

ANNEX E-8 .

METHOD 7060A

ARSENIC (ATOMIC ABSORPTION FURNACE TECHNIQUE)

1.0 SCOPE AND APPLICATION

1.1 Method **7060A** is an atomic absorption procedure approved for determining the concentration of arsenic **in** wastes, mobility procedure **extracts**, soils, and ground water. **All** samples must be subjected to **an** appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis by Method **7060A**, samples must be prepared in order to convert organic forms of arsenic to **inorganic** forms, to **minimize** organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix. Aqueous samples are subjected to the acid digestion procedure described in **this** method. Sludge samples are prepared using the procedure described in **Method 3050A**.

2.2 Following the appropriate dissolution of the sample, a representative aliquot of the digestate is spiked with a nickel titrate solution and is placed manually or by **means** of an automatic sampler into a graphite **tube** furnace. The sample aliquot is then slowly evaporated to dryness, charred (**ashed**), and atomized. The absorption of hollow cathode or **EDL** radiation during **atomization** will **be** proportional to the arsenic concentration. Other **modifiers** may be used in place of **nickel** nitrate if the analyst documents the chemical **and concentration** used.

2.3 The typical detection limit for water samples using this method is 1 **ug/L**. This detection limit may not be achievable when analyzing waste samples.

3.0 INTERFERENCES

3.1 Elemental arsenic and many of its compounds are volatile; therefore, samples may be subject to losses of arsenic during sample **preparation**. Spike samples and relevant standard reference materials should be processed to determine if the chosen dissolution method is appropriate.

3.2 Likewise, caution must be employed during the selection of **temperature** and times for the dry and char (ash) cycles. A nickel nitrate solution must be added to all digestates prior to analysis to minimize **volatilization** losses during **drying** and **ashing**.

3.3 In addition to the **normal** interferences experienced during graphite furnace analysis, arsenic analysis can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Arsenic analysis is **particularly** susceptible to these problems because of its low analytical wavelength (193.7 nm). **Simultaneous** background correction must be employed to avoid erroneously high results. Aluminum is a severe positive interferent in the analysis of arsenic, especially using D2 arc background **correction**. **Zeeman** background correction is very useful in this situation.

3.4 If the **analyte** is not completely **volatilized and** removed from the furnace during **atomization**, memory effects will occur. If this situation is detected by means of blank burns, the tube should be cleaned by operating the furnace at full power at regular intervals in the analytical scheme.

4.0 APPARATUS AND MATERIALS

4.1 **Griffin beaker** or equivalent: 250 mL.

4.2 Class A Volumetric flasks: 10-mL.

4.3 Atomic absorption **spectrophotometer**: Single or dual channel, single- or double-beam instrument having a grating **monochromator**, photo-multiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for **simultaneous** background correction and interfacing with a **strip-chart** recorder.

4.4 Arsenic hollow cathode lamp, or **electrodeless** discharge lamp (**EDL**): **EDLs** provide better **sensitivity** for arsenic analysis.

4.5 Graphite furnace: Any graphite furnace device with the appropriate temperature and timing controls.

4.6 Data systems recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and so that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can easily be **recognized**.

4.7 **Pipets**: Microliter with disposable tips. Sizes **can** range from 5 to 1,000 **uL**, as **required**.

5.0 REAGENTS

5.1 **Reagent water**: Water should be monitored for impurities. **All** references to water **will** refer to reagent water.

5.2 Concentrated nitric acid: Acid should be analyzed to determine levels of impurities. If a method blank using the acid is **<MDL**, the acid can be used.

5.3 Hydrogen peroxide (30%): Oxidant should be analyzed to determine levels of impurities. If a method blank using the **H2O2** is **<MDL**, the acid can be used.

5.4 Arsenic standard **stock** solution (1,000 **mg/L**): Either procure a certified aqueous standard from a supplier and **verify** by comparison with a **second** standard, or dissolve 1.3209 g of **arsenic trioxide (As2O3)**, analytical reagent grade) or equivalent in 100 **mL** of reagent water **containing** 4 g **NaOH**. Acidify the solution with **20 mL** concentrated **HN03** and dilute to 1 liter (1 **mL** = 1 mg As).

5.5 Nickel **nitrate** solution (5%): Dissolve 24.7809 g of **ACS** reagent grade **Ni(NO3)2·6H2O** or equivalent in reagent water and dilute to 100 **mL**.

5.6 Nickel nitrate solution (1%): Dilute 20 **mL** of the 5 % nickel nitrate to 100 **mL** with reagent water.

5.7 **Arsenic** working standards: Prepare dilutions of the stock solution to be used as **calibration** standards at the time of the analysis. Withdraw appropriate **aliquots** of the stock solution, add **concentrated HNO3**, 30 % **H2O2**, and 5 % nickel nitrate solution. Amounts added should be representative of the concentrations found in the **samples**. Dilute to 100 **mL** with reagent water.

6.0 **SAMPLE** COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nme of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and reagent water. Plastic and glass containers **are** both suitable.

6.3 Special containers (e.g., containers used for **volatile** organic analysis) may have to be used **if** very volatile arsenic compounds are to be analyzed.

6.4 Aqueous samples must **be** acid&d to a **pH** of **<2** with **nitric** acid and refrigerated prior to analysis.

6.5 Although waste samples do not need to be **refrigerated** sample handling and storage must comply with the minimum requirements established in **Chapter** One.

7.0 PROCEDURE

7.1 Sample preparation: Aqueous samples should be prepared in the manner described in Paragraphs 7.1.1-7.1.3. Sludge-type samples should be prepared **according** to Method 3050A. The applicability of a sample-preparation technique to a new matrix type must be demonstrated by analyzing **spiked** samples **and/or** relevant standard reference materials.

7.1.1 Transfer a known volume of well-mixed sample to a **250-mL Griffin** beaker or equivalent; add 2 **mL** of 30% **H2O2** and sufficient concentrated **HN03** to result in **an** acid concentration of 1% (v/v). Heat, until digestion is complete, at **~ 95 °C** or **until** the volume is slightly less than **50 mL**.

7.1.2 Cool and bring back to 50 **mL** with **reagent** water.

7.1.3 **Pipet 5 mL** of this digested solution into a **10-mL** volumetric flask, add 1 **mL** of the 1 A nickel nitrate solution, and dilute to 10 **mL** with reagent water. **The** sample is now ready for injection **into** the furnace.

