Vol. 56 No. 137 Wednesday, July 17, 1991 p 32688

>>>> This article, FR94, is divided into five files. This is File A: Corrections for Part 261 through Part 270. Technical Amendments to Part 261 through 3.2.7.4 of Appendix IX to Part 266. <<<<

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Parts 260, 261, 264, 265, 266, 270, and 271

[EPA/OSW-FR-91- SWH-FRL-39689]

Burning of Hazardous Waste in Boilers and Industrial Furnaces

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule: corrections; technical amendments.

SUMMARY: On February 21, 1991, the Environmental Protection Agency (EPA) published a final rule to regulate air emissions from the burning of hazardous waste in boilers and industrial furnaces (56 FR 7134). Today's notice corrects typographical and editorial errors that appeared in the regulatory text, including corrections to appendices II and III, and adds two appendices, appendix IX and appendix X, to part 266. Appendices IX and X were not ready at the time of publication; therefore, a note was placed in the appropriate location in the rule to inform readers that these appendices were to be published at a later date. Copies of these appendices were, however, made available to the public through the RCRA Docket maintained at EPA and through the National Technical Information Service (NTIS).

EFFECTIVE DATE: The effective date of the rule remains August 21, 1991.

FOR FURTHER INFORMATION CONTACT: For general information, contact the RCRA Hotline at (800) 424-9346 (toll-free) or (703) 920-9810. For more specific aspects of the final rule, contact Shiva Garg, Office of Solid Waste (0S-322), U.S. Environmental Protection Agency, 401 M Street, SW., Washington, DC 20460 (703) 308-8460.

>>>> Preamble has not been included in this file. <<<<

A. Technical Corrections

In rule document number 91-2667, beginning on page 7134 in the Federal Register published on Thursday, February 21, 1991, make the following corrections:

PART 261-[AMENDED]

1. On page 7206, third column, in amendment 2 to part 261, add the following at the beginning of line 3 of the amendatory language of § 261.2: ", paragraph (d)(3) as (d)(4) and paragraph (d)(4) as (d)(5)". The corrected amendatory language will read as follows:

"2. Section 261.2 is amended by redesignating paragraph (d)(2) as (d)(3), paragraph (d)(3) as (d)(4), and paragraph (d)(4) as (d)(5), and adding a new paragraph (d)(2) to read as follows:"

PART 266-[AMENDED]

§ 266.100 [Corrected]

2. On page 7208, second column, in § 266.100(b)(2), line 3, replace the period after "recovery" with a semicolon.

3. On page 7208, second column, in § 266.100(b)(3), line 7, change "§ 261.5 of this chapter." to "§ 261.5 of this chapter; and ".

§ 266.102 [Corrected]

4. On page 7209, second column, in § 266.102(b)(1), line 12, change "for the Evaluation of Solid Waste" to "for Evaluating Solid Waste".

5. On page 7210, first column, in § 266.102, the paragraph designated as "(d)(4)(iii)(D)" should be designated as "(d)(4)(iv)".

6. On page 7210, third column, in § 266.102(e)(4)(i)(C), line 1, insert "A" between "(C)" and "sampling".

7. On page 7211, second column, in § 266.102(e)(6) heading, line 1, change "paramenters" to "parameters".

8. On page 7211, third column, in § 266.102(e)(6)(i)(B)(1)(ii), line 2, change "means" to "mean".

9. On page 7211, third column, at two locations: In lines 30 and 59, the number "2" in each subparagraph (2) heading of § 266.102(e)(6)(i)(B) and § 266.102(e)(6)(ii)(B), respectively, should be italicized.

10. On page 7211, third column, in § 266.102(e)(6)(ii), line 10, change "opeator" to "operator".

11. On page 7211, third column, in § 266.102(e)(6)(ii)(B)(2), lines 3 and 4, change "arithmetic mean of the most recent one hour block average for the average period" to "arithmetic mean of one hour block averages for the averaging period".

12. On page 7212, first column, in § 266.102(e)(6)(iv)(B), line 15, delete the comma between "§ 266.106(f)" and "need".

§ 266.103 [Corrected]

13. On page 7213, first column, in § 266.103(a)(1)(ii), line 7, insert "or" between "burn" and "to".

14. On page 7214, first column, in § 266.103(b)(2)(ii)(A), line 4, insert "and" between "silver" and "thallium".

15 On page 7214, first column, in § 266.103(b)(2)(ii)(B), line 3, the reference to "(b)(ii)(A)" should read "(b)(2)(ii)(A)".

16. On page 7214, first column, in § 266.103(b)(2)(ii)(D), line 4, the reference to "(b)(ii)(B) or (b)(ii)(C)" should read "(b)(2)(ii)(B) or (b)(2)(ii)(C)".

17. On page 7214, second column, line 3, in § 266.103(b)(2)(iv), change "paticulate" to "particulate".

18. On page 7214, second column, in § 266.103(b)(2)(v)(A)(5), line 2, change "eqivalent" to "equivalent".

19. On page 7214, second column, in § 266.103(b)(2)(v)(A)(5), line 4, replace the period after "facility" with a semicolon.

20. On page 7214, second column, in § 266.103(b)(2)(vi), line 3, change "HC1" to "HCl".

21. On page 7214, third column, in § 266.103(b)(3)(ii), line 2, replace the semicolon after "streams" with a colon.

22. On page 7215, first column, in § 266.103(b)(5)(ii)(B), line 1, change "meat" to "meet".

23. On page 7215, first column, at two locations: in lines 38 and 66, the number "1" in each subparagraph (1) heading of § 266.103(b)(5)(i)(B) and § 266.103(b)(5)(ii)(B), respectively, should be italicized.

24. On page 7215, first column, line 46, the number "2" in the subparagraph (2) heading of § 266.103(b)(5)(i)(B) should be italicized.

25. On page 7215, second column, line 4, the number "2" in the subparagraph (2) heading of § 266.103(b)(5)(ii)(B) should be italicized.

26. On page 7215, second column, in § 266.103(b)(5)(ii)(B)(2), line 3 and 4, change "arithmetic mean of the most recent one hour block averages" to "arithmetic mean of one hour block averages".

27. On page 7215, second column, in § 266.103(b)(6) introductory text, lines 2 and 3, change "[the effective date of this rule]" to "August 21, 1991".

28. On page 7216, first column, in § 266.103(c), lines 1 and 2, delete "On or before August 21, 1992", capitalize the "t" in "the", and insert "on or before August 21, 1992" in line 12 between "Director" and "a".

29. On page 7216, first column, in § 266.103(c)(1), line 10, add "and all applicable emissions standards" after "limits".

30. On page 7216, second column, in § 266.103(c)(1)(iv), line 3, change "light-weighted" to "light-weight".

31. On page 7216, second column, in § 266.103(c)(1)(ix), line 8, replace the semicolon after "(e))" with a colon.

32. On page 7216, third column, in § 266.103(c)(1)(xi), lines 1, change "system" to "systems", and in line 7, replace the semicolon after "(e))" with a colon.

33. On page 7216, third column, in § 266.103(c)(1)(xii), line 8, replace the semicolon after "(e))" with a colon.

34. On page 7217, third column, in § 266.103(c)(4)(i)(C), line 2, change "test" to "testing".

35. On page 7217, third column, in § 266.103(c)(4)(ii)(B)(5), line 3, change "averge" to "average".

36. On page 7218, second column, in § 266.103(c)(4)(iv)(C)(2)(ii), lines 3 and 4, change "the arithmetic mean of the most recent one hour block averages for the averaging period" to "arithmetic mean of one hour block averages for the averaging period".

37. On page 7218, third column, in § 266.103(c)(7)(i)(A), line 3, change "(1)" (one) to "(1)" (lower case "el").

38. On page 7218, third column, in § 266.103(c)(7)(i)(B), delete the last word "to" in line 1 and replace by "only for purposes of compliance testing (and pretesting to prepare for compliance testing)".

39. On page 7219, first column, in § 266.103(c)(7)(ii)(B)(1)(ii), line 7, insert a period after "HCl/Cl_2".

40. On page 7219, first column, line 18, the number "2" in the subparagraph (2) heading of § 266.103(c)(7)(ii)(B) should be italicized.

41. On page 7219, third column, in § 266.103(g)(1), line 1, change "or" to "of".

§ 266.104 [Corrected]

42. On page 7220, second column, in § 266.104(a)(1), change the equation:

$$DRE = \left[\frac{1-W_{out}}{W_{in}} \right] X\ 100$$

to:

$$DRE = \left[1-\frac{W_{out}}{W_{in}} \right] X\ 100$$

43. On page 7220, third column, in § 266.104(a)(3), in line 12, change "tetrra-," to "tetra-," and in line 16, the reference to "paragraph (a)" should read "paragraph (a)(1)".

44. On page 7220, third column, in § 266.104(b)(2), lines 5 through 7, change "in Hazardous Waste Incinerators, Boilers, and Industrial Furnaces" to "for Incinerators, Boilers, and Industrial Furnaces Burning Hazardous Waste".

45. On page 7221, first column, in § 266.104(c)(3), lines 3 through 6, change "Performance Specifications for Continuous Emission Monitoring of Hydrocarbons for Incinerators, Boilers and Industrial Furnaces" to "Performance Specifications for Continuous Emission Monitoring of Hydrocarbons for Incinerators, Boilers, and Industrial Furnaces Burning Hazardous Waste"; and in line 7, insert "and" between "CO" and "oxygen".

46. On page 7221, second column, line 3, in § 266.104(e)(1), insert "(PCDDs)" after "Dibenzo-p-Dioxins", and in line 6, replace the period at the end of the sentence after "part" with a semicolon.

47. On page 7221, second column, in § 266.104(e)(2), line 13, replace the period at the end of the sentence after "TCDD" with a semicolon.

48. On page 7221, second column, in § 266.104(e)(4), line 6, insert a before "2.2".

49. On page 7221, third column, in § 266.104(f)(3)(iii), line 4, change "conducts" to "conduct".

50. On page 7222, first column, the paragraph designated as "(g)2." of § 266.104, should be designated as "(g)(2)".

§ 266.106 [Corrected]

51. On page 7222, second column, in § 266.106(a), line 9, change "for Evaluation Solid Waste" to "for Evaluating Solid Waste".

52. On page 7222, third column, in the equation after line 3, in § 266.106(b)(2)(i), change "<1.0" to " ≤ 1.0", and change "n = numer of carcinogenic metals" to "n = number of carcinogenic metals".

53. On page 7222, third column, in § 266.106(b)(2)(ii)(B), line 2, insert "as defined in § 266.102(e)(6)(ii)" between "hours" and "with".

54. On page 7222, third column, in § 266.106(b)(5), line 7, insert "shall be used" at the end of the sentence before the period.

55. On page 7223, first column, in § 266.106(b)(6), in the equation after line 15, change lines 5 and 6 of the equation that read "K = physical stack height (meters); K = stack gas flow rate (m^3/second); and" to read: "H = physical stack height (meters); V = stack gas flow rate (m^3/second); and".

56. On page 7223, second column, in § 266.106(c)(2), in the equation after line 12, delete the minus sign after "AER(i)".

57. On page 7223, third column, the equation in § 266.106(d)(3) is revised to read as below:

$$\sum_{i=1}^{n} \frac{\text{Predicted Ambient Concentration}_{(i)}}{\text{Risk-Specific Dose}_{(i)}} \leq 1.0$$

58. On page 7224, first column, in § 266.106(e), in line 8, change "each" to "a", and in line 10, change "levels" to "level".

59. On page 7224, first column, in § 266.106(f)(2)(ii) introductory text, delete the semicolon after "metal".

60. On page 7224, second column, line 3, in § 266.106(f)(2)(ii)(B), change "ratio" to "ratios".

§ 266.107 [Corrected]

61. On page 7224, second column, in § 266.107(a), line 4, change "provided by paragraphs (b), (c), or (d) of" to "provided by paragraph (b) or (c) of".

62. On page 7224, third column, in § 266.107(b)(2) heading, line 1, change "screen" to "screening".

63. On page 7224, third column, in § 266.107(b)(3), line 9, change "screen" to "screening".

64. On page 7225, first column, in § 266.107(d), change the paragraphs designated as "(i)" and "(ii)" to "(1)" and "(2)".

65. On page 7225, first column, in § 266.107(e), in line 3, insert "limit" between "screening" and "provided", and in line 4, the reference to "Appendix I" should read "Appendix II".

66. On page 7225, first column, in § 266.107(h), line 5, change "his" to "this".

§ 266.108 [Corrected]

67. On page 7225, first column, in § 266.108(a), in the heading, line 1, change "Exemption" to "Exempt", and in the introductory text, line 5, change "section" to "subpart".

68. On page 7225, second column, first column of the table entitled "Exempt Quantities for Small Quantity Burner Exemption" in § 266.108(a)(1), insert "to" between "10.0" and "11.9".

69. On page 7225, second column, in § 266.108(c), in the equation after line 7, change "<1.0" to "≤1.0", and in the line after the equation that reads "Allowable Quantity Burned, means the", delete the comma after "Burned".

§ 266.109 [Corrected]

70. On page 7225, third column, in § 266.109(a)(1)(i), in line 4, change "of" to "on", and in line 8, delete the apostrophe after "fuel" and replace it with an end quotation mark.

71. On page 7226, first column, in § 266.109(a)(2)(iv) introductory text, line 3, change the reference to "(a)(iii)" to "(a)(2)(iii)".

72. On page 7226, first column, in § 266.109(a)(2)(iv)(A), line 2, change "componds" to "compounds".

73. On page 7226, first column, in § 266.109(b)(2), line 2, add "or adjusted Tier I" between "I" and "metals".

§ 266.110 [Corrected]

74. On page 7226, third column, line 4, in § 266.110(f)(3), insert "the" between "of" and "fuel".

75. On page 7227, third column, line 11, "§ 266.122" should correctly be designated as "§ 266.112".

Part 266, Appendix I [Amended]

76. On page 7228, in appendix I to part 266, in the title for Table I-A, change "Carcinogenic" to "Noncarcinogenic".

77. On page 7230, in appendix I to part 266, Table I-D: under "Values for use in rural areas", the first column "Beryllium" is moved so that it appears as the fifth column under "Values for use in urban areas". Also under "Values for use in urban areas", in second column under "Arsenic g/hr", change "9.6E + 01" corresponding to a terrain adjusted eff. stack ht. of 16 meters to "9.6E-01".

78. On page 7230, in appendix I to part 266, Table I-E, in column 2, change "4.3-01" corresponding to "Terrain adjusted eff. stack ht. (m)" of 12 meters in the first column to "4.3E-01".

79. On page 7231, appendix II to part 266 is corrected to read as follows:

Appendix II.-Tier I Feed Rate Screening Limits for Total Chlorine

	Noncomplex Terrain		Complex Terrain
Terrain-adjusted effective stack height (m)	Urban (g/hr)	Rural (g/hr)	(g/hr)
4	8.2E + 01	4.2E + 01	1.9E + 01
6	9.1E + 01	4.8E + 01	2.8E + 01
8	1.0E + 02	5.3E + 01	4.1E + 01
10	1.2E + 02	6.2E + 01	5.8E + 01
12	1.3E + 02	7.7E + 01	7.2E + 01
14	1.5E + 02	9.1E + 01	9.1E + 01
16	1.7E + 02	1.2E + 02	1.1E + 02

18	1.9E + 02	1.4E + 02	1.2E + 02
20	2.1E + 02	1.8E + 02	1.3E + 02
22	2.4E + 02	2.3E + 02	1.4E + 02
24	2.7E + 02	2.9E + 02	1.6E + 02
26	3.1E + 02	3.7E + 02	1.7E + 02
28	3.5E + 02	4.7E + 02	1.9E + 02
30	3.9E + 02	5.8E + 02	2.1E + 02
35	5.3E + 02	9.6E + 02	2.6E + 02
40	6.2E + 02	1.4E + 03	3.3E + 02
45	8.2E + 02	2.0E + 03	4.0E + 02
50	1.1E + 03	2.6E + 03	4.8E + 02
55	1.3E + 03	3.5E + 03	6.2E + 02
60	1.6E + 03	4.6E + 03	7.7E + 02
65	2.0E + 03	6.2E + 03	9.1E + 02
70	2.3E + 03	7.2E + 03	1.1E + 03
75	2.5E + 03	8.6E + 03	1.2E + 03
80	2.9E + 03	1.0E + 04	1.3E + 03
85	3.3E + 03	1.2E + 04	1.4E + 03
90	3.7E + 03	1.4E + 04	1.6E + 03
95	4.2E + 03	1.7E + 04	1.8E + 03
100	4.8E + 03	2.1E + 04	2.0E + 03
105	5.3E + 03	2.4E + 04	2.3E + 03
110	6.2E + 03	2.9E + 04	2.5E + 03
115	7.2E + 03	3.5E + 04	2.8E + 03
120	8.2E + 03	4.1E + 04	3.2E + 03

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80. On pages 7231 and 7232, appendix III to part 266 is corrected to read as follows:

Appendix III.-Tier II Emission Rate Screening Limits for Free Chlorine and Hydrogen Chloride

	Noncomplex terrain				Complex terrain	
Terrain- and adjusted effective stack height (m)	Values for urban areas		Values for rural areas		Values for use in urban rural areas	
	$C1_2$ (g/hr)	HC1 (g/hr)	$C1_2$ (g/hr)	HC1 (g/hr)	$C1_2$ (g/hr)	HC1 (g/hr)
4	8.2E + 01	1.4E + 03	4.2E + 01	7.3E + 02	1.9E + 01	3.3E + 02
6	9.1E + 01	1.6E + 03	4.8E + 01	8.3E + 02	2.8E + 01	4.9E + 02
8	1.0E + 02	1.8E + 03	5.3E + 01	9.2E + 02	4.1E + 01	7.1E + 02
10	1.2E + 02	2.0E + 03	6.2E + 01	1.1E + 03	5.8E + 01	1.0E + 03
12	1.3E + 02	2.3E + 03	7.7E + 01	1.3E + 03	7.2E + 01	1.3E + 03
14	1.5E + 02	2.6E + 03	9.1E + 01	1.6E + 03	9.1E + 01	1.6E + 03
16	1.7E + 02	2.9E + 03	1.2E + 02	2.0E + 03	1.1E + 02	1.8E + 03
18	1.9E + 02	3.3E + 03	1.4E + 02	2.5E + 03	1.2E + 02	2.0E + 03
20	2.1E + 02	3.7E + 03	1.8E + 02	3.1E + 03	1.3E + 02	2.3E + 03
22	2.4E + 02	4.2E + 03	2.3E + 02	3.9E + 03	1.4E + 02	2.4E + 03
24	2.7E + 02	4.8E + 03	2.9E + 02	5.0E + 03	1.6E + 02	2.8E + 03
26	3.1E + 02	5.4E + 03	3.7E + 02	6.5E + 03	1.7E + 02	3.0E + 03
28	3.5E + 02	6.0E + 03	4.7E + 02	8.1E + 03	1.9E + 02	3.4E + 03
30	3.9E + 02	6.9E + 03	5.8E + 02	1.0E + 04	2.1E + 02	3.7E + 03
35	5.3E + 02	9.2E + 03	9.6E + 02	1.7E + 04	2.6E + 02	4.6E + 03
40	6.2E + 02	1.1E + 04	1.4E + 03	2.5E + 04	3.3E + 02	5.7E + 03
45	8.2E + 02	1.4E + 04	2.0E + 03	3.5E + 04	4.0E + 02	7.0E + 03
50	1.1E + 03	1.8E + 04	2.6E + 03	4.6E + 04	4.8E + 02	8.4E + 03

55	1.3E + 03	2.3E + 04	3.5E + 03	6.1E + 04	6.2E + 02	1.1E + 04
60	1.6E + 03	2.9E + 04	4.6E + 03	8.1E + 04	7.7E + 02	1.3E + 04
65	2.0E + 03	3.4E + 04	6.2E + 03	1.1E + 05	9.1E + 02	1.6E + 04
70	2.3E + 03	3.9E + 04	7.2E + 03	1.3E + 05	1.1E + 03	1.8E + 04
75	2.5E + 03	4.5E + 04	8.6E + 03	1.5E + 05	1.2E + 03	2.0E + 04
80	2.9E + 03	5.0E + 04	1.0E + 04	1.8E + 05	1.3E + 03	2.3E + 04
85	3.3E + 03	5.8E + 04	1.2E + 04	2.2E + 05	1.4E + 03	2.5E + 04
90	3.7E + 03	6.6E + 04	1.4E + 04	2.5E + 05	1.6E + 03	2.9E + 04
95	4.2E + 03	7.4E + 04	1.7E + 04	3.0E + 05	1.8E + 03	3.2E + 04
100	4.8E + 03	8.4E + 04	2.1E + 04	3.6E + 05	2.0E + 03	3.5E + 04
105	5.3E + 03	9.2E + 04	2.4E + 04	4.3E + 05	2.3E + 03	3.9E + 04
110	6.2E + 03	1.1E + 05	2.9E + 04	5.1E + 05	2.5E + 03	4.5E + 04
115	7.2E + 03	1.3E + 05	3.5E + 04	6.1E + 05	2.8E + 03	5.0E + 04
120	8.2E + 03	1.4E + 05	4.1E + 04	7.2E + 05	3.2E + 03	5.6E + 04

Part 266, Appendix IV [Amended]

81. On page 7232, in appendix IV to part 266, first column of the table, change "Methyl Ethyl Katone" to "Methyl Ethyl Ketone", and in line 34, change "Metyl Parathion" to "Methyl Parathion".

