the average velocity of the flue gas. The average flue gas velocity is calculated from the average pitot tube pressure (ΔP), the average flue gas temperature, the wet molecular weight, and the absolute static pressure. Temperature and ΔP profile data was obtained by traversing the outlet stack prior to the each test run. The number of sampling points required to measure the average gas velocity was determined using the procedures in EPA Method 1. The sampling points and their distances from the duct wall is a function of the proximity of the sampling location to the nearest upstream and downstream flow disturbance.

Temperature and pressure profile data was measured at each of the sampling points using an S-type pitot tube. A calibrated aneroid barometer was used to obtain barometric pressure readings each day. The static gas pressures at the inlet and outlet locations were measured by disconnecting one side of the S-type pitot and then rotating the pitot so that it was perpendicular to the gas flow.

The standard flue gas flow rate at the inlet location was not measured; but was calculated by correcting the measured standard flue gas flow rate with difference between the inlet and outlet oxygen concentration. This calculation corrects for the dilution air from the shaft cooling that is entering the duct between the inlet and outlet

locations. Isokinetic sampling was achieved by measuring the pitot tube pressure (ΔP) at a single point at regular intervals during sampling. The isokinetic calculations were performed using these measured values.

5.2.6.2 Flue Gas Molecular Weight Determination - The integrated sampling technique described in EPA Method 3 was used to obtain composite flue gas samples at the inlet and outlet locations for fixed gas (O_2 , CO_2) analysis. A small diaphragm pump and a stainless steel probe were used to extract a single-point flue gas sample which was collected in a Tedlar bag. Moisture was removed from the gas sample by a water-cooled condenser so that the fixed gas analysis is on a dry basis.

The composition of the gas sample was determined using an Orsat analyzer only when there was problems with the CEMS. When using the Orsat, if more than six passes were required to obtain a constant ($\leq 0.3\%$ difference, absolute) reading for either O_2 or CO_2 , the appropriate absorbing solution was replaced. The SO_2 concentration was well below the level at which correction of the CO_2 concentration is required (5,000 ppm).

5.2.6.3 Flue Gas Moisture Determination - The moisture content of the flue gas was determined using the methodology described in EPA Method 4. Based on this method, a known volume of particulate-free gas was pulled through a chilled impinger train. The quantity of condensed water was determined gravimetrically and then related to the volume of gas sampled to determine the moisture content.

During this project, the moisture content of the flue gas was determined simultaneously during the operation of the manual sampling trains. The volume of solution in the impingers used with these trains was determined before and after sampling. The volume increase in water was then related to the volume of gas sampled to calculate the moisture content.

5.2.7 Process Samples

Samples of sludge feed, bottom ash, and scrubber inlet (influent) and outlet (effluent) water were collected during the flue gas sampling. These process samples were composites of grab samples collected at regular intervals and combined after the run was completed. All process samples were stored in 500-ml polyethylene sample containers prepared according to EPA Protocol C.

The sludge feed sampling begin approximately 30 min prior to the start of the flue gas sampling to account for the residence time of the sludge in the furnace. The sludge feed samples for metals analysis were collected from the feed conveyor at 30-min intervals. The volume of each grab sample was approximately 250-ml. The grab samples were combined and homogenized in a plastic bucket using a mortar mixer for at least 10 min. From the homogenized mixture, two 500-ml portions were taken and saved for metals analysis, proximate and ultimate analyses, and analyses of moisture content and heating value.

Ash samples were collected from the bottom hearth of the incinerator. A 1-L grab sample was taken once during each test run using a scoop. The total mass flow of the ash discharge was estimated based on the sludge feed rate and the sludge ash content.

Scrubber inlet and outlet water samples consisted of the composite of two equal grab samples collected during each run. These samples were thoroughly mixed before aliquots were taken for analysis.

5.3 PROCESS DATA

Incinerator and control system operating parameters were monitored during all manual test runs to characterize the system operations. The parameters monitored are presented in Table 5-9.

TABLE 5-9. PROCESS MONITORING DATA

Parameter	Frequency of Readings	Source of Readings
<u>Incinerator Operating Data</u>		
Hearth Temperatures	30 minutes	Plant operating log
Furnace Discharge Temp	30 minutes	Plant operating log
Incinerator Outlet O ₂	Continuous	Entropy CEMSs
Auxiliary fuel usage	As used	Plant operating log
<u>Sludge Feed Characteristics</u>		
Moisture (wt %)	Once per run	Entropy analysis
Volatiles (wt %)	Once per run	Entropy analysis
Heating Value	Once per run	Entropy analysis
<u>Scrubber System Operating Data</u>		
Delta P (in. H ₂ O)	30 minutes	Plant operating log
Scrubber Inlet Temp (°F)	30 minutes	Plant operating log
Scrubber Outlet Temp (°F)	15 minutes	Plant operating log

6.0 ANALYTICAL PROCEDURES

The laboratory activities for this program involved (1) analytical procedures designed to speciate chromium and nickel compounds based on their valency state; (2) analysis of selected samples for arsenic, beryllium, cadmium, chromium, lead, and nickel. The sample matrices included flue gas samples, sludge samples, bottom ash samples, and scrubber water samples. Sludge samples were also subjected to moisture, proximate and ultimate analyses, and heating value determination. A summary of the analytical methods employed is provided in Table 6-1. Each of these methods are described briefly in the sections below and detailed procedures are included in Volume IV, Appendices.

6.1 CHROMIUM SPECIATION AND ANALYSES

Several analytical procedures were employed to speciate chromium compounds in the samples to determine the ratio of hexavalent chromium (Cr^{+6}) to total chromium (Cr). Since the hexavalent chromium filter train analytical results were not released for publication in this report and were not conducted under the RREL contracts, the analytical techniques will not be discussed. Flow diagrams for application of these procedures are provided in Figure 6-1 for impinger train samples. Samples from the impinger train were analyzed using ion chromatography with a Cr^{+6} -specific post column reaction (IC/PCR) for Cr^{+6} performed by Entropy and inductively-coupled argon plasma emission spectroscopy (ICAP) for total Cr performed by Research Triangle Institute (RTI). Entropy also performed gamma emission measurements of labeled hexavalent chromium ($^{51}\text{Cr}^{+6}$) spiked into samples to monitor conversion of chromium species that may occur during sampling, sample handling, and sample preparation.

TABLE 6-1. SUMMARY OF SAMPLING AND ANALYTICAL METHODS

Sample Type	Parameter	Analysis Method
Flue Gas	• Total chromium, Cr ^{+6a,b}	IC/PCR, gamma counter ICAP/AAS, ICP/MS, XANES
	• Total nickel, nickel subsulfides ^c	EPA Draft Method, ICAP/AAS, XANES
	• Particulates, metals ^d	Gravimetric, ICAP/AAS
Solid/Liquid	• Feed sludge	----- ^e
	• Scrubber water: inlet	----- ^d
	outlet	----- ^d
	• Bottom ash	----- ^d

^aRecirculating train for hexavalent chromium, with 0.1 N KOH impinger solution.