7.2 The **193.7-nm** wavelength line and a background correction system **are** required. Follow the manufacturer's suggestions for all other **spectrophotometer** parameters.

7.3 Furnace parameters suggested by the manufacturer should be employed as **guidelines**. Because temperature-sensing **mechanisms** and temperature controllers can vary between **instruments** or with time, the **validity** of the furnace parameters must be periodically **confirmed** by **systematically altering** the furnace parameters while analyzing a standard. **In** this manner, losses of **analyte** due to overly high temperature settings or losses **in** sensitivity due to less than optimum settings can be minimized. **Similar verification** of furnace parameters may be required for complex sample matrices.

7.4 Inject a **measured** microliter aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and **reanalyzed**. The use of multiple injections can improve accuracy and help detect furnace **pipetting** errors.

8.0 QUALITY CONTROL

8.1 Refer to section 8.0 of **Method 7000A**.

9.0 METHOD PERFORMANCE

9.1 **Precision and** accuracy data are available in Method 206.2 of Methods for Chemical Analysis of Water and Wastes.

9.2 The optimal **concentration** range for aqueous samples using this method is 5- 100 **ug/L**. **Concentration** ranges for non-aqueous samples will vary with matrix type.

9.3 The data shown in Table 1 were obtained from **records** of state and contractor laboratories. The data are intended to show the precision of the combined sample preparation and analysis method.

10.0 REFERENCES

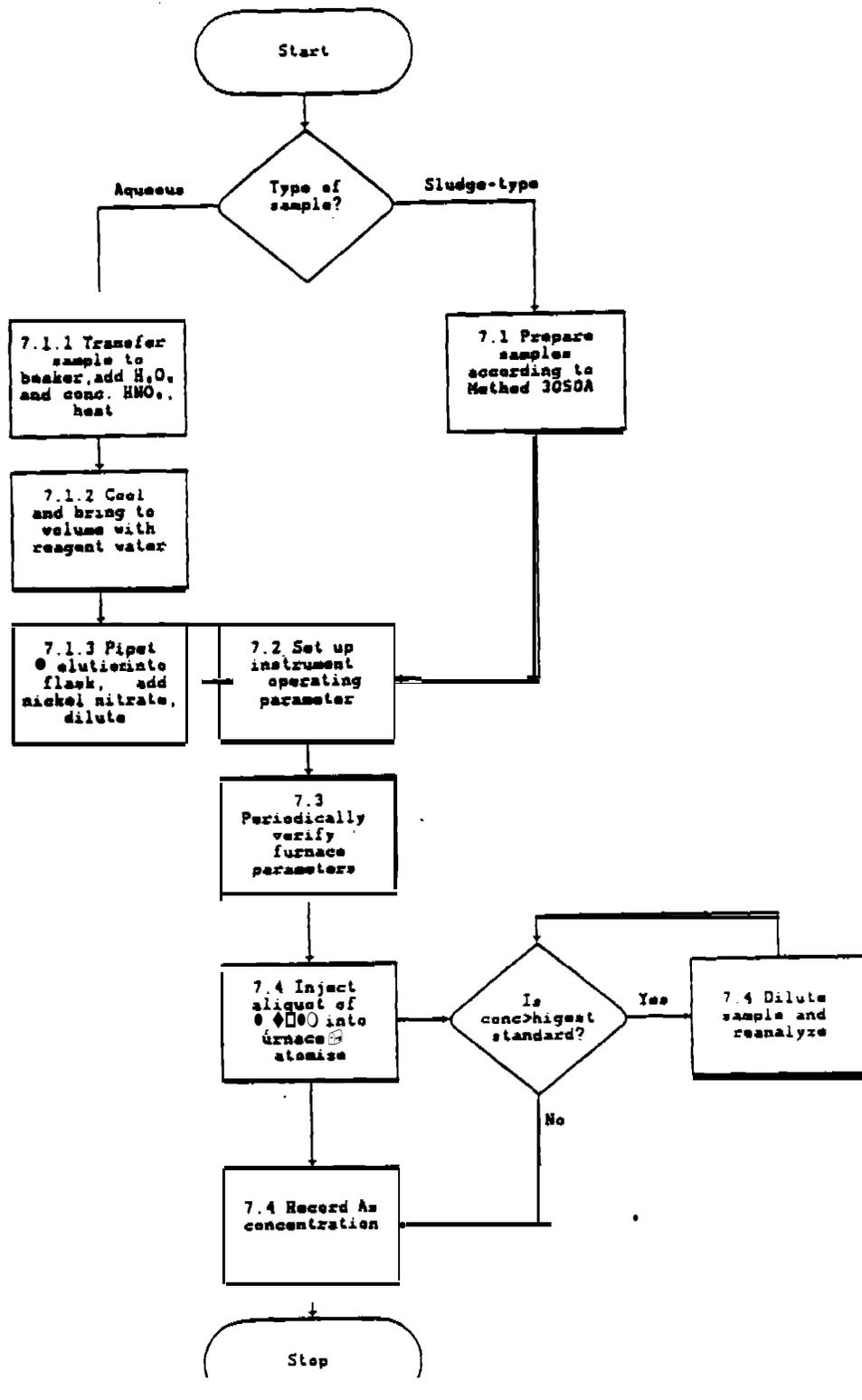
1. Methods for **Chemical Analysis** of Water and Wastes, **EPA-600/4-82-055**, December 1982, Method 206.2.
2. **Gaskill, A.**, Compilation and Evaluation of RCRA Method performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.

TABLE 1. METHOD PERFORMANCE DATA

Sample Matrix	Preparation Method	Laboratory Replicates
Contaminated soil	3050	2.0, 1.8 ug/g
Oily soil	3050	3.3, 3.8 ug/g
NBS SRM 1646 Estuarine sediment	3050	8.1, a.33 ug/g ^a
Emission control dust	3050	430, 350 ug/g

^aBias of -30 and -28% from expected, respectively.

