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APPENDICES

APPENDIX A

Table A-1 Brands and masses of fluorescent lamps

Brand	Sample number	Mass with caps (g)	Mass without caps (g)	Mass of caps (g)
#1	1	187.1	183.4	3.7
	2	193.1	188.2	4.9
	3	189.7	185.2	4.5
	4	187.5	182.7	4.8
	5	190.3	185.6	4.7
	6	190.2	185.3	4.9
	7	193.0	188.0	5.0
	8	195.5	189.1	6.4
	9	193.9	189.6	4.3
	10	190.1	185.4	4.7
#2	1	193.6	188.1	5.5
	2	191.0	185.3	5.7
	3	185.0	179.4	5.6
	4	185.3	179.4	5.9
	5	198.4	191.6	6.8
	6	201.6	195.6	6.0
	7	194.8	188.5	6.3
	8	198.2	192.4	5.8
	9	203.4	196.8	6.6
	10	197.2	191.2	6.0
#3	1	165.7	160.4	5.3
	2	163.7	158.9	4.8
	3	177.0	171.9	5.1
	4	160.3	155.3	5.0
	5	165.3	160.7	4.6
#4	1	174.6	169.4	5.2
	2	187.0	181.5	5.5
	3	189.3	183.6	5.7
	4	189.4	183.0	6.4
	5	196.3	190.5	5.8

Table A-1 Brands and masses of fluorescent lamps (cont.)

Brand	Sample number	Mass with caps (g)	Mass without caps (g)	Mass of caps (g)
#5	1	193.4	188.6	4.8
	2	186.8	181.8	5.0
	3	190.5	184.2	6.3
	4	196.6	191.9	4.7
	5	196.0	190.0	6.0
	6	198.5	193.7	4.8
	7	197.9	193.0	4.9
	8	191.3	186.1	5.2
	9	188.1	182.8	5.3
	10	196.6	191.2	5.4
#6	1	195.1	187.8	7.3
	2	194.7	188.9	5.8
	3	192.8	185.2	7.6
	4	194.0	186.9	7.1
	5	194.6	187.3	7.3
	6	195.3	187.8	7.5
	7	190.0	185.3	4.7
	8	192.1	186.1	6.0
	9	192.9	187.3	5.6
	10	193.7	188.9	4.8
	Mean	190.0	184.4	5.6
	SD	9.4	9.1	0.9
	Range	160.3-203.4	155.3-196.8.	3.7-7.6

Table A-2 Amounts of mercury vapor remained in the crushing unit

Sodium sulfide times the stoichiometric amount of Hg vapor	Mercury concentration (mg/m^3)				
	Sam#1	Sam#2	Sam#3	Average	SD
Water	9.16	6.52	8.76	8.15	1.42
1	6.94	5.26	7.02	6.40	1.00
1.25	1.30	2.74	1.46	1.83	0.79
1.5	4.28	5.11	3.97	4.46	0.59
1.75	0.81	0.84	0.66	0.77	0.09
2.0	5.10	7.16	3.59	5.29	1.79
2.5	2.62	3.83	1.83	1.76	1.01

TableA- 3 Mercury concentrations leached from composite samples

Samples	Mercury concentration (mg/l)
1	0.038
2	0.044
3	0.044
4	0.050
5	0.052
6	0.054
7	0.084
8	0.031
9	0.068
10	0.071
11	0.060
12	0.055
Average	0.054
Median	0.053
SD	0.015

Table A-4 Mercury concentrations leached from stabilised/solidified wastes

Sodium sulfide concentration (times)	W/C ratio	Mercury concentration (mg/l)				
		#1	#2	#3	Average	SD
Water	0.5	0.031	0.018	0.005	0.018	0.013
	0.75	0.027	0.019	0.011	0.019	0.008
	1.0	0.035	0.019	0.016	0.023	0.010
Na_2S (1.00)	0.5	0.020	0.019	0.017	0.018	0.002
	0.75	0.025	0.016	0.016	0.019	0.005
	1.0	0.029	0.035	0.020	0.028	0.007
Na_2S (1.25)	0.5	0.040	0.053	0.052	0.048	0.007
	0.75	0.072	0.055	0.045	0.057	0.013
	1.0	0.094	0.069	0.061	0.075	0.017
Na_2S (1.50)	0.5	0.055	0.063	0.054	0.057	0.005
	0.75	0.080	0.084	0.050	0.071	0.019
	1.0	0.099	0.109	0.056	0.088	0.028
Na_2S (1.75)	0.5	0.060	0.100	0.034	0.065	0.034
	0.75	0.091	0.129	0.077	0.099	0.027
	1.0	0.089	0.098	0.080	0.089	0.009
Na_2S (2.00)	0.5	0.018	0.004	0.021	0.014	0.009
	0.75	0.032	0.047	0.017	0.032	0.015
	1.0	0.030	0.039	0.016	0.028	0.011
Na_2S (2.50)	0.5	0.016	0.049	0.015	0.026	0.019
	0.75	0.019	0.042	0.023	0.028	0.012
	1.0	0.044	0.080	0.040	0.055	0.022

Table A-5 pH of extraction fluid before rotating

Sodium sulfide concentration (times)	W/C ratio	pH of extraction fluid				
		#1	#2	#3	Average	SD
Water	0.5	11.91	11.93	11.20	11.68	0.42
	0.75	11.80	11.94	12.13	11.95	0.16
	1	11.82	11.90	12.08	11.93	0.13
Na_2S (1.00)	0.5	11.89	11.88	12.03	11.93	0.08
	0.75	11.85	11.94	12.07	11.95	0.11
	1	11.72	11.95	12.08	11.92	0.18
Na_2S (1.25)	0.5	11.78	11.97	12.03	11.93	0.13
	0.75	11.82	11.90	12.07	11.93	0.13
	1	11.79	11.87	12.07	11.91	0.15
Na_2S (1.50)	0.5	11.78	11.85	12.08	11.90	0.16
	0.75	11.68	11.88	12.11	11.89	0.22
	1	11.82	11.92	12.06	11.93	0.12
Na_2S (1.75)	0.5	11.70	11.86	12.09	11.88	0.20
	0.75	11.78	11.95	11.99	11.91	0.11
	1	11.87	11.82	12.10	11.93	0.15
Na_2S (2.00)	0.5	11.90	11.84	12.08	11.94	0.12
	0.75	11.87	11.81	12.04	11.91	0.12
	1	11.88	11.87	12.09	11.95	0.12
Na_2S (2.50)	0.5	11.95	11.82	12.17	11.98	0.17
	0.75	11.82	11.83	12.08	11.91	0.15
	1	12.01	11.88	12.08	11.99	0.10