^bMethod 5-type sampling train for chromium.

^cMethod 5-type sampling train for nickel.

^dMetals analysis included at a minimum chromium, nickel, arsenic, lead, cadmium, beryllium, and mercury.

^eThe sludge samples were analyzed for metals, moisture, proximate and ultimate analysis, and heating value by the methods described in Section 5.0.

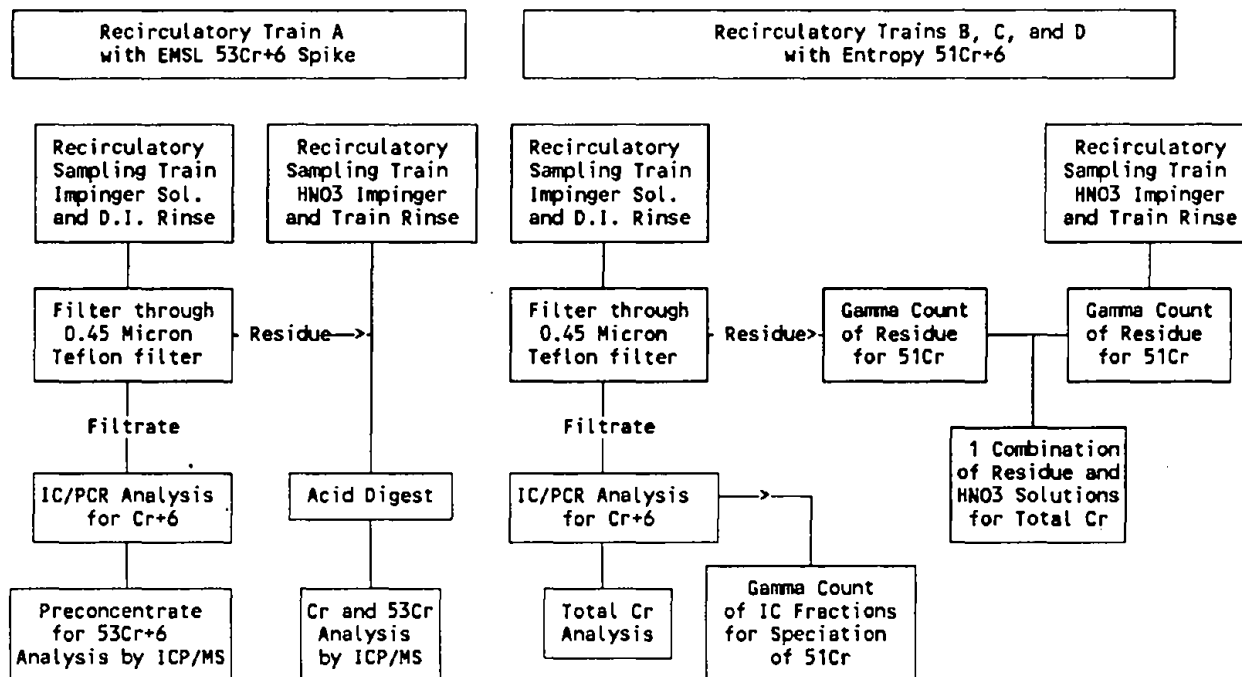


Figure 6-1. Analytical protocol for quadruplicate recirculatory train hexavalent chromium sampling at outlet location.

6.1.1 IC/PCR Analysis for Hexavalent Chromium

The IC/PCR analysis for Cr^{+6} was performed by Entropy on the recirculatory (RC) impinger train samples. Samples consisted of alkaline impinger solutions from the recirculatory impinger train.

Entropy performed on-site filtration of the alkaline impinger samples employing an all-Teflon pressure filtration device and a Teflon membrane filter with a 0.45 micron pore size (see Figure 6-1). The filtrate was analyzed for Cr^{+6} . For samples with the ^{51}Cr spike, the gamma emissions from the filter residue were measured before combining the residue with the HNO_3 solutions for digestion prior to total Cr analysis.

In all cases, the resulting filtrates were analyzed by the IC/PCR method. To determine the ratio of the soluble $^{51}\text{Cr}^{+3}$ and $^{51}\text{Cr}^{+6}$ species in Entropy-spiked samples, 0.5 ml fractions were collected during the IC/PCR analysis, and measured for the gamma emissions for each fraction.

The IC/PCR system was calibrated with a series of three Cr^{+6} standard solutions with concentrations ranging from 1.0-to-100 ng/ml, prepared fresh daily from a working standard. The laboratory verified the concentration of their working standard solution by ICAP analysis or ICP/MS analysis for total Cr. A calibration check sample was analyzed with every ten samples.

6.1.2 ICAP Analysis for Total Chromium

For RC trains, residue samples from the filtration of the alkaline impinger solution were analyzed with the corresponding HNO_3 solutions and rinses for total chromium (see Figure 6-1). Where appropriate, an aliquot of HNO_3 solutions and rinses was first measured for gamma emissions prior to the sample being reduced to near dryness. After being reduced to near dryness, the HNO_3 sample was combined with residue sample for HNO_3/HF digestion.

Sludge samples, bottom ash samples, and scrubber water samples were analyzed

for total Cr during the ICAP analysis for the other target metals, described in Subsections 6.3.2, 6.3.3, and 6.3.4, respectively.

6.1.3 XANES Analysis for Chromium Speciation

XANES spectroscopy can determine the chemical state of an element without the necessity for chemical pretreatment which may alter the chemical state. XANES spectroscopy requires a high intensity X-ray source provided by synchrotron radiation. For this test program, Brigham Young University (BYU) arranged for access to the Brookhaven synchrotron. Eight-hour irradiation times were required to obtain spectra for samples in the 300-to-1000 ug/g concentration range. The irradiation was performed at an electron energy of 3.0 GeV with a current of approximately 50 mA. The X-ray beam is monochromatized with a double crystal silicon spectrometer and a 1-mm (vertical dimension) entrance slit which, with this configuration, produces a resolution of approximately 0.4 eV at the vanadium K edge at 5.465 KeV.

A 4-in² section of each filter sample was placed in the sample chamber at an angle 45 degrees to the X-ray beam, and the sample chamber was purged with helium. The sample spectra was measured by the fluorescence extended X-ray absorbance fine structure technique with a fluorescence detector.

Reference spectra obtained from standards with known ratios of Cr⁺⁶ to total Cr. A separate report prepared by BYU is presented in Volume IV Site 6 Draft Test Report, Appendices.