METHOD 7060A
 ARSENIC (ATOMIC ABSORPTION, FURNACE TECHNIQUE)



Final Rpt, Kuwait Oil Fire HRA No. 39-26-L192-91, 5 May - 3 Dec 91

ANNEX E-9

E-9- 1

METHOD 7131A

CADMIUM (ATOMIC ABSORPTION, FURNACE TECHNIQUE)

1.0 SCOPE **AND** APPLICATION

1.1 See Section 1.0 of Method **7000A**.

2.0 SUMMARY OF **METHOD**

2.1 See Section 2.0 of Method 7000A.

3.0 **INTERFERENCES**

3.1 See Section 3.0 of Method 7000A if interferences are suspected.

3.2 In addition to the **normal** interferences experienced during graphite furnace analysis- cadmium analysis **can** suffer from severe **nonspecific** absorption and light **scattering caused** by matrix components during atomization. Simultaneous background **correction** is required to avoid erroneously high results.

3.3 **Excess** chloride may cause premature **volatilization** of **cadmium**. Ammonium phosphate used as a **matrix modifier** minimizes this loss. Other **modifiers** may be used as long as it **is** documented with the **type** of suppressant and concentration.

3.4 Many plastic **pipet** tips (yellow) contain cadmium. Use "**cadmiumfree**" tips.

4.0 APPARATUS AND **MATERIALS**

4.1 For basic apparatus, see Section 4.0 of Method 7000A.

4.2 Instrument parameters (general):

4.2.1 Drying time and temp: 30 **sec** at 125°C.

4.2.2 Ashing time and temp: 30 **sec** at 500°C.

4.2.3 Atomizing time and temp: 10 **sec** at 1900°C.

4.2.4 Purge gas: Argon.

4.2.5 Wavelength: 228.8 **nm**.

4.2.6 Background correction: Required.

4.2.7 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE: The above concentration values and instrument conditions are for a **Perkin-Elmer HGA-2100**, based on the use of a **20- μ L** injection, continuous-flow purge gas, and nonpyrolytic graphite. Smaller sizes of furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above-recommended settings.

5.0 REAGENTS

5.1 See Section 5.0 of Method 7000A.

5.2 **Preparation** of standards:

5.2.1 Stock solution: Dissolve 1.000 g of cadmium metal (analytical reagent grade) in 20 mL of **1:1 HNO₃** and dilute to 1 liter with reagent water. Alternatively, procure a **certified** standard from a supplier and verify by comparison with a second **standard**.

5.2.2 Prepare dilutions of the stock cadmium solution to be used as calibration standards at the time of analysis. To each 100 mL of standard and sample **alike** add 2.0 mL of the ammonium phosphate solution. The calibration standards should be prepared to contain 0.5% (v/v) HNO₃.

5.2.3 Ammonium phosphate solution (40%): Dissolve 40 g of ammonium phosphate, **(NH₄)₂HPO₄** (analytical reagent grade), in reagent water and dilute to 100 mL.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 **See** Chapter Three; Section 3.1.3, Sample Handling and Preservation.

7.0 PROCEDURE

7.1 Sample preparation: The procedures for preparation of the sample are given in Chapter **Three**, Section 3.2.

7.2 See Method **7000A**, Paragraph 7.3, **Furnace** Procedure. The calculation is given in Method **7000A**, **Paragraph** 7.4.

8.0 QUALITY CONTROL

8.1 See Section 8.0 of Method 7000A.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 213.2 of Methods for Chemical Analysis of Water and Wastes.

9.2 The performance characteristics for an aqueous sample free of interferences are:

Optimum **concentration range**: OS-10 **ug/L**.

Detection limit: 0.1 **ug/L**.

9.3 The data shown in Table 1 were obtained from records of state and contractor laboratories. The data are intended to show the precision of the combined sample **preparation** and analysis method.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, **EPA-600/4-82-055**, December 1982, Method 213.2.
2. **Gaskill, A.**, Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. **68-01-7075**, September 1986.

TABLE 1. METHOD PERFORMANCE DATA

Sample Matrix	Preparation Method	Laboratory Replicates
Lagoon soil	3050	0.10, 0.095 ug/g
NBS SRM 1646 Estuarine sediment	3050	0.35 ug/g*
Solvent extract of oily waste	3030	1.39, 1.09 ug/L

*Bias of -3% from expected value.



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ANNEX E-10

E-10-1

MERCURY IN SEDIMENT

Method 245.5 (Manual Cold Vapor Technique)

1. Scope and **Application**

- 1.1 This **procedure**⁽¹⁾ measures total **mercury** (organic + inorganic) in soils, sediments, bottom deposits and sludge type **materials**.
- 1.2 The range of the method is 0.2 to 5 **µg/g**. The **range** may be extended above or below the normal range by **increasing** or decreasing sample size or **through** instrument and recorder control.

2. Summary of **Method**

2.1 A weighed portion of the sample is digested in aqua **regia** for 2 minutes at 95 °C, followed by oxidation with potassium **permanganate**. Mercury in the digested sample is then measured by the conventional cold vapor technique.

2.2 An alternate **digestion**⁽²⁾ involving the use of an autoclave is described in (8.2).

3. Sample Handling and preservation

3.1 Because of the extreme **sensitivity** of the **analytical** procedure and the omnipresence of mercury, care must be taken to avoid **extraneous** contamination. Sampling devices and sample containers should be ascertained to **be** free of mercury; the sample should not **be** exposed to any condition in the laboratory that may result in contact or air-borne mercury **contamination**.

3.2 While the sample may be analyzed without drying, it **has** been found to be more convenient to **analyze a dry sample**. Moisture may be driven off **in** a drying oven at a **temperature** of 60 °C. No mercury losses have been **observed** by using this drying step. The dry sample should **be** pulverized and thoroughly mixed before the **aliquot** is weighed.

4. Interferences

4.1 The same types of interferences that may occur in water samples are also possible with sediments, i.e., sulfides, high copper, high chlorides, etc.

4.2 Volatile materials which absorb at 253.7 **nm** will cause a positive interference. In order to remove any interfering volatile materials, the head space in the BOD bottle should be purged before the addition of **stannous** sulfate.

5. Apparatus

5.1 **Atomic Absorption Spectrophotometer** (See Note 1): Any atomic absorption unit having an open sample **presentation area** in which to mount the absorption **cell** is suitable. Instrument settings **recommended** by the particular manufacturer should **be** followed.

NOTE 1: Instruments designed **specifically** for the measurement of mercury using the cold vapor technique are commercially available and may be substituted for the atomic absorption **spectrophotometer**.