Table A-6 pH of extraction fluid after rotating for 18 hours

Sodium sulfide concentration (times)	W/C ratio	pH of extraction fluid				
		#1	#2	#3	Average	SD
Water	0.5	12.48	12.62	12.56	12.55	0.07
	0.75	12.48	12.63	12.54	12.55	0.08
	1	12.48	12.62	12.53	12.54	0.07
Na_2S (1.00)	0.5	12.48	12.63	12.54	12.55	0.07
	0.75	12.48	12.64	12.55	12.55	0.08
	1	12.46	12.63	12.52	12.53	0.08
Na_2S (1.25)	0.5	12.47	12.64	12.64	12.59	0.10
	0.75	12.49	12.64	12.55	12.56	0.08
	1	12.48	12.64	12.64	12.58	0.09
Na_2S (1.50)	0.5	12.47	12.67	12.55	12.57	0.10
	0.75	12.49	12.49	12.56	12.51	0.05
	1	12.48	12.48	12.48	12.48	0.00
Na_2S (1.75)	0.5	12.48	12.673	12.557	12.57	0.10
	0.75	12.48	12.66	12.55	12.56	0.09
	1	12.45	12.64	12.45	12.51	0.11
Na_2S (2.00)	0.5	12.45	12.65	12.56	12.55	0.10
	0.75	12.50	12.66	12.55	12.57	0.08
	1	12.50	12.65	12.56	12.57	0.08
Na_2S (2.50)	0.5	12.49	12.669	12.56	12.57	0.09
	0.75	12.47	12.67	12.56	12.56	0.10
	1	12.52	12.52	12.55	12.53	0.02

Figure A-1 Discarded used fluorescent lamps

Figure A-2 Sorbent sample solution

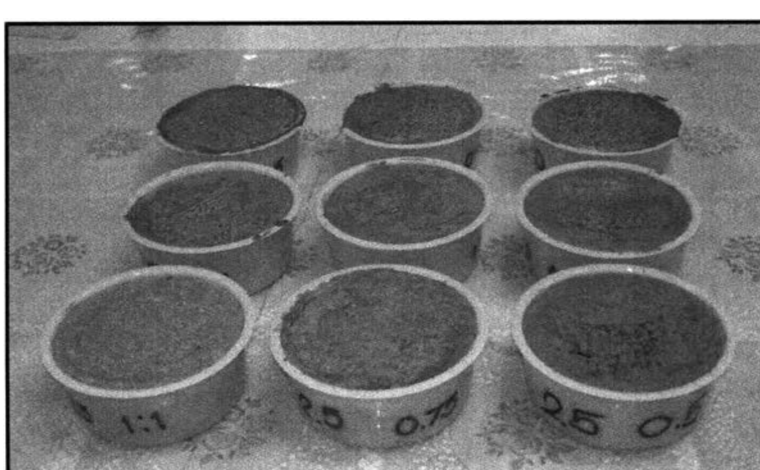

Figure A-3 Solidified wastes

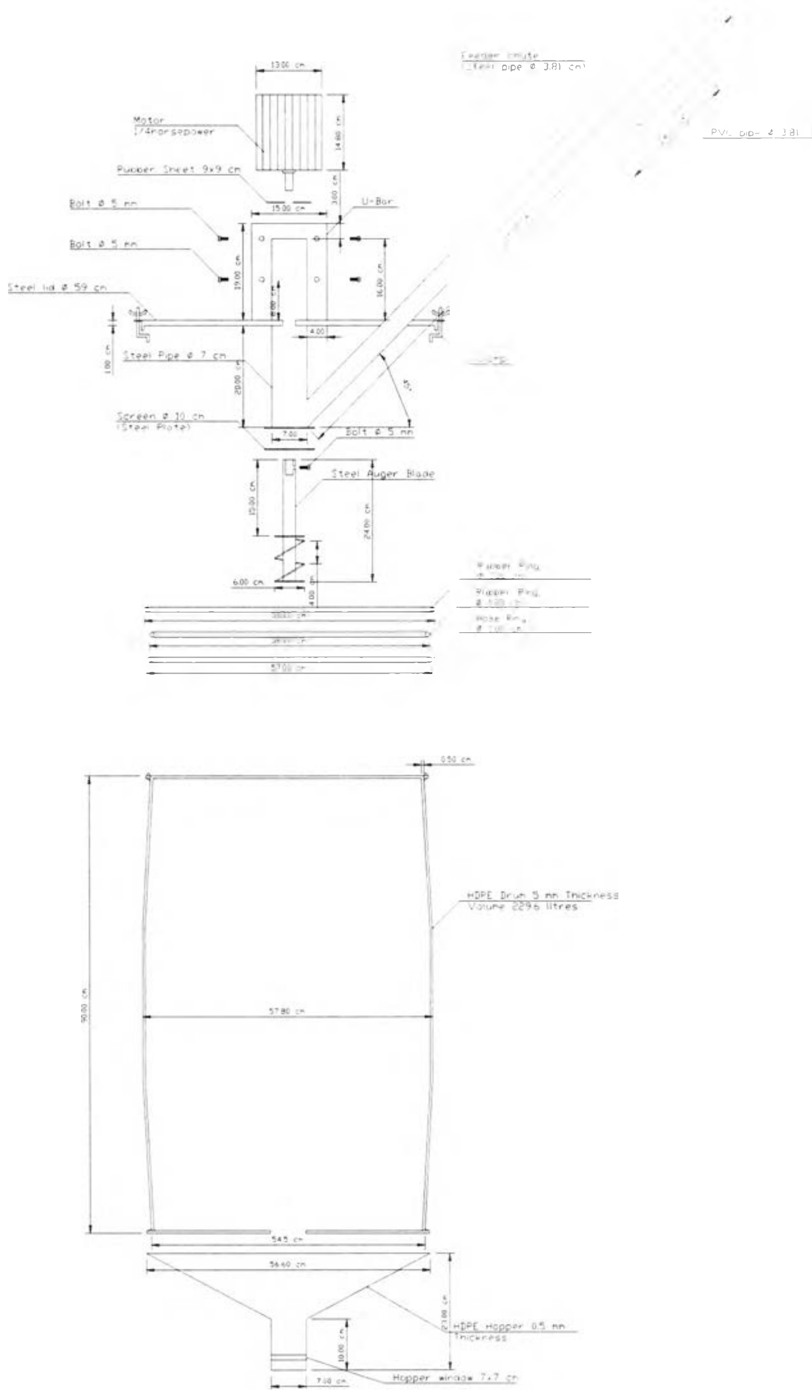

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Figure A-4 Detail of the fluorescent lamp crushing unit