6.2 NICKEL SPECIATION AND ANALYSIS

Two different procedures were employed to speciate nickel compounds in samples to determine the ratio of nickel subsulfide (Ni₃S₂) to total nickel (Ni). The first procedure, X-ray absorption near-edge structure (XANES) analysis, were performed by BYU. The second procedure, to be performed by Dr. Zatka, employed the Nickel Producers Environmental Research Association (NiPERA) method. The analytical

protocol for the inlet sampling location and outlet location are presented in Figure 6-2 and Figure 6-3, respectively.

6.2.1 XANES Analysis for Nickel Speciation

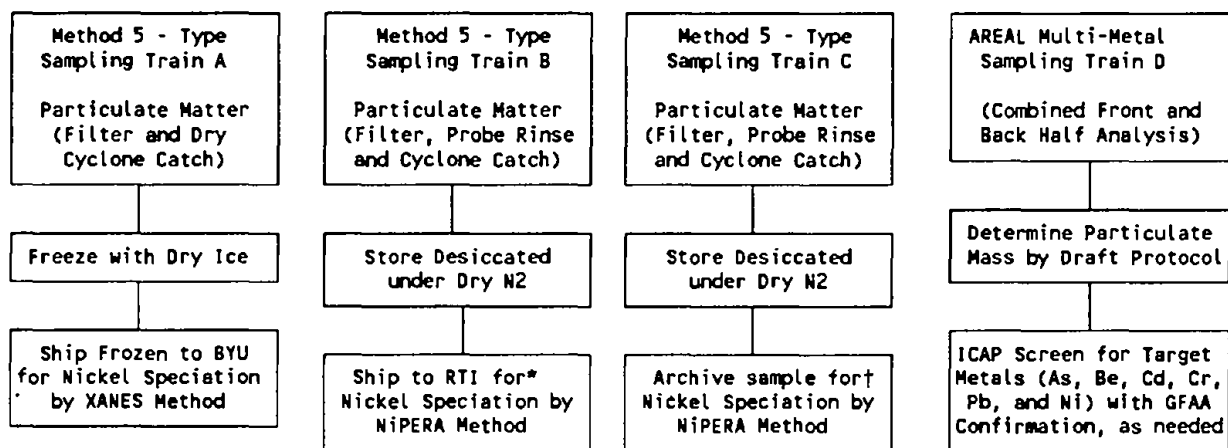
XANES spectroscopy was employed by BYU to determine the ratio of Ni_3S_2 to total Ni. The analytical procedure was identical to the procedure described in Subsection 6.1.3 for chromium speciation, with the exception that reference spectra was determined on standards with known ratios of Ni_3S_2 to total Ni. The detection limit for Ni_3S_2 by XANES is reported to be 100 ug/g for a one hour irradiation.

6.2.2 NiPERA Method for Nickel Speciation

The NiPERA sequential leaching method was employed by Dr. Zatka to determine the ratio of sulfidic nickel species, Ni_3S_2 and nickel sulfide (NiS), to total Ni. The NiPERA method is not capable of speciating between Ni_3S_2 and NiS. The NiPERA method involved two sequential leachings of the solid sample with a series of solutions with increasing oxidation strength. The leaching procedure was performed in an all Teflon vacuum filtration device fitted with a cellulose membrane filter with a 0.2 micron pore size. The water soluble Ni species was leached during the first step and the sulfidic Ni species was leached during the second step.

For this test program, the first and second leach solutions were collected separately. The leached residue was digested prior to Ni analysis following the SW-846, Method 3050 procedure described below in Subsection 6.3.2.

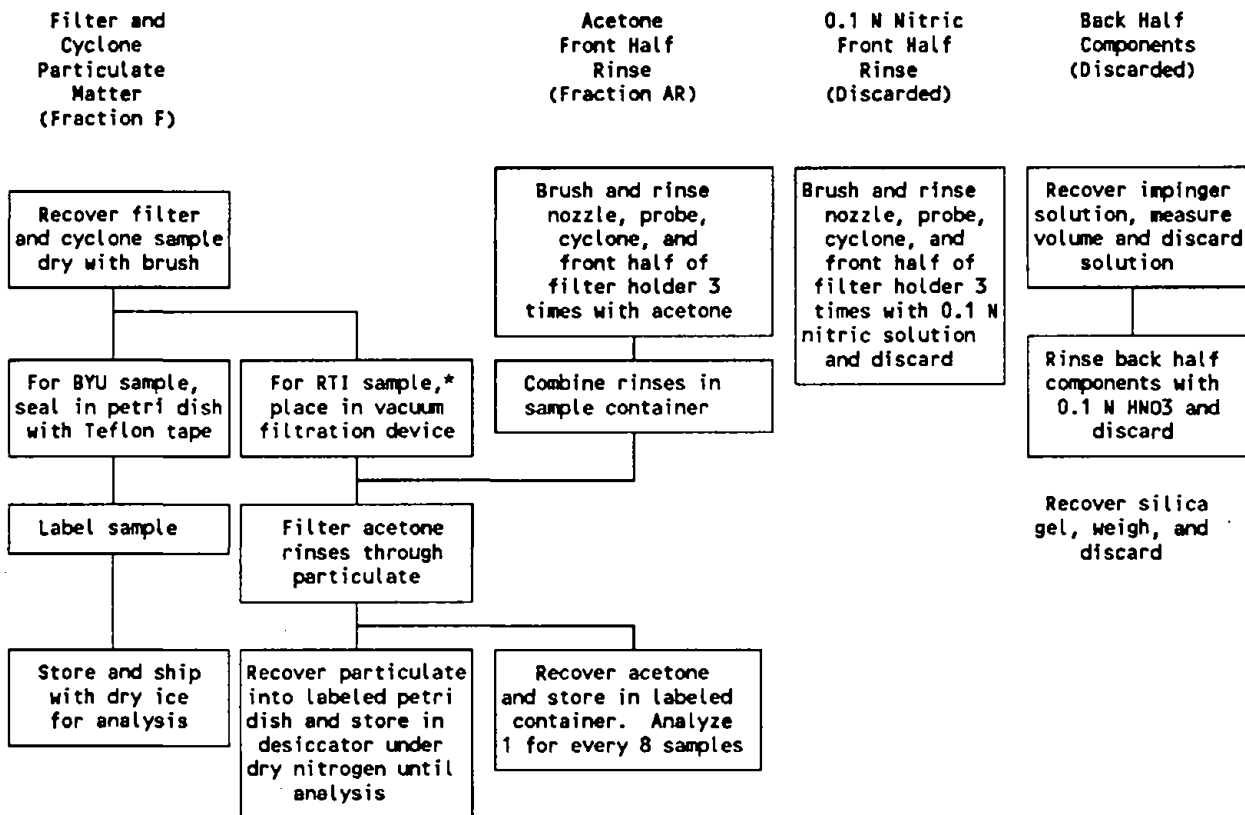
The three Ni subsamples were analyzed for total Ni by atomic absorption (AA) analysis. The AA was calibrated with a series of seven Ni standard solutions ranging in concentration from 0.5-to-20 ug/ml. An interference check sample was analyzed prior to sample analysis, and a calibration check sample was analyzed with every 10 samples. A reagent blank was carried through the procedure and analyzed with the actual samples.