Part 266, Appendix VII [Amended]

82. On page 7234, first column, in appendix VII to part 266, in the table entitled "Metals-TCLP Extract Concentration Limits": in the heading in the third column of the table, replace "Concentration limits (mg/kg)" with "Concentration limits (mg/L)"; and add the following at the end of the table:

Thallium 7440-28-0 7 X E + 00

83. On page 7234, third column, in appendix VII to part 266, in the table entitled "Nonmetals-Residue Concentration Limits-Continued", delete 8 lines beginning with "Thallium" and ending with "Thallium(l) sulfate.

Part 266, Appendix VIII [Amended]

84. On page 7235, in appendix VIII to part 266, first column of the table entitled "PICS Found in Stack Effluents", change "roform" to "chloroform" and "robenzene" to "chlorobenzene".

PART 270-[AMENDED]

§ 270.22 [Corrected]

85. On page 7235, second column, in § 270.22(a)(2)(ii)(B), line 12, change "Test Methods for the Evaluation of" to "Test Methods for Evaluating".

86. On page 7235, second column, in § 270.22(a)(2)(ii)(C), line 5, the reference to "(a)(1)(ii)(B)" should read "(a)(2)(ii)(B)".

87. On page 7236, first column, in § 270.22(a)(5)(vii), line 4, change "feestocks" to "feedstocks".

88. On page 7236, first column, in § 270.22(a)(6), line 1, change "trail" to "trial".

89. On page 7236, second column, in § 270.22(a)(6), line 8, change "from from" to "from" i.e. delete one "from" as it is duplicative.

90. On page 7236, second column, in § 270.22(b)(1), line 2, change "minimze" to "minimize".

§ 270.42 [Corrected]

91. On page 7237, first column, in § 270.42(g)(1) introductory text, line 3, change "wates" to "wastes".

92. On page 7237, first column, in § 270.42(g)(1)(i), line 5, change "effetive" to "effective".

93. On page 7237, second column, in § 270.42(g)(1)(iv), in lines 1 and 2, delete "In the case of Classes 2 and 3 modifications,"; in line 2, capitalize the "t" in "the"; and insert "Class 2 or 3" between "complete" and "modification" so that paragraph (iv) reads as follows: "The permittee also submits a complete Class 2 or 3 modification request within 180 days of the effective date of the rule listing or identifying the waste, or subjecting the unit to RCRA Subtitle C management standards;".

94. On page 7237, in appendix I to § 270.42, in line 1 of "L.5.", replace the period after "requirements" with a colon.

95. On page 7237, third column, in amendment 4 to part 270, the amendatory language is corrected to read as follows: "4. In § 270.42, appendix I is amended by revising the heading of L and items 1 through 4, 5a, 6, 7b, and 8 to read as follows:"

§ 270.66 [Corrected]

96. On page 7237, third column, line 2, in § 270.66(b)(1), change "operation" to "operational".

97. On page 7238, second column, in § 270.66(b)(4), line 8, change "107" to "266.107".

98. On page 7238, at two locations, lines 13, 14, and 15, in § 270.66(c)(2)(i) and in lines 5 and 6 in § 270.66(c)(2)(ii), change "Test Methods for the Evaluation of Solid Waste" to "Test Methods for Evaluating Solid Waste".

99. On page 7238, third column, in § 270.66(c)(3)(vi), line 3, delete "and".

100. On page 7238, third column, in § 270.66(c)(3)(vii), line 1, insert "air" between "any" and "pollution".

101. On page 7239, second column, in § 270.66(f)(3), line 10, replace the period after "standard" with a semicolon.

102. On page 7239, second column, in § 270.66(f)(8), line 5, change "is" to "in".

§ 270.33 [Corrected]

103. On page 7239, third column, in § 270.73 at two locations, in paragraph (f), line 2, and in paragraph (g), line 3, change "as" to "has".

104. On page 7240, in § 271.1(j), Table 1, third column, replace "[insert FR page numbers]" with "56 FR 7134-7240".

B. Technical Amendments

For the reasons set out in the preamble, 40 CFR part 261 is amended as follows:

PART 261-IDENTIFICATION AND LISTING OF HAZARDOUS WASTE

1. The authority citation for part 261 continues to read as follows:

Authority: 42 U.S.C. 6905, 6912(a), 6921, 6922, and 6938.

§ 261.3 [Amended]

2. In § 261.3(c)(2)(ii)(8), line 3 is amended by replacing "by § 261.6(a)(3) (v) through (ix)" with "by § 261.6(a)(3) (v) through (viii)".

§ 261.6 [Amended]

3. In § 261.6(a)(2), line 4, the letter "G" is amended to read "H". The entire line should now read as: "subparts C through H of part 266 of".

4. In § 261.6(a)(2)(ii), line 5, the reference to "subpart D" should be replaced by "subpart H".

For the reasons set out in the preamble, 40 CFR part 265 is amended as follows:

PART 265-INTERIM STATUS STANDARDS FOR OWNERS AND OPERATORS OF HAZARDOUS WASTE TREATMENT, STORAGE, AND DISPOSAL FACILITIES

1. The authority citation for part 265 continues to read as follows:

Authority: 42 U.S.C. 6905, 6912(a), 6924, 6925, 6935.

§ 265.370 [Amended]

2. § 265.370 is amended by deleting the period (.) at the end and replacing it with the following: ", and subpart H of part 266, if the unit is a boiler or an industrial furnace as defined in § 260.10."

For the reasons set out in the preamble, 40 CFR part 270 is amended as follows:

PART 270-EPA ADMINISTERED PERMIT PROGRAMS: THE HAZARDOUS WASTE PERMIT PROGRAM

1. The authority citation for part 270 continues to read as follows:

Authority: 42 U.S.C. 6905, 6912, 6924, 6925, 6927, 6939, 6974.

§ 270.1 [Amended]

2. § 270.1(b) is amended by replacing "40 CFR part 265" in line 44 by "40 CFR parts 265 and 266".

§ 270.42 [Amended]

3. Section 270.42(c)(1)(iv) is revised to read as follows:

(c) * * *

(1) * * *

(iv) Provides the applicable information required by 40 CFR 270.13 through 270.22, 270.62, 270.63, and 270.66.

* * * * *

For the reasons set out in the preamble, 40 CFR part 266 is amended as follows:

PART 266-STANDARDS FOR THE MANAGEMENT OF SPECIFIC HAZARDOUS WASTES AND SPECIFIC TYPES OF HAZARDOUS WASTE MANAGEMENT FACILITIES

1. The authority citation for part 266 continues to read as follows:

Authority: Secs. 1006, 2002(a), 3004, and 3014 of the Solid Waste Disposal Act, as amended (42 U.S.C. 6905, 6912(a), 6924, and 6934).

§ 266.4 [Amended]

2. Section 266.40(c) is amended by replacing "subpart D" in line 6 by "subpart H."

3. Section 266.40(d) is amended by replacing "subpart D" in line 4 by "subpart H".

4. Part 266 is amended by adding two appendices, appendices IX and X as follows:

Appendix IX to Part 266-Methods Manual for Compliance With the BIF Regulations

Burning Hazardous Waste in Boilers and Industrial Furnaces

Table of Contents

Section 1.0 INTRODUCTION

This document presents required methods for demonstrating compliance uith U.S. Environmental Protection Agency regulations for boilers and industrial furnaces (BIFs) burning hazardous waste (see 40 CFR part 266, subpart H). Included in this document are:

1. Performance Specifications for Continuous Emission Monitoring (CEM) of Carbon Monoxide, Oxygen, and Hydrocarbons in Stack Gases.

2. Sampling and Analytical (S&A) Methods for Multiple Metals, Hexavalent Chromium, HCl and Chlorine, Polychlorinated Dibenzo-p-dioxins and Dibenzofurans, and Aldehydes and Ketones.

3. Procedures for Estimating the Toxicity Equivalency of Chlorinated Dibenzo-p-dioxin and Dibenzofuran Congeners.

4. Hazardous Waste Combustion Air Quality Screening Procedures (HWCAQSP).

5. Simplified Land Use Classification Procedure for Compliance vith Tier I and Tier II Limits.

6. Statistical Methodology for Bevill Residue Determinations.

7. Procedures for Determining Default Values for Air Pollution Control System Removal Efficiencies.

8. Procedures for Determining Default Values for Partitioning of Metals, Ash, and Total Chloride/Chlorine.

9. Alternate Methodology for Implementing Metals Controls.

Additional methods referenced in subpart H of part 266 but not included in this document can be found in 40 CFR parts 60 and 61, and "Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods" (SW-846).

The CEM performance specifications of section 2.0, the S&A methods of section 3.0 and the toxicity equivalency procedure for dioxins and furans of section 4.0 are required procedures for determining compliance with BIF regulations. The CEM performance specifications and the S&A methods are interim. The finalized CEM performance specifications and methods will be published in SW-846 or 40 CFR parts 60 and 6l.

SECTION 2.0 PERFORMANCE SPECIFICATIONS FOR CONTINUOUS EMISSION MONITORING SYSTEMS

2.l Performance Specifications for Continuous Emission Monitoring of Carbon Monoxide and Oxygen for Incinerators, Boilers, and Industrial Furnaces Burning Hazardous Waste

2.1.1 Applicability and Principle

2.1.1.1 Applicability. These performance specifications apply to carbon monoxide (CO) and oxygen (O_2) continuous emission monitoring systems (CEMSs) installed on incinerators, boilers, and industrial furnaces burning hazardous waste. The specifications include procedures which are intended to be used to evaluate the acceptability of the CEMS at the time of its installation or whenever specified in regulations or permits. The procedures are not designed to evaluate CEMS performance over an extended period of time. The source owner or operator is responsible for the proper calibration, maintenance, and operation of the CEMS at all times.

2.1.1.2 Principle. Installation and measurement location specifications, performance and equipment specifications, test and data reduction procedures, and brief quality assurance guidelines are included in the specifications. Calibration drift, relative accuracy, calibration error, and response time tests are conducted to determine conformance of the CEMS with the specifications.

2.1.2 Definitions

2.1.2.1 Continuous Emission Monitoring System (CEMS). A continuous monitor is one in which the sample to be analyzed passes the measurement section of the analyzer without interruption, and which evaluates the detector response to the sample at least once each 15 seconds and computes and records the results at least every 60 seconds. A CEMS consists of all the equipment used to acquire data and includes the sample extraction and transport hardware, the analyzer(s), and the data recording/processing hardware and software.

2.1.2.2 Monitoring System Types. The specifications require CEMSs capable of accepting calibration gases. Alternative system designs may be used if approved by the Regional Administrator. There are two basic types of monitoring systems: extractive and in-situ.

2.1.2.2.1 Extractive. Systems that use a pump or other mechanical, pneumatic, or hydraulic means to draw a sample of the stack or flue gas and convey it to a remotely located analyzer.

2.1.2.2.2 In-situ. Systems that perform an analysis without removing a sample from the stack. Point in-situ analyzers place the sensing or detecting element directly in the flue gas stream. Cross-stack in-situ analyzers measure the parameter of interest by placing a source beam on one side of the stack and the detector (in single-pass instruments) or a retroreflector (in double-pass instruments) on the other side, and measuring the parameter of interest (e.g., CO) by the attenuation of the beam by the gas in its path.

2.1.2.3 Instrument Measurement Range. The difference between the minimum and maximum concentration that can be measured by a specific instrument. The minimum is often stated or assumed to be zero and the range expressed only as the maximum.

2.1.2.4 Span or Span Value. Full scale instrument measurement range.

2.1.2.5 Calibration Drift (CD). The difference in the CEMS output readings from the established reference value after a stated period of operation during which no unscheduled maintenance, repair, or adjustment takes place. A CD test is performed to demonstrate the stability of the CEMS calibration over time.

2.1.2.6 Response Time. The time interval between the start of a step change in the system input (e.g., change of calibration gas) and the time when the data recorder displays 95 percent of the final value.

2.1.2.7 Accuracy. A measure of agreement between a measured value and an accepted or true value, expressed as the percentage difference between the true and measured values relative to the true value. For these performance specifications, accuracy is checked by conducting a calibration error (CE) test and a relative accuracy (RA) test. Certain facilities, such as those using solid waste or batch-fed processes, may observe long periods of almost no CO emissions with brief, high-level CO emission spikes. These facilities, as well as facilities whose CO emissions never exceed 5-10 ppm, may need to be exempted from the RA requirement because the RA test procedure cannot ensure acquisition of meaningful test results under these conditions. An alternative procedure for accuracy determination is described in section 2.1.9.

2.1.2.8 Calibration Error (CE). The difference between the concentration indicated by the CEMS and the known concentration of the cylinder gas. A CE test procedure is performed to document the accuracy and linearity of the monitoring equipment over the entire measurement range.

2.1.2.9 Relative Accuracy (RA). A comparison of the CEMS response to a value measured by a performance test method (PTM). The PA test is used to validate the calibration technique and verify the ability of the CEMS to provide representative and accurate measurements.

2.1.2.10 Performance Test Method (PTM). The sampling and analysis procedure used to obtain reference measurements for comparison to CEMS measurements. The applicable test methods are Method 10, 10A, or 10B (for the determination of CO) and Method 3 or 3A (for the determination of 0^2). These methods are found in 40 CFR part 60, appendix A.

2.1.2.11 Performance Specification Test (PST) Period. The period during which CD, CE, response time, and RA tests are conducted.

2.1.2.12 Centroidal Area. A concentric area that is geometrically similar to the stack or duct cross section and is no greater than 1 percent of the stack or duct cross-sectional area.

2.1.3 Installation and Measurement Location Specifications

2.1.3.1 CEMS Installation and Measurement Locations. The CEMS shall be installed in a location in which measurements representative of the source's emissions can be obtained. The optimum location of the sample interface for the CEMS is determined by a number of factors, including ease of access for calibration and maintenance, the degree to which sample conditioning will be required, the degree to which it represents total emissions, and the degree to which it represents the combustion situation in the firebox. The location should be as free from in-leakage influences as possible and reasonably free from severe flow disturbances. The sample location should be at least two equivalent duct diameters downstream from the nearest control device, point of pollutant generation, or other point at which a change in the pollutant concentration or emission rate occurs and at least 0.5 diameter upstream from the exhaust or control device. The equivalent duct diameter is calculated as per 40 CFR part 60, appendix A, method 1, section 2.1. If these criteria are not achievable or if the location is otherwise less than optimum, the possibility of stratification should be checked as described in Section 2.1.3.3 to determine whether the location would cause failure of the relative accuracy test.

2.1.3.1.1 For extractive or point in-situ CEMSs, the measurement point should be within or centrally located over the centroidal area of the stack or duct cross section.

2.1.3.1.2 For cross-stack CEMSs, the effective measurement path should (1) have at least 70 percent of the path within the inner 50 percent of the stack or duct cross-sectional area or (2) be centrally located over any part of the centroidal area.

2.1.3.1.3 Both the CO and O_2 monitors should be installed at the same general location. If this is not possible, they may be installed at different locations if the effluent gases at both sample locations are not stratified and there is no in-leakage of air between sampling locations.

2.1.3.2 Performance Test Method (PTM) Measurement Location and Traverse Points.

2.1.3.2.1 Select an accessible PTM measurement point at least two equivalent diameters downstream from the nearest control device, the point of CO generation, or other point at which a change in the CO concentration may occur, and at least a half equivalent diameter upstream from the effluent exhaust or control device. When pollutant concentration changes are due solely to diluent leakage (e.g., air heater leakages) and CO and O_2 are simultaneously measured at the same location, one half diameter may be used in place of two equivalent diameters. The CEMS and PTM locations need not be the same.

2.1.3.2.2 Select traverse points that ensure acquisition of representative samples over the stack or duct cross section. At a minimum, establish a measurement line that passes through the centroidal area in the direction of any expected stratification. If this line interferes with the CEMS measurements, displace the line up to 30 cm (or 5 percent of the equivalent diameter of the cross section, whichever is less) from the centroidal area. Locate three traverse points at 17, 50, and 83 percent of the measurement line. If the measurement line is no longer than 2.4 meters and pollutant stratification is not expected, the tester may choose to locate the three traverse points on the line at 0.4, 1.2, and 2.0 meters from the stack or duct wall. This option must not be used at a site located within eight equivalent diameters downstream of a flow disturbance. The tester may select other traverse points, provided that they can be shown to the satisfaction of the Administrator to provide a representative sample over the stack or duct cross-section. Conduct all necessary PTM tests within 3 cm of the selected traverse points. Sampling must not be performed within 3 cm of the duct or stack inner wall.

2.1.3.3 Stratification Test Procedure. Stratification is defined as a difference in excess of 10 percent between the average concentration in the duct or stack and the concentration at any point more than 1.0 meter from the duct or stack wall. To determine whether effluent stratification exists, a dual probe system should be used to determine the average effluent concentration while measurements at each traverse point are being made. One probe, located at the stack or duct centroid, is used as a stationary reference point to indicate the change in effluent concentration over time. The second probe is used for sampling at the traverse points specified in method 1, appendix A, 40 CFR part 60. The monitoring system samples sequentially at the reference and traverse points throughout the testing period for five minutes at each point.

2.1.4 CEMS Performance and Equipment Specifications

Table 2.1-1 summarizes the performance specifications for the CEMSs. Two sets of standards for CO are given; one for low-range and another for high-range measurements. The high-range specifications relate to measurement and quantification of short duration high concentration peaks, while the low-range specifications relate to the overall average operating condition of the burning device. The dual-range specifications can be met by using (1) one analyzer for each range, (2) a dual range unit, or (3) a single measurement range instrument capable of meeting both specifications with a single unit. Adjustments cannot be made to the analyzer between determinations of low- and high-level accuracy within the single measurement range. In the second case, when the concentration exceeds the span of the lower range, the data acquisition system recorder shall switch to the high range automatically.

2.1.4.1 CEMS Span Value. In order to measure high and low concentrations with the same or similar degree of accuracy, the maximum ranges (span values) are specified for low and high range analyzers. The span values are listed in Table 2.1-2. Tier I and Tier II format definitions are established in 40 CFR part 266, subpart H.

Table 2.1-1-Performance Specifications of CO and O_2 Monitors

	CO monitors		
Parameter	Low range	High range	O_2 monitors
Calibration drift 24 hours.	$\leq$6 ppm[1]	$\leq$90 ppm	$\leq$0.5% O_2
Calibration error.	$\leq$10 ppm[1]	$\leq$150 ppm	$\leq$0.5% O_2
Response time.	$\leq$2 min	$\leq$2 min	$\leq$2 min
Relative accuracy[2].	(3)	(3)	(incorporated in CO RA calculation)

[1] For Tier II, CD and CE are $\leq$3% and $\leq$5% of twice the permit limit, respectively.
[2] Expressed as the sum of the mean absolute value plus the 95% confidence interval of a series of measurements.
[3] The greater of 10% of PTM or 10 ppm.

Table 2.1-2-CEMS Span Values for CO and O_2 Monitors

	CO monitors		
	Low range (ppm)	High range (ppm)	O_2 monitors (percent)
Tier I rolling average format.	200	3,000	25
Tier II rolling average format.	2 X permit limit.	3,000	25

2.1.4.2 Daily Calibration Gas Values. The owner or operator must choose calibration gas concentrations (or calibration filters for in-situ systems) that include zero and high-level calibration values for the daily calibration checks. For a single measurement range monitor, three CO calibration gas concentrations (or calibration filters for in-situ systems) shall be used, i.e., the zero and high-level concentrations of the low-range CO analyzer and the high-level concentration of the high-range CO analyzer.

2.1.4.2.1 The zero level for the CO or O_2 analyzer may be between zero and 20 percent of the span value, e.g., 0-40 ppm for low-range CO analyzer, 0-600 ppm for the high-range CO analyzer, and 0-5 percent for the O_2 analyzer (for Tier I).

2.1.4.2.2 The high-level concentration for the CO or O_2 analyzer shall be between 50 and 90 percent of the span value, i.e., 100-180 ppm for the low-range CO analyzer, 1500-2700 ppm for the high-range CO analyzer, and 12.5-22.5 percent O_2 for the O_2 analyzer.

2.1.4.3 Data Recorder Scale. The strip chart recorder, computer, or digital recorder must be capable of recording all readings within the CEMS's measurement range and shall have a resolution of 0.5 percent of span value, i.e., 1 ppm CO for low-range CO analyzer, 15 ppm CO for high-range CO analyzer, and 0.1 percent O_2 for the O_2 analyzer.

2.1.4.4 Response Time. The response time for the CO or O_2 monitor shall not exceed 2 minutes to achieve 95 percent of the final stable value.