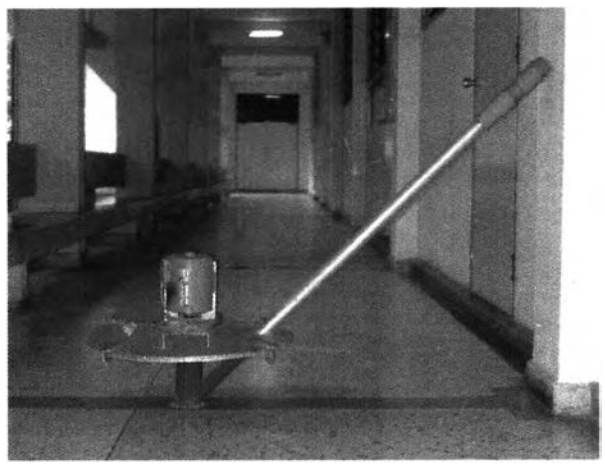


Figure A-5 A drum-mounted crusher

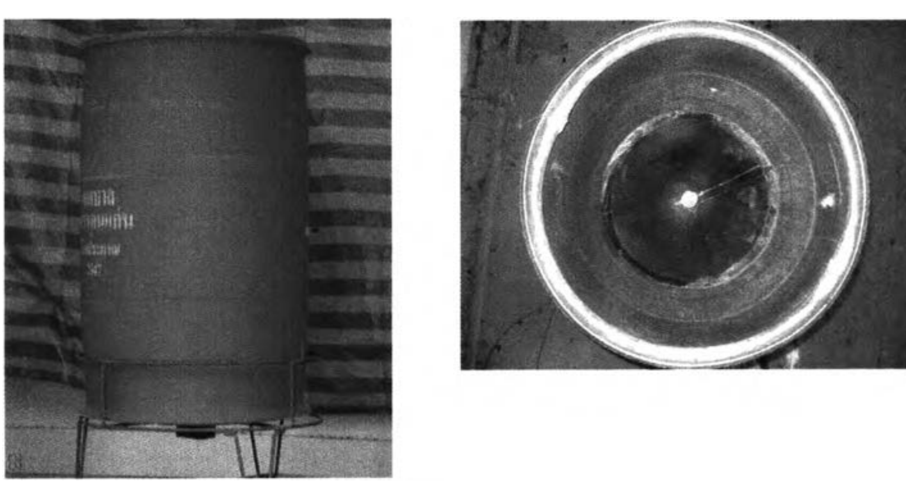
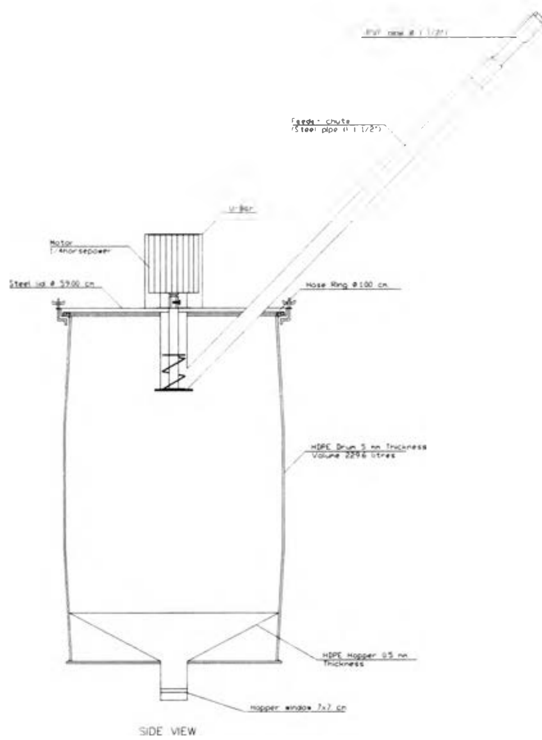


Figure A-6 A drum container



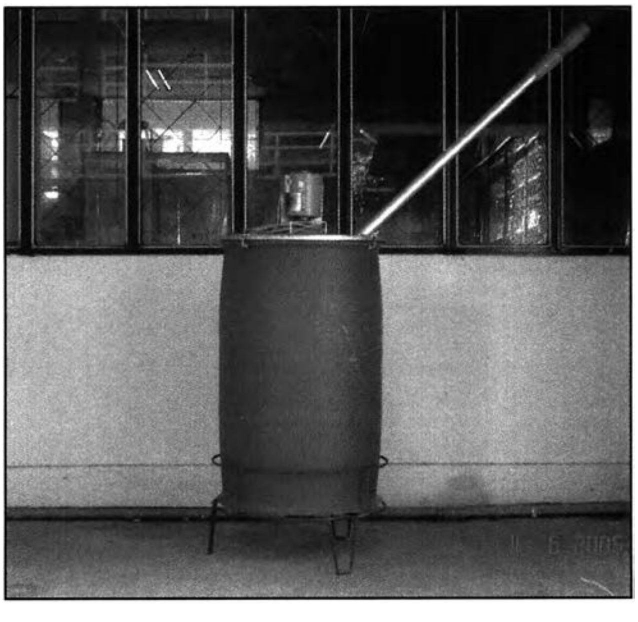
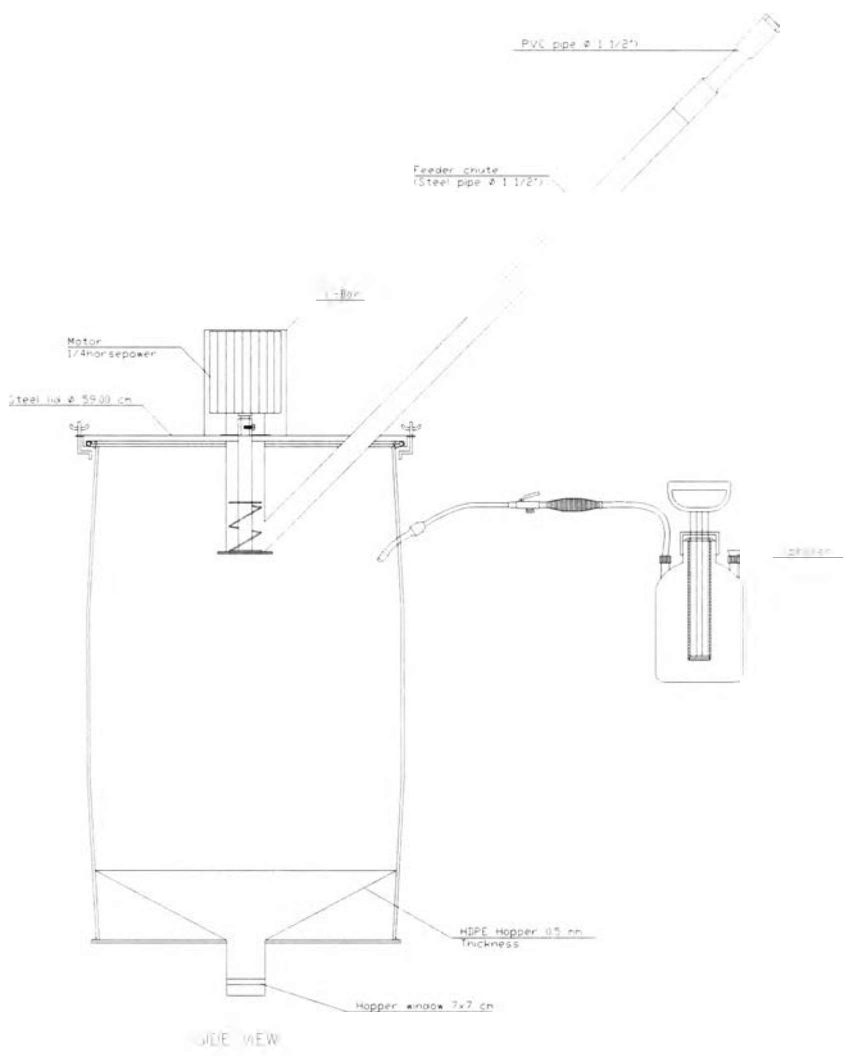