* RTI unable to get reliable results.

† Archived samples were sent to Zarka for sample analyses.

Figure 6-2. Analytical protocol for quadruplicate nickel sampling at the scrubber inlet sampling location.



* RTI unable to get reliable results, archived samples sent to Zarka

Figure 6-3. Analytical protocol for quadruplicate nickel sampling at the scrubber outlet sampling location.

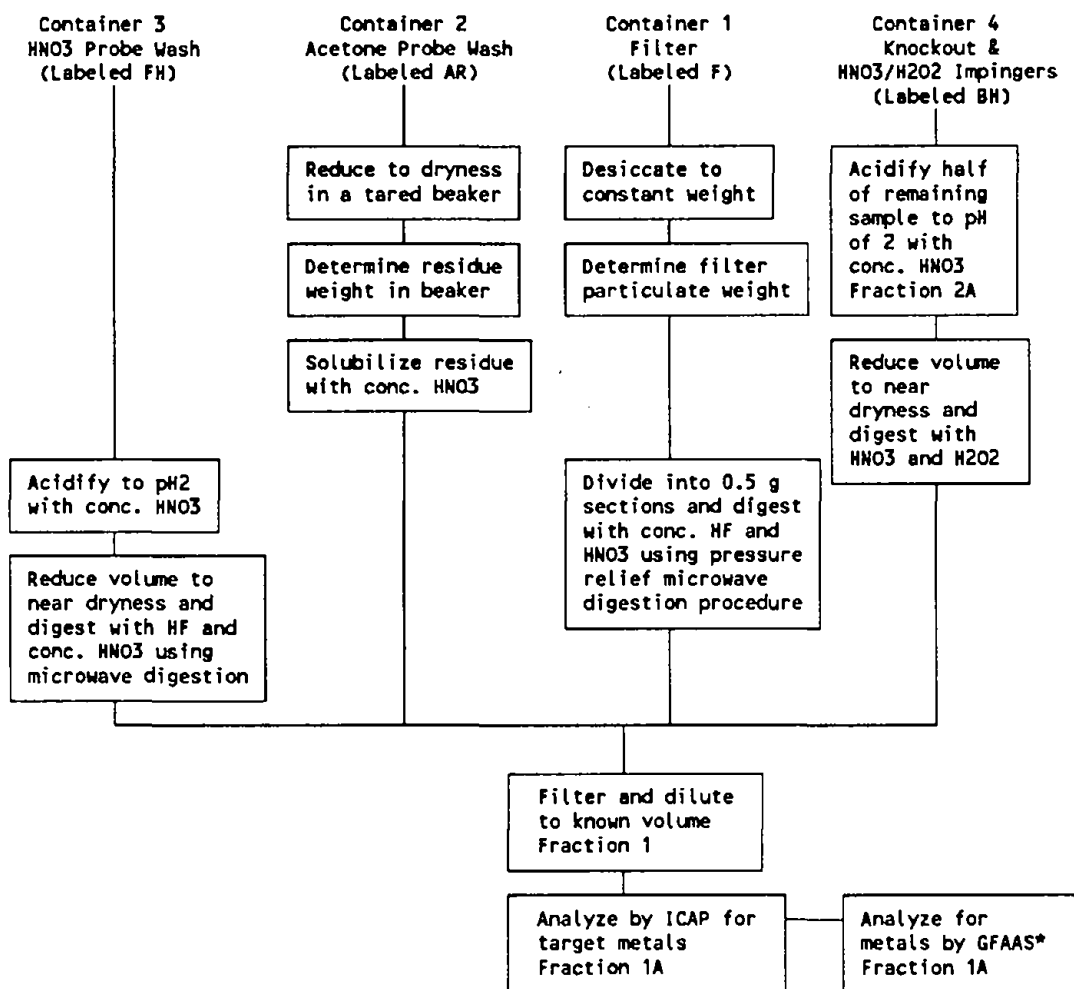
6.3 MULTIPLE METALS ANALYSIS

Analysis of flue gas samples, dewatered sludge samples, incinerator bottom ash, and scrubber water samples for the target metals: arsenic (As), beryllium (Be), cadmium (Cd), chromium (Cr), lead (Pb), and nickel (Ni), employed matrix-specific preparation and digestion followed by ICAP analysis. All prepared sample solutions were initially archived for possible reanalysis for As and Pb by graphite furnace atomic absorption spectroscopy (GFAAS). Since the sample, sludge, and ash could determine the results for Cd, Cr, Pb, and Ni, no samples were reanalyzed by GFAAS.

6.3.1 Flue Gas Samples

Flue gas samples were analyzed for the target metals following the procedures described in the draft AREAL procedure. A copy of the draft method is provided in Volume IV Site 6 Draft Test Report, Appendices, and an analytical flow chart is provided in Figure 6-4. The particulate mass was determined for the front half portion of the sampling train. The particulate matter was subjected to microwave HNO_3 /HF digestion in a pressure relief vessel. The nitric acid/hydrogen peroxide impinger solution and nitric acid rinses were reduced to near dryness and digested with HNO_3 . The front and back half digestates were combined for a single ICAP analysis for As, Be, Cd, Cr, Pb, and Ni. A portion of the digestate was initially archived for possible reanalysis of As and Pb by GFAAS. Since all metals of interest were detected, GFAAS was not performed.

The ICAP was calibrated with a series of five standard solutions containing the target metals ranging in concentrations from 0-to-100 ug/ml (depending on the element). The Cr and Ni standards were prepared in one solution and the As, Be, Cd, and Pb was prepared in a second solution. An interference check sample was analyzed prior to sample analysis, and a calibration check sample was analyzed with every 10 samples. A reagent blank was carried through the procedure and analyzed with the actual samples.



* Analysis by for metals found at less than the ICAP working level.

Figure 6-4. Sample preparation and analysis scheme for multiple metals trains.

6.3.2 Dewatered Sludge Samples

Dewatered sludge samples were analyzed for the target metals after determination of the moisture and ash content, heating value, and proximate and ultimate analyses following ASTM D3174, D3175, D3177, D3178, D3179, and D2361. A dried portion of the sludge sample was subjected to microwave HNO_3 /HF digestion in a pressure relief vessel identical to the flue gas particulate samples described above. This digestion procedure was chosen to provide for comparison of the metals in the sludge with the flue gas samples and the bottom ash samples, described below. The digestion solution was analyzed by ICAP following the procedures described for the flue gas samples and archived for possible GFAAS analysis.