5.2 Mercury Hollow Cathode Lamp: Westinghouse WL-22847, argon **filled**, or equivalent.

5.3 Recorder: Any multi-range variable speed recorder that is compatible with the **W** detection system is suitable.

5.4 Absorption Cell: Standard spectrophotometer cells 10 cm long, having quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 1" O.D. X 4-1/2". The ends are ground perpendicular to the longitudinal **axis** and quartz windows (1" diameter X 1/16" thickness) are cemented **in** place. Gas inlet and **outlet** ports (also of plexiglass but 1/4" O.D.) are attached approximately 1/2" **from** each end. **The** cell is **strapped** to a **burner** for support and aligned in the light **beam** to give the **maximum transmittance**.

NOTE 2: Two 2" X 2" **cards** with one inch diameter holes may be placed over each end of the cell to assist **in** positioning the cell for maximum transmittance.

5.5 Air Pump: Any peristaltic pump capable of delivering 1 liter of **air** per minute may be used. A **Masterflex** pump with electronic speed control has been found to be satisfactory. (Regulated compressed air can be used in an open one-pass system.)

5.6 **Flowmeter**: Capable of measuring an air flow of 1 liter per minute.

5.7 Aeration Tubing: Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return. Straight **glass** tubing terminating in a coarse porous **frit** is used for **sparging** air into the sample.

5.8 Drying Tube: 6" X 3/4" diameter tube **containing** 20 **g** of magnesium perchlorate (**See Note 3**). **The apparatus** is assembled as shown in the accompanying **diagram**.

NOTE 3: **In** place of the magnesium **perchlorate** drying tube, a small reading lamp with **60W** bulb may **be** used to prevent condensation of moisture inside **the** cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature **in** the cell about 10°C above ambient.

6. Reagents

6.1 If Aqua **Regia**: **Prepare** immediately before use by carefully adding three volumes of **conc. HCl** to one volume of **conc. HNO₃**.

6.2 Sulfuric Acid, 0.5 N: Dilute 14.0 ml of **conc.** sulfuric acid to 1 liter.

6.3 **Stannous** Sulfate: Add 25 **g** **stannous** sulfate to 250 ml of 0.5 N sulfuric acid (6.2). This mixture is a suspension and should be **stirred** continuously during use.

6.4 Sodium Chloride-Hydroxylami Sulfate Solution: Dissolve 12 **g** of sodium chloride and 12 **g** of hydroxylamine sulfate **in** distilled water and dilute to 100 ml. NOTE 4: A 10% solution of **stannous** chloride may be substituted for (6.3) and hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate in **(6.4)**.

6.5 Potassium **Permanganate**: 5% solution, **w/v**. Dissolve 5 **g** of potassium **permanganate** in 100 ml of **distilled** water.

Stock Mercury Solution: Dissolve 0.1354 **g** of mercuric chloride in 75 ml of distilled **water**. Add 10 ml of **conc. nitric** acid and adjust the volume to 100.0 ml. 1.0 **ml** = 1.0 mg **Hg**.

6.7 Working Mercury Solution: Make successive dilutions of the stock mercury solution (6.6) to obtain a working standard containing 0.1 $\mu\text{g/ml}$. This working standard and the dilution of the stock mercury solutions should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15 % nitric acid. This acid should be added to the flask as needed before the addition of the **aliquot**.

7. Calibration

7.1 Transfer 0, 0.5, 1.0, 2.0, 5.0 and 10 ml **aliquots** of the working mercury solution (6.7) containing 0 to 1.0 μg of mercury to a series of 300 ml BOD bottles. Add enough **distilled** water to each bottle to make a total volume of 10 ml. Add 5 ml of aqua **regia** (6.1) and heat 2 minutes **in** a water bath at 95 °C. Allow the **sample** to cool and add 50 ml distilled water and 15 ml of **KMnO₄** solution (6.5) to each bottle and return to the water bath for 30 minutes. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate solution (6.4) to reduce the excess **permanganate**. Add 50 ml of distilled water. Treating each bottle individually, add 5 ml of stannous **sulfate** solution (6.3) and immediately attach the bottle to the aeration **apparatus**. At this point, the sample is allowed to **stand** quietly without manual agitation. The circulating pump, which has **previously** been adjusted to rate of 1 liter per minute, is allowed to run continuously. The absorbance, as exhibited either on the **spectrophotometer** or the recorder, will increase and **reach** maximum within 30 seconds, **As soon as** the recorder **pen** levels off, approximately 1 minute, open the bypass valve and continue the aeration until the absorbance **returns** to its minimum value (See Note 5). Close the bypass valve, remove the **fritted** tubing from the BOD bottle and continue the aeration. **Proceed** with the **standards** and construct a **standard curve** by plotting **peak** height versus micrograms of mercury.

NOTE 5: Because of the toxic nature of mercury vapor precaution must be taken to avoid its inhalation. Therefore, a bypass has been included **in** the system to either vent the mercury vapor into an exhaust hood or pass the vapor through some absorbing media, such as:

- a) equal volumes of 0.1 N **KMnO₄** and 10% **H₂SO₄**
- b) 0.25% iodine in a 3% **KI** solution.

A specially treated charcoal that will absorb mercury vapor is also available from **Barnebey** and Cheney, E. 8th Ave., and North Cassidy St., Columbus, Ohio 43219, Cat. #580-13 or #580-22.

8. Procedure

8.1 Weigh **triplicate** 0.2 g portions of dry sample and place in bottom of a BOD bottle. **Add 5 ml of distilled water and 5 ml of aqua regia (6.1). Heat 2 minutes in a** water bath at 95 °C. Cool, add 50 ml distilled water and 15 ml potassium **permanganate** solution (6.5) to each sample bottle. Mix thoroughly and place in the water bath for 30 minutes at 95 °C. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate (6.4) to **reduce** the excess **permanganate**. Add 55 ml of distilled water. **Treating** each bottle individually, add 5 ml of stannous **sulfate** (6.3) and immediately **attach** the bottle to the aeration **apparatus**. Continue as described under (7.1).

8.2 An alternate digestion procedure employing an autoclave may also be used. In this method 5 ml of **conc. H₂SO₄** and 2 ml of **conc. HNO₃** are added to the 0.2 g of sample. 5 ml of saturated **KMnO₄** solution is added and the bottle covered with a piece of aluminum foil, The samples are **autoclaved** at 121 °C and 15 lbs. for 15 minutes. Cool, make up to a volume of 100 ml with distilled water and add 6 ml of sodium **chloride-hydroxylamine** sulfate solution (6.4) to reduce the excess **permanganate**. Purge the **dead air** space and continue as **described** under (7.1).