Figure A-7 The fluorescent lamp crushing unit



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APPENDIX B

Calculation for the amounts of sodium sulfide for spraying to capture Hg vapor from broken lamps

Amounts of mercury vapor released from crushed spent lamps

Sample number	mg/set
#1	5.56
#2	6.91
#3	7.00
Mean	6.49
SD	0.81

Remark: 3 lamps/set

Molecular weight

Hg = 200.59

S = 32

Use sodium sulfide in form of Na2S.9H2O

Reaction equation

$Hg^{2+}_{(aq)}$ + $S^{2-}_{(aq)}$ -----------> $HgS_{(s)}$

1 mole : 1 mole 1mole

200.59 mg: 32 mg 232.59 mg

6.49 mg : ? mg

Therefore use S = 6.49x32/200.59 = 1.04 mg

And S in form of Na2S.9H2O = 1.04 x 240.18/32 = 7.8 mg

One times stoichiometric amount = 7.8 mg

The amounts of Na2S.9H2O for spraying

1.0 Times	1.25 Times	1.5 Times	1.75 Times	2.0 Times	2.5 Times
7.8 mg	9.75 mg	11.7 mg	13.65 mg	15.6 mg	19.5 mg

MERCURY VAPOR IN WORKPLACE ATMOSPHERES

Method no.:	ID-140
Matrix:	Air
OSHA Permissible Exposure Limits	
Mercury Vapor (Final Rule Limit):	0.05 mg/m^3 (TWA)
Mercury (Transitional Limit):	0.1 mg/m^3 as total mercury (TWA)
Collection Device:	A passive or an active sampling device are available. Both devices use Hydrar[R] or hopcalite as the solid sorbent.
Recommended Sampling Rate	
Passive Dosimeter:	0.020 L/min (@ 20 °C and 101 kPa)
Active Sampler:	0.20 L/min
Recommended Air Volume Range	
Passive Dosimeter:	9.6 L
Active Sampler:	3 to 100 L
Analytical Procedure:	The sorbent is digested using nitric acid and hydrocloric acid. The mercury in the sample is reduced to elemental mercury using stannous chloride and analyzed using a cold vapor-atomic absorption spectrophotometer
Detection Limit	
Qualitative Passive Dosimeter:	0.002 mg/m^3 for a 240-min (4.8 L)sample
Active Sampler:	0.00067 mg/m^3 for a 75-min (15 L) sample
Quantitative	
Passive Dosimeter:	0.004 mg/m^3 for a 240-min (4.8 L) sample
Active Sampler:	0.0013 mg/m^3 for a 75-min (15 L) sample
Precision and Accuracy	
Passive Dosimeter Validation Range:	0.061 to 0.20 mg/m^3
CV_T(pooled)	0.039
Bias	+0.008
Overall Error	±8.6%
Method Classification:	Validated Method
Date (Date Revised)	1987 (June, 1991)

1.Introduction

This method describes the collection of airborne elemental mercury in a passive dosimeter or active sampling device and subsequent analysis using a cold vapor-atomic absorption spectrophotometer (CV-AAS).

1.1. Principle

The mercury dosimeter samples the workplace atmosphere by controlled diffusion into the badge while the active sampler uses a calibrated sampling pump. The mercury vapor entering either passive or active device is collected on a solid sorbent (Hydrar^R or hopcalite) which has an irreversible affinity for mercury (8.1., 8.2.). After sample collection the sorbent is initially dissolved with concentrated nitric acid and then hydrochloric acid. Stannous chloride is added to an aliquot of the sample to generate mercury vapor. This vapor is then driven into an absorption cell of a flameless atomic absorption spectrophotometer for analysis.

1.2. History

Previously, mercury samples were collected on iodine-impregnated charcoal contained in glass tubes. The treated charcoal was analyzed for mercury by placing it in a tantalum sampling boat and then heating to drive the mercury vapor into the beam of an atomic absorption spectrophotometer (8.3.). The amount of mercury was determined by absorbance at253.7 nm. The detection limit was approximately 0.1 μg. Drawbacks with this method were:

a. The mercury vapor and hence the entire sample was immediately lost into the surrounding atmosphere

b. The method was imprecise at lower sample loadings (8.4.)

c. The analytical technique was somewhat tedious

Hopcalite solid sorbent (8.5.) was substituted in place of the iodine-impregnated charcoal for mercury vapor sampling. Previously, hopcalite had been used in respirator cartridges for carbon monoxide and consisted of oxides of copper, manganese, cobalt, and silver (8.6.). Analysis of recent batches of hopcalite used for mercury collection indicate the composition was mainly oxides of manganese and copper.

Hydrar^R has been used as a substitute for collecting mercury vapor and is very similar in composition to hopcalite. A ceramic material, insoluble in nitric and hydrochloric acid, is present in the Hydrar^R but not in the hopcalite.

1.3. Advantages and Disadvantages

1.3.1. These sampling and analytical techniques have adequate sensitivity for measuring workplace atmospheric concentrations of elemental mercury.