6.3.3 Incinerator Bottom Ash Samples

Incinerator bottom ash samples were analyzed for the target metals including Hg after determination of the moisture content following ASTM D3174. The procedures used were the same as described above for the sludge samples.

6.3.4 Scrubber Water Samples

Portions of the inlet and outlet scrubber water samples were acidified with HNO_3 and reduced to near dryness on a hot plate. If any solids remain after the initial digestion, the sample was subjected to the microwave HNO_3 /HF digestion described above. The digested solutions were analyzed by ICAP for all the target metals except Hg following the procedures described for the flue gas samples; a portion of the solution was initially archived for possible GFAAS analysis. No additional GFAAS analyses were required.

6.4 SLUDGE SAMPLE ANALYSES

Dewatered sludge samples were subjected to the following: moisture analysis, proximate and ultimate analyses. Ultimate and proximate analyses were combinations of measurements performed with the following ASTM procedures; ash by ASTM D3174, volatile matter by ASTM D3175, carbon and hydrogen by ASTM D3178, nitrogen by ASTM D3179, chlorine by ASTM D2361, and sulfur by D3177. These procedures are detailed in the ASTM methods and the methods are provided in Volume IV Site 6 Draft Report, Appendices. The heating value was calculated from the carbon and hydrogen content determined by ASTM D3178.

7.0 QUALITY ASSURANCE AND QUALITY CONTROL

This section discusses the quality assurance and quality control (QA/QC) program implemented for the sewage sludge incineration test program and the QA/QC results for the Site 6 test. The objectives of and basic activities for the QA/QC program are briefly discussed in the section below. Summaries of the QC data and QA audit data are presented in Sections 7.2 through 7.4.

7.1 QA/QC PROGRAM OBJECTIVES

For any environmental measurement, a degree of uncertainty exists in the data generated due inherent limitations of the measurement systems employed. To assess the quality of the data and to establish limitations on the ultimate use of the data, a comprehensive QA/QC program was implemented for this test effort. The objective of the QA/QC program was to produce complete, representative, and comparable data of known quality. To meet these objectives, a thorough Quality Assurance Project Plan (QAPP), integrated with the sampling and analysis plan, was prepared. All elements of the QAPP were implemented during the sampling and analytical phases of the sewage sludge incinerator test program for Site 6. In the QAPP, the specific EPA methods, other standard test methods, and state-of-the-art sampling/analytical procedures to be employed and QC activities performed were described.

The terms used to define the QA/QC objectives established for the test program are defined as follows:

- (1) Data Quality: The total of features and characteristics of a product (measurement data) that determine its ability to satisfy a given purpose. These characteristics are defined as follows:

- Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation (or the relative standard deviation). Various measures of precision exist depending upon the prescribed conditions.
 - Accuracy - The degree of agreement of a measurement (or an average of measurements of the same parameter), X , with an accepted reference or true value, T , is usually expressed as the difference between two values, $X - T$, or the difference as a percentage of the reference or true value, $100 (X - T)/T$, and sometimes expressed as a ratio, X/T . Accuracy is a measure of the bias in a system.
 - Completeness - A measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under correct normal conditions.
 - Comparability - A measure of the confidence with which one data set can be compared with another.
 - Representativeness - The degree to which data accurately and precisely represent a characteristic of a population, variations of a parameter at a sampling point, or an environmental condition.
- (2) Quality Control: The overall system of activities whose purpose is to provide a quality product or service: for example, the routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.

(3) Quality Assurance: A system of activities whose purpose is to provide assurance that the overall quality control is in fact being done effectively.

- It is the total integrated program for assuring the reliability of monitoring and measurement data.
- It is the system for integrating the quality planning, quality assessment, and quality improvement efforts of various groups in an organization to enable operations to meet user requirements at an economical level. In pollution measurement systems, quality assurance is concerned with the activities that have an important effect on the quality of the pollutant measurements, as well as the establishment of methods and techniques to measure the quality of the pollution measurements. The more authoritative usage differentiates between "quality assurance" and "quality control," where quality assurance is the "system of activities to provide assurance that the quality control system is performing adequately."

The QAPP emphasized: (1) adherence to the prescribed sampling procedures, (2) careful documentation of sample collection and analytical data, (3) the use of chain-of-custody records, (4) adherence to prescribed analytical procedures, and (5) implementation of independent systems audits and performance audits. These QA/QC efforts provide a mechanism to control data quality within acceptable limits and provide the necessary information to assess the quality of the data.

The data quality objectives for the measurement parameters are presented in Table 7-1. These data quality objectives are for analysis of the samples collected during emission testing at the individual sites. Where possible, the precision and accuracy for the measurement parameters were obtained from the specified methods or from EPA collaborative tests. This type of data was not available for the determination of metals in the flue gas and solid samples.

TABLE 7-1. PRECISION, ACCURACY AND COMPLETENESS OBJECTIVES

Parameter	Precision ^a (%)	Accuracy ^a (%)	Completeness ^b (%)
Total particulate (EPA Method 5)	± 11	± 10	90
Nickel/metals distribution in particulate	50 ^c	NA	90
Cr ⁺⁶ distribution in particulate	50 ^c	NA	90
Flue gas total metal	NA	NA	90
Continuous Emission Monitoring (O ₂ , CO ₂ , CO, THC, NO _x , SO ₂)	± 20 ^e	± 20 ^f	90
Feed sludge: Metals/Cr/Ni	NA	NA	90
Velocity/volumetric flow rate (Methods 1&2)	± 6	± 10	95
Fixed gases/molecular weight (Method 3)	± 10 ^f	± 20 ^g	90
Moisture (EPA Method 4)	± 20	± 10	90
Flue gas temperature (thermocouple)	± 2°F	± 5°F	90
Scrubber Water Influent and Effluent: Metals/Cr/Ni	NA	NA	90

^aWhen possible, precision and accuracy based on collaborative tests results.

^bValid data percentage of total tests conducted.

^cEPA collaborative test data not available.

^ePercent difference for duplicate analyses, where

$$\text{Percent Difference} = \frac{\text{First Value} - \text{Second Value}}{0.5 (\text{First} + \text{Second Values})} \times 100$$

^fCoefficient of variation (CV) determined from daily analyses of a control sample, where

$$\% \text{ CV} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

^gRelative error (%) derived from audit analyses, where

$$\text{Percent Error} = \frac{\text{Measured Value} - \text{Theoretical Value}}{\text{Theoretical Value}} \times 100$$

NA = Not applicable. For precision, because multiple samples not to be taken or analyzed; for accuracy, because audit samples not available.