2.1.4.5 Calibration Drift. The CEMS must allow the determination of CD at the zero and high-level values. The CD must be determined separately for CO and O_2 monitors in terms of concentration. The CO CEMS calibration response must not drift or deviate from the reference value of the calibration gas (or calibration filters for in-situ systems) by more than 3 percent of the span value after each 24-hour period of the 7-day test, i.e., 6 ppm CO for the low-range analyzer (Tier I) and 90 ppm for the high-range analyzer, at both zero and high levels. The O_2 monitor calibration response must not drift or deviate from the reference value by more than 0.5 percent O_2 at both zero and high levels.

2.l.4.6 Relative Accuracy. The result of the PA test of the CO CEMS (which incorporates the O_2 monitor) must be no greater than 10 percent of the mean value of the PTM results or must be within 10 ppm CO of the PTM results, whichever is less restrictive. The ppm CO concentration shall be corrected to 7 percent O_2 before calculating the RA.

2.1.4.7 Calibration Error. The mean difference between the CEMS and reference values at all three test points (see Table 2.1-3) must be no greater than 5 percent of span value for CO monitors (i.e., 10 ppm CO for low range Tier I CO analyzers and 150 ppm CO for high range CO analyzers) and 0.5 percent for O_2 analyzers.

2.1.4.8 Measurement and Recording Frequency. The sample to be analyzed shall pass through the measurement section of the analyzer without interruption. The detector shall measure the sample concentration at least once every 15 seconds. An average emission rate shall be computed and recorded at least once every 60 seconds.

2.1.4.9 Hourly Rolling Average Calculation. The CEMS shall calculate every minute an hourly rolling average, which is the arithmetic mean of the 60 most recent 1-minute average values.

2.1.4.10 Retest. If the CEMS produces results within the specified criteria, the test is successful. If the CEMS does not meet one or more of the criteria, the necessary corrections must be made and the performance tests repeated.

2.1.5 Test Periods

2.1.5.1 Pretest Preparation Period. Install the CEMS, prepare the PTM test site according to the specifications in section 2.1.3, and prepare the CEMS for operation and calibration according to the manufacturer's written instructions. A pretest conditioning period similar to that of the 7-day CD test is recommended to verify the operational status of the CEMS.

2.1.5.2 Calibration Drift Test Period. While the facility is operating under normal conditions, determine the CD at 24-hour intervals for seven consecutive days according to the procedure given in section 2.1.6.1. All CD determinations must be made following a 24-hour period during which no unscheduled maintenance, repair, or adjustment takes place. If the combustion unit is taken out of service during the test period, record the onset and duration of the downtime and continue the calibration drift test when the unit resumes operation.

2.1.5.3 Relative Accuracy Test Period. Conduct the RA test according to the procedure in section 2.1.6.4 while the facility is operating under normal conditions. RA testing for CO and O_2 shall be conducted simultaneously so that the results can be calculated for CO corrected to 7 percent O_2. The RA test shall be conducted during the CD test period. It is emphasized that during the CD test period, no adjustments or repairs may be made to the CEMS other than routine calibration adjustments performed immediately following the daily CD determination.

2.1.5.4 Calibration Error Test and Response Time Test Periods. Conduct the CE and response time tests during the CD test period.

2.1.6 Performance Specification Test Procedures

2.1.6.1 Calibration Drift Test.

2.1.6.1.1 Sampling Strategy. Conduct the CD test for all monitors at 24-hour intervals for seven consecutive days using calibration gases at the two (or three, if applicable) concentration levels specified in section 2.1.4.2. Introduce the calibration gases into the sampling system as close to the sampling probe outlet as practical. The gas shall pass through all filters, scrubbers, conditioners, and other CEMS components used during normal sampling. If periodic automatic or manual adjustments are made to the CEMS zero and calibration settings, conduct the CD test immediately before these adjustments, or conduct it in such a way that the CD can be determined. Record the CEMS response and subtract this value from the reference (calibration gas) value. To meet the specification, none of the differences shall exceed the limits specified in Table 2.1-1.

2.1.6.1.2 Calculations. Summarize the results on a data sheet. An example is shown in Figure 2.1-1. Calculate the differences between the CEMS responses and the reference values.

2.1.6.2 Response Time. Check the entire CEMS including sample extraction and transport, sample conditioning, gas analyses, and the data recording.

2.1.6.2.1 Introduce zero gas into the system. For extractive systems, introduce the calibration gases at the probe as near to the sample location as possible. For in-situ system, introduce the zero gas at a point such that all components active in the analysis are tested. When the system output has stabilized (no change greater than 1 percent of full scale for 30 seconds), switch to monitor stack effluent and wait for a stable value. Record the time (upscale response time) required to reach 95 percent of the final stable value.

2.1.6.2.2 Next, introduce a high-level calibration gas and repeat the above procedure. Repeat the entire procedure three times and determine the mean upscale and downscale response times. The longer of the two means is the system response time.

2.1.6.3 Calibration Error Test Procedure.

2.1.6.3.1 Sampling Strategy. Challenge each monitor (both low- and high-range CO and O_2) with zero gas and EPA Protocol 1 cylinder gases at three measurement points within the ranges specified in Table 2.1-3.

Table 2.1-3-Calibration Error Concentration Ranges for Tier I

	GAS Concentration Ranges		
	CO, ppm		
Measurement point	Low range [1]	High range	O_2 percent
1	0-40	0-600	0-2
2	60-80	900-1200	8-10
3	140-160	2100-2400	14-16

[1] For Tier II, the CE specifications for the low-range CO CEMS are 0-20%, 30-40%, and 70-80% of twice the permit limit.

>>>> See the accompanying hardcopy volume for non-machine-readable data that appears at this point. <<<<

2.1.6.3.1.1 If a single measurement range is used, the calibration gases used in the daily CD checks (if they are Protocol 1 cylinder gases and meet the criteria in section 2.1.6.3.1) may be used for determining CE.

2.1.6.3.1.2 Operate each monitor in its normal sampling mode as nearly as possible. The calibration gas shall be injected into the sample system as close to the sampling probe outlet as practical and should pass through all CEMS components used during normal sampling. Challenge the CEMS three non-consecutive times at each measurement point and record the responses. The duration of each gas injection should be sufficient to ensure that the CEMS surfaces are conditioned.

2.1.6.3.2 Calculations. Summarize the results on a data sheet. An example data sheet is shown in Figure 2.1-2. Average the differences between the instrument response and the certified cylinder gas value for each gas. Calculate three CE results (five CE results for a single-range CO CEMS) according to Equation 5 (section 2.1.7.5). No confidence coefficient is used in CE calculations.

2.1.6.4 Relative Accuracy Test Procedure.

2.1.6.4.1 Sampling Strategy for PTM tests. Conduct the PTM tests in such a way that they will yield measurements representative of the emissions from the source and can be correlated to the CEMS data. Although it is preferable to conduct the CO, diluent, and moisture (if needed) simultaneously, moisture measurements that are taken within a 60-minute period which includes the simultaneous CO and O_2 measurements may be used to calculate the dry CO concentration.

Note: At times, CEMS RA tests may be conducted during incinerator performance tests. In these cases, PTM results obtained during CEMS RA tests may be used to determine compliance with incinerator emissions limits as long as the source and test conditions are consistent with the applicable regulations.

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2.1.6.4.2 Performance Test Methods.

2.1.6.4.2.1 Unless otherwise specified in the regulations, method 3 or 3A and method 10, 10A, or 10B (40 CFR part 60, appendix A) are the test methods for O_2 and CO, respectively. Make a sample traverse of at least 21 minutes, sampling for 7 minutes at each of three traverse points (see section 3.2).

2.1.6.4.2.2 When the installed CEMS uses a nondispersive infrared (NDIR) analyzer, method 10 shall use the alternative interference trap specified in section 10.1 of the method. An option, which may be approved by the Administrator in certain cases, would allow the test to be conducted using method 10 without the interference trap. Under this option, a laboratory interference test is performed for the analyzer prior to the field test. The laboratory interference test includes the analysis of SO_2, NO, and CO_2 calibration gases over the range of expected effluent concentrations. Acceptable performance is indicated if the CO analyzer response to each of the gases is less than 1 percent of the applicable measurement range of the analyzer.

2.1.6.4.3 Number of PTM Tests. Conduct a minimum of nine sets of all necessary PTM tests. If more than nine sets are conducted, a maximum of three sets may be rejected at the tester's discretion. The total number of sets used to determine the RA must be greater than or equal to nine. All data, including the rejected data, must be reported.

2.1.6.4.4 Correlation of PTM and CEMS Data. The time and duration of each PTM test run and the CEMS response time should be considered in correlating the data. Use the CEMS final output (the one used for reporting) to determine an integrated average CO concentration for each PTM test run. Confirm that the pair of results are on a consistent moisture and O_2 concentration basis. Each integrated CEMS value should then be compared against the corresponding average PTM value. If the CO concentration measured by the CEMS is normalized to a specified diluent concentration, the PTM results shall be normalized to the same value.

2.1.6.4.5 Calculations. Summarize the results on a data sheet. Calculate the mean of the PTM values and calculate the arithmetic differences between the PTM and the CEMS data sets. The mean of the differences, standard deviation, confidence coefficient, and CEMS RA should be calculated using Equations 1 through 4.

2.1.7 Equations

2.1.7.1 Arithmetic Mean (p). Calculate , p of the difference of a data set using Equation 1.

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where: n = Number of data points.

n

d_i = Algebraic sum of the individual difference d_i.

E

i = 1

When the mean of the differences of pairs of data is calculated, correct the data for moisture, if applicable.

2.1.7.2 Standard Deviation (S_d). Calculate S_d using Equation 2.

>>>> See the accompanying hardcopy volume for non-machine-readable data that appears at this point. <<<<

2.1.7.3 Confidence Coefficient (CC). Calculate the 2.5 percent error CC (one-tailed) using Equation 3.

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where:

$t_{0.975}$ = t-value (see Table 2.1-4).

Table 2.1-4-t-Values

n^a	$t_{0.975}$	n^a	$t_{0.975}$	n^a	$t_{0.975}$
2	12.706	7	2.447	12	2.201
3	4.303	8	2.365	13	2.179
4	3.182	9	2.306	14	2.160
5	2.776	10	2.662	15	2.145
6	2.571	11	2.228	16	2.131

[a] The values in this table are already corrected for n-1 degrees of freedom. Use n equal to the number of individual values.

2.1.7.4 Relative Accuracy. Calculate the RA of a set of data using Equation 4.

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where:

p = Absolute value of the mean of the differences (Equation 1).
CC = Absolute value of the confidence coefficient (Equation 3).

PTM = Average reference value.

2.1.7.5 Calibration Error. Calculate CE using Equation 5.

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where:

p = Mean difference between CEMS response and the known reference concentration.

2.1.8 Reporting

At a minimum, summarize in tabular form the results of the CD, RA, response time, and CE test, as appropriate. Include all data sheets, calculations, CEMS data records, and cylinder gas or reference material certifications.

2.1.9 Alternative Procedure

2.1.9.1 Alternative RA Procedure Rationale. Under some operating conditions, it may not be possible to obtain meaningful results using the RA test procedure. This includes conditions where consistent, very low CO emissions or low CO emissions interrupted periodically by short duration, high level spikes are observed. It may be appropriate in these circumstances to waive the PTM RA test and substitute the following procedure.

2.1.9.2 Alternative RA Procedure. Conduct a complete CEMS status check following the manufacturer's written instructions. The check should include operation of the light source, signal receiver, timing mechanism functions, data acquisition and data reduction functions, data recorders, mechanically operated functions (mirror movements, calibration gas valve operations, etc.), sample filters, sample line heaters, moisture traps, and other related functions of the CEMS, as applicable. All parts of the CEMS must be functioning properly before the RA requirement can be waived. The instruments must also have successfully passed the CE and CD requirements of the performance specifications. Substitution of the alternative procedure requires approval of the Regional Administrator.

2.1.10 Quality Assurance (QA)

Proper calibration, maintenance, and operation of the CEMS is the responsibility of the owner or operator. The owner or operator must establish a QA program to evaluate and monitor CEMS performance. As a minimum, the QA program must include:

2.1.10.1 A daily calibration check for each monitor. The calibration must be adjusted if the check indicates the instrument's CD exceeds the specification established in section 2.1.4.5. The gases shall be injected as close to the probe as possible to provide a check of the entire sampling system. If an alternative calibration procedure is desired (e.g., direct injections or gas cells), subject to Administrator approval, the adequacy of this alternative procedure may be demonstrated during the initial 7-day CD test. Periodic comparisons of the two procedures are suggested.

2.1.10.2 A daily system audit. The audit must include a review of the calibration check data, an inspection of the recording system, an inspection of the control panel warning lights, and an inspection of the sample transport and interface system (e.g., flowmeters, filters), as appropriate.

2.1.10.3 A quarterly calibration error (CE) test. Quarterly RA tests may be substituted for the CE test when approved by the Director on a case-by-case basis.

2.1.10.4 An annual performance specification test.

2.1.11 References

1. Jahnke, James A. and G.J. Aldina, "Handbook: Continuous Air Pollution Source Monitoring Systems," U.S. Environmental Protection Agency Technology Transfer, Cincinnati, Ohio 45268, EPA-625/6-79-005, June 1979.

2. "Gaseous Continuous Emissions Monitoring Systems-Performance Specification Guidelines for SO_2, NO_x, CO_2, O_2, and TRS." U.S. Environmental

Protection Agency OAQPS, ESED, Research Triangle Park, North Carolina 27711, EPA-450/3-82-026, October 1982.

3. "Quality Assurance Handbook for Air Pollution Measurement Systems: Volume I. Principles." U.S. Environmental Protection Agency ORD/EMSL, Research Triangle Park, North Carolina, 27711, EPA-600/9-76-006, December 1984.

4. Michie, Raymond, M. Jr., et. al., "Performance Test Results and Comparative Data for Designated Reference Methods for Carbon Monoxide," U.S. Environmental Protection Agency ORD/EMSL, Research Triangle Park, North Carolina, 27711, EPA-600/S4-83-013, September 1982.

5. Ferguson, B.B., R.E. Lester, and W.J. Mitchell, "Field Evaluation of Carbon Monoxide and Hydrogen Sulfide Continuous Emission Monitors at an Oil Refinery," U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 27711, EPA-600/4-82-054, August 1982.

2.2 Performance Specifications for Continuous Emission Monitoring of Hydrocarbons for Incinerators, Boilers, and Industrial Furnaces Burning Hazardous Waste

2.2.1 Applicability and Principle

2.2.1.1 Applicability. These performance specifications apply to hydrocarbon (HC) continuous emission monitoring systems (CEMSs) installed on incinerators, boilers, and industrial furnaces burning hazardous waste. The specifications include procedures which are intended to be used to evaluate the acceptability of the CEMS at the time of its installation or whenever specified in regulations or permits. The procedures are not designed to evaluate CEMS performance over an extended period of time. The source owner or operator is responsible for the proper calibration, maintenance, and operation of the CEMS at all times.

2.2.1.2 Principle. A gas sample is extracted from the source through a heated sample line and heated filter (except as provided by section 2.2.10) to a flame ionization detector (FID). Results are reported as volume concentration equivalents of propane. Installation and measurement location specifications, performance and equipment specifications, test and data reduction procedures, and brief quality assurance guidelines are included in the specifications. Calibration drift, calibration error, and response time tests are conducted to determine conformance of the CEMS with the specifications.

2.2.2 Definitions

2.2.2.1 Continuous Emission Monitoring System (CEMS). The total equipment used to acquire data, which includes sample extraction and transport hardware, analyzer, data recording and processing hardware, and software. The system consists of the following major subsystems:

2.2.2.1.1 Sample Interface. That portion of the system that is used for one or more of the following: Sample acquisition, sample transportation, sample conditioning, or protection of the analyzer from the effects of the stack effluent.

2.2.2.1.2 Organic Analyzer. That portion of the system that senses organic concentration and generates an output proportional to the gas concentration.

2.2.2.1.3 Data Recorder. That portion of the system that records a permanent record of the measurement values. The data recorder may include automatic data reduction capabilities.

2.2.2.2 Instrument Measurement Range. The difference between the minimum and maximum concentration that can be measured by a specific instrument. The minimum is often stated or assumed to be zero and the range expressed only as the maximum.

2.2.2.3 Span or Span Value. Full scale instrument measurement range.

2.2.2.4 Calibration Gas. A known concentration of a gas in an appropriate diluent gas.

2.2.2.5 Calibration Drift (CD). The difference in the CEMS output readings from the established reference value after a stated period of operation during which no unscheduled maintenance, repair, or adjustment takes place. A CD test is performed to demonstrate the stability of the CEMS calibration over time.

2.2.2.6 Response Time. The time interval between the start of a step change in the system input (e.g., change of calibration gas) and the time when the data recorder displays 95 percent of the final value.

2.2.2.7 Accuracy. A measurement of agreement between a measured value and an accepted or true value, expressed as the percentage difference between the true and measured values relative to the true value. For these performance specifications, accuracy is checked by conducting a calibration error (CE) test.

2.2.2.8 Calibration Error (CE). The difference between the concentration indicated by the CEMS and the known concentration of the cylinder gas. A CE test procedure is performed to document the accuracy and linearity of the monitoring equipment over the entire measurement range.

2.2.2.9 Performance Specification Test (PST) Period. The period during which CD, CE, and response time tests are conducted.

2.2.2.10 Centroidal Area. A concentric area that is geometrically similar to the stack or duct cross section and is no greater than 1 percent of the stack or duct cross-sectional area.

2.2.3 Installation and Measurement Location Specifications

2.2.3.1 CEMS Installation and Measurement Locations. The CEMS shall be installed in a location in which measurements representative of the source's emissions can be obtained. The optimum location of the sample interface for the CEMS is determined by a number of factors, including ease of access for calibration and maintenance, the degree to which sample conditioning will be required, the degree to which it represents total emissions, and the degree to which it represents the combustion situation in the firebox. The location should be as free from in-leakage influences as possible and reasonably free from severe flow disturbances. The sample location should be at least two equivalent duct diameters downstream from the nearest control device, point of pollutant generation, or other point at which a change in the pollutant concentration or emission rate occurs and at least 0.5 diameter upstream from the exhaust or control device. The equivalent duct diameter is calculated as per 40 CFR part 60, appendix A, method 1, section 2.1. If these criteria are not achievable or if the location is otherwise less than optimum, the possibility of stratification should be investigated as described in section

2.2.3.2. The measurement point shall be within the centroidal area of the stack or duct cross section.

2.2.3.2 Stratification Test Procedure. Stratification is defined as a difference in excess of 10 percent between the average concentration in the duct or stack and the concentration at any point more than 1.0 meter from the duct or stack wall. To determine whether effluent stratification exists, a dual probe system should be used to determine the average effluent concentration while measurements at each traverse point are being made. One probe, located at the stack or duct centroid, is used as a stationary reference point to indicate the change in effluent concentration over time. The second probe is used for sampling at the traverse points specified in 40 CFR Part 60 appendix A, method 1. The monitoring system samples sequentially at the reference and traverse points throughout the testing period for five minutes at each point.

2.2.4 CEMS Performance and Equipment Specifications

If this method is applied in highly explosive areas, caution and care shall be exercised in choice of equipment and installation.

2.2.4.1 Flame Ionization Detector (FID) Analyzer. A heated FID analyzer capable of meeting or exceeding the requirements of these specifications. Heated systems shall maintain the temperature of the sample gas between 150 °C (300 °F) and 175 °C (350 °F) throughout the system. This requires all system components such as the probe, calibration valve, filter, sample lines, pump, and the FID to be kept heated at all times such that no moisture is condensed out of the system.

Note: As specified in the regulations, unheated HC CEMs may be considered an acceptable interim alternative monitoring technique. For additional notes, see section 2.2.10. The essential components of the measurement system are described below:

2.2.4.1.1 Sample Probe. Stainless steel, or equivalent, to collect a gas sample from the centroidal area of the stack cross-section.

2.2.4.1.2 Sample Line. Stainless steel or Teflon tubing to transport the sample to the analyzer.

Note: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.2.4.1.3 Calibration Valve Assembly. A heated three-way valve assembly to direct the zero and calibration gases to the analyzer is recommended. Other methods, such as quick-connect lines, to route calibration gas to the analyzers are applicable.

2.2.4.1.4 Particulate Filter. An in-stack or out-of-stack sintered stainless steel filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter must be heated.

2.2.4.1.5 Fuel. The fuel specified by the manufacturer (e.g., 40 percent hydrogen/60 percent helium, 40 percent hydrogen/60 percent nitrogen gas mixtures, or pure hydrogen) should be used.

2.2.4.1.6 Zero Gas. High purity air with less than 0.1 parts per million by volume (ppm) HC as methane or carbon equivalent or less than 0.1 percent of the span value, whichever is greater.

2.2.4.1.7 Calibration Gases. Appropriate concentrations of propane gas (in air or nitrogen). Preparation of the calibration gases should be done according to the procedures in EPA Protocol 1. In addition, the manufacturer of the cylinder gas should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change by more than ± 2 percent from the certified value.

2.2.4.2 CEMS Span Value. 100 ppm propane.

2.2.4.3 Daily Calibration Gas Values. The owner or operator must choose calibration gas concentrations that include zero and high-level calibration values.