1.3.2. The passive dosimeter used for collection of mercury vapor is small, lightweight, and requires no sampling pumps. Also, the dosimeter housing is reusable; therefore, cost per measurement is kept to a minimum.

1.3.3. The collected mercury sample is stable for at least 30 days.

1.3.4. Sample preparation for analysis involves simple procedures.

1.3.5. Either sampling device can be analyzed in any laboratory equipped with a CV-AAS.

1.3.6. A disadvantage with the passive dosimeter is particulate compounds cannot be collected with the device. A separate sampling pump and collection media should be used for particulate collection.

1.3.7. Another disadvantage with the dosimeter is sample rate dependence on face velocity. The dosimeter should not be used in areas where the air velocity is greater than 229 m/min (750 ft/min) since erratic increases in sampling rate may occur.

1.3.8. A disadvantage with the active device is the dependence on a calibrated pump to take the sample.

1.4. Toxic Effects (This section is for information only and should not be taken as a basis for OSHA policy.)
Exposure to elemental mercury vapor can occur via the respiratory tract and skin. Possible symptoms from an acute exposure include severe nausea, vomiting, abdominal pain, bloody diarrhea, kidney damage, and death. These symptoms usually present themselves within 10 days of exposure. Potential symptoms from a chronic exposure include inflammation of the mouth and gums, excessive salivation, loosening of the teeth, kidney damage, muscle tremors, jerky gait, spasms of the extremities, personality changes, depression, irritability, and nervousness (8.7., 8.8.).

1.5. Workplace Exposure

Occupations with potential exposure to mercury and its compounds are listed (8.8.):

amalgam makers	fur processors
bactericide makers	gold extractors
barometer makers	histology technicians
battery makers, mercury	ink makers
boiler makers	insecticide makers
bronzers	investment casting workers
calibration instrument makers	jewelers
cap loaders, percussion	laboratory workers, chemical
carbon brush makers	lampmakers, fluorescent
caustic soda makers	manometer makers

ceramic workers	mercury workers
chlorine makers	miners, mercury
dental amalgam makers	neon light makers
dentists	paint makers
direct current meter workers	paper makers
disinfectant makers	percussion cap makers
disinfectors	pesticide workers
drug makers	photographers
dye makers	pressure gage makers
electric apparatus makers	refiners, mercury
electroplaters	seed handlers
embalmers	silver extractors
explosive makers	switch makers, mercury
farmers	tannery workers
fingerprint detectors	taxidermists
fireworks makers	textile printers
fungicide makers	thermometer makers
fur preservers	wood preservative workers

1.6. Properties (8.7., 8.8.)

Elemental mercury (CAS No. 7439-97-6) is a silver-white, heavy, mobile, liquid metal at room temperature. Some physical properties and data for mercury are:

Atomic Number	80
Atomic Symbol	Hg
Atomic Weight	200.61
Freezing Point	-38.87 °C
Boiling Point	356.90 °C
Density	13.546 g/mL (20 °C)
Synonyms	Quicksilver, Hydrargyrum

The high vapor pressure of mercury at normal temperatures combined with the potential toxicity makes good control measures necessary to avoid exposure. Also, the concentration of mercury vapor in the air rapidly increases as the temperature increases. To illustrate, listed below are vapor pressures of mercury, and mercury concentrations of air after saturation with mercury vapor at different temperatures:

Vapor Pressure-Saturation Concentration of Mercury at Various Temperatures

Temperature °C	Temperature °F	Vapor Pressure (torr)	Mercury Concentration ($\mu g/m^3$)
0	32.0	0.000185	2,180
10	50.0	0.000490	5,880
20	68.0	0.001201	13,200
24	75.2	0.001691	18,300
28	82.4	0.002359	25,200
30	86.0	0.002777	29,500
32	89.6	0.003261	34,400
36	96.8	0.004471	46,600
40	104.0	0.006079	62,600

2. Range

2.1. The qualitative and quantitative detection limits for the analytical procedure are 0.01 μg and 0.02 μg mercury, respectively (8.9.).

2.2. Working Range

The range of the analytical procedure has been determined to be 0.1 to 2 μg mercury. Using the analytical conditions specified, a nonlinear response was noted above 2 μg.

3. Method Performance

3.1. The SKC Hydrar[R] gas monitoring dosimeter badge for mercury (SKC Inc., Eighty Four, PA) was evaluated at 80% RH and 25 °C over the range of 0.061 to 0.203 mg/m^3 using a dynamic generation system (8.2.). The pooled coefficient of variation (CV_T) for badge samples taken in this concentration range was 0.039. The average recovery was 100.8% and the overall error was ±8.6%. In a separate study, active samplers were spiked with mercury in the range of 1 to 2.5 μg. The mean recovery of these 125 quality control samples was 96.9% with a CV_1 of 0.106 (8.10.).

3.2. In storage stability studies, the mean recoveries of Hydrar[R] samples analyzed 5, 14, and 30 days after collection were within ±10% of the known generated concentration (8.2.).

3.3. The Hydrar[R] active sampling device was compared using linear regression statistics to the dosimeter in a field study (8.11.). The dosimeter results agreed well with the active sampler and are summarized below (Note: A correlation coefficient and slope = 1 would indicate ideal agreement):

Number of paired samples	(N)	= 26
Concentration Range		= 0.01 to 0.7 mg/m^3
Correlation coefficient	(r)	= 0.985
Intercept	(a)	= 0.017
Slope	(b)	= 0.960
Standard deviation of the slope	(Sb)	= 0.038

4. Interferences

4.1. Sampling:

Particulate mercury compounds are a positive interference; however, the badge does not sample particulates and the glass wool of the active sampler prevents particulate from entering the sorbent. Chlorine in the sampled air does not interfere when using Hydrar^R or hopcalite sorbent. The chlorine does react with available mercury vapor in the air to presumably form mercuric chloride (8.12.). Workplaces containing both chlorine and mercury should be sampled for both mercury vapor and particulate.

4.2. Analysis:

Organic-free deionized water should be used during sample and standard preparation. Any compound with the same absorbance wavelength as mercury (253.7 nm) can be a positive interference. Some volatile organic compounds (i.e. benzene, toluene, acetone, carbon tetrachloride) absorb at this wavelength and are considered analytical interferences. They occur as contaminants in the reagents used during sample preparation. These compounds are not expected to be retained on Hydrar^R or hopcalite during sample collection. Analytical interferences are rendered insignificant by using organic-free deionized water and at least reagent grade chemicals or by blank subtraction.

Increasing the concentration of nitric acid in the samples or standards appears to produce an elevated background signal. The nitric acid concentration in the samples and standards should not be greater than 10%.