7.2 FLUE GAS SAMPLING AND ANALYSIS QC RESULTS

Quality control activities for flue gas sampling include: (1) equipment calibrations, (2) glassware and equipment cleaning, (3) procedural checks during sampling and sample recovery, (4) sample custody procedures, (5) procedural checks during sample analysis, and (6) the use of labeled surrogates, field blanks, laboratory blanks, QC check samples, matrix spikes, and duplicate analyses. The QC results for these activities are discussed in this section, with activities generally applicable to all the flue gas sampling methods discussed in Section 7.2.1 and method specific results discussed separately in Sections 7.2.2 and 7.2.3.

7.2.1 General Flue Gas Sampling Quality Control

For all of the flue gas sampling methods, pre-test calibrations were performed on the sampling nozzles, pitot tubes, temperature sensors, and analytical balances. Both pre- and post-test calibrations were performed on all dry gas meters employed during flue gas sampling. All equipment met the criteria specified in the QAPP for this program. The post-test calibrations for the all dry gas meters employed during sampling were within the specified 5% agreement with the pre-test calibrations.

All sampling train glassware and Teflon components, sample containers, and sampling tools were precleaned initially with soap and water followed by a DI water and 0.1 N nitric acid rinse, and a final DI water rinse. During on-site testing, all sampling train glassware was capped with Parafilm or Teflon tape prior to and immediately after each test run. A clean, dust-free environment was maintained on-site for sampling train assembly and sample recovery.

QC activities during flue gas sampling included:

- Visual equipment inspection;
- collection of sample train blanks;
- ensuring the proper location and number of traverse points;

- conducting pre-test and post-test pitot tube and sample train leak checks;
- maintaining proper temperature at the sample probe (if applicable), filter housing, and impinger train outlet;
- maintaining isokinetic sampling rates; and
- recording all data on preformatted field data sheets and noting any unusual occurrences on a test log sheet.

Leak check and isokinetic calculation results are presented separately for each method in the sections that follow. The QC criterion for leak checks was a rate less than or equal to 0.02 cubic feet per minute at 15 in Hg vacuum for the pre-test check and, for the post-test check, at the highest vacuum encountered during sampling. For isokinetic sampling, the QC criterion was to have the average sampling rate within 10% of isokinetic.

Detailed procedures for sample recovery, specific for each method and described in the QAPP, were followed. Graphic flow charts of each procedure were readily available in the recovery area.

Sample custody procedures employed for all flue gas samples and process samples emphasized proper labeling and preparation of chain-of-custody records for transfer of the samples to the different laboratories involved in the test program. Pre-printed labels were prepared with a unique alphanumeric code for each sample collected or generated during sample recovery. Samples were also logged in a master logbook. Each sample container was sealed with a custody seal to ensure sample integrity. Samples were stored and shipped to the laboratories following the method-specific procedures. Upon receipt of the samples, each laboratory logged the samples into their own sample custody system and stored the samples under the prescribed conditions.

7.2.2 Sampling and Analysis for Particulate, Total Metals and Nickel/Nickel Subsulfide

Sampling and analysis for particulate matter and total metals followed the draft EPA method, "Methodology for the Determination of Trace Metals Emissions in

Exhaust Gases from Stationary Source Combustion Processes." Sampling for nickel and nickel subsulfide followed EPA Method 5 for sample collection and the NiPERA method for sample analyses. Quadruplicate sampling trains were employed with three sampling trains used to collect samples for nickel speciation (Trains A, B and C) and a single train used to collect particulate matter/total metals sample (Train D). The results of the QC operations during sampling and analysis are presented in the following sections.

7.2.2.1 Sampling Operations - Isokinetic calculation and leak check results for the scrubber outlet sampling location are summarized in Table 7-2. Nine of the 28 sampling trains operated at the scrubber outlet for collection of particulate matter/total metals and nickel/nickel subsulfide samples were less than 90% isokinetic. However, all of these runs were within 6% of the allowable. All the sampling trains (Train D) operated to collect the particulate matter/total metals met the isokinetic criterion. The nickel speciation results from Train A were not reported due to analytical problems. The samples from Train B were sent to BYU for XANES and EXAFS analyses. The samples from Train C were speciated by Dr. Vladimir Zatka, the developer of the NiPERA analytical method. The instrumental techniques performed by BYU confirmed the wet chemical techniques conducted by Dr. Zatka.

Although some bias due to particle size differentiation may have been introduced by nonisokinetic sampling, it is generally accepted that very small particles are emitted from venturi scrubbers, and isokinetic sampling is not as critical under these conditions.

The post-test leak check results for all 28 outlet sampling trains met the QC criteria.

At the inlet sampling location, isokinetic sampling was not performed due to the duct configuration. The post-test leak check results for all 28 inlet sampling trains met the QC criteria.

TABLE 7-2. ISOKINETICS AND LEAK CHECK SUMMARY; SITE 6, OUTLET LOCATION, PARTICULATE MATTER/TOTAL METALS AND NICKEL/NICKEL SUBSULFIDE SAMPLING

Run No.	Train No.	Percent Isokinetics	Leak Rate (cfm)	Vacuum (in. Hg)
2	A	95.4	0.006	3
2	B	96.5	0.004	2
2	C	88.1	0.007	4
2	D	93.6	0.002	2
4	A	95.2	0.002	8
4	B	93.1	0.004	8
4	C	87.9	0.000	9
4	D	94.2	0.005	9
5	A	89.9	0.005	7
5	B	90.2	0.008	6
5	C	84.1	0.002	6
5	D	101.6	0.010	9
6	A	92.6	0.019	12
6	B	92.9	0.017	9
6	C	87.1	0.015	14
6	D	92.1	0.002	11
8	A	95.7	0.004	8
8	B	87.7	0.012	8
8	C	94.6	0.009	13
8	D	99.2	0.009	11
10	A	82.2	0.013	15
10	B	94.0	0.009	15
10	C	86.7	0.016	15
10	D	94.0	0.018	15
12	A	95.2	0.006	4
12	B	93.3	0.012	8
12	C	88.6	0.007	8
12	D	97.4	0.009	5

7.2.2.2 Sample Analysis - Analytical results for the metals field recovery blank, laboratory blanks, and the audit sample are presented in Table 7-3. The field recovery blank was collected from an outlet sampling train previously used to collect a sample. After normal sample recovery, the train was prepared as if to collect another sample, taken to the outlet sampling location, and leak checked. The field recovery blank train was left at the outlet location during sampling, and then recovered following the normal sample recovery procedure. The field recovery blank and reagent blanks had non-detectable quantities of all target metals except chromium. Chromium was found in both reagent blanks and the field recovery blank, but the quantity was less than those found in the field samples. An average value of 1.1 ug was used to correct the field sample results, as well as the audit sample result.

Calibration check samples were analyzed with every ten samples. The results for the calibration check samples were all within 10% of the expected value.

The audit samples results were generally biased low ranging from -9.1% to -23.0% less than the true audit value. The audit sample analyzed was provided by EPA's Quality Assurance Division in Research Triangle Park, NC.