2.2.4.3.1 The zero level may be between 0 and 20 ppm (zero and 20 percent of the span value).

2.2.4.3.2 The high-level concentration shall be between 50 and 90 ppm (50 and 90 percent of the span value).

2.2.4.4 Data Recorder Scale. The strip chart recorder, computer, or digital recorder must be capable of recording all readings within the CEMS's measurement range and shall have a resolution of 0.5 ppm (0.5 percent of span value).

2.2.4.5 Response Time. The response time for the CEMS must not exceed 2 minutes to achieve 95 percent of the final stable value.

2.2.4.6 Calibration Drift. The CEMS must allow the determination of CD at the zero and high-level values. The CEMS calibration response must not differ by more than ± 3 ppm (± 3 percent of the span value) after each 24-hour period of the 7-day test at both zero and high levels.

2.2.4.7 Calibration Error. The mean difference between the CEMS and reference values at all three test points listed below shall be no greater than 5 ppm (± 5 percent of the span value).

2.2.4.7.1 Zero Level. Zero to 20 ppm (0 to 20 percent of span value).

2.2.4.7.2 Mid-Level. 30 to 40 ppm (30 to 40 percent of span value).

2.2.4.7.3 High-Level. 70 to 80 ppm (70 to 80 percent of span value).

2.2.4.8 Measurement and Recording Frequency. The sample to be analyzed shall pass through the measurement section of the analyzer without interruption. The detector shall measure the sample concentration at least once every 15 seconds. An average emission rate shall be computed and recorded at least once every 60 seconds.

2.2.4.9 Hourly Rolling Average Calculation. The CEMS shall calculate every minute an hourly rolling average, which is the arithmetic mean of the 60 most recent 1-minute average values.

2.2.4.10 Retest. If the CEMS produces results within the specified criteria, the test is successful. If the CEMS does not meet one or more of the criteria, necessary corrections must be made and the performance tests repeated.

2.2.5 Performance Specification Test (PST) Periods

2.2.5.1 Pretest Preparation Period. Install the CEMS, prepare the PTM test site according to the specifications in section 2.2.3, and prepare the

CEMS for operation and calibration according to the manufacturer's written instructions. A pretest conditioning period similar to that of the 7-day CD test is recommended to verify the operational status of the CEMS.

2.2.5.2 Calibration Drift Test Period. While the facility is operating under normal conditions, determine the magnitude of the CD at 24-hour intervals for seven consecutive days according to the procedure given in section 2.2.6.1. All CD determinations must be made following a 24-hour period during which no unscheduled maintenance, repair, or adjustment takes place. If the combustion unit is taken out of service during the test period, record the onset and duration of the downtime and continue the CD test when the unit resumes operation.

2.2.5.3 Calibration Error Test and Response Time Test Periods. Conduct the CE and response time tests during the CD test period.

2.2.6 Performance Specification Test Procedures

2.2.6.1 Calibration Drift Test.

2.2.6.1.1 Sampling Strategy. Conduct the CD test at 24-hour intervals for seven consecutive days using calibration gases at the two daily concentration levels specified in section 2.2.4.3. Introduce the two calibration gases into the sampling system as close to the sampling probe outlet as practical. The gas shall pass through all CEM components used during normal sampling. If periodic automatic or manual adjustments are made to the CEMS zero and calibration settings, conduct the CD test immediately before these adjustments, or conduct it in such a way that the CD can be determined. Record the CEMS response and subtract this value from the reference (calibration gas) value. To meet the specification, none of the differences shall exceed 3 ppm.

2.2.6.1.2 Calculations. Summarize the results on a data sheet. An example is shown in Figure 2.2-1. Calculate the differences between the CEMS responses and the reference values.

2.2.6.2 Response Time. The entire system including sample extraction and transport, sample conditioning, gas analyses, and the data recording is checked with this procedure.

2.2.6.2.1 Introduce the calibration gases at the probe as near to the sample location as possible. Introduce the zero gas into the system. When the system output has stabilized (no change greater than 1 percent of full scale for 30 sec), switch to monitor stack effluent and wait for a stable value. Record the time (upscale response time) required to reach 95 percent of the final stable value.

2.2.6.2.2 Next, introduce a high-level calibration gas and repeat the above procedure. Repeat the entire procedure three times and determine the mean upscale and downscale response times. The longer of the two means is the system response time.

2.2.6.3 Calibration Error Test Procedure.

2.2.6.3.1 Sampling Strategy. Challenge the CEMS with zero gas and EPA Protocol 1 cylinder gases at measurement points within the ranges specified in section 2.2.4.7.

2.2.6.3.1.1 The daily calibration gases, if Protocol 1, may be used for this test.

>>>> See the accompanying hardcopy volume for non-machine-readable data that appears at this point. <<<<

2.2.9 Quality Assurance (QA)

Proper calibration, maintenance, and operation of the CEMS is the responsibility of the owner or operator. The owner or operator must establish a QA program to evaluate and monitor CEMS performance. As a minimum, the QA program must include:

2.2.9.1 A daily calibration check for each monitor. The calibration must be adjusted if the check indicates the instrument's CD exceeds 3 ppm. The gases shall be injected as close to the probe as possible to provide a check of the entire sampling system. If an alternative calibration procedure is desired (e.g., direct injections or gas cells), subject to Administrator approval, the adequacy of this alternative procedure may be demonstrated during the initial 7-day CD test. Periodic comparisons of the two procedures are suggested.

2.2.9.2 A daily system audit. The audit must include a review of the calibration check data, an inspection of the recording system, an inspection of the control panel warning lights, and an inspection of the sample transport and interface system (e.g., flowmeters, filters), as appropriate.

2.2.9.3 A quarterly CE test. Quarterly RA tests may be substituted for the CE test when approved by the Director on a case-by-case basis.

2.2.9.4 An annual performance specification test.

2.2.10 Alternative Measurement Technique

The regulations allow gas conditioning systems to be used In conjunction with unheated HC CEMs during an interim period. This gas conditioning may include cooling to not less than 40 °F and the use of condensate traps to reduce the moisture content of sample gas entering the FID to less than 2 percent. The gas conditioning system, however, must not allow the sample gas to bubble through the condensate as this would remove water soluble organic compounds. All components upstream of the conditioning system should be heated as described in section 2.2.4 to minimize operating and maintenance problems.

2.2.11 References

1. Measurement of Volatile Organic Compounds-Guideline Series. U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 27711, EPA-450/2-78-041, June 1978.

2. Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Continuous Source Emission Monitors (Protocol No. 1). U.S. Environmental Protection Agency ORD/EMSL, Research Triangle Park, North Carolina, 27711, June 1978.

3. Gasoline Vapor Emission Laboratory Evaluation-Part 2. U.S. Environmental Protection Agency, OAQPS, Research Triangle Park, North Carolina, 27711, EMB Report No. 76-GAS-6, August 1975.

Section 3.0 SAMPLING AND ANALYTICAL METHODS

3.1 Methodology for the Determination of Metals Emissions in Exhaust Gases from Hazardous Waste Incineration and Similar Combustion Processes

3.1.1 Applicability and Principle

3.1.1.1 Applicability. This method is being developed for the determination of total chromium (Cr), cadmium (Cd), arsenic (As), nickel (Ni), manganese (Mn), beryllium (Be), copper (Cu), zinc (Zn), lead (Pb), selenium (Se), phosphorus (P), thallium (Tl), silver (Ag), antimony (Sb), barium (Ba), and mercury (Hg) stack emissions from hazardous waste incinerators and similar combustion processes. This method may also be used for the determination of particulate emissions following the procedures and precautions described. Modifications to the sample recovery and analysis procedures described in this protocol for the purpose of determining particulate emissions may potentially impact the front-half mercury determination. Mercury emissions should be determined using EPA method 101A given in 40 CFR part 61.

3.1.1.2 Principle. The stack sample is withdrawn isokinetically from the source, with particulate emissions collected in the probe and on a heated filter and gaseous emissions collected in a series of chilled impingers containing an aqueous solution of dilute nitric acid combined with dilute hydrogen peroxide in each of two impingers, and acidic potassium permanganate solution in each of two impingers. Sampling train components are recovered and digested in separate front- and back-half fractions. Materials collected in the sampling train are digested with acid solutions to dissolve organics and to remove organic constituents that may create analytical interferences. Acid digestion is performed using conventional Parr® Bomb or microwave digestion techniques. The nitric acid and hydrogen peroxide impinger solution, the acidic potassium permanganate impinger solution, the HCl rinse solution, and the probe rinse and digested filter solutions are analyzed for mercury by cold vapor atomic absorption spectroscopy (CVAAS). The nitric acid and hydrogen peroxide solution and the probe rinse and digested filter solutions of the train catches are analyzed for Cr, Cd, Ni, Mn, Be, Cu, Zn, Pb, Se, P, Tl, Ag, Sb, Ba, and As by inductively coupled argon plasma emission spectroscopy (ICAP) or atomic absorption spectroscopy (AAS). Graphite furnace atomic absorption spectroscopy (GFAAS) is used for analysis of antimony, arsenic, cadmium, lead, selenium, and thallium, if these elements require greater analytical sensitivity than can be obtained by ICAP. Additionally, if desired, the tester may use AAS for analyses of all metals if the resulting in-stack method detection limits meet the goal of the testing program. For convenience, aliquots of each digested sample Fraction 1A plus Fraction 2A can be combined proportionally with respect to the original Fraction 1 (normally diluted to 300 ml following digestion and prior to analysis) section 3.1.5.3.3; and concentrated Fraction 2A (normally diluted to 150 ml following digestion and prior to analysis) section 3.1.5.3.4.1 or 3.1.5.3.4.2 for a single analytical determination. The efficiency of the analytical procedure is quantified by the analysis of spiked quality control samples containing each of the target metals and/or other quality assurance measures, as necessary, including actual sample matrix effects checks.

3.1.2 Range, Sensitivity, Precision, and Interferences

3.1.2.1 Range. For the analyses described in this methodology and for similar analyses, the ICAP response is linear over several orders of magnitude. Samples containing metal concentrations in the nanograms per milliliter (ng/ml) to micrograms per milliliter (µg/ml) range in the analytical finish solution can be analyzed using this technique. Samples containing greater than approximately 50 µg/ml of chromium, lead, or arsenic should be diluted to that level or lower for final analysis. Samples containing greater than approximately 20 µg/ml of cadmium should be diluted to that level before analysis.

3.1.2.2 Analytical Sensitivity. ICAP analytical detection limits for the sample solutions (based on SW-846, method 6010) are approximately as follows: Sb (32 ng/ml), As (53 ng/ml), Ba (2 ng/ml), Be (0.3 ng/ml), Cd (4 ng/ml), Cr (7 ng/ml), Cu (6 ng/ml), Pb (42 ng/ml), Mn (2 ng/ml), Ni (15 ng/ml), P (75 ng/ml), Se (75 ng/ml), Ag (7 ng/ml), T1 (40 ng/ml), and Zn (2 ng/ml). The actual method detection limits are sample dependent and may vary as the sample matrix may affect the limits. The analytical detection limits for analysis by direct aspiration AAS (based on SW-846, Method 7000 series) are approximately as follows: Sb (200 ng/ml), As (2 ng/ml), Ba (100 ng/ml), Be (5 ng/ml), Cd (5 ng/ml), Cr (50 ng/ml), Cu (20 ng/ml), Pb (100 ng/ml), Mn (10 ng/ml), Ni (40 ng/ml), Se (2 ng/ml), Ag (10 ng/ml), Tl (100 ng/ml), and Zn (5 ng/ml). The detection limit for mercury by CVAAS is approximately 0.2 ng/ml). The use of GFAAS can give added sensitivity compared to the use of direct aspiration AAS for the following metals: Sb (3 ng/ml), As (1 ng/ml), Be (0.2 ng/ml), Cd (0.1 ng/ml), Cr (1 ng/ml), Pb (1 ng/ml), Se (2 ng/ml), and Tl (1 ng/ml).

Using (1) the procedures described in this method, (2) the analytical detection limits described in the previous paragraph, (3) a volume of 300 ml, Fraction 1, for the front half and 150 ml, Fraction 2A, for the back-half samples, and (4) a stack gas sample volume of 1.25 m^3 , the corresponding instack method detection limits are presented in Table A-1 and calculated as shown:

$$\frac{A \times B}{C} = D$$

where:

A = analytical detection limit, μg/ml.
B = volume of sample prior to aliquot for analysis, ml.
C = stack sample volume, dscm (dsm^3).
D = in-stack detection limit, μg/m^3.

Values in Table 3.1-1 are calculated for the front and back half and/or the total train.

To ensure optimum sensitivity in obtaining the measurements, the concentrations of target metals in the solutions are suggested to be at least ten times the analytical detection limits. Under certain conditions, and with greater care in the analytical procedure, this concentration can be as low as approximately three times the analytical detection limit. In all cases, on at least one sample (run) in the source test and for each metal analyzed, repetitive analyses, method of standard additions (MSA), serial dilution, or matrix spike addition, etc., shall be used to establish the quality of the data.

Actual in-stack method detection limits will be determined based on actual source sampling parameters and analytical results as described above. If required, the method in-stack detection limits can be made more sensitive than those shown in Table A-I for a specific test by using one or more of the following options:

- A 1-hour sampling run may collect a stack gas sampling volume of about 1.25 m^3. If the sampling time is increased and 5 m^3 are collected, the in-stack method detection limits would be one fourth of the values shown in Table A-I (this means that with this change, the method is four times more sensitive than a 1-hour run. Larger sample volumes (longer runs) would make it even more sensitive).

- The in-stack detection limits assume that all of the sample is digested (with exception of the aliquot for mercury) and the final liquid volumes for analysis are 300 ml, Fraction 1 for the front half and 150 ml, Fraction 2A, for the back-half sample. If the front-half volume is reduced from 300 ml to 30 ml, the front-half in-stack detection limits would be one tenth of the values shown above (ten times more sensitive). If the back-half volume is reduced from 150 ml to 25 ml, the in-stack detection limits would be one sixth of the above values. Matrix effects checks are necessary on analyses of samples and typically are of greater significance for samples that have been concentrated to less than the normal original sample volume. Reduction to a volume of less than 25 ml may not allow redissolving of the residue and may increase interference by other compounds.

- When both of the above two improvements are used on one sample at the same time, the resultant improvements are multiplicative. For example, where stack gas volume is increased by a factor of five and the total liquid sample digested volume of both the front and back halves is reduced by a factor of six, the in-stack method detection limit is reduced by a factor of thirty (the method is thirty times more sensitive).

Table 3. 1-1-In-Stack Method Detection Limits (ug/m^3) for Train Fractions Using ICAP and AAS

Metal	Front-half fraction 1 probe and filter	Back-half fraction 2 impingers 1-3	Back-half fractions "Hg, only" impingers 4-6	Total train
Antimony	7.7 (0.7)*	3.8 (0.4)*		11.5 (1.1)*
Arsenic	12.7 (0.3)*	6.4 (0.1)*		19.1 (0.4)*
Barium	0.5	0.3		0.8
Beryllium	0.07 (0.05)*	0.04 (0.03)*		0.11 (0.08)*
Cadmium	1.0 (0.02)*	0.5 (0.01)*		1.5 (0.03)*
Chromium	1.7 (0.2)*	0.8 (0.1)*		2.5 (0.3)*
Copper	1.4	0.7		2.1
Lead	10.1 (0.2)*	5.0 (0.1)*		15.1 (0.3)*
Manganese	0.5 (0.2)*	0.2 (0.1)*		0.7 (0.3*)
Mercury	0.6**	3.0**	2.0**	5.6**
Nickel	3.6	1.8		5.4
Phosphorus	18	9		27
Selenium	18 (0.5)*	9 (0.3)*		27 (0.8)*
Silver	1.7	0.9		2.6
Thallium	9.6 (0.2)*	4.8 (0.1)*		14.4 (0.3)*
Zinc	0.5	0.3		0.8

( )* Detection limit when analyzed by GFAAS.

** Detection limit when analyzed by CVAAS, estimated for Back Half and Total Train.

Note: Actual method in-stack detection limits will be determined based on actual source sampling parameters and analytical results as described earlier in this section.

- Conversely, reducing stack gas sample volume and increasing sample liquid volume will increase in-stack detection limits (the method would then be less sensitive). The front-half and back-half samples (Fractions 1A plus and 2A) can be combined proportionally (see section 3.1.1.2 of this methodology) prior to analysis. The resultant liquid volume (excluding the mercury fractions, which must be analyzed separately) is recorded. Combining the sample as described does not allow determination (whether front or back half) of where in the train the sample was captured. The in-stack method detection limit then becomes a single value for all metals except mercury, for which the contribution of the mercury fractions must be considered.

- The above discussion assumes no blank correction. Blank corrections are discussed later in this method.

3.1.2.3 Precision. The precisions (relative standard deviation) for each metal detected in a method development test at a sewage sludge incinerator, are as follows: Sb (12.7%), As (13.5%), Ba (20.6%), Cd (11.5%), Cr (11.2%), Cu (11.5%), Pb (11.6%), P (14.6%), Se (15.3%), T1 (12.3%), and Zn (11.8%). The precision for nickel was 7.7% for another test conducted at a source simulator. Beryllium, manganese, and silver were not detected in the tests; however, based on the analytical sensitivity of the ICAP for these metals, it is assumed that their precisions should be similar to those for the other metals, when detected at similar levels.

3.1.2.4 Interferences. Iron can be a spectral interference during the analysis of arsenic, chromium, and cadmium by ICAP. Aluminum can be a spectral interference during the analysis of arsenic and lead by ICAP. Generally, these interferences can be reduced by diluting the sample, but this increases the method detection limit (in-stack detection limit). Refer to EPA method 6010 (SW-846) or the other analytical methods used for details on potential interferences for this method. The analyst must eliminate or reduce interferences to acceptable levels. For all GFAAS analyses, matrix modifiers should be used to limit interferences, and standards should be matrix matched.

3.1.3 Apparatus

3.1.3.1 Sampling Train. A schematic of the sampling train is shown in Figure 3.1-1. It is similar to the 40 CFR part 60, appendix A method 5 train. The sampling train consists of the following components:

3.1.3.1.1 Probe Nozzle (Probe Tip) and Borosilicate or Quartz Glass Probe Liner. Same as method 5, sections 2.1.1 and 2.1.2, except that glass nozzles are required unless an alternate probe tip prevents the possibility of contamination or interference of the sample with its materials of construction. If a probe tip other than glass is used, no correction (because of any effect on the sample by the probe tip) of the stack sample test results can be made.

3.1.3.1.2 Pitot Tube and Differential Pressure Gauge. Same as method 2, sections 2.1 and 2.2, respectively.

3.1.3.1.3 Filter Holder. Glass, same as method 5, section 2.1.5, except that a Teflon filter support or other non-metallic, non-contaminating support must be used to replace the glass frit.

3.1.3.1.4 Filter Heating System. Same as method 5, section 2.1.6.

3.1.3.1.5 Condenser. The following system shall be used for the condensation and collection of gaseous metals and for determining the moisture content of the stack gas. The condensing system should consist of four to seven impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. The first impinger is optional and is recommended as a moisture knockout trap for use during test conditions which require such a trap. The first impinger shall be appropriately-sized, if necessary, for an expected large moisture catch and generally constructed as described for the first impinger in method 5, paragraph 2.1.7. The second impinger (or the first HNO_3/H_2O_2 impinger) shall also be constructed as described for the first impinger in method 5. The third impinger (or the second HNO_3/H_2O_2 impinger) shall be the same as the Greenburg Smith impinger with the standard tip described as the second impinger in method 5, paragraph 2.1.7. All other impingers used in the methods train are the same as the first HNO_3/H_2O_2 impinger described in this paragraph. In summary, the first impinger which may be optional as described in this methodology shall be empty, the second and third shall contain known quantities of a nitric acid/hydrogen peroxide solution (section 3.l.4.2.1), the fourth shall be empty, the fifth and sixth shall contain a known quantity of acidic potassium permanganate solution (section 3.1.4.2.2), and the last impinger shall contain a known quantity of silica gel. A thermometer capable of measuring to within 1°C (2°F) shall be placed at the outlet of the last impinger. When the moisture knockout impinger is not needed, it is removed from the train and the other impingers remain the same. If mercury analysis is not to be performed, the potassium permanganate impingers and the empty impinger preceding them are removed.

>>>> See the accompanying hardcopy volume for non-machine-readable data that appears at this point. <<<<

3.1.3.1.6 Metering System, Barometer, and Gas Density Determination Equipment. Same as method 5, sections 2.1.8 through 2.1.10, respectively.

3.1.3.1.7 Teflon Tape. For capping openings and sealing connections, if necessary, on the sampling train.

3.1.3.2 Sample Recovery. Same as method 5, sections 2.2.1 through 2.2.8 (Nonmetallic Probe-Liner and Probe-Nozzle Brushes or Swabs, Wash Bottles, Sample Storage Containers, Petri Dishes, Glass Graduated Cylinder, Plastic Storage Containers, Funnel and Rubber Policeman, and Glass Funnel), respectively, with the following exceptions and additions:

3.1.3.2.1 Nonmetallic Probe-Liner and Probe-Nozzle Brushes or Swabs. For quantitative recovery of materials collected in the front half of the sampling train: Description of acceptable all-Teflon component brushes or swabs is to be included in EPA's Emission Measurement Technical Information Center (EMTIC) files.