9. Calculation

9.1 **Measure** the peak height of the **unknown** from the chart and read the mercury value **from** the standard curve.

9.2 Calculate the mercury concentration in the sample by the formula:

$$\mu\text{g Hg/g} = \frac{\text{UP Hg in the aliquot}}{\text{wt of the aliquot in gms}}$$

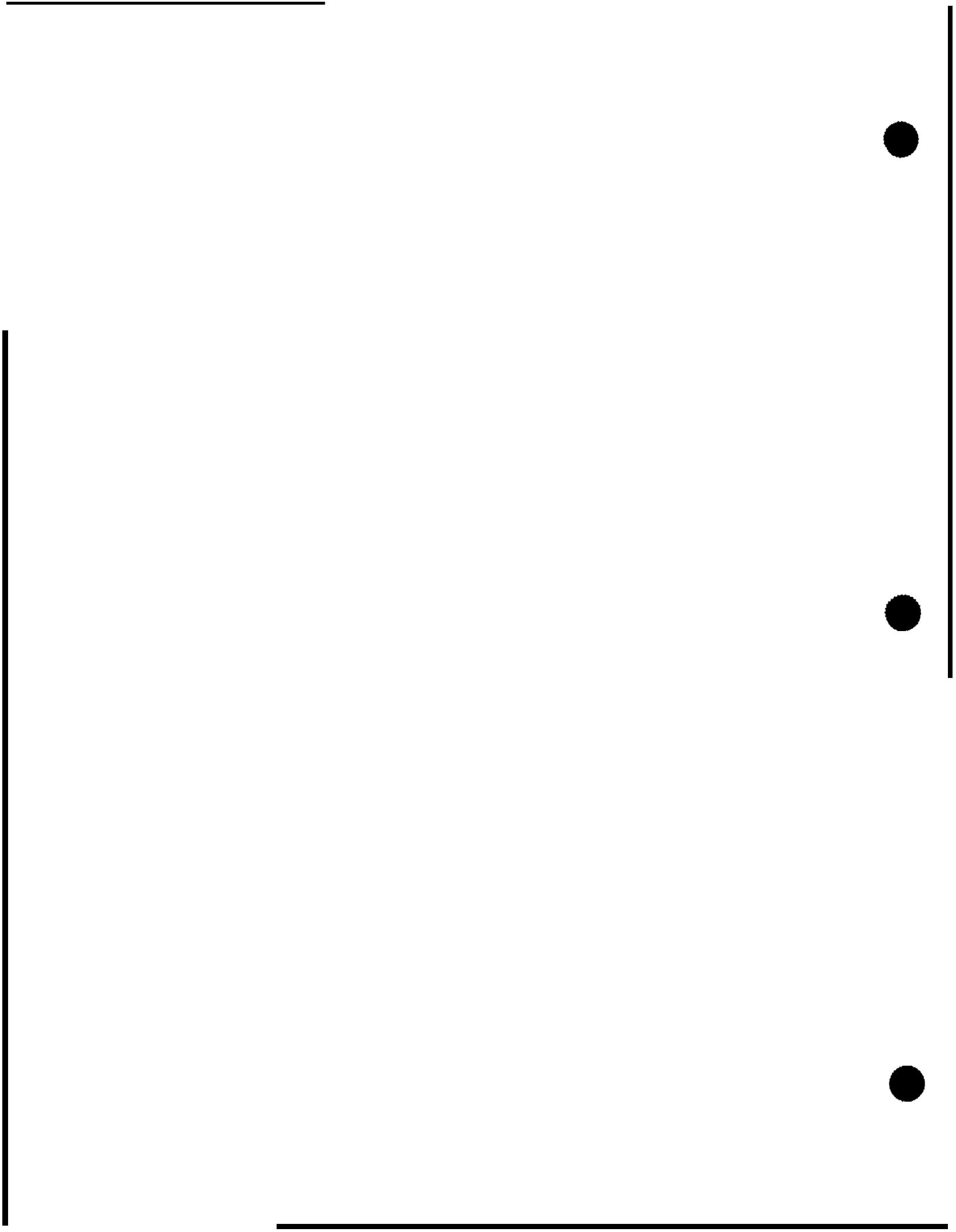
9.3 **Report** mercury concentrations as follows: Below 0.1 **ug/gm**, < 0.1; between 0.1 and 1 **ug/gm**, to the nearest 0.01 ug; between 1 and 10 **ug/gm**, to nearest 0.1 ug; **above 10 ug/gm**, to nearest ug.

10. **Precision** and Accuracy

10.1 The following standard deviations on replicate sediment samples were recorded at the **indicated** levels; 0.29 **ug/g ± 0.02** and 0.82 **ug/g** f0.03; **Recovery** of mercury at these levels, added as methyl mercuric chloride, was 97% and **94%**, respectively.

Bibliography

- 1 Bishop, J.N., "Mercury in Sediments", Ontario Water Resources Comm., Toronto, Ontario, Canada, 19711
2. Salma, M., private communication, **EPA Cal/Nev** Basin Office, Almeda, California.



Final Rpt, Kuwait Oil Fire HRA No. 39-26-L192-91, 5 May - 3 Dec 91

ANNEX E-11

E-11-1

HSHB-LR Radiological and Inorganic Chemistry Division
Metals Analysis Branch
SOP No. MDF 14.1
Disk Name /TAB/HIVOL.MDP
Effective Date rev 1 Oct91

METALS ON HIGH VOLUME AIR FILTERS

Ambient air-suspended particulate matter is collected on a glass fiber filter for twenty four hours using a high volume **airsampler**. **Metals** in the particulate matter is **solubilized** by extraction with hot nitric acid and **analyzed** using **AA** or ICP. An example, the range is from 0.0.3 to 7.5 **ug Pb/cubic** meter assuming an upper **linear** range of analysis of 15 ppm and an air volume of 2400 cubic meters.

PROCEDURE:

Cut a 1" x 10 strip from the **folded edge** of the **filter** using a **straight** edge and a stainless steel **surgical** blade. (Other non-contaminating cutting procedures may be used.) Metals in ambient particulate matter collected on glass fiber filters has been shown to be uniformly distributed across the filter, suggesting that the position of the strip is unimportant. Other 'studies, however, have shown that if the sampling site is **near** a roadway, the lead is not uniformly distributed across the **filter** and therefore, in this case, additional **strips** at different positions within the **filter** should be **analyzed**.