7.2.3 Total Chromium and Hexavalent Chromium Sampling and Analysis

Sampling and analysis for total chromium (Cr) and hexavalent chromium (Cr⁺⁶) was performed following the procedures in the draft EPA method, "Determination of Hexavalent Chromium from Stationary Sources." The QC activities for the Cr/Cr⁺⁶ testing performed at Site 6 are discussed in the following sections.

7.2.3.1 Sampling Operations - Isokinetic and leak check results for the chromium sampling at the scrubber outlet sampling location are summarized in Table 7-4. Fifteen of the 20 sampling trains operated at the scrubber for collection of Cr/Cr⁺⁶ samples were less than 90% isokinetic. Since the outlet sampling location was downstream of a venturi scrubber which tends to emit small particles, the non-isokinetic sampling should

TABLE 7-3. QC RESULTS FOR FIELD RECOVERY BLANKS, REAGENT BLANKS, AND AUDIT SAMPLES

Metal	Field Recovery Blank (ug)	Reagent Blanks (ug)		Audit Sample (ug)		Percent Error
		Blank 1	Blank 2	Found	Actual	
Arsenic	ND*	ND	ND	<23	9.6	---
Beryllium	ND	ND	ND	4.4	4.85	-9.3
Cadmium	ND	ND	ND	7.7	10.0	-23.0
Chromium	1.0	1.0	1.3	9.5 ^b	10.3	-6.8
Lead	ND	ND	ND	43.1	50.4	-14.5
Nickel	ND	ND	ND	22.4	25.2	-11.1

*Not Detected

^bBlank corrected by 1.1 ug (average value detected in blanks)

Table 7-4. ISOKINETICS AND LEAK CHECK SUMMARY; SITE 6, OUTLET LOCATION, TOTAL CHROMIUM AND HEXAVALENT CHROMIUM SAMPLING

Run No.	Train No.	Percent Isokinetics	Leak Rate (cfm)	Vacuum (in. Hg)
3	A	74.6	0.100	12
3	B	51.7	0.006	11
3	C	59.1	0.009	18
3	D	65.1	0.008	14
7	A	90.2	0.001	15
7	B	93.6	0.002	7
7	C	88.0	0.004	15
7	D	67.8	0.015	15
9	A	81.8	0.008	13
9	B	88.8	0.009	11
9	C	84.2	0.011	14
9	D	88.6	0.009	12
11	A	68.2	0.006	8
11	B	89.8	0.001	14
11	C	87.5	0.003	13
11	D	78.7	0.001	13
13	A	79.1	0.020	8
13	B	95.3	0.001	9
13	C	94.5	0.021	10
13	D	95.1	0.019	13

not have caused a significant bias. The low isokinetic sampling resulted from use of prototype Teflon sampling nozzles which were larger in diameter than desirable and required a higher sampling rate to achieve isokinetic sampling. The pressure drop created by the Teflon aspirator used to recirculate the absorbing solution prevented sampling at the higher isokinetic rate when higher velocities occurred in the stack.

The post-test leak check results for 19 of the 20 outlet sampling trains met the QC criteria, with one train having a leak rate of 0.021 cfm.

At the inlet sampling location, isokinetic sampling was not performed due to the duct configuration. The post-test leak check results for all 20 inlet sampling trains met the QC criteria.

7.2.3.2 Sample Analysis - Neither Cr or Cr⁺⁶ were detected in any of the reagent blanks submitted for analysis. All analyses for Cr⁺⁶ were performed in duplicate with the percent deviation of duplicate samples being less than 5%. The Cr⁺⁶ preconcentration curve was linear from 0.071 ppb to 0.749 ppb, with a maximum percent deviation of - 10.7%.

To determine the extent of Cr⁺⁶ conversion that occurred during sampling, a radioactive Cr⁺⁶ surrogate, ⁵¹Cr⁺⁶, was added to the absorbing solution in each sampling train prior to sampling. The ⁵¹Cr⁺⁶ spike was recovered and analyzed by ion chromatography with a post column reaction (IC/PCR). The IC fractions were collected and counted in a gamma counter, along with the filter and rinse samples. The ⁵¹Cr⁺⁶ recoveries are shown in Table 7-5.

The surrogate recoveries ranged from 33.9% to 98.8% for the total recovery of ⁵¹Cr⁺⁶, when the chromium in the acid rinses were counted. Recoveries without including the radioactivity in the 0.1 N HNO₃ rinse ranged from 75.4% to 98.9%. The degree of conversion appeared to increase during the test program when the radioactivity in the 0.1 N HNO₃ rinses are included. A black film accumulated in the Teflon components during the test program that was not removed by the specified sample recovery procedures. The black film may have been responsible in part for the gradual increase in the amount of radioactivity measured in the 0.1 N HNO₃ rinses.

TABLE 7-5. RECOVERIES OF $^{51}\text{Cr}^{+6}$ SURROGATE

Run No.	Train No.	$^{51}\text{Cr}^{+6}$ Surrogate Recoveries (percent of total)		
		Soluble ^a	Total ^b	Without HNO_3 Rinse
3	A	99.5	96.5	98.8
3	B	99.3	98.8	98.9
3	C	81.0	74.8	75.4
3	D	91.8	81.7	87.3
7	A	83.3	71.8	81.9
7	B	88.3	74.8	87.4
7	C	85.4	79.8	84.7
7	D	91.0	90.0	90.4
9	C	94.4	68.3	93.9
9	D	97.4	82.8	96.7
11	C	95.3	53.7	94.6
11	D	97.6	97.1	97.3
13	C	94.9	33.9	92.7
13	D	97.6	43.3	96.0
Field Blank		96.5	NA ^c	96.5
Reagent Blank		99.0	NA	99.0

^aRadioactivity in soluble NaOH fraction coeluting with native Cr^{+6} .

^bTotal $^{51}\text{Cr}^{+6}$ in soluble fraction divided by total radioactivity.

^cNot applicable.

7.2.4 Continuous Emission Monitoring

Continuous emission monitoring (CEM) was performed at the scrubber inlet and scrubber outlet for O₂, CO₂, CO, SO₂, and NO_x. Total hydrocarbons were also monitored at the scrubber outlet on a conditioned (cold) sample. Instrument calibrations were performed at the beginning of each test day, between sampling runs, and at the conclusion of each test day. Instrument drift checks, the comparison of the post-test measurement of zero and span gases to the pre-test values, were performed for each run, and are summarized in Table 7-6. All CEM data were drift corrected assuming linear drift. Data from an instrument with drift exceeding 20% during a test period was invalidated.

The drift for both the zero and span were within 1% for almost every run. Because the monitoring data was not intended to standard setting purposes, four calibration gases were used for O₂, CO₂, CO, SO₂, and NO_x, and three calibration gases were used in the direct calibration check. Two calibration gases were used for the drift test after test run, and no performance audit was conducted. All the data meets the requirements of Method 3A, 6C, 7E, 10, and 25A.