3.1.3.2.2 Sample Storage Containers. Glass bottles with Teflon-lined caps which are non-reactive to the oxidizing solutions, with a capacity of 1000- and 500-ml, shall be used for $KMnO_4$-containing samples and blanks. Polyethylene bottles may be used for other sample types.

3.1.3.2.3 Graduated Cylinder. Glass or equivalent.

3.1.3.2.4 Funnel. Glass or equivalent.

3.1.3.2.5 Labels. For identification of samples.

3.1.3.2.6 Polypropylene Tweezers and/or Plastic Gloves. For recovery of the filter from the sampling train filter holder.

3.1.3.3 Sample Preparation and Analysis. For the analysis, the following equipment is needed:

3.1.3.3.1 Volumetric Flasks, 100-ml, 250-ml, and 1000-ml. For preparation of standards and sample dilution.

3.1.3.3.2 Graduated Cylinders. For preparation of reagents.

3.1.3.3.3 Parr^R Bombs or Microwave Pressure Relief Vessels with Capping Station (GEM Corporation model or equivalent).

3.1.3.3.4 Beakers and Watchglasses. 250-ml beakers for sample digestion with watchglasses to cover the tops.

3.1.3.3.5 Ring Stands and Clamps. For securing equipment such as filtration apparatus.

3.1.3.3.6 Filter Funnels. For holding filter paper.

3.1.3.3.7 Whatman 541 Filter Paper (or equivalent). For filtration of digested samples.

3.1.3.3.8 Disposable Pasteur Pipets and Bulbs.

3.1.3.3.9 Volumetric Pipets.

3.1.3.3.10 Analytical Balance. Accurate to within 0.1 mg.

3.1.3.3.11 Microwave or Conventional Oven. For heating samples at fixed power levels or temperatures.

3.1.3.3.12 Hot Plates.

3.1.3.3.13 Atomic Absorption Spectrometer (AAS). Equipped with a background corrector.

3.1.3.3.13.1 Graphite Furnace Attachment. With antimony, arsenic, cadmium, lead, selenium, thallium hollow cathode lamps (HCLs) or electrodeless discharge lamps (EDLs). (Same as EPA SW-846 methods 7041 (antimony), 7060 (arsenic), 7131 (cadmium), 7421 (lead), 7740 (selenium), and 7841 (thallium).)

3.1.3.3.13.2 Cold Vapor Mercury Attachment. With a mercury HCL or EDL. The equipment needed for the cold vapor mercury attachment includes an air recirculation pump, a quartz cell, an aerator apparatus, and a heat lamp or desiccator tube. The heat lamp should be capable of raising the ambient temperature at the quartz cell by 10°C such that no condensation forms on the wall of the quartz cell. (Same as EPA method 7470.)

3.1.3.3.14 Inductively Coupled Argon Plasma Spectrometer. With either a direct or sequential reader and an alumina torch. (Same as EPA method 6010.)

3.1.4 Reagents

The complexity of this methodology is such that to obtain reliable results, the testers (Including analysts) should be experienced and knowledgeable in source sampling, in handling and preparing (including mixing) reagents as described, and using adequate safety procedures and protective equipment in performing this method, including sampling, mixing reagents, digestions, and analyses. Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on

Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

3.1.4.1 Sampling. The reagents used in sampling are as follows:

3.1.4.1.1 Filters. The filters shall contain less than 1.3 μg/in^2 of each of the metals to be measured. Analytical results provided by filter manufacturers are acceptable. However, if no such results are available, filter blanks must be analyzed for each target metal prior to emission testing. Quartz fiber or glass fiber (which meet the requirement of containing less than 1.3 μg/in^2 of each metal) filters without organic binders shall be used. The filters should exhibit at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D2986-7l (incorporated by reference). For particulate determination in sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3, as described in EPA method 5. Quartz fiber filters meeting these requirements are recommended for use in this method.

3.1.4.1.2 Water. To conform to ASTM Specification Dl193.77, Type II (incorporated by reference). If necessary, analyze the water for all target metals prior to field use. All target metal concentrations should be less than 1 ng/ml.

3.1.4.1.3 Nitric Acid. Concentrated. Baker Instra-analyzed or equivalent.

3.1.4.1.4 Hydrochloric Acid. Concentrated. Baker Instra-analyzed or equivalent.

3.1.4.1.5 Hydrogen Peroxide, 30 Percent (V/V).

3.1.4.1.6 Potassium Permanganate.

3.1.4.1.7 Sulfuric Acid. Concentrated.

3.1.4.1.8 Silica Gel and Crushed Ice. Same as method 5, sections 3.1.2 and 3.1.4, respectively.

3.1.4.2 Pretest Preparation for Sampling Reagents.

3.1.4.2.1 Nitric Acid (HNO_3)/Hydrogen Peroxide (H_2O_2) Absorbing Solution, 5 Percent HNO_3/10 Percent H_2O_2. Carefully with stirring, add 50 ml of concentrated HNO_3 to a 1000-ml volumetric flask containing approximately 500 ml of water, and then, carefully with stirring, add 333 ml of 30 percent H_2O_2. Dilute to volume (1000 ml) with water. Mix well. The reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.2.2 Acidic Potassium Permanganate ($KMnO_4$) Absorbing Solution, 4 Percent $KMnO_4$ (W/V), 10 Percent H_2SO_4 (V/V). Prepare fresh daily. Mix carefully, with stirring, 100 ml of concentrated H_2SO_4 into 800 ml of water, and add water with stirring to make a volume of 1 L: This solution is 10 percent H_2SO_4 (V/V). Dissolve, with stirring, 40 g of $KMnO_4$ into 10 percent H_2SO_4 (V/V) and add 10 percent H_2SO_4 (V/V) with stirring to make a volume of 1 L: this is the acidic potassium permanganate absorbing solution. Prepare and store in glass bottles to prevent degradation. The reagent shall contain less than 2 ng/ml of Hg.

Precaution: To prevent autocatalytic decomposition of the permanganate solution, filter the solution through Whatman 541 filter paper. Also, due to the potential reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottle; these bottles shall not be fully filled and shall be vented both to relieve potential excess pressure and prevent explosion due to pressure buildup. Venting is required, but should not allow contamination of the sample; a No. 70-72 hole drilled in the container cap and Teflon liner has been used.

3.1.4.2.3 Nitric Acid, 0.1 N. With stirring, add 6.3 ml of concentrated HNO_3 (70 percent) to a flask containing approximately 900 ml of water. Dilute to 1000 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.2.4 Hydrochloric Acid (HCl), 8 N. Make the desired volume of 8 N HCl in the following proportions. Carefully with stirring, add 690 ml of concentrated HCl to a flask containing 250 ml of water. Dilute to 1000 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of Hg.

3.1.4.3 Glassware Cleaning Reagents.

3.1.4.3.1 Nitric Acid, Concentrated. Fisher ACS grade or equivalent.

3.1.4.3.2 Water. To conform to ASTM Specifications D1193-77, Type II.

3.1.4.3.3 Nitric Acid, 10 Percent (V/V). With stirring, add 500 ml of concentrated HNO_3 to a flask containing approximately 4000 ml of water. Dilute to 5000 ml with water. Mix well. Reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.4 Sample Digestion and Analysis Reagents.

3.1.4.4.1 Hydrochloric Acid, Concentrated.

3.1.4.4.2 Hydrofluoric Acid, Concentrated.

3.1.4.4.3 Nitric Acid, Concentrated. Baker Instra-analyzed or equivalent.

3.1.4.4.4 Nitric Acid, 50 Percent (V/V). With stirring, add 125 ml of concentrated HNO_3 to 100 ml of water. Dilute to 250 ml with water. Mix well. Reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.4.5 Nitric Acid, 5 Percent (V/V). With stirring, add 50 ml of concentrated HNO_3 to 800 ml of water. Dilute to 1000 ml with water. Mix well. Reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.4.6 Water. To conform to ASTM Specifications D1193-77, Type II.

3.1.4.4.7 Hydroxylamine Hydrochloride and Sodium Chloride Solution. See EPA method 7470 for preparation.

3.1.4.4.8 Stannous Chloride. See method 7470.

3.1.4.4.9 Potassium Permanganate, 5 Percent (W/V). See method 7470.

3.1.4.4.10 Sulfuric Acid, Concentrated.

3.1.4.4.11 Nitric Acid, 50 Percent (V/V).

3.1.4.4.12 Potassium Persulfate, 5 Percent (W/V). See Method 7470.

3.1.4.4.13 Nickel Nitrate, $Ni(NO_3)_2 . 6H_2O$.

3.1.4.4.14 Lanthanum, Oxide, La_2O_3.

3.1.4.4.15 AAS Grade Hg Standard, 1000 µg/ml.

3.1.4.4.16 AAS Grade Pb Standard, 1000 µg/ml.

3.1.4.4.17 AAS Grade As Standard, 1000 µg/ml.

3.1.4.4.18 AAS Grade Cd Standard, 1000 µg/ml.

3.1.4.4.19 AAS Grade Cr Standard, 1000 µg/ml.

3.1.4.4.20 AAS Grade Sb Standard, 1000 µg/ml.

3.1.4.4.21 AAS Grade Ba Standard, 1000 µg/ml.

3.1.4.4.22 AAS Grade Be Standard, 1000 µg/ml.

3.1.4.4.23 AAS Grade Cµ Standard, 1000 µg/ml.

3.1.4.4.24 AAS Grade Mn Standard, 1000 µg/ml.

3.1.4.4.25 AAS Grade Ni Standard, 1000 µg/ml.

3.1.4.4.26 AAS Grade P Standard, 1000 µg/ml.

3.1.4.4.27 AAS Grade Se Standard, 1000 µg/ml.

3.1.4.4.28 AAS Grade Ag Standard, 1000 µg/ml.

3.1.4.4.29 AAS Grade T1 Standard, 1000 µg/ml.

3.1.4.4.30 AAS Grade Zn Standard, 1000 µg/ml.

3.1.4.4.31 AAS Grade Al Standard, 1000 µg/ml.

3.1.4.4.32 AAS Grade Fe Standard, 1000 µg/ml.

3.1.4.4.33 The metals standards may also be made from solid chemicals as described in EPA Method 200.7. EPA SW-846 Method 7470 or Standard Methods for the Analysis of Water and Wastewater, 15th Edition, Method 303F should be referred to for additional information on mercury standards.

3.1.4.4.34 Mercury Standards and Quality Control Samples. Prepare fresh weekly a 10 µg/ml intermediate mercury standard by adding 5 ml of 1000 µg/ml mercury stock solution to a 500-ml volumetric flask; dilute with stirring to 500 ml by first carefully adding 20 ml of 15 percent HNO_3 and then adding water to the 500-ml volume. Mix well. Prepare a 200 ng/ml working mercury standard solution fresh daily: Add 5 ml of the 10 µg/ml intermediate standard to a 250-ml volumetric flask and dilute to 250 ml with 5 ml of 4 percent $KMnO_4$, 5 ml of 15 percent HNO_3, and then water. Mix well. At least six separate aliquots of the working mercury standard solution should be used to prepare the standard curve. These aliquots should contain 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the working standard solution containing 0, 200, 400, 600, 800, and 1000 ng mercury, respectively. Quality control samples should be prepared by making a separate 10 µg/ml standard and diluting until in the range of the calibration.

3.1.4.4.35 ICAP Standards and Quality Control Samples. Calibration standards for ICAP analysis can be combined into four different mixed standard solutions as shown below.

Mixed Standard Solutions for ICAP Analysis

Solution	Elements
I	As, Be, Cd, Mn, Pb, Se, Zn.
II	Ba, Cu, Fe.
III	Al, Cr, Ni.
IV	Ag, P, Sb, Tl.

Prepare these standards by combining and diluting the appropriate volumes of the 1000 µg/ml solutions with 5 percent nitric acid. A minimum of one standard and a blank can be used to form each calibration curve. However, a separate quality control sample spiked with known amounts of the target metals in quantities in the midrange of the calibration curve should be prepared. Suggested standard levels are 25 µg/ml for Al, Cr, and Pb, 15 µg/ml for Fe, and 10 µg/ml for the remaining elements. Standards containing less than 1 µg/ml of metal should be prepared daily. Standards containing greater than 1 µg/ml of metal should be stable for a minimum of 1 to 2 weeks.

3.1.4.4.36 Graphite Furnace AAS Standards. Antimony, arsenic, cadmium, lead, selenium, and thallium. Prepare a 10 µg/ml standard by adding 1 ml of 1000 µg/ml standard to a 100-ml volumetric flask. Dilute with stirring to 100 ml with 10 percent nitric acid. For graphite furnace AAS, the standards must be matrix matched. Prepare a 100 ng/ml standard by adding 1 ml of the 10 µg/ml standard to a 110-ml volumetric flask and dilute to 100 ml with the appropriate matrix solution. Other standards should be prepared by dilution of the 100 ng/ml standards. At least five standards should be used to make up the standard curve. Suggested levels are 0, 10, 50, 75, and 100 ng/ml. Quality control samples should be prepared by making a separate 10 µg/ml standard and diluting until it is in the range of the samples. Standards containing less than 1 µg/ml of metal should be prepared daily. Standards containing greater than 1 µg/ml of metal should be stable for a minimum of 1 to 2 weeks.

3.1.4.4.3 Matrix Modifiers.

3.1.4.4.37.1 Nickel Nitrate, 1 Percent (V/V). Dissolve 4.956 g of $Ni(NO_3)_2$. $6H_2O$ in approximately 50 ml of water in a 100-ml volumetric flask. Dilute to 100 ml with water.

3.1.4.4.37.2 Nickel Nitrate, 0.1 Percent (V/V). Dilute 10 ml of the 1 percent nickel nitrate solution from section 4.4.37.1 above to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for As.

3.1.4.4.37.3 Lanthanum. Carefully dissolve 0.5864 g of La_2O_3 in 10 ml of concentrated HNO_3 and dilute the solution by adding it with stirring to approximately 50 ml of water, and then dilute to 100 ml with water. Mix well. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for Pb.

3.1.5 Procedure

3.1.5.1 Sampling. The complexity of this method is such that, to obtain reliable results, testers and analysts should be trained and experienced with the test procedures, including source sampling, reagent preparation and handling, sample handling, analytical calculations, reporting, and descriptions specifically at the beginning of and throughout section 3.1.4 and all other sections of this methodology.

3.1.5.1.1 Pretest Preparation. Follow the same general procedure given in method 5, section 4.1.1, except that, unless particulate emissions are to be determined, the filter need not be desiccated or weighed. All sampling train glassware should first be rinsed with hot tap water and then washed in hot soapy water. Next, glassware should be rinsed three times with tap water, followed by three additional rinses with water. All glassware should then be soaked in a 10 percent (V/V) nitric acid solution for a minimum of 4 hours, rinsed three times with water, rinsed a final time with acetone, and allowed to air dry. All glassware openings where contamination can occur should be covered until the sampling train is assembled for sampling.

3.1.5.1.2 Preliminary Determinations. Same as method 5, section 4.1.2.

3.1.5.1.3 Preparation of Sampling Train. Follow the same general procedures given in method 5, section 4.1.3, except place 100 ml of the nitric acid/hydrogen peroxide solution (section 3.1.4.2.1) in each of the two HNO_3/H_2O_2 impingers as shown in Figure 3.1-1 (normally the second and third impingers), place 100 ml of the acidic potassium permanganate absorbing solution (section 3.1.4.2.2) in each of the two permanganate impingers as shown in Figure A-1, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the last impinger. Alternatively, the silica gel may be weighed directly in the impinger just prior to train assembly.

Several options are available to the tester based on the sampling requirements and conditions. The use of an empty first impinger can be eliminated if the moisture to be collected in the impingers will be less than approximately 100 ml. If necessary, use as applicable to this methodology the procedure described in section 7.1.1 of EPA method 101A, 40 CFR part 61, appendix B, to maintain the desired color in the last permanganate impinger.

Retain for reagent blanks volumes of the nitric acid/hydrogen peroxide solution per section 3.1.5.2.9 of this method and of the acidic potassium permanganate solution per section 3.1.5.2.10. These reagent blanks should be labeled and analyzed as described in section 3.1.7. Set up the sampling train as shown in Figure 3.1-1, or if mercury analysis is not to be performed in the train, then it should be modified by removing the two permanganate impingers and the impinger preceding the permanganate impingers. If necessary to ensure leak-free sampling train connections and prevent contamination Teflon tape or other non-contaminating material should be used instead of silicone grease.

Precaution: Extreme care should be taken to prevent contamination within the train. Prevent the mercury collection reagent (acidic potassium permanganate) from contacting any glassware of the train which is washed and analyzed for Mn. Prevent hydrogen peroxide from mixing with the acidic potassium permanganate.

Mercury emissions can be measured, alternatively, in a separate train which measures only mercury emissions by using EPA method 101A with the modifications described below (and with the further modification that the permanganate containers shall be processed as described in the precaution in section 3.1.4.2.2 and the note in section 3.1.5.2.5 of this methodology). This alternative method is applicable for measurement of mercury emissions, and it may be of special interest to sources which must measure both mercury and manganese emissions.

Section 7.2.1 of method 101A shall be modified as follows after the 250 to 400-ml $KMnO_4$ rinse:

To remove any precipitated material and any residual brown deposits on the glassware following the permanganate rinse, rinse with approximately 100 ml of deionized distilled water, and add this water rinse carefully assuring transfer of all loose precipitated materials from the three permanganate impingers into the permanganate Container No. 1. If no visible deposits remain after this water rinse, do not rinse with 8 N HCl. However, if deposits do remain on the glassware after this water rinse, wash the impinger surfaces with 25 ml of 8 N HCl, and place the wash in a separate sample container labeled Container No. 1.A. containing 200 ml of water as follows. Place 200 ml of water in a sample container labeled Container No. 1.A. Wash the impinger walls and stem with the HCl by turning the impinger on its side and rotating it so that the HCl contacts all inside surfaces. Use a total of only 25 ml of 8 N HCl for rinsing all permanganate impingers combined. Rinse the first impinger, then pour the actual rinse used for the first impinger into the second impinger for its rinse, etc. Finally, pour the 25 ml of 8 N HCl rinse carefully with stirring into Container No. 1.A. Analyze the HCl rinse separately by carefully diluting with stirring the contents of Container No. 1.A. to 500 ml with deionized distilled water. Filter (if necessary) through Whatman 40 filter paper, and then analyze for mercury according to section 7.4, except limit the aliquot size to a maximum of 10 ml. Prepare and analyze a water diluted blank 8 N HCl sample by using the same procedure as that used by Container No. 1.A., except add 5 ml of 8 N HCl with stirring to 40 ml of water, and then dilute to 100 ml with water. Then analyze as instructed for the sample from Container No. 1.A. Because the previous separate permanganate solution rinse (section 7.2.1) and water rinse (as modified in these guidelines) have the capability to recover a very high percentage of the mercury from the permanganate impingers, the amount of mercury in the HCl rinse in Container No. 1.A. may be very small, possibly even insignificantly small. However, add the total of any mercury analyzed and calculated for the HCl rinse sample Container No. 1.A. to that calculated from the mercury sample from section 7.3.2 which contains the separate permanganate rinse (and water rinse as modified herein) for calculation of the total sample mercury concentration.

3.1.5.1.4 Leak-Check Procedures. Follow the leak-check procedures given in method 5, section 4.1.4.1 (Pretest Leak-Check), section 4.1.4.2 (Leak-Checks During the Sample Run), and section 4.1.4.3 (Post-Test Leak-Checks).

3.1.5.1.5 Sampling Train Operation. Follow the procedures given in method 5, section 4.1.5. For each run, record the data required on a data sheet such as the one shown in Figure 5-2 of method 5.

3.1.5.1.6 Calculation of Percent Isokinetic. Same as method 5, section 4.1.6.

3.1.5.2 Sample Recovery. Begin cleanup procedures as soon as the probe is removed from the stack at the end of a sampling period.

The probe should be allowed to cool prior to sample recovery. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a rinsed, non-contaminating cap over the probe nozzle to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling. This normally causes a vacuum to form in the filter holder, thus causing the undesired result of drawing liquid from the impingers into the filter.

Before moving the sampling train to the cleanup site, remove the probe from the sampling train and cap the open outlet. Be careful not to lose any condensate that might be present. Cap the filter inlet where the probe was fastened. Remove the umbilical cord from the last impinger and cap the impinger. Cap off the filter holder outlet and impinger inlet. Use noncontaminating caps, whether ground-glass stoppers, plastic caps, serum caps, or Teflon tape to close these openings.

Alternatively, the train can be disassembled before the probe and filter holder/oven are completely cooled, if this procedure is followed: Initially disconnect the filter holder outlet/impinger inlet and loosely cap the open ends. Then disconnect the probe from the filter holder or cyclone inlet and loosely cap the open ends. Cap the probe tip and remove the umbilical cord as previously described.

Transfer the probe and filter-impinger assembly to a cleanup area that is clean and protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly and note any abnormal conditions. The sample is recovered and treated as follows (see schematic in Figure 3.1-2). Ensure that all items necessary for recovery of the sample do not contaminate it.