5. Sampling

[Note: A prefilter assembly, consisting of a mixed-cellulose ester filter in a polystyrene cassette, can be used with the active samplers. Although a significant loss of mercury vapor, presumably due to the prefilter assembly, has been noted when using this type of sampling train (8.12.), these results were not duplicated in a series of recent experiments(8.13.).]

5.1. Equipment

Either tubes or dosimeters can be used to collect mercury vapor. The dosimeter should not be used when:

1. The air velocity of the sampling site is greater than 229 m/min (750 ft/min)
2. The operation being sampled is characterized by extremely poor hygienic practices and splashing of mercury on the badge may occur
3. Determination of compliance to the Transitional Permissible Exposure Limit (PEL) to total mercury is necessary and mercury particulate appears to be present in the workplace atmosphere

The tube can be used to determine compliance with the Transitional PEL of 0.1 mg/m^3 (*TWA*) **as total mercury (vapor + particulate)**. The badge can only collect mercury vapor. For Ceiling exposures to particulate mercury, or for wipe and bulk sampling and analysis consult reference 8.14. for further information.

5.1.1. *PASSIVE DOSIMETER:*

Gas monitoring dosimeter badge and pouch containing a $Hydrar^R$ capsule [badge - cat. no. 520-03, pouch - cat. no. 520-02 (SKC Inc., Eighty Four, PA)]. The capsule contains 800 mg of sorbent.

5.1.2. *ACTIVE SAMPLER:*

$Hydrar^R$ or hopcalite sampling tubes (cat. no. 226-17-1 or 226-17-lA, SKC, Inc., Eighty Four, PA). These are 6-mm o.d. × 70-mm long glass tubes which contain 200 mg of sorbent.

Note: Before use, the active sampling tubes must be examined for movement of the the solid sorbent into the glass wool. See Section 5.3.1. for further details.

5.1.3. Sampling pumps capable of sampling at 0.2 liters per minute (L/min).
5.1.4. Assorted flexible tubing.
5.1.5. Stopwatch and bubble tube or meter for pump calibration.

5.2. Sampling Procedure - *PASSIVE DOSIMETER*

5.2.1. Assemble the components of the mercury monitoring badge according to manufacturer instructions (8.1.).

Note: A foam insert must be placed in the Model 520-03 dosimeter to hold the capsule in place (8.13.).

5.2.2. Record the sampling start time, sampling site temperature, and atmospheric pressure. Remove the protective cap and then place the dosimeter in the breathing zone of the employee. The suggested sampling time for the dosimeter is 8 h.

5.2.3. Immediately after sampling, carefully remove the sorbent capsule from the dosimeter and place it in the sorbent pouch. Fold the pouch top twice and press it flat to seal the capsule inside the pouch. Record the sampling stop time, final temperature, and atmospheric pressure. Calculate and record the total sampling time, average temperature, and pressure.

5.3. Sampling Procedure - *ACTIVE SAMPLER*

5.3.1. Calibrate each personal sampling pump with an active sampler in-line using a flow rate of about 0.2 L/min.

Note: A prefilter assembly consisting of a mixed-cellulose ester filter, polystyrene cassette, and a ***minimum*** amount of Tygon tubing can be used if:

a. particulate mercury compounds may present a problem during sampling or

b. the hopcalite or Hydrar^R contained in the active sampling tube has migrated to the glass wool plug.

Before use, the active sampling tubes must be examined for movement of the solid sorbent into the glass wool. Certain lots of Hydrar^R or hopcalite have been noted as being very friable or having a sorbent particle-size range small enough as to allow migration. This movement can easily be noted - the glass wool in the sampling tube appears somewhat discolored (darkened) from the small sorbent particles. If sorbent migration has occurred, a prefilter assembly is recommended. The recommended sampling flow rate is also 0.2 L/min with the prefilter-sampling tube-pump assembly.

5.3.2. Connect a sampling tube (or sampling assembly) to a calibrated pump using flexible tubing. If a prefilter is used, connect it to the sampling tube with a minimum amount of Tygon tubing. Connect the other end of the sampling tube to the pump. Place the sampling tube (or assembly) in the breathing zone and the pump in an appropriate position on the employee.
5.3.3. Use an air volume in the range of 3 to 100 L to collect the mercury in the workplace air. Record the total volume.
5.3.4. Replace the plastic end caps on the active sampler after sampling is completed.

5.4. Sample Shipment

5.4.1. Securely wrap each sorbent pouch or active sampling tube end-to-end with an OSHA Form 21 sample seal. Also seal and prepare cassettes if a prefilter assembly was used.

5.4.2. Submit at least one blank sample with each set of samples. The blank sample should be handled in the same manner as the other samples except that an air sample is not taken.
5.4.3. Request the laboratory to analyze the samples for mercury. Submit any pertinent sampling information to the lab. *Record if a prefilter assembly was used.*
5.4.4. Ship the sealed pouches and used dosimeter housings, or active sampling tubes to the laboratory in appropriate containers as soon as possible. The filter/cassette assembly can also be submitted for mercury particulate analysis; however, sampling periods may be longer than reflected in exposure regulations. The PEL for mercury particulate is a Ceiling(8.14.) and the vapor is a TWA PEL.

6. Analysis

6.1. Safety Precautions
6.1.1. Wear safety glasses, lab coat, and gloves at all times.
6.1.2. Handle acid solutions with care. Avoid direct contact of acids with work area surfaces, eyes, skin, and clothes. Flush acid solutions which contact the skin or eyes with copious amounts of cold water.
6.1.3. Prepare solutions containing hydrochloric acid in an exhaust hood and store in narrow-mouthed bottles.
6.1.4. Keep B.O.D. bottles containing stannous chloride/hydrochloric acid solutions capped when not in use to prevent inhalation of noxious vapors.
6.1.5. Exercise care when using laboratory glassware. Do not use chipped pipets, volumetric flasks, beakers or any glassware with sharp edges exposed.
6.1.6. Never pipet by mouth.
6.1.7. When scoring the glass of active samplers to remove the sorbent before analysis, score with care. Apply only enough pressure to scratch a clean mark on the glass. Use a paper towel or cloth to support the opposite side while scoring. Moisten the mark with DI H_2O and wrap the tube in cloth before breaking. If the tube does not break easily, re-score. Dispose of glass in a waste receptacle specifically designed and designated for broken-glass.
6.1.8. Always purge the mercury from the CV-AAS into an exhaust vent.
6.1.9. Occasionally monitor the CV-AAS for mercury vapor leaks using an appropriate direct reading instrument.
6.2. Equipment - Cold Vapor Analysis
(Note: Specific equipment is listed for illustration only)
6.2.1. Atomic absorption spectrophotometer (model 503, Perkin-Elmer, Norwalk, CT).
6.2.2. Mercury hollow cathode lamp or electrodeless discharge lamp and power supply.
6.2.3. Biological Oxygen Demand (B.O.D.) bottles, borosilicate glass, 300 mL.
6.2.4. Peristaltic pump, 1.6 to 200 mL range, and controller, 1-100 rpm range (Masterflex model 7553-30 with model 7015 head, Cole-Parmer, Chicago, IL).
6.2.5. Quartz absorption cell, 22-mm (7/8 in) o.d. × 152-mm (6 in) long (part no. 303-3101, Perkin-Elmer).
6.2.6. Heating tape.
6.2.7. Variable transformer 50-60 Hz, 10 A, 120 V input, 0-140 V output, 1.4 kW (Superior Electric, Bristol, CT).