7.3 PROCESS SAMPLE ANALYSIS QC RESULTS/METAL ANALYSIS

Samples of sludge feed, bottom ash, scrubber influent water, and scrubber effluent water were collected during each test run. These process samples were grab samples collected at regular intervals, and combined after each test to form composite samples. QC results for analyses performed on these samples are presented in this section.

Quality control indicators for process samples are method blanks and calibration checks during analysis.

None of the six target metals were detected in the method blanks for the sludge feed, bottom ash, scrubber influent, and scrubber effluent samples.

Calibration check samples were analyzed with every ten samples. The results for the calibration check samples for the sludge feed and bottom ash were all within 10% of

TABLE 7-6. SUMMARY OF CEM DRIFT CHECKS

Date and Run No.		CEM Location		Instrument Zero and Span Drift (% of span)										Cold THC	
				O ₂		CO ₂		O		SO ₂		NO _x			
				Zero	Span	Zero	Span	Zero	Span	Zero	Span	Zero	Span	Zero	Span
10/09/89	Inlet	0.0	-0.8	0.0	0.5	-0.1	-0.1	0.5	0.3	0.5	0.7	0.9	1.2		
Run 03	Outlet	0.0	0.0	1.0	0.0	-0.2	-2.4	0.4	0.7	0.2	0.6				
10/09/89	Inlet	0.8	0.0	0.0	0.0	-0.9	-3.0	0.3	0.9	0.3	-0.9	0.3	0.3		
Run 04	Outlet	0.4	0.4	0.0	1.0	-1.5	-6.3	0.3	-0.3	0.2	-0.2				
10/10/89	Inlet	0.4	-0.4	0.0	0.0	-0.1	0.8	0.3	0.2	0.0	-2.0	0.0	-0.6		
Run 05	Outlet	0.4	-0.8	0.5	0.5	-0.1	-1.0	0.5	-0.2	0.1	0.5				
10/10/89	Inlet	0.4	0.4	0.0	-0.5	0.0	0.0	-0.7	1.2	0.2	-1.2	-0.2	-0.9		
Run 06	Outlet	-0.4	0.4	0.0	-0.5	-0.3	-1.2	-0.2	0.2	0.5	-0.1				
10/10/89	Inlet	-0.8	-0.8	0.0	0.0	0.0	0.0	0.5	-0.3	0.3	2.9	0.5	1.2		
Run 07	Outlet	0.0	0.0	-0.5	2.0	0.1	0.7	0.6	0.0	-0.1	-0.7				

(Continued)

TABLE 7-6. (Continued)

Date and Run No.		CEM Location		Instrument Zero and Span Drift (% of span)										Cold THC	
				O ₂		CO ₂		CO		SO ₂		NO _x			
				Zero	Span	Zero	Span	Zero	Span	Zero	Span	Zero	Span		
10/11/89	Inlet	0.4	-0.4	0.0	0.5	-0.1	-1.0	0.4	-0.5	0.0	-0.5	0.0	-0.7		
Run 08	Outlet	0.0	-1.2	1.0	0.5	-0.6	-1.5	0.4	0.3	0.2	-0.7				
10/11/89	Inlet	-0.4	0.0	-0.5	-0.5	0.0	1.1	-0.8	0.3	0.0	0.8	-0.2	-0.6		
Run 09	Outlet	0.0	0.0	0.5	0.0	0.0	0.7	0.3	0.7	0.1	0.0				
10/12/89	Inlet	0.0	-0.4	0.0	0.0	-0.1	-0.1	0.3	1.0	-0.1	-0.5	0.1	-0.3		
Run 10	Outlet	0.0	-0.8	0.5	1.0	-0.3	-1.6	0.2	0.5	0.3	0.6				
10/12/89	Inlet	0.4	0.0	0.0	-0.5	0.0	0.1	-0.2	0.1	-0.2	-0.4	0.0	-0.5		
Run 11	Outlet	0.0	0.0	0.5	-0.5	-0.2	-0.2	0.0	-0.3	0.2	-0.1				
10/12/89	Inlet	0.0	0.0	0.0	0.5	0.0	-0.1	0.2	0.3	0.4	0.6	Not conducted			
Run 12	Outlet	0.0	0.4	1.0	0.5	0.3	0.7	-0.7	-0.7	-0.5	-0.5				
10/13/89	Inlet	0.0	-0.8	0.0	0.0	0.0	-0.9	0.2	-0.5	0.3	-0.4	0.0	-0.6		
Run 13	Outlet	0.0	-0.4	-0.5	0.0	-0.4	-1.6	1.1	1.4	0.4	0.2				

the expected value, except for one analysis where lead measured 14% higher than the expected value. Calibration check samples for the scrubber water samples were all within 10% of the expected value.

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TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA/600/R-92/003c		2.		3. PB92-151570	
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7. AUTHOR(S) William G. DeWees, Robin R. Segall F. Michael Lewis				6. PERFORMING ORGANIZATION CODE	
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16. ABSTRACT Site 6, a multiple hearth furnace was tested under two operating conditions, normal combustion was compared with improved combustion conditions as indicated by reduced CO and THC emissions. The effect of lime conditioning on the conversion of total chromium in the sludge to hexavalent chromium emissions was also a primary concern at Site 6. Secondary objectives included comparing the results for chromium and nickel subspecies determined by different analytical procedures, gathering data on other metals and inorganic/organic gases in incinerator emissions, and assessing pollutant removal efficiencies by measuring emissions at both the inlet and outlet to the control system. The Site 6 plant treats 30 million gallons a day of municipal and industrial wastewater. The blended primary/secondary sludge is dewatered to approximately 28% solids using recessed plate cloth filters. The metal mass emissions rate for the outlet runs averaged: As - not detected (< 508 mg/hr), Be - not detected (< 2.2 mg/hr), Cd - 1,450 mg/hr, Cr - 83.3 mg/hr, Pb - 21,100 mg/hr, and Ni - 73.9 mg/hr. The particulate mass emission rates averaged 42 kg/hr and 0.7 kg/hr, respectively for the inlet and outlet. A positive correlation between the CO/CO ₂ ratios (an indication of combustion conditions) and the hexavalent to total chromium ratio was demonstrated for the outlet location. At low CO levels (good combustion) the ratio of hexavalent chromium to trivalent chromium was highest, with approximately 10% of the total chromium in the form of hexavalent chromium. It was anticipated that the nickel subsulfide emissions from multiple hearth incinerators would constitute less than 1% of the total nickel emissions. A wet chemical analysis indicated that within the detection limit (< 10%), no nickel subsulfide was present.					
17. KEY WORDS AND DOCUMENT ANALYSIS					
a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS		c. COSATI Field/Group	
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