3.1.5.2.1 Container No. 1 (Filter). Carefully remove the filter from the filter holder and place it in its identified petri dish container. Acid-washed polypropylene or Teflon coated tweezers or clean, disposable surgical gloves rinsed with water and dried should be used to handle the filters. If it is necessary to fold the filter, make certain the particulate cake is inside the fold. Carefully transfer the filter and any particulate matter or filter fibers that adhere to the filter holder gasket to the petri dish by using a dry (acid-cleaned) nylon bristle brush. Do not use any metalcontaining materials when recovering this train. Seal the labeled petri dish.

>>>> See the accompanying hardcopy volume for non-machine-readable data that appears at this point. <<<<

3.1.5.2.2 Container No. 2 (Acetone Rinse).

Note: Perform section 3.1.5.2.2 only if determination of particulate emissions are desired in addition to metals emissions. If only metals emissions are desired, skip section 3.1.5.2.2 and go to section 3.1.5.2.3. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter and any condensate from the probe nozzle, probe fitting (plastic such as Teflon, polypropylene, etc. fittings are recommended to prevent contamination by metal fittings; further, if desired, a single glass piece consisting of a combined probe tip and probe liner may be used, but such a single glass piece is not a requirement of this methodology), probe liner, and front half of the filter holder by washing these components with 100 ml of acetone and placing the wash in a glass container.

Note: The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows: Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a nonmetallic brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse the sample-exposed, inside parts of the fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Allow the acetone to drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washings to the container. Follow the acetone rinse with a nonmetallic probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed through the probe three times or more until none remains in the probe liner on visual inspection. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

Clean the inside of the front half of the filter holder by rubbing the surfaces with a nonmetallic nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. After all acetone washings and particulate matter have been collected in the sample container tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container clearly to identify its contents.

3.1.5.2.3 Container No. 3 (Probe Rinse). Keep the probe assembly clean and free from contamination as described in section 3.1.5.2.2 of this method during the 0.1 N nitric acid rinse described below. Rinse the probe nozzle and fitting probe liner, and front half of the filter holder thoroughly with 100 ml of 0.1 N nitric acid and place the wash into a sample storage container.

Note: The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Perform the rinses as applicable and generally as described in method 12, section 5.2.2. Record the volume of the combined rinse. Mark the height of the fluid level on the outside of the storage container and use this mark to determine if leakage occurs during transport. Seal the container and clearly label the contents. Finally, rinse the nozzle, probe liner, and front half of the filter holder with water followed by acetone and discard these rinses.

3.1.5.2.4 Container No. 4 (Impingers 1 through 3, HNO_3/H_2O_2 Impingers and Moisture Knockout Impinger, when used, Contents and Rinses). Due to the potentially large quantity of liquid involved, the tester may place the impinger solutions from impingers 1 through 3 in more than one container. Measure the liquid in the first three impingers volumetrically to within 0.5 ml using a graduated cylinder. Record the volume of liquid present. This information is required to calculate the moisture content of the sampled flue gas. Clean each of the first three impingers, the filter support, the back half of the filter housing, and connecting glassware by thoroughly rinsing with 100 ml of 0.1 N nitric acid using the procedure as applicable and generally as described in method 12, section 5.2.4.

Note: The use of exactly 100 ml of 0.1 N nitric acid rinse is necessary for the subsequent blank correction procedures. Combine the rinses and impinger solutions, measure and record the volume. Mark the height of the fluid level on the outside of the container to determine if leakage occurs during transport. Seal the container and clearly label the contents.

3.1.5.2.5 Container Nos. 5A, 5B, and 5C. 5A (0.1 N HNO_3), 5B ($KMnO_4/H_2SO_4$ absorbing solution), and 5C (8 N HCl rinse and dilution). (As described previously at the end of section 3.1.3.1.5 of this method, if mercury is not being measured in this train, then impingers 4, 5, and 6, as shown in Figure 3.1-2, are not necessary and may be eliminated.) Pour all the liquid, if any, from the impinger which was empty at the start of the run and which immediately precedes the two permanganate impingers (normally impinger No. 4) into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Place the liquid in Sample Container No. 5A. Rinse the impinger (No. 4) with 100 ml of 0.1 N HNO_3 and place this into Container No. 5A.

Pour all the liquid from the two permanganate impingers into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Place this $KMnO_4$ absorbing solution stack sample from the two permanganate impingers into Container No. 5B. Using 100 ml total of fresh acidified potassium permanganate solution, rinse the two permanganate impingers and connecting glass pieces a minimum of three times and place the rinses into Container No. 5B, carefully ensuring transfer of all loose precipitated materials from the two impingers into Container No. 5B. Using 100 ml total of water, rinse the permanganate impingers and connecting glass pieces a minimum of three times, and place the rinses into Container 5B, carefully ensuring transfer of all loose precipitated material, if any, from the two impingers into Container No. 5B. Mark the height of the fluid level on the outside of the bottle to determine if leakage occurs during transport. See the following note and the precaution in paragraph 3.1.4.2.2 and properly prepare the bottle and clearly label the contents.

Note: Due to the potential reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottles. These bottles shall not be completely filled and shall be vented to relieve potential excess pressure. Venting is required. A No. 70-72 hole drilled in the container cap and Teflon liner has been used.

If no visible deposits remain after the above described water rinse, do not rinse with 8 N HCl. However, if deposits do remain on the glassware after this water rinse, wash the impinger surfaces with 25 ml of 8 N HCl, and place the wash in a separate sample container labeled Container No. 5C containing 200 ml of water as follows: Place 200 ml of water in a sample container labeled Container No. 5C. Wash the impinger walls and stem with the HCl by turning the impinger on its side and rotating it so that the HCl contacts all inside surfaces. Use a total of only 25 ml of 8 N HCl for rinsing both permananate impingers combined. Rinse the first impinger, then pour the actual rinse used for the first impinger into the second impinger for its rinse. Finally, pour the 25 ml of 8 N HCl rinse carefully with stirring into Container No. 5C. Mark the height of the fluid level on the outside of the bottle to determine if leakage occurs during transport.

3.1.5.2.6 Container No. 6 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its impinger to its original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger.

The small amount of particles that may adhere to the impinger wall need not be removed. Do not use water or other liquids to transfer the silica gel since weight gained in the silica gel impinger is used for moisture calculations. Alternatively, if a balance is available in the field, record the weight of the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

3.1.5.2.7 Container No. 7 (Acetone Blank). If particulate emissions are to be determined, at least once during each field test, place a 100-ml portion of the acetone used in the sample recovery process into a labeled container for use in the front-half field reagent blank. Seal the container.

3.1.5.2.8 Container No. 8A (0.1 N Nitric Acid Blank). At least once during each field test, place 300 ml of the 0.1 N nitric acid solution used in the sample recovery process into a labeled container for use in the front-half and back-half field reagent blanks. Seal the container. Container No. 8B (water blank). At least once during each field test, place 100 ml of the water used in the sample recovery process into a labeled Container No. 8B. Seal the container.

3.1.5.2.9 Container No. 9 (5% Nitric Acid/10% Hydrogen Peroxide Blank). At least once during each field test, place 200 ml of the 5% nitric acid/10% hydrogen peroxide solution used as the nitric acid impinger reagent into a labeled container for use in the back-half field reagent blank. Seal the container.

3.1.5.2.10 Container No. 10 (Acidified Potassium Permanganate Blank). At least once during each field test, place 100 ml of the acidified potassium permanganate solution used as the impinger solution and in the sample recovery process into a labeled container for use in the back-half field reagent blank for mercury analysis. Prepare the container as described in section 3.1.5.2.5.

Note: Due to the potential reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottles. These bottles shall not be completely filled and shall be vented to relieve potential excess pressure. Venting is required. A No. 70-72 hole drilled in the container cap and Teflon liner has been used.

3.1.5.2.11 Container No. 11 (8 N HCl Blank). At least once during each field test, perform both of the following: Place 200 ml of water into a sample container. Pour 25 ml of 8N HCl carefully with stirring into the 200 ml of water in the container. Mix well and seal the container.

3.1.5.2.12 Container No. 12 (Filter Blank). Once during each field test, place three unused blank filters from the same lot as the sampling filters in a labeled petri dish. Seal the petri dish. These will be used in the front-half field reagent blank.

3.1.5.3 Sample Preparation. Note the level of the liquid in each of the containers and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. A diagram illustrating sample preparation and analysis procedures for each of the sample train components is shown in Figure 3.1-3.

3.1.5.3.1 Container No. 1 (Filter). If particulate emissions are being determined, then desiccate the filter and filter catch without added heat and weigh to a constant weight as described in section 4.3 of method 5. For analysis of metals, divide the filter with its filter catch into portions containing approximately 0.5 g each and place into the analyst's choice of either individual microwave pressure relief vessels or Parr® Bombs. Add 6 ml of concentrated nitric acid and 4 ml of concentrated hydrofluoric acid to each vessel. For microwave heating, microwave the sample vessels for approximately 12-15 minutes in intervals of 1 to 2 minutes at 600 Watts. For conventional heating, heat the Parr Bombs at 140°C (285°F) for 6 hours. Cool the samples to room temperature and combine with the acid digested probe rinse as required in section 3.1.5.3.3, below.

>>>> See the accompanying hardcopy volume for non-machine-readable data that appears at this point. <<<<

Notes: 1. Suggested microwave heating times are approximate and are dependent upon the number of samples being digested. Twelve to 15 minute heating times have been found to be acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by sorbent reflux within the vessel.

2. If the sampling train uses an optional cyclone, the cyclone catch should be prepared and digested using the same procedures described for the filters and combined with the digested filter samples.

3.1.5.3.2 Container No. 2 (Acetone Rinse). Note the level of liquid in the container and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ± 1 ml or gravimetrically to ± 0.5 g. Transfer the contents to an acid-cleaned, tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. If particulate emissions are being determined, desiccate for 24 hours without added heat, weigh to a constant weight according to the procedures described in section 4.3 of method 5, and report the results to the nearest 0.1 mg. Redissolve the residue with 10 ml of concentrated nitric acid and, carefully with stirring, quantitatively combine the resultant sample including all liquid and any particulate matter with Container No. 3 prior to beginning the following section 3.1.5.3.3.

3.1.5.3.3 Container No. 3 (Probe Rinse). The pH of this sample shall be 2 or lower. If the pH is higher, the sample should be acidified to pH 2 by the careful addition with stirring of concentrated nitric acid. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Digest the sample in microwave vessels or Parr® Bombs by quantitatively transferring the sample to the vessel or bomb, by carefully adding the 6 ml of concentrated nitric acid and 4 ml of concentrated hydrofluoric acid and then continuing to follow the procedures described in section 3.1.5.3.1; then combine the resultant sample directly with the acid digested portions of the filter prepared previously in section 3.1.5.3.1. The resultant combined sample is referred to as Fraction 1 precursor. Filter the combined solution of the acid digested filter and probe rinse samples using Whatman 541 filter paper. Dilute to 300 ml (or the appropriate volume for the expected metals concentration) with water. This dilution is Fraction 1. Measure and record the volume of the Fraction 1 solution to within 0.1 ml. Quantitatively remove a 50-ml aliquot and label as Fraction 1B. Label the remaining 250-ml portion as Fraction 1A. Fraction 1A is used for ICAP or AAS analysis. Fraction 1B is used for the determination of front-half mercury.

3.1.5.3.4 Container No. 4 (Impingers 1-3). Measure and record the total volume of this sample (Fraction 2) to within 0.5 ml. Remove a 75- to 100-ml aliquot for mercury analysis and label as Fraction 2B. Label the remaining portion of Container No. 4 as aliquot Fraction 2A. Aliquot Fraction 2A defines the volume of 2A prior to digestion. All of the aliquot Fraction 2A is digested to produce concentrated Fraction 2A. Concentrated Fraction 2A defines the volume of 2A after digestion which is normally 150 ml. Only concentrated Fraction 2A is analyzed for metals (except that it is not analyzed for mercury). The Fraction 2B aliquot should be prepared and analyzed for mercury as described in section 3.1.5.4.3. Aliquot Fraction 2A shall be pH 2 or lower. If necessary, use concentrated nitric acid, by careful addition and stirring, to lower aliquot Fraction 2A to pH 2. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Next follow either the conventional or microwave digestion procedures described in sections 3.1.5.3.4.1 and 3.1.5.3.4.2, below.

3.1.5.3.4.1 Conventional Digestion Procedure. Add 30 ml of 50 percent nitric acid and heat for 30 minutes on a hot plate to just below boiling. Add 10 ml of 3 percent hydrogen peroxide and heat for 20 more minutes. Add 50 ml of hot water and heat the sample for an additional 20 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water. This dilution is concentrated Fraction 2A. Measure and record the volume of the Fraction 2A solution to within 0.1 ml.

3.1.5.3.4.2 Microwave Digestion Procedure. Add 10 ml of 50 percent nitric acid and heat for 6 minutes in intervals of 1 to 2 minutes at 600 Watts. Allow the sample to cool. Add 10 ml of 3 percent hydrogen peroxide and heat for 2 more minutes. Add 50 ml of hot water and heat for an additional 5 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water. This dilution is concentrated Fraction 2A. Measure and record the volume of the Fraction 2A solution to within 0.1 ml.

Note: All microwave heating times given are approximate and are dependent upon the number of samples being digested at a time. Heating times as given above have been found acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by solvent reflux within the vessel.

3.1.5.3.5 Container Nos. 5A, 5B, and 5C (Impingers 4, 5, and 6). Keep these samples separate from each other and measure and record the volumes of 5A and 5B separately to within 0.5 ml. Dilute sample 5C to 500 ml with water. These samples 5A, 5B, and 5C are referred to respectively as Fractions 3A, 3B, and 3C. Follow the analysis procedures described in section 3.1.5.4.3.

Because the permanganate rinse and water rinse have the capability to recover a high percentage of the mercury from the permanganate impingers, the amount of mercury in the HCl rinse (Fraction 3C) may be very small, possibly even insignificantly small. However, as instructed in this method, add the total of any mercury measured in and calculated for the HCl rinse (Fraction 3C) to that for Fractions 1B, 2B, 3A, and 3B for calculation of the total sample mercury concentration.

3.1.5.3.6 Container No. 6 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

3.1.5.4 Sample Analysis. For each sampling train, seven individual samples are generated for analysis. A schematic identifying each sample and the prescribed sample preparation and analysis scheme is shown in Figure 3.1-3. The first two samples, labeled Fractions 1A and 1B, consist of the digested samples from the front half of the train. Fraction 1A is for ICAP or AAS analysis as described in sections 3.1.5.4.1 and/or 3.1.5.4.2. Fraction 1B is for determination of front-half mercury as described in section 3.1.5.4.3.

The back half of the train was used to prepare the third through seventh samples. The third and fourth samples, labeled Fractions 2A and 2B, contain the digested samples from the moisture knockout, if used, and HNO_3/H_2O_2 Impingers 1 through 3. Fraction 2A is for ICAP or AAS analysis. Fraction 2B will be analyzed for mercury.

The fifth through seventh samples, labeled Fractions 3A, 3B, and 3C, consist of the impinger contents and rinses from the empty and permanganate impingers 4, 5, and 6. These samples are analyzed for mercury as described in section 3.1.5.4.3. The total back-half mercury catch is determined from the sum of Fraction 2B and Fractions 3A, 3B, and 3C.

3.1.5.4.1 ICAP Analysis. Fraction 1A and Fraction 2A are analyzed by ICAP using EPA SW-846 method 6010 or method 200.7 (40 CFR 136, appendix C). Calibrate the ICAP, and set up an analysis program as described in method 6010 or method 200.7. The quality control procedures described in section 3.1.7.3.1 of this method shall be followed. Recommended wavelengths for use in the analysis are listed below:

Element	Wavelength (nm)
Aluminum	308.215
Antimony	206.833
Arsenic	193.696
Barium	455.403
Beryllium	313.042
Cadmium	226.502
Chromium	267.716
Copper	324.754
Iron	259.940
Lead	220.353
Manganese	257.610
Nickel	231.604
Phosphorus	214.914
Selenium	196.026
Silver	328.068
Thallium	190.864
Zinc	213.856

The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.

Initially, analyze all samples for the desired target metals (except mercury) plus iron and aluminum. If iron and aluminum are present in the sample, the sample may have to be diluted so that each of these elements is at a concentration of less than 50 ppm to reduce their spectral interferences on arsenic, cadmium, chromium, and lead.

Note. When analyzing samples in a hydrofluoric acid matrix, an alumina torch should be used; since all front-half samples will contain hydrofluoric acid, use an alumina torch.

3.1.5.4.2 AAS by Direct Aspiration and/or Graphite Furnace. If analysis of metals in Fraction 1A and Fraction 2A using graphite furnace or direct aspiration AAS is desired, Table 3.1-2 should be used to determine which techniques and methods should be applied for each target metal. Table 3.1-2 should also be consulted to determine possible interferences and techniques to be followed for their minimization. Calibrate the instrument according to section 3.1.6.3 and follow the quality control procedures specified in section 3.1.7.3.2.

Table 3.1-2-Applicable Techniques, Methods, and Minimization of Interference for AAS Analysis

Metal	Technique	SW-846 Method No.	Wavelength (nm)	Interferences	
				Cause	Minimization
Sb	Aspiration	7040	217.6	1000 mg/mL Pb Ni, Cu, or acid	Use secondary wavelength of 231.1 nm; match sample & standards' acid concentration or use nitrous oxide/acetylene flame.
Sb	Furnace	7041	217.6	High Pb	Secondary wavelength or Zeeman correction.
As	Furnace	7060	193.7	Arsenic volatization	Spiked samples and add nickel nitrate solution to digestates prior to analysis.
				Aluminum	Use Zeeman background correction.
Ba	Aspiration	7080	553.6	Calcium	High hollow cathode current and narrow band set.
				Barium ionization	2 mL of KC1 per 100 mL of sample.
Be	Aspiration	7090	234.9	500 ppm A1	Add 0.1% fluoride.
				High Mg and Si	Use method of standard additions.
Be	Furnace	7091	234.9	Be in optical path	Optimize parameters to minimize effects.
Cd	Aspiration	7130	228.8	Absorption and light scattering	Background correction is required.
Cd	Furnace	7131	228.8	As above	As above.
				Excess chloride	Ammonium phosphate used as a matrix modifier.
				Pipet tips	Use cadmium-free tips.
Cr	Aspiration	7190	357.9	Alkali metal	KC1 ionization suppressant in samples and standards.

				Absorption and scatter	Consult manufacturer's literature.
Cr	Furnace	7191	357.9	200 mg/L Ca and P	All calcium nitrate for a known constant effect and to eliminate effect of phosphate.
Cu	Aspiration	7210	324.7	Absorption and scatter	Consult manufacturer's manual.
Fe	Aspiration	7380	248.3	Contamination	Great care taken to avoid contamination.
Pb	Aspiration	7420	283.3	217.0 nm alternate	Background correction required.
Pb	Furnace	7421	283.3	Poor recoveries	Matrix modifier, add 10 uL of phosphorus acid to 1 mL of prepared sample in sampler cup.
Mn	Aspiration	7460	279.5	403.1 nm alternate	Background correction required.
Ni	Aspiration	7520	232.0	352.4 nm alternate	Background correction required.
				Fe, Co, and Cr	Matrix matching or nitrous-oxide/acetylene flame.
				Nonlinear response	Sample dilution or use 352.3 nm line.
Se	Furnace	7740	196.0	Volatility	Spike samples and reference materials and add nickel nitrate to minimize volatilization.
				Adsorption & scatter	Background correction is required and Zeeman background correction can be useful.
Ag	Aspiration	7760	328.1	Adsorption & scatter	Background correction is required.
				AgC1 insolube	Avoid hydrochloric acid unless silver is in solution as a

					chloride complex.
				Viscosity	Sample and standards monitored for aspiration rate.
Tl	Aspiration	7840	276.8		Background correction is required. Hydrochloric acid should not be used.
Tl	Furnace	7841	276.8	Hydrochloric acid or chloride	Background correction is required. Verify that losses are not occurring for volatization by spiked samples or standard addition; Palladium is a suitable matrix modifier.
Zn	Aspiration	7950	213.9	High Si, Cu, & P	Strontium removes Cu and phosphate.
				Contamination	Great care taken to avoid contamination.