Fold the selected 2" strip **in** half twice and place it **in** a 60 ml teflon container with a screw lid. Add 30 ml of 3 **M³ HNO₃**; cover the sample. (The acid should **COMPLETELY** cover the sample. Screw the lid on the container and place it on a hot plate with the temperature set low enough to heat the sample for 45 minutes **WITHOUT BOILING** OF evaporating the sample to dryness.

CAUTION: Nitric acid fumes are toxic.

Remove the teflon container from the hot plate and cool to room temperature.

Quantitatively transfer the sample in the following **manner**: Decant as much liquid as possible from the container into a 125 ml erlenmeyer flask, leaving the filter in the teflon container. Rinse the teflon lid into the **container** using 3 **small** (2-3 ml) portions of deionized water. Add enough deionized water to cover the filter in the teflon container.

³ 3 M **HNO₃**: Add 192 ml of concentrated nitric acid to about 600 ml of deionized water in a liter volumetric flask. Shake **well**, cool and dilute to volume.

SOP No. MDF 14.1
Disk Name /TAB/HIVOL.MDP
Effective Date rev 1 Oct 91

Cover the container tightly, **shake** it and allow to stand for a minimum of 15 minutes. THIS IS A CRITICAL STEP which cannot **be** omitted since **it** ensures maximum diffusion of the **HNO₃** digestate into the rinse water. At the end of this time, decant as above and **repeat** the process. Filter the combined rinsing through a **0.45u** membrane into a filtering flask. Rinse the erlenmeyer with three small portions of deionized water onto the membrane **filter**, transfer the fiber glass (Hi-Vol) filter and vacuum to dryness, and rinse the teflon lid, container and, **finally**, the filter support and funnel into the **filter** flask:

KEEP THE VOLUME OF **FILTRATE** BELOW 100 ML (ABOUT 80-85 ML)! Transfer the **filtrate** quantitatively to a **100** ml volumetric flask, rinsing filtering flask, and bring to volume. Shake well and pour into a clean polyethylene bottle. The sample is ready for analysis.

CALCULATIONS:

Take the concentration of the solution and multiply by **.4** will give the amount in **mg/filter**. (One quarter of the filter was brought up to 100 **mL** vol).

REFERENCE:

40 CFR Part **50**, **Appendix G**, as reported in **ENVIRONMENT REPORTER**, Bureau of National **Affairs**, **1977**, pages 1278 through 1281.

PREPARATION: This SOP was prepared by Toni Bishop.

DISTRIBUTION: This SOP will be filed with **MAB's** branch chief and distributed to all trained personnel for this operation.

REVIEW and APPROVAL: This SOP will be reviewed yearly for modifications and any changes will be approved by **MAB s** branch chief.

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Chief, Metals Analysis Branch



Final Rpt, Kuwait Oil Fire, HRA No. 39-26-L192-91, 5 May - 3 Dec 91

ANNEX E-12

E-12-1

METHOD 200.7

DETERMINATION OF **METALS** AND TRACE **ELEMENTS IN WATER AND WASTES** BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION **SPECTROMETRY**

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Technology **Applications**, Inc.

Revision 3.3
April 1991

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
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METHOD 200.7

DETERMINATION OF METALS AND **TRACE** ELEMENTS BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION **SPECTROMETRY**

1. SCOPE AND APPLICATION

- 1.1 This **method** provides **procedures** for determination of dissolved elements in ground waters, surface waters, and drinking water supplies. It may also be used for determination of total recoverable element concentrations in these waters and **wastewaters** and, with the exception of **silica**, in sediments, sludges and solid waste samples.
- 1.2 Dissolved elements are determined after suitable **filtration** and acid preservation. Acid digestion procedures are required prior to the determination of total recoverable elements. To reduce potential interferences, dissolved solids should be < 0.2% (w/v), (Sect. 4.1.2).
- 1.3 **Estuarine** water may be analyzed by this method, however, **matrix** matched **standards** or the method of standard addition (Sect. 9.8) must be used following sample **preparation** (Sect. 11.2.2). **Prepared** samples may require **dilution** prior to **analysis** to avoid physical interferences (Sect. **4.1.2**) and problematic operation of the sample introduction system.
- 1.4 This method is applicable to the following **analytes**:

<u>Analyte</u>		Chemical Abstract Service <u>Registry Numbers (CASRN)</u>
Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Boron	(B)	7440-42-8
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-47-3
cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Lithium	(Li)	7439-93-1
Magnesium	(Mg)	7439-954

Manganese	(Mn)	7439-965
Mercury	(Hg)	7439-97-6
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Phosphorus	(P)	7723-14-0
Potassium	(K)	7440-09-7
Selenium	(Se)	778249-2
Silica	(SiO₂)	7631-86-9
Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Strontium	(Sr)	7440-24-6
Thallium	(Tl)	7440-28-0
Tin	(Sn)	7440-31-5
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

Listed in Table 1 are the recommended **wavelengths** for these **analytes** along with adjacent locations for background correction. Also listed in Table 1 **are** typical instrument detection limits (**IDLs** Sect. 3.3) determined using reagent acid ASTM type I water and conventional pneumatic **nebulization** sample introduction into the **plasma**. **These IDLs** are intended as a guide and may vary for each laboratory depending on instrumentation and selected **operating** conditions. Wavelengths and background **corrector**, locations other **than** those recommended may be substituted if they **provide** the needed sensitivity, and **are** properly corrected for inter-element **spectral** interferences.