6.2.8. Tygon peristaltic pump tubing (part no. N06409-15, Cole-Parmer) and glass tubing.

6.2.9. Aerator (part no. 0303-3102, Perkin-Elmer).
6.2.10. Chart recorder.
6.2.11. Desiccant (Drierite, W.A. Hammond Drierite Co., Xenia, OH).
6.2.12. Volumetric flasks, volumetric pipets, beakers, and other laboratory glassware.
6.2.13. Automatic pipets, adjustable, 0.1 to 5.0 mL range (models P-1000 and P-5000, Rainin Instruments Co., Woburn, MA).
6.2.14. Glass tube scorer, or needle, 21 to 25 gauge - for removing metal screens in dosimeters or glass wool from tubes. A piece of bent wire can also be used.
6.2.15. Exhaust vent.

6.3. Reagents - All reagents should be at least reagent grade.

Stannous chloride, ($SnCl_2$)
6.3.1. Deionized water (DI H_2O), organic-free.
6.3.2. Hydrochloric acid (HCl), concentrated (36.5 to 38%), with a mercury concentration less than 0.005 ppm.
6.3.3. Mercury standard stock solution, 1,000 µg/mL: Use a commercially available certified standard or, alternatively, dissolve 1.0798 g of dry mercuric oxide (HgO) in 50 mL of 1:1 hydrochloric acid and then dilute to 1 L with DI H_2O. Store this reagent in a dark environment, preferably in an amber colored container.
6.3.4. Nitric acid (HNO_3), concentrated (69 to 71%), with a mercury concentration less than 0.005 ppm.
6.3.5. Nitric acid, 1:1: Carefully add equal portions of concentrated HNO_3 and DI H_2O.
6.3.6. Nitric acid, 10%: Carefully add 100 mL concentrated HNO_3 to 900 mL DI H_2O.
6.3.7. Stannous chloride ($SnCl_2$) solution, 10%: Dissolve 20 g $SnCl_2$ in 100 mL concentrated HCl. Slowly and carefully pour this solution into 100 mL DI H_2O and then mix well. Transfer and store the final solution in a capped B.O.D. bottle to prevent oxidation. Prepare this solution before each new analysis.
6.4. Glassware Preparation
6.4.1. Clean the B.O.D. bottles and stoppers with 1:1 HNO_3 and thoroughly rinse with DI H_2O prior to use.
6.4.2. Rinse all other glassware with 10% nitric acid and then with DI H_2O prior to use. Air dry all 50-mL volumetric flasks to be used in sample preparation.
6.5. Standard Preparation
6.5.1. Prepare a 1 µg/mL mercury standard by making appropriate ten-fold serial dilutions of the 1,000 µg/mL mercury standard stock solution with 10% HNO_3.
6.5.2. Prepare working mercury standards (ranging from 0.1 to 2.0 µg) and reagent blanks *immediately prior to use*. A few standards at each concentration should be made. Add an appropriate aliquot of the 1 µg/mL standard to a clean B.O.D. bottle containing enough 10% HNO_3 to bring the total volume to 100 mL. A suggested dilution scheme is given:

Mercury Standard (μg)	Aliquot (mL)*	Final Volume (mL)
Reagent Blank	0	100
0.1	0.1	100
0.2	0.2	100
0.5	0.5	100
1.0	1.0	100
1.5	1.5	100
2.0	2.0	100

* Aliquot taken from 1 μg/mL standard prepared in Section 6.5.1.

6.6. Sample Preparation [Note: A hooked needle or piece of fine wire is useful to remove the dosimeter screen or glass wool (active sampler) and the sorbent particles.]

6.6.1. *DOSIMETER*

Open each sample pouch and remove the sorbent capsule. Carefully remove the screen from the top of the capsule without losing any sorbent. Carefully pour the sorbent into a clean, dry 50-mL flask without spilling any. Discard the screen and empty capsule.

6.6.2. *ACTIVE SAMPLER*

Score the tube with a glass tube cutter (also see Section 6.1.7.) and then break open the front section of the tube above the glass wool. An alternative approach to scoring and breaking is to carefully remove the glass wool with a bent wire or needle.

a. *If a prefilter was not used* during sampling, place the glass wool and sorbent into separate 50-mL volumetric flasks.

b. *If a prefilter was used* and the glass wool appears to contain hopcalite or Hydrar, the glass wool can be analyzed along with the sorbent. Carefully transfer the glass wool and sorbent to a 50-mL volumetric flask without losing any of the particles.

6.6.3. *Prefilter*
Prepare and analyze any prefilters according to reference 8.14.

6.6.4. Add 2.5 mL of concentrated HNO_3 followed by 2.5 mL concentrated HCl to each volumetric flask [Note: To minimize any loss of mercury through a change in oxidation state, the HNO_3 is added before the HCl (8.5.)].

6.6.5. Gently swirl the sample occasionally for approximately 1 h. If Hydrar[R] was used to collect the sample, the dark brown solution will also contain some undissolved clear to white-tan colored ceramic material.

6.6.6. Carefully dilute to a 50-mL total volume with DI H_2O. The final *sorbent* sample solution will be light blue or blue-green. This is a good place to stop if the analysis cannot be completed the same day.