3.1.5.4.3 Cold Vapor AAS Mercury Analysis. Fraction 1B, Fraction 2B, and Fractions 3A, 3B, and 3C should be analyzed separately for mercury using cold vapor atomic absorption spectroscopy following the method outlined in EPA SW-846 method 7470 or in Standard Methods for Water and Wastewater Analysis, 15th Edition, Method 303F. Set up the calibration curve (zero to 1000 ng) as described in SW-846 method 7470 or similar to method 303F, using 300-ml BOD bottles instead of Erlenmeyers. Dilute separately, as described below, a 1 ml to 10 ml aliquot of each original sample to 100 ml with water. Record the amount of the aliquot used for dilution to 100 ml. If no prior knowledge exists of the expected amount of mercury in the sample, a 5-ml aliquot is suggested for the first dilution to 100 ml and analysis. To determine the stack emission value for mercury, the amount of the aliquot of the sample used for dilution and analysis is dependent on the amount of mercury in the aliquot: The total amount of mercury in the aliquot used for analysis shall be less than 1 μg, and within the range (zero to 1000 ng) of the calibration curve. Place each sample aliquot into a separate 300-ml BOD bottle and add enough Type II water to make a total volume of 100 ml. Then analyze the 100 ml for mercury by adding to it sequentially the sample preparation solutions and performing the sample preparation and analysis as described in the procedures of SW-846 method 7470 or method 303F. If, during the described analysis, the reading maximum(s) are off-scale (because the aliquot of the original sample analyzed contained more mercury than the maximum of the calibration range) including the analysis of the 100-ml dilution of the 1-ml aliquot of the original sample causing a reading maximum which is off-scale, then perform the following: Dilute the original sample (or a portion of it) with 0.15% HNO_3 in water (1.5 ml concentrated HNO_3 per liter aqueous solution) so that when a 1-ml to 10-ml aliquot of the dilution of the original sample is then further diluted to 100 ml in the BOD bottle, and analyzed by the procedures described above, it will yield an analysis within the range of the calibration curve.

3.1.6 Calibration

Maintain a laboratory log of all calibrations.

3.1.6.1 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of method 5: Probe Nozzle (section 5.1); Pitot Tube (section 5.2); Metering System (section 5.3); Probe Heater (section 5.4); Temperature Gauges (section 5.5); Leak-Check of the Metering System (section 5.6); and Barometer (section 5.7).

3.1.6.2 Inductively Coupled Argon Plasma Spectrometer Calibration. Prepare standards as outlined in section 3.1.4.4. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures using the above standards. The instrument calibration should be checked once per hour. If the instrument does not reproduce the concentrations of the standard within 10 percent, the complete calibration procedures should be performed.

3.1.6.3 Atomic Absorption Spectrometer-Direct Aspiration, Graphite Furnace and Cold Vapor Mercury Analyses. Prepare the standards as outlined in section 3.1.4.4. Calibrate the spectrometer using these prepared standards. Calibration procedures are also outlined in the EPA methods referred to in Table 3.1-2 and in SW-846 Method 7470 or Standard Methods for Water and Wastewater, 15th Edition, method 303F (for mercury). Each standard curve should be run in duplicate and the mean values used to calculate the calibration line. The instrument should be recalibrated approximately once every 10 to 12 samples.

3.1.7 Quality Control

3.1.7.1 Sampling. Field Reagent Blanks. When analyzed, the blank samples in Container Numbers 7 through 12 produced previously in sections 3.1.5.2.7 through 3.1.5.2.12, respectively, shall be processed, digested, and analyzed as follows: Digest and process one of the filters from Container No. 12 per section 3.1.5.3.1, 100 ml from Container No. 7 per section 3.1.5.3.2, and 100 ml from Container No. 8A per section 3.1.5.3.3. This produces Fraction Blank 1A and Fraction Blank 1B from Fraction Blank 1. (If desired, the other two filters may be digested separately according to section 3.1.5.3.1, diluted separately to 300 ml each, and analyzed separately to produce a blank value for each of the two additional filters. If these analyses are performed, they will produce two additional values for each of Fraction Blank 1A and Fraction Blank 1B. The three Fraction Blank 1A values will be calculated as three values of M_{fhb} in Equation 3 of section 3.1.8.4.3, and then the three values shall be totalled and divided by 3 to become the value M_{fhb} to be used in the computation of M_t by Equation 3. Similarly, the three Fraction Blank 1B values will be calculated separately as three values, totalled, averaged, and used as the value for Hg_{fhb} in Equation 8 of section 3.1.8.5.3. The analyses of the two extra filters are optional and are not a requirement of this method, but if the analyses are performed, the results must be considered as described above.) Combine 100 ml of Container No. 8A with 200 ml of the contents of Container No. 9 and digest and process the resultant volume per section 3.1.5.3.4. This produces concentrated Fraction Blank 2A and Fraction Blank 2B from Fraction Blank 2. A 100-ml portion of Container No. 8A is Fraction Blank 3A. Combine 100 ml of the contents of Container No. 10 with 33 ml of the contents of Container No. 8B. This produces Fraction Blank 3B (use 400 ml as the volume of Fraction Blank 3B when calculating the blank value. Use the actual volumes when calculating all the other blank values). Dilute 225 ml of the contents of Container No. 11 to 500 ml with water. This produces Fraction Blank 3C. Analyze Fraction Blank 1A and Fraction Blank 2A per section 3.1.5.4.1 and/or 3.1.5.4.2. Analyze Fraction Blank 1B, Fraction Blank 2B, and Fraction Blanks 3A, 3B, and 3C per section 3.1.5.4.3. The analysis of Fraction Blank 1A produces the front-half reagent blank correction values for the metals except mercury; the analysis of Fraction Blank 1B produces the front-half reagent blank correction value for mercury. The analysis of concentrated Fraction Blank 2A produces the back-half reagent blank correction values for the metals except mercury, while separate analysis of Fraction Blanks 2B, 3A, 3B, and 3C produce the back-half reagent blank correction value for mercury.

3.1.7.2 An attempt may be made to determine if the laboratory reagents used in section 3.1.5.3 caused contamination. They should be analyzed by the procedures in section 3.1.5.4. The Administrator will determine whether the laboratory blank reagent values can be used in the calculation of the stationary source test results.

3.1.7.3 Quality Control Samples. The following quality control samples should be analyzed.

3.1.7.3.1 ICAP Analysis. Follow the quality control shown in section 8 of method 6010. For the purposes of a three-run test series, these requirements have been modified to include the following: Two instrument check standard runs, two calibration blank runs, one interference check sample at the beginning of the analysis (must be within 25% or analyze by the method of standard additions), one quality control sample to check the accuracy of the calibration standards (must be within 25% of calibration), and one duplicate analysis (must be within 10% of average or repeat all analyses).

3.1.7.3.2 Direct Aspiration and/or Graphite Furnace AAS Analysis for antimony, arsenic, barium, beryllium, cadmium, copper, chromium, lead, nickel, manganese, mercury, phosphorus, selenium, silver, thallium, and zinc. All samples should be analyzed in duplicate. Perform a matrix spike on at least one front-half sample and one back-half sample or one combined sample. If recoveries of less than 75 percent or greater than 125 percent are obtained for the matrix spike, analyze each sample by the method of standard additions. A quality control sample should be analyzed to check the accuracy of the calibration standards. The results must be within 10% or the calibration repeated.

3.1.7.3.3 Cold Vapor AAS Analysis for Mercury. All samples should be analyzed in duplicate. A quality control sample should be analyzed to check the accuracy of the calibration standards (within 15% or repeat calibration). Perform a matrix spike on one sample from the nitric impinger portion (must be within 25% or samples must be analyzed by the method of standard additions). Additional information on quality control can be obtained from EPA SW-846 method 7470 or in Standard Methods for the Examination of Water and Wastewater, 15th Edition, method 303F.

3.1.8 Calculations

3.1.8.1 Dry Gas Volume. Using the data from this test, calculate $V_{m(std)}$, the dry gas sample volume at standard conditions as outlined in Section 6.3 of Method 5.

3.1.8.2 Volume of Water Vapor and Moisture Content. Using the data obtained from this test, calculate the volume of water vapor $V_{w(std)}$ and the moisture content B_{ws} of the stack gas. Use Equations 5-2 and 5-3 of Method 5.

3.1.8.3 Stack Gas Velocity. Using the data from this test and Equation 2-9 of Method 2, calculate the average stack gas velocity.

3.1.8.4 Metals (Except Mercury) in Source Sample.

3.1.8.4.1 Fraction 1A, Front Half, Metals (except Hg). Calculate separately the amount of each metal collected in Fraction 1 of the sampling train using the following equation:

$$M_{fh} = C_{a1}\ F_d\ V_{soln,1} \qquad \text{Eq. } 1^{*}$$

[*] If Fractions 1A and 2A are combined, proportional aliquots must be used. Appropriate changes must be made in Equations 1-3 to reflect this approach.

where:

M_{fh} = total mass of each metal (except Hg) collected in the front half of the sampling train (Fraction 1), μg.

C_{a1} = concentration of metal in sample Fraction 1A as read from the standard curve (μg/ml).

F_d = dilution factor (F_d = the inverse of the fractional portion of the concentrated sample in the solution actually used in the instrument to produce the reading C_{a1}. For example, when 2 ml of Fraction 1A are diluted to 10 ml, $F_d = 5$).

$V_{soln,1}$ = total volume of digested sample solution (Fraction 1), ml.

3.1.8.4.2 Fraction 2A, Back Half, Metals (except Hg). Calculate separately the amount of each metal collected in Fraction 2 of the sampling train using the following equation:

$$M_{bh} = C_{a2}F_aV_a \qquad \text{Eq. } 2^{*}$$

where:

M_{bh} = total mass of each metal (except Hg) collected in the back half of the sampling train (Fraction 2), μg.

C_{a2} = concentration of metal in sample concentrated Fraction 2A, as read from the standard curve (μg/ml).

F_a = aliquot factor, volume of Fraction 2 divided by volume of aliquot Fraction 2A (see section 3.1.5.3.4).

V_a = total volume of digested sample solution (concentrated Fraction 2A), ml (see section 3.1.5.3.4.1 or 3.1.5.3.4.2, as applicable).

3.1.8.4.3 Total Train, Metals (except Hg). Calculate the total amount of each of the quantified metals collected in the sampling train as follows:

$$M_t = (M_{fh} - M_{fhb}) + (M_{bh} - M_{bhb}) \qquad \text{Eq. } 3^{*}$$

where:

M_t = total mass of each metal (separately stated for each metal) collected in the sampling train, μg.

M_{fhb} = blank correction value for mass of metal detected in front-half field reagent blank, μg.

M_{bhb} = blank correction value for mass of metal detected in back-half field reagent blank, μg.

Note: If the measured blank value for the front half (m_{fhb}) is in the range 0.0 to A μg (where A μg equals the value determined by multiplying 1.4 μg per square inch (1.4 μg/in^2) times the actual area in square inches (in^2)

of the filter used in the emission sample) m_{fhb} may be used to correct the emission sample value (m_{fh}); if m_{fhb} exceeds A µg, the greater of the two following values (either I. or II.) may be used:

I. A µg, or

II. the lesser of (a) m_{fhb}, or (b) 5 percent of m_{fh}.

If the measured blank value for the back half (m_{bhb}) is in the range of 0.0 to 1 µg, m_{bhb} may be used to correct the emission sample value (m_{bh}); if m_{bhb} exceeds 1 µg, the greater of the two following values may be used: 1 µg or 5 percent of m_{bh}.

3.1.8.5 Mercury in Source Sample.

3.1.8.5.1 Fraction 1B, Front Half, Hg. Calculate the amount of mercury collected in the front half, Fraction 1, of the sampling train using the following equation:

$$Hg_{fh} = \frac{Q_{fh}}{V_{f1B}} \times V_{soln,1} \qquad \text{Eq. 4}$$

where:

Hg_{fh} = total mass of mercury collected in the front half of the sampling train (Fraction 1), µg.
Q_{fh} = quantity of mercury in analyzed sample, µg.
$V_{soln,1}$ = total volume of digested sample solution (Fraction 1), ml.
V_{f1B} = volume of Fraction 1B analyzed, ml.

See the following notice.

Note: V_{f1B} is the actual amount of Fraction 1B analyzed. For example, if 1 ml of Fraction lB were diluted to 100 ml to bring it into the proper analytical range, and 1 ml of the 100-ml dilution were analyzed, V_{f1B} would be 0.01 ml.

3.1.8.5.2 Fraction 2B and Fractions 3A, 3B, and 3C, Back Half, Hg. Calculate the amount of mercury collected in Fractions 2 using Equation 5 and in Fractions 3A, 3B, and 3C using Equation 6. Calculate the total amount of mercury collected in the back half of the sampling train using Equation 7.

$$Hg_{bh2} = \frac{Q_{bh2}}{V_{f2B}} \times V_{soln,2} \qquad \text{Eq. 5}$$

where:

Hg_{bh2} = total mass of mercury collected in Fraction 2, µg.
Q_{bh2} = quantity of mercury in analyzed sample, µg.
$V_{soln,2}$ = total volume of Fraction 2, ml.
V_{f2B} = volume of Fraction 2B analyzed, ml (see the following note).

Note: V_{f2B} is the actual amount of Fraction 2B analyzed. For example, if 1 ml of Fraction 2B were diluted to 10 ml to bring it into the proper analytical range, and 5 ml of the 10-ml dilution was analyzed, V_{f2B} would be 0.5.

Use Equation 6 to calculate separately the back-half mercury for Fractions 3A, then 3B, then 3C.

$$Hg_{bh3(A,B,C)} = \frac{Q_{bh3(A,B,C)}}{V_{f3(A,B,C)}} \quad X \quad V_{soln,3(A,B,C)} \qquad \text{Eq.6}$$

where:

$Hg_{bh3(A,B,C)}$ = total mass of mercury collected separately in Fraction 3A, 3B, or 3C, µg.
$Q_{bh3(A,B,C)}$ = quantity of mercury in separately analyzed samples, µg.
$V_{f3(A,B,C)}$ = volume of Fraction 3A, 3B, or 3C analyzed, ml (see Note in sections 3.1.8.5.1 and 3.1.8.5.2, and calculate similarly).
$V_{soln,3(A,B,C)}$ = total volume of Fraction 3A, 3B, or 3C, ml.

$$Hg_{bh} = Hg_{bh2} + Hg_{bh3A} + Hg_{bh3B} + Hg_{bh3C} \qquad \text{Eq. 7}$$

where:

Hg_{bh} = total mass of mercury collected in the back half of the sampling train, µg.

3.1.8.5.3 Total Train Mercury Catch. Calculate the total amount of mercury collected in the sampling train using Equation 8.

$$Hg_{t} = (Hg_{fh} - Hg_{fhb}) + (Hg_{bh} - Hg_{bhb}) \qquad \text{Eq. 8}$$

where:

Hg_{t} = total mass of mercury collected in the sampling train, µg.
Hg_{fhb} = blank correction value for mass of mercury detected in front-half field reagent blank, µg.
Hg_{fhb} = blank correction value for mass of mercury detected in back-half field reagent blanks, µg.

Note: If the total of the measured blank values (Hg_{fhb} + Hg_{bhb}) is in the range of 0 to 6 µg, then the total may be used to correct the emission sample value (Hg_{fh} + Hg_{bh}); if it exceeds 6 µg, the greater of the following two values may be used; 6 µg or 5 percent of the emission sample value (Hg_{fh} + Hg_{bh}).

3.1.8.6 Metal Concentration of Stack Gas. Calculate each metal separately for the cadmium, total chromium, arsenic, nickel, manganese, beryllium, copper, lead, phosphorus, thallium, silver, barium, zinc, selenium, antimony, and mercury concentrations in the stack gas (dry basis, adjusted to standard conditions) as follows:

$$C_{s} = K_{4}(M_{t}/V_{m(std)}) \qquad \text{Eq. 9}$$

where:

C_{s} = concentration of each metal in the stack gas, mg/dscm.
K_{4} = 10^{-3}mg/µg.
M_{t} = total mass of each metal collected in the sampling train, µg; (substitute Hg_{t} for M_{t} for the mercury calculation).
$V_{m(std)}$ = volume of gas sample as measured by the dry gas meter, corrected to dry standard conditions, dscm.

3.1.8.7 Isokinetic Variation and Acceptable Results. Same as method 5, sections 6.11 and 6.12, respectively.

3.1.9 Bibliography

3.1.9.1 Method 303F in Standard Methods for the Examination of Water and Wastewater, 15th Edition, 1980. Available from the American Public Health Association. 1015 18th Street NW., Washington, DC 20036.

3.1.9.2 EPA Methods 6010, 7000, 7041, 7060, 7131, 7421. 7470, 7740. and 7841. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods SW-846, Third Edition. September 1988. Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Washington, DC 20460.

3.1.9.3 EPA Method 200.7, Code of Federal Regulations, title 40, part 136, appendix C. July 1, 1987.

3.1.9.4 EPA Methods 1 through 5, and 12 Code of Federal Regulations, title 40, part 60, appendix A, July 1, 1987.

3.2 Determination of Hexavalent Chromium Emissions from Stationary Sources (Method Cr^{+6})

3.2.1 Applicability and Principle

3.2.1.1 Applicability. This method applies to the determination of hexavalent chromium (Cr^{+6}) emissions from hazardous waste incinerators. municipal waste combustors, sewage sludge incinerators, and boilers and industrial furnaces. With the approval of the Administrator, this method may also be used to measure total chromium. The sampling train, constructed of Teflon components, has only been evaluated at temperatures less than 300 °F. Trains constructed of other materials, for testing at higher temperatures, are currently being evaluated.

3.2.1.2 Principle. For incinerators and combustors, the Cr^{+6} emissions are collected isokinetically from the source. To eliminate the possibility of Cr^{+6} reduction between the nozzle and impinger, the emission samples are collected with a recirculatory train where the impinger reagent is continuously recirculated to the nozzle. Recovery procedures include a post-sampling purge and filtration. The impinger train samples are analyzed for Cr^{+6} by an ion chromatograph equipped with a post-column reactor and a visible wavelength detector. The IC/PCR separates the Cr^{+6} as chromate ($CrO^{4+6=}$) from other components in the sample matrices that may interfere with the Cr^{+6}-specific diphenylcarbazide reaction that occurs in the post-column reactor. To increase sensitivity for trace levels of chromium, a preconcentration system is also used in conjunction with the IC/PCR.

3.2.2 Range, Sensitivity, Precision, and Interference

3.2.2.1 Range. Employing a preconcentration procedure, the lower limit of the detection range can be extended to 16 nanograms per dry standard cubic meter (ng/dscm) with a 3 dscm gas sample (0.1 ppb in solution). With sample dilution, there is no upper limit.

3.2.2.2 Sensitivity. A minimum detection limit of 8 ng/dscm with a 3 dscm gas sample can be achieved by preconcentration (0.05 ppb in solution).

3.2.2.3 Precision. The precision of the IC/PCR with sample preconcentration is 5 to 10 percent. The overall precision for sewage sludge incinerators emitting 120 ng/dscm of Cr^{+6} and 3.5 µg/dscm of total chromium is

25% and 9% for Cr^{+6} and total chromium, respectively; for hazardous waste incinerators emitting 300 ng/dscm of Cr^{+6} it is 20 percent.

3.2.2.4 Interference. Components in the sample matrix may cause Cr^{+6} to convert to trivalent chromium (Cr^{+3}) or cause Cr^{+3} to convert to Cr^{+6} A post-sampling nitrogen purge and sample filtration are included to eliminate many of these interferences. The chromatographic separation of Cr^{+6} using ion chromatography reduces the potential for other metals to interfere with the post-column reaction. For the IC/PCR analysis, only compounds that coelute with Cr^{+6} and affect the diphenylcarbazide reaction will cause interference. Periodic analysis of deionized (DI) water blanks is used to demonstrate that the analytical system is essentially free from contamination. Sample cross-contamination that can occur when high-level and low-level samples or standards are analyzed alternately is eliminated by thorough purging of the sample loop. Purging can easily be achieved by increasing the injection volume of the samples to ten times the size of the sample loop.

3.2.3 Apparatus

3.2.3.1 Sampling Train. Schematics of the recirculating sampling trains employed in this method are shown in Figures 3.2-1 and 3.2-2. The recirculatory train is readily assembled from commercially available components. All portions of the train in contact with the sample are either glass, quartz, Tygon, or Teflon, and are to be cleaned as per subsection 3.2.5.1.1.

The metering system is identical to that specified by Method 5 (see section 3.8.1); the sampling train consists of the following components:

>>>> See the accompanying hardcopy volume for non-machine-readable data that appears at this point. <<<<

3.2.3.1.1 Probe Nozzle. Glass or Teflon with a sharp, tapered leading edge. The angle of taper shall be ≤30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (1/8 to 1/2 in) (or larger if higher volume sample trains are used) inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in). Each nozzle shall be calibrated according to the procedures outlined in section 3.2.6.

3.2.3.1.2 Teflon Aspirator or Pump/Sprayer Assembly. Teflon aspirator capable of recirculating absorbing reagent at 50 ml/min while operating at 0.75 cfm. Alternatively, a pump/sprayer assembly may be used instead of the Teflon aspirator. A Teflon union-T is connected behind the nozzle to provide the absorbing reagent/sample gas mix; a peristaltic pump is used to recirculate the absorbing reagent at a flow rate of at least 50 ml/min. Teflon fittings, Teflon ferrules. and Teflon nuts are used to connect a glass or Teflon nozzle. recirculating line. and sample line to the Teflon aspirator or union-T. Tygon, C-flex[**] or other suitable inert tubing for use with peristaltic pump.

[*] Note: Mention of trade names or specific product does not constitute endorsement by the Environmental Protection Agency.

3.2.3.1.3 Teflon Sample Line. Teflon, 3/8" outside diameter (OD) and 1/4" inside diameter (ID), or 1/2" OD x 3/8" ID, of suitable length to connect aspirator (or T-union) to first Teflon impinger.