- 1.5 Specific instrumental **operating** conditions are given in Table 4. However, because of the differences between various makes and models of spectrometers, the analyst should follow the instrument manufacturer's instructions and if possible, approximate the recommended conditions given (Table 4).
- 1.6 When using this method for determination of boron and **silica** in aqueous samples, only plastic, Teflon or quark **labware** should be used from time of sample collection to completion of analysis. For accurate determinations of boron **in** solid sample extracts at **concentrations** below 100 **mg/kg**, only quartz **beakers** should be used in the digestion with immediate transfer of au extract **aliquot** to a plastic **centrifuge tube** following dilution of the digestate to volume. For these determinations, **borosilicate** glass must not be used in order to avoid sample contamination of these analytes from the glass.
- 1.7 This method is **applicable** to analysis of drinking water for the determination of primary and secondary **contaminant** metals. However, it can only be used for compliance monitoring of a drinking water **contaminant** when listed in the Federal Register as an approved method **and laboratory performance data** meet the required

method detection limit (**MDL**) or practical quantification limit (**PQL**) established by the Office of Ground Water and Drinking Water. All drinking water samples must be **pretreated** with acid prior to analysis. When pneumatic **nebulization** is used for these determinations, certain **analytes** require 4X **preconcentration** prior to analysis instead of the 2X **preconcentration** procedure given in Sect. 11.2.1 of this method. Analytes requiring 4X **preconcentration** are noted in the Federal Register at the time the **method** is promulgated.

- 1.8 **This** method is suitable for determination of silver in aqueous samples containing concentrations up to 0.1 **mg/L**. For the analysis of wastewater samples containing higher concentrations of silver, **succeeding** smaller volume, well mixed **aliquots** should be prepared until the analysis solution contains < 0.1 **mg/L** silver.
- 1.9 The sample **preparation** procedures given in Sects. 11.2 and 11.3 will **solubilize** and hold in solution only minimal concentrations of barium, as barium sulfate. In addition, the stability of **solubilized** barium is **greatly affected** when free sulfate is available in solution. The concentration of barium that will remain in solution decreases as the free sulfate concentration **increases**. [For example, when a 100 **mL aliquot** of drinking water containing 60 **mg/L** sulfate was **fortified** with 5 mg of **BaSO₄** salt (equivalent to 59 **mg/L** Ba in the 2X analysis solution) only 33 **mg/L** Ba was **initially** solubilized using the procedure given in Sect. 11.2.1. Upon standing one week, the barium concentration **decreased** to 12 **mg/L**. When 100 **mL** of deionized distilled water was **fortified**, the entire 5 mg of **BaSO** was solubilized and remained in solution over the same time period.] For more accurate **determinations** of barium in samples having **varying** and unknown **concentrations** of sulfate, samples should be analyzed as soon as possible after sample preparation is completed.
- 1.10 With the exception of **estuarine** waters, once the samples have been collected, approximately 20 samples including the mandatory quality control samples can be analyzed using this **method** during a 1.5 work day period.

2. SUMMARY OF METHOD

- 2.1 This method describes a technique for **simultaneous** or sequential multielement determination of **metals** and trace elements in solution. The basis of the method is the measurement of atomic emission by an optical **spectrometric** technique. Samples are **nebulized** and the aerosol that is produced is **transported** to the plasma torch where desolvation and excitation **occur**. Characteristic atomic-line emission spectra **are produced** by a radio-frequency inductively coupled plasma (**ICP**). The spectra are dispersed by a grating spectrometer, and **line** intensities are monitored by a photosensitive device (e.g., **photomultiplier** tube or diode array). **Photocurrents** from the photosensitive device are **processed** and controlled by a computer system. A background correction technique is required to compensate

for variable background contribution to the determination of the **analytes**. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must either be free of **spectral interference** or adequately corrected to reflect the same change in background **intensity** as occurs at the analyte wavelength measured. Background correction is not **required in** cases of line **broadening** where a background correction measurement would **actually** degrade the **analytical** result. The possibility of additional interferences named **in** Sect. 4.1 (and tests for their presence as described in Sect. 4.2) should also be recognized and appropriate corrections made.

3. DEFINITIONS

- 3.1 **DISSOLVED** - The **concentration** of analyte that will pass through a **0.45- μ m** membrane filter assembly, prior to sample **acidification**.
- 3.2 **TOTAL RECOVERABLE** - The concentration of **an** analyte determined in an **unfiltered** sample following **treatment** by refluxing with hot, dilute mineral acid.
- 3.3 **INSTRUMENTAL DETECTION LIMIT (IDL)** - The concentration equivalent to the **analyte** signal which is equal to three times the standard deviation of a series of 10 **replicate measurements** of a **reagent** blank signal at the same wavelength.
- 3.4 **METHOD DETECTION LIMIT (MDL)** - The minimum concentration of an **analyte** that **can be** identified, measured and **reported** with 99% **confidence** that the **analyte concentration** is **greater** than zero (Sect. 10.2.2).
- 3.5 **LINEAR DYNAMIC RANGE (LDR)** - The concentration range over which the analytical curve remains linear (Sect. 10.2.3).
- 3.6 **METHOD OF STANDARD ADDITION** - **The** standard addition technique involves the use of the unknown and the unknown plus a known amount of standard (Sect. 9.8.1).
- 3.7 **LABORATORY REAGENT BUNK (LRB) (preparation blank)** - **An aliquot** of reagent water that is **treated** exactly as a sample including **exposure to all** glassware, equipment, **reagents**, and acids **that** are used with other samples. The **LRB** is used to determine if **method analytes or** other interferences are present in the laboratory environment, the reagents or **apparatus** (Sects. 7.5.2 and 10.3.1).
- 3.8 **CALIBRATION BLANK** - A volume of ASTM type I water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to calibrate the ICP instrument (Sect. 7.5.1).

- 3.9 **STOCK STANDARD SOLUTION** - A concentrated solution containing one analyte prepared in the **laboratory** using assayed reference materials or purchased from a reputable commercial source (Sect. 7.3). Stock standard solutions are used to prepare calibration solutions and other **needed** analyte solutions.
- 3.10 **CALIBRATION STANDARD (CAL)** - A solution prepared from the dilution of stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte **concentration** (Sect. 7.4).
- 3.11 **LABORATORY PERFORMANCE CHECK SOLUTION (LPC)** - A solution of method analytes, used to evaluate the performance of the instrument system with respect to a defined set of method criteria (Sects. 7.8 and 9.6).
- 3.12 **PLASMA SOLUTION** - A solution that is used to determine the optimum height above the work wil for viewing the plasma (Sects. 7.6 and 9.3 .3).
- 3.13 **TUNING SOLUTION** - A solution which is used to determine acceptable instrument performance prior to calibration and sample analyses (Sects. 7.7 and 9.4).
- 3.14 **SPECTRAL INTERFERENCE CHECK SOLUTION (SIC)** - A solution of selected method analytes of higher level **concentrations** which is used to evaluate the procedural routine for correcting known interelement **spectral interferences** with respect to a defined set of method criteria (Sects. 7.9 and 9.7).
- 3.15 **LABORATORY FORTIFIED BUNK (LFB)** - An aliquot of reagent water to which known quantities of the method analytes **are** added in the laboratory. The **LFB** is analyzed exactly like a sample, and its purpose **is** to determine whether method performance is within acceptable control limits (Sects. 7.11 and 10.3.2).
- 3.16 **LABORATORY FORTIFIED SAMPLE MATRIX (LFM)** - An **aliquot of an** environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix **contributes** bias to the **analytical** results. The background **concentrations** of the **analytes** in the sample matrix must be determined in a **separate** aliquot and the measured values in the LFM corrected for the **concentrations** found (Sect. 10.4).
- 3.17 **FIELD DUPLICATES (FD1 AND FD2)** - Two **separate** samples collected at the same time and place under identical circumstances and **treated** exactly the same throughout field and laboratory procedures. Analyses of **FD1** and FD2 give a **measure** of the precision associated with sample collection, preservation, and storage, **as** well as with laboratory procedure.