6.7. Analysis - Instrument Parameters

6.7.1. Set up the CV-AAS
6.7.2. Wrap the heating tape around the quartz cell and then turn on the variable transformer. The heat setting on the tape should be sufficient to prevent water vapor condensation in the absorption cell.
6.7.3. Place the aerator in a B.O.D. bottle which contains approximately ½ to 1 inch of desiccant. Operate the peristaltic pump for approximately 30 min at full speed to remove any water vapor from the system.
6.7.4. Operate the hollow cathode or electrodeless discharge mercury lamp at the manufacturer's recommended current or power rating.
6.7.5. Use the following settings (Note: The mentioned instrument settings are for specific models used at the OSHA-SLCAL. If instrumentation other than what is specified in Section 6.2. is used, please consult the instrument manufacturer's recommendations.):

Atomic Absorption Spectrophotometer:

Slit	0.7 nm
Signal	Repeat Mode
Function	ABS
Mode	ABS
Range	UV
Wavelength	253.7 nm
Filter	Out
EM Chopper	Off
Phase	Normal

Strip Chart Recorder:

Chart Speed	5 mm/min
Chart Range	10 mV

6.7.6. Optimize the ENERGY meter reading at 253.7 nm.
6.7.7. Align the beam of the mercury lamp so it passes directly through the center of the quartz cell windows. This can be accomplished by adjusting the burner height, depth, and angle knobs to give a minimum ABSORBANCE reading.

6.7.8. Operate the peristaltic pump at full speed. Rinse the aerator with DI H_2O and insert it into a holder in the exhaust vent.

6.7.9. Perform the following steps to obtain a baseline signal near an absorbance of zero:

1. start the chart recorder,
2. set the spectrophotometer absorbance reading to zero,
3. wait until the baseline stops drifting,
4. set the reading to zero again

6.8. Analysis

6.8.1. Samples: *Immediately before analyzing*, transfer an appropriate aliquot of the sample solution to a clean B.O.D. bottle containing enough 10% HNO_3 solution to bring the total volume to 100 mL. The transfer must be done with a volumetric pipet.
6.8.2. Standards: *Immediately before analyzing*, prepare standards according to instructions listed in Section 6.5.2.
6.8.3. Deliver 5 mL of the 10% $SnCl_2$ solution with an automatic pipet to a B.O.D. bottle containing a standard, reagent blank, or sample to be analyzed. Immediately place the aerator into the solution with the peristaltic pump operating at full speed.
6.3.4. Record the maximum absorbance reading and label the signal produced on the strip chart.
6.8.5. Stop the pump, remove the B.O.D. bottle from the CV-AAS and stopper it. Rinse the aerator with DI H_2O and insert it into a holder in the exhaust vent. Turn the pump on at full speed until the CV-AAS system is purged of mercury and the baseline returns to zero.
6.8.6. If the absorbance reading of a sample is greater than the highest standard *at any time* during analysis, *immediately* remove the B.O.D. bottle from the CV-AAS. Purge the system following the procedure listed in Section 6.8.5. Take a smaller aliquot or dilute the high concentration sample and re-analyze. Make any necessary sample dilutions with 10% HNO_3 and use the appropriate dilution factor when calculating results.
6.8.7. Repeat Sections 6.8.3. through 6.8.5. for each prepared standard, reagent blank, or sample.
6.9. Analytical Recommendations
6.9.1. It is recommended to analyze the reagent blank, lowest, and highest standard two or three times each to check for contamination, reproducibility, and sensitivity before starting the sample analysis. A 2.0-µg mercury standard should give a three-quarter to full-scale deflection on the chart recorder and an absorbance unitreading of about 0.850 when using the equipment and conditions specified. The lowest and highest standard should provide a linear response and the lowest standard should be at least two to three times the blank signal.
6.9.2. It is also recommended to analyze an entire series of standards (including the reagent blank) at the beginning and end of the sample analysis to ensure standard readings are reproducible. As a general guideline, standard readings should be within ±10% throughout the analysis.

6.9.3. A standard near the concentration range of the samples should be analyzed after every four to five samples.
6.9.4. Quality control (QC) samples should be prepared and analyzed using the same matrix and analytical conditions as the samples. If possible, the QC samples should be generated from an independent source.
6.9.5. Approximately 10% of the samples should be reanalyzed.

7. Calculations

7.1. Use a least squares regression program to plot a concentration-response curve of peak absorbance versus the amount (µg) of mercury in each standard.
7.2. Determine the amount (µg) of mercury, A, corresponding to the peak absorbance in each analyzed sample aliquot from this curve.
7.3. Calculate the total amount (µg) of mercury, W, in each sorbent or glass wool sample:

$$W = \frac{(A)\,(\text{sample volume, mL})\,(DF)}{(\text{aliquot, mL})}$$

Where:

DF = Dilution Factor (if none, DF = 1)
7.4. A blank correction is made for each sample (Note: When using the reagents and conditions specified, previous blank results have been less than 1 µg). Calculate the concentration of mercury in each sorbent or glass wool sample:

$$\text{mercury mg/m} = \frac{W - Wb}{\text{air volume, L}}$$

Where:

Wb = Total µg of mercury in the blank sample.
Air vol = Sampling time × flow rate (for ACTIVE SAMPLERS)
(Note: For PASSIVE DOSIMETERS, the sampling rate is affected by temperature and pressure. To correct for this, use:

$$\text{Air vol} = ST \times 0.020 \times (T_1/T_2)^{1.5} \times (P_2/P_1)$$

Where:

ST = Sampling time (min)
0.020 = Sampling rate (L/min) at 20 °C and 760 torr
T_1 = Sampling site temperature (K)
T_2 = 293 K
P_1 = Sampling site pressure (torr)
P_2 = 760 torr

7.5. Reporting Results to the Industrial Hygienist

For PASSIVE DOSIMETER samples, report results to the industrial hygienist as mg/m^3 mercury vapor.

For ACTIVE SAMPLERS, report results as:

a. mg/m^3 mercury vapor
b. mg/m^3 total mercury

For mercury vapor result a): *If a prefilter was used* and the glass wool and sorbent were combined:

mercury vapor = glass wool + sorbent

The prefilter (if used) was present during sampling to assure that mercury particulate was not trapped in the glass wool.

For total mercury result (b): The sum of the mercury found in the sorbent (vapor), glass wool, and prefilter (if used) for each active sampler is considered. This result is used to determine compliance to the Transitional PEL for total mercury. The Transitional PEL considers both the vapor and particulate fractions of mercury.

Any mercury particulate found on the prefilter can be assessed for compliance with the Ceiling PEL for mercury. See reference 8.14. for further details.

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BIOGRAPHY

Mr. Worawit Intrchom was born on December 14, 1973 in Phichit province, Thailand. He graduated with a Bachelor's degree from the Faculty of Public Health, Khon Kaen University in 1996. He has worked as an environmental official at the Office of Natural Resources and Environmental Policy and Planning since 1999 to the present. He studied for his Master's degree of Science in Environmental Management (Inter-Department), Chulalongkorn University, Thailand from May, 2003 to October, 2005.