3.2.3.1.4 Teflon Recirculation Line. Teflon, 1/4" O.D. and 1/8" I.D., of suitable length to connect first impinger to aspirator (or T-union).

3.2.3.1.5 Teflon Impingers. Four Teflon Impingers; Teflon tubes and fittings, such as made by Savillex** can be used to construct impingers 2" diameter by 12" long, with vacuum-tight 3/8" O.D. Teflon compression fittings. Alternatively, standard glass impingers that have been Teflon-lined, with Teflon stems and U-tubes, may be used. Inlet fittings on impinger top to be bored through to accept 3/8" O.D. tubing as impinger stem. The second and third 3/8" OD Teflon stem has a 1/4" OD Teflon tube, 2" long. inserted at its end to duplicate the effects of the Greenburg-Smith impinger stem. The first impinger stem should extend 2" from impinger bottom, high enough in the impinger reagent to prevent air from entering recirculating line; the second and third impinger stems should extent to 1/2" from impinger bottom. The first impinger should include a 1/4" O.D. Teflon compression fitting for recirculation line. The fourth impinger serves as a knockout impinger.

3.2.3.1.6 Glass Impinger. Silica gel impinger. Vacuum-tight impingers, capable of containing 400 g of silica gel, with compatible fittings. The silica gel impinger will have a modified stem (1/2" ID at tip of stem).

3.2.3.1.7 Thermometer, (identical to that specified by Method 5) at the outlet of the silica gel impinger, to monitor the exit temperature of the gas.

3.2.3.1.8 Metering System, Barometer, and Gas Density Determinations Equipment. Same as method 5, sections 2.1.8 through 2.1.10, respectively.

3.2.3.2 Sample Recovery. Clean all items for sample handling or storage with 10% nitric acid solution by soaking, where possible, and rinse thoroughly with DI water before use.

3.2.3.2.1 Nitrogen Purge Line. Inert tubing and fittings capable of delivering 0 to 1 scf/min (continuously adjustable) of nitrogen gas to the impinger train from a standard gas cylinder (see Figure 3.2.3). Standard 3/8-inch Teflon tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve may be used.

>>>> See the accompanying hardcopy volume for non-machine-readable data that appears at this point. <<<<

3.2.3.2.2 Wash bottles. Two polyethylene wash bottles, for DI water and nitric rinse solution.

3.2.3.2.3 Sample Storage Containers. Polyethylene, with leak-free screw cap, 500-ml or 1000-ml.

3.2.3.2.4 1000-ml Graduated Cylinder.

3.2.3.2.5 Plastic Storage Containers. Air tight containers to store silica gel.

3.2.3.2.6 Funnel and Rubber Policeman. To aid in transfer of silica gel from impinger to storage container; not necessary if silica gel is weighed directly in the impinger.

3.2.3.2.7 Balance.

3.2.3.3 Sample Preparation for Analysis. Sample preparation prior to analysis includes purging the sample train immediately following the sample run. and filtering the recovered sample to remove particulate matter immediately following recovery.

3.2.3.3.1 Beakers, Funnels, Volumetric Flasks, Volumetric Pipets, and Graduated Cylinders. Assorted sizes, Teflon or glass, for preparation of samples, sample dilution, and preparation of calibration standards. Prepare initially following procedure described in section 3.2.5.1.3 and rinse between use with 0.1 N HNO_3 and DI water.

3.2.3.3.2 Filtration Apparatus. Teflon, or equivalent, for filtering samples, and Teflon filter holder. Teflon impinger components have been found to be satisfactory as a sample reservoir for pressure filtration using nitrogen.

3.2.3.4 Analysis.

3.2.3.4.1 IC/PCR System. High performance liquid chromatograph pump, sample injection valve, post-column reagent delivery and mixing system, and a visible detector, capable of operating at 520 nm, all with a non-metallic (or inert) flow path. An electronic recording integrator operating in the peak area mode is recommended, but other recording devices and integration techniques are acceptable provided the repeatability criteria and the linearity criteria for the calibration curve described in section 3.2.5.5 can be satisfied. A sample loading system will be required if preconcentration is employed.

3.2.3.4.2 Analytical Column. A high performance ion chromatograph (HPIC) non-metallic column with anion separation characteristics and a high loading capacity designed for separation of metal chelating compounds to prevent metal interference. Resolution described in section 3.2.5.4 must be obtained. A non-metallic guard column with the same ion-exchange material is recommended.

3.2.3.4.3 Preconcentration Column. An HPIC non-metallic column with acceptable anion retention characteristics and sample loading rates as described in section 3.2.5.5.

3.2.3.4.4 0.45 um filter cartridge. For the removal of insoluble material. To be used just prior to sample injection/analysis.

3.2.4 Reagents

All reagents should, at a minimum, conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. All prepared reagents should be checked by IC/PCR analysis for Cr^{+6} to ensure that contamination is below the analytical detection limit for direct injection or, if selected, preconcentration. If total chromium is also to be determined, the reagents should also be checked by the analytical technique selected to ensure that contamination is below the analytical detection limit.

3.2.4.1 Sampling.

3.2.4.1.1 Water. Deionized water. It is recommended that water blanks be checked prior to preparing sampling reagents to ensure that the Cr^{+6} content is less than the analytical detection limit.

3.2.4.1.2 Potassium Hydroxide, 0.1 N. Add 5.6 gm of KOH(s) to approximately 900 ml of DI water and let dissolve. Dilute to 1000 ml with DI water.

Note: At sources with high concentrations of acids and/or SO_2, the concentration of KOH should be increased to 0.5 N to ensure that the pH of the solution is above 8.5 after sampling.

3.2.4.1.3 Silica Gel and Crushed Ice. Same as Method 5, sections 3.1.2 and 3.1.4, respectively.

3.2.4.2 Sample Recovery. The reagents used in sample recovery are as follows:

3.2.4.2.1 Water. Same as subsection 3.2.4.1.1.

3.2.4.2.2 Nitric Acid, 0.1 N. Add 6.3 ml of concentrated HNO_3 (70 percent) to a graduated cylinder containing approximately 900 ml of DI water. Dilute to 1000 ml with DI water, and mix well.

3.2.4.2.3 pH Indicator Strip. pH indicator capable of determining pH of solution between the pH range of 7 and 12, at 0.5 pH intervals.

3.2.4.3 Sample Preparation

3.2.4.3.1 Water. Same as subsection 3.2.4.1.1.

3.2.4.3.2 Nitric Acid, 0.1 N. Same as subsection 3.2.4.2.2.

3.2.4.3.3 Filters. Acetate membrane, or equivalent, filters with 0.45 micrometer or smaller pore size to remove insoluble material.

3.2.4.4 Analysis.

3.2.4.4.1 Chromatographic Eluent. The eluent used in the analytical system is ammonium sulfate based. It is prepared by adding 6.5 ml of 29 percent ammonium hydroxide (NH_4OH) and 33 grams of ammonium sulfate ($(NH_4)_2SO_4$) to 500 ml of DI water. The mixture should then be diluted to 1 liter with DI water and mixed well. Other combinations of eluents and/or columns may be employed provided peak resolution, as described in section 3.2.5.4, repeatability and linearity, as described in section 3.2.6.2, and analytical sensitivity are acceptable.

3.2.4.4.2 Post-Column Reagent. An effective post-column reagent for use with the chromatographic eluent described in section 3.2.4.4.1 is a diphenylcarbazide (DPC) based system. Dissolve 0.5 g of 1.5-diphenylcarbazide (DPC) in 100 ml of ACS grade methanol. Add to 500 ml of degassed DI water containing 50 ml of 96 percent spectrophotometric grade sulfuric acid. Dilute to 1 liter with degassed DI water.

3.2.4.4.3 Cr^{+6} Calibration Standard. Prepare Cr^{+6} standards from potassium dichromate ($K_2Cr_2O_7$, FW 294.19). To prepare a 1000 µg/ml Cr^{+6} stock solution, dissolve 2.829 g of dry $K_2Cr_2O_7$ in 1 liter of DI water. To prepare working standards, dilute the stock solution to the chosen standard concentrations for instrument calibration with 0.05 N KOH to achieve a matrix similar to the actual field samples.

3.2.4.4.4 Performance Audit Sample. A performance audit sample shall be obtained from the Quality Assurance Division of EPA and analyzed with the field samples. The mailing address to request audit samples is: U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Quality Assurance Division, Source Branch. Mail Drop 77-A, Research Triangle Park, North Carolina 27711.

The audit sample should be prepared in a suitable sample matrix at a concentration similar to the actual field samples.

3.2.5 Procedure

3.2.5.1 Sampling. The complexity of this method is such that to obtain reliable results, testers should be trained and experienced with test procedures.

3.2.5.1.1 Pretest Preparation. All components shall be maintained and calibrated according to the procedures described in APTD-0576, unless otherwise specified herein.

Rinse all sample train components from the glass nozzle up to the silica gel impinger and sample containers with hot tap water followed by washing with hot soapy water. Next, rinse the train components and sample containers three times with tap water followed by three rinses with DI water. All the components and containers should then be soaked overnight, or a minimum of 4 hours, in a 10 percent (v/v) nitric acid solution, then rinsed three times with DI water. Allow the components to air dry prior to covering all openings with Parafilm, or equivalent.

3.2.5.1.2 Preliminary Determinations. Same as method 5, section 4.1.2.

3.2.5.1.3 Preparation of Sampling Train. Measure 300 ml of 0.1 N KOH into a graduated cylinder (or tare-weighed precleaned polyethylene container). Place approximately 150 ml of the 0.1 N KOH reagent in the first Teflon impinger. Split the rest of the 0.1 N KOH between the second and third Teflon impingers. The next Teflon impinger is left dry. Place a preweighed 200-to 400-g portion of indicating silica gel in the final glass impinger. (For sampling periods in excess of two hours, or for high moisture sites. 400-g of silica gel is recommended.)

Retain reagent blanks of the 0.1 N KOH equal to the volumes used with the field samples.

3.2.5.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, section 4.1.4.1 (Pretest Leak-Check), Section 4.1.4.2 (Leak-Checks During the Sample Run), and Section 4.1.4.3 (Post-Test Leak-Checks).

3.2.5.1.5 Sampling Train Operation. Follow the procedures given in method 5, section 4.1.5. The sampling train should be iced down with water and ice to ensure heat transfer with the Teflon impingers.

Note: If the gas to be sampled is above 200 °F, it may be necessary to wrap three or four feet of the Teflon sample and recirculating lines inside the ice bath to keep the recirculated reagent cool enough so it does not turn to steam.

For each run, record the data required on a data sheet such as the one shown in Figure 5.2 of method 5.

At the end of the sampling run, determine the pH of the reagent in the first impinger using a pH indicator strip. The pH of the solution shall be greater than 8.5.

3.2.5.1.6 Calculation of Percent Isokinetic. Same as method 5, section 4.1.6.

3.2.5.2 Post-Test Nitrogen Purge. The nitrogen purge is used as a safeguard against the conversion of hexavalent chromium to the trivalent oxidation state. The purge is effective in the removal of SO_2 from the impinger contents.

Attach the nitrogen purge line to the input of the impinger train. Check to ensure the output of the impinger train is open, and that the recirculating line is capped off. Open the nitrogen gas flow slowly and adjust the delivery rate to 10 L/min. Check the recirculating line to ensure that the pressure is not forcing the impinger reagent out through this line. Continue the purge under these conditions for one-half hour, periodically checking the flow rate.

3.2.5.3 Sample Recovery. Begin cleanup procedures as soon as the train assembly has been purged at the end of the sampling run. The probe assembly may be disconnected from the sample train prior to sample purging.

The probe assembly should be allowed to cool prior to sample recovery. Disconnect the umbilical cord from the sample train. When the probe assembly can be safely handled, wipe off all external particulate matter near the tip of the nozzle, and cap the nozzle prior to transporting the sample train to a cleanup area that is clean and protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly and note any abnormal conditions.

3.2.5.3.1 Container No. 1 (Impingers 1 through 3). Disconnect the first impinger from the second impinger and disconnect the recirculation line from the aspirator or peristaltic pump. Drain the Teflon impingers into a precleaned graduated cylinder or tare-weighed precleaned polyethylene sample container and measure the volume of the liquid to within 1 ml or 1 g. Record the volume of liquid present as this information is required to calculate the moisture content of the flue gas sample. If necessary, transfer the sample from the graduated cylinder to a precleaned polyethylene sample container. With DI water, rinse four times the insides of the glass nozzle, the aspirator, the sample and recirculation lines, the impingers, and the connecting tubing, and combine the rinses with the impinger solution in the sample container.

3.2.5.3.2 Container No. 2 (HNO_3 rinse optional for total chromium). With 0.1 N HNO_3, rinse three times the entire train assembly, from the nozzle to the fourth impinger and combine the rinses into a separate precleaned polyethylene sample container for possible total chromium analysis. Repeat the rinse procedure a final time with DI water, and discard the water rinses. Mark the height of the fluid level on the container or, alternatively if a balance is available, weigh the container and record the weight to permit determination of any leakage during transport. Label the container clearly to identify its contents.

3.2.5.3.3 Container No. 3 (Silica Gel). Note the color of the indicating silica gel to determine if it has been completely spent. Quantitatively transfer the silica gel from its impinger to the original container, and seal the container. A funnel and a rubber policeman may be used to aid in the transfer. The small amount of particulate that may adhere to the impinger wall need not be removed. Do not use water or other liquids to transfer the silica gel. Alternatively, if a balance is available in the field, record the weight of the spent silica gel (or the silica gel plus impinger) to the nearest 0.5 g.

3.2.5.3.4 Container No. 4 (0.1 N KOH Blank). Once during each field test, place a volume of reagent equal to the volume placed in the sample train into a precleaned polyethylene sample container, and seal the container. Mark the height of the fluid level on the container or, alternatively if a balance is available, weigh the container and record the weight to permit determination of any leakage during transport. Label the container clearly to identify its contents.

3.2.5.3.5 Container No. 5 (DI Water Blank). Once during each field test, place a volume of DI water equal to the volume employed to rinse the sample train into a precleaned polyethylene sample container, and seal the container. Mark the height of the fluid level on the container or, alternatively if a balance is available, weigh the container and record the weight to permit determination of any leakage during transport. Label the container clearly to identify its contents.

3.2.5.3.6 Container No. 6 (0.1 N HNO_3 Blank). Once during each field test if total chromium is to be determined, place a volume of 0.1 N HNO_3 reagent equal to the volume employed to rinse the sample train into a pre-cleaned polyethylene sample container, and seal the container. Mark the height of the fluid level on the container or, alternatively if a balance is available, weigh the container and record the weight to permit determination of any leakage during transport. Label the container clearly to identify its contents.

3.2.5.4 Sample Preparation. For determination of Cr^{+6}, the sample should be filtered immediately following recovery to remove any insoluble matter. Nitrogen gas may be used as a pressure assist to the filtration process (see Figure Cr^{+6}-4).

Filter the entire impinger sample through a 0.45-micrometer acetate filter (or equivalent), and collect the filtrate in a 1000-ml graduated cylinder. Rinse the sample container with DI water three separate times, pass these rinses through the filter, and add the rinses to the sample filtrate. Rinse the Teflon reservoir with DI water three separate times, pass these rinses through the filter, and add the rinses to the sample. Determine the final volume of the filtrate and rinses and return them to the rinsed polyethylene sample container. Label the container clearly to identify its contents. Rinse the Teflon reservoir once with 0.1 N HNO_3 and once with DI water and discard these rinses.

If total chromium is to be determined, quantitatively recover the filter and residue and place them in a vial. (The acetate filter may be digested with 5 ml of 70 percent nitric acid; this digestion solution may then be diluted with DI water for total chromium analysis.)

>>>> See the accompanying hardcopy volume for non-machine-readable data that appears at this point. <<<<

Note: If the source has a large amount of particulate in the effluent stream, testing teams may wish to filter the sample twice, once through a 2 to 5-micrometer filter, and then through the 0.45-micrometer filter.

3.2.5 4.1 Container 2 (HNO_3 rinse, optional for total chromium). This sample shall be analyzed in accordance with the selected procedure for total chromium analysis. At a minimum, the sample should be subjected to a digestion procedure sufficient to solubilize all chromium present.

3.2.5.4.2 Container 3 (Silica Gel). Weigh the spent silica gel to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

3.2.5.5 Sample analysis. The Cr^{+6} content of the sample filtrate is determined by ion chromatography coupled with a post-column reactor (IC/PCR). To increase sensitivity for trace levels of chromium, a preconcentration system is also used in conjunction with the IC/PCR.

Prior to preconcentration and/or analysis, all field samples will be filtered through a 0.45-µ filter. This filtration should be conducted just prior to sample injection/analysis.

The preconcentration is accomplished by selectively retaining the analyte on a solid absorbent (as described in 3.2.3.4.3), followed by removal of the analyte from the absorbent. The sample is injected into a sample loop of the desired size (repeated loadings or larger size loop for greater sensitivity) and the Cr^{+6} is collected on the resin bed of the column. When the injection valve is switched. the eluent displaces the concentrated Cr^{+6} sample moving it off the preconcentration column and onto the IC anion separation column. After separation from other sample components, Cr^{+6} forms a specific complex in the post.column reactor with a diphenylcarbazide reaction solution, and the complex is then detected by visible absorbance at a wavelength of 520 nm. The amount of absorbance measured is proportional to the concentration of the Cr^{+6} complex formed. The IC retention time and absorbance of the Cr^{+6} complex is compared with known Cr^{+6} standards analyzed under identical conditions to provide both qualitative and quantitative analyses.

Prior to sample analysis, establish a stable baseline with the detector set at the required attenuation by setting the eluent flowrate at approximately 1 ml/min and post-column reagent flowrate at approximately 0.5 ml/min.

Note: As long as the ratio of eluent flowrate to PCR flowrate remains constant, the standard curve should remain linear. Inject a sample of DI water to ensure that no Cr^{+6} appears in the water blank.

First, inject the calibration standards prepared, as described in section 3.2.4.4.4, to cover the appropriate concentration range, starting with the lowest standard first. Next. inject, in duplicate, the performance audit sample, followed by the 0.1 N KOH field blank and the field samples. Finally, repeat the injection of the calibration standards to allow for compensation of instrument drift. Measure areas or heights of the Cr^{+6}/DPC complex chromatogram peak. The response for replicate, consecutive injections of samples must be within 5 percent of the average response, or the injection should be repeated until the 5 percent criterion can be met. Use the average response (peak areas or heights) from the duplicate injections of calibration standards to generate a linear calibration curve. From the calibration curve, determine the concentration of the field samples employing the average response from the duplicate injections.

The results for the analysis of the performance audit sample must be within 10 percent of the reference value for the field sample analysis to be valid.

3.2.6 Calibration. Maintain a written log of all calibration activities.

3.2.6.1 Sample Train Calibration. Calibrate the sample train components according to the indicated sections of method 5: Probe Nozzle (section 5.1); Pitot Tube (section 5.2); Metering System (section 5.3); Temperature Gauges (section 5.5); Leak-Check of the Metering System (section 5.6); and Barometer (section 5.7).

3.2.6.2 Calibration Curve for the IC/PCR. Prepare working standards from the stock solution described in section 3.2.4.4.4 by dilution with a DI water solution to approximate the field sample matrix. Prepare at least four standards to cover one order of magnitude that bracket the field sample concentrations. Run the standards with the field samples as described in section 3.2.5.5. For each standard, determine the peak areas (recommended) or the peak heights, calculate the average response from the duplicate injections, and plot the average response against the Cr^{+6} concentration in µg/L. The individual responses for each calibration standard determined before and after field sample analysis must be within 5 percent of the average response for the analysis to be valid. If the 5 percent criteria is exceeded, excessive drift and/or instrument degradation may have occurred, and must be corrected before further analyses are performed.

Employing linear regression, calculate a predicted value for each calibration standard with the average response for the duplicate injections. Each predicted value must be within 7 percent of the actual value for the calibration curve to be considered acceptable. If not acceptable, remake and/or rerun the calibration standards. If the calibration curve is still unacceptable. reduce the range of the curve.

3.2.7 Calculations

3.2.7.1 Dry Gas Volume. Using the data from the test, calculate $V_{m(std)}$, the dry gas sample volume at standard conditions as outlined in Section 6.3 of Method 5.

3.2.7.2 Volume of Water Vapor and Moisture Content. Using the data from the test, calculate $V_{w(std)}$ and B_{ws}, the volume of water vapor and the moisture content of the stack gas, respectively, using Equations 5-2 and 5-3 of Method 5.

3.2.7.3 Stack Gas Velocity. Using the data from the test and Equation 2-9 of Method 2, calculate the average stack gas velocity.

3.2.7.4 Total µg Cr^{+6} per Sample. Calculate as described below:

$$m = (S-B) \times V_{1s} \times d$$

where:

m = Mass of Cr^{+6} in the sample, µg.
S = Concentration of sample, µg Cr^{+6}/ml.
B = Concentration of blank, µg Cr^{+6}/ml.
V_{1s} = Volume of sample after filtration, ml.
d = Dilution factor (1 if not diluted).

>>>> End of File FR94A. This article is continued in File FR94B. <<<<