- 3.18 **QUALITY CONTROL SAMPLE (QCS)** - A solution of method **analytes** of known concentrations which is used to fortify an aliquot of **LRB** matrix. The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance (Sects. 7.12 and 10.2.4).

4. INTERFERENCES

- 4.1 Several **types** of interference effects may **contribute** to inaccuracies in the determination of an **analyte** by ICP-AES. They **can be** summarized as follows:
- 4.1.1 Spectral interferences - **Can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) background contribution from stray light from the line emission of high concentration elements.**1 The **first** of these **effects** can **be compensated** by utilizing a computer correction of raw data, requiring monitoring and measurement of the interfering element. 3 The second effect may require selection of **an** alternative wavelength. The third and fourth effects can usually be **compensated** by a background **correction** adjacent to the **analyte line**.

Given in Table 3 **is a** listing of the interelement **spectral** interferences that can occur between **method analytes** when using the recommended wavelengths and locations for background **corrections** listed in Table 1. Table 3 is not a complete listing of all possible **interelement** interferences; however, those not included are **interferences** from elements either not readily **solubilized** by the sample preparation **procedures described in this method or from elements rare in nature**. The **correction** factors listed in Table 3 indicate the magnitude of the interference. The factors were experimentally determined at **EMSL-Cincinnati** using an instrument with a **specified** wavelength **dispersion** of 0.53 nm/mm and a **spectral bandpass** resolution of 0.036 nm in the **first** order. The factors have been rounded to the **tenth-thousand** place or reported to one **significant** number. The listing is presented as a guide for users of this method for determining interelement interference effects. The reader is cautioned that **other** analytical systems may exhibit somewhat different levels of interference than those shown in Table 3 and that the **interference** effects must be evaluated for each individual instrumental system.

The **correction** factors given in Table 3 were **determined** by analyzing single element solutions of each interfering element. The **concentration** of each single element solution was within the **LDR** of that element. For most elements a 100 mg/L solution was used with the **numerical** value of most **correction** factors being **confirmed** by analyzing lesser dilutions of the single element solution. **Because** Ca, Fe, **Mg** and Na **can** normally **be** present at concentrations in excess of 100 mg/L, the interferences attributed to these elements were determined at

concentrations **near** their **linear** limits. The criteria for listing a spectral interference was an

apparent analyte concentration from the interfering single element solution that was outside the 95 % confidence interval estimates for the determined MDL **limits**⁴ of the analyte using the 2x **preconcentration** procedure described in Sect. 11.2.1 (See Table 2). The **correction** factor was calculated by dividing the blank subtracted apparent analyte concentration by the determined concentration of the interfering element.

Positive values in Table 3 are interferences that occur on the wavelength **peaks**, while negative values indicate **an** interference at the location used for background **correction**. In practice, during analysis, the **correction** factor is used to calculate the apparent concentration from interfering element and is then subtracted from the instrumental analyte concentration to determine the net, or sample analyte concentration (while positive values are subtracted, negative values are actually added). Without these **corrections** when interference effects are present, either false positive or false negative determinations **will** result. Also, the reliability of an applied correction depends on the variance surrounding the measurement of the interfering element. **As** the **concentration** of the **interfering** element **increase**, the variance increases; this is reflected in the calculated apparent analyte concentration. **Extreme** caution should be exercised when reporting analyte concentrations where the **apparent** analyte concentration from an interfering element accounts for 90% of the measured **analyte** concentration. Once a routine procedure for correcting interelement **spectral** interferences has been established, it should be periodically tested to evaluate its **operational** effectiveness and **continued** reliability (Sect. 7.9).

4.1.2 Physical **interferences** - Are generally considered to be effects associated with the sample **nebulization** and transport **processes**. Such **properties as** change in viscosity and **surface** tension **can** cause **significant** inaccuracies especially in samples which may **contain** high dissolved solids and/or high acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by sample **dilution and/or** utilization of standard addition techniques (Sect. 9.8). Another problem which can occur from high dissolved solids is salt buildup at the tip of the **nebulizer**. This affects aerosol flow rate causing instrumental drift. Wetting the argon prior to **nebulization**, use of a tip washer, or sample dilution have been used to control this problem. **Also**, it has been reported that better control of the argon flow rate improves **instrument** performance. This is accomplished with the use of **mass** flow controllers.

4.1.3 Chemical Interferences - Are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if **observed** they **can** be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by buffering the sample, matrix matching, or standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

- 4.1.4 Memory interferences - Result when **analytes** in a previous sample contribute to the signals measured in a current sample. Memory effects can result from sample deposition on the uptake tubing to the **nebulizer** or from build-up of sample material in the plasma torch and spray chamber. The site where these effects **occur** is dependent on the element and can be **minimized** by flushing the system with a rinse blank between samples (Sect. 7.5.3). The possibility of memory interferences should be recognized within an **analytical** run and suitable rinse times should be used to **reduce** them. The rinse times necessary for a particular element should be **estimated** prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to either their **LDRs** or concentrations ten times those **usually** encountered. The aspiration time should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the **required** rinse time is established, this method recommends a **rinse** period of 60 sec between samples and standards. If a memory interference is suspected, the sample should be reanalyzed after a long rinse period.
- 4.2 **The** occurrence of interferences described in Sects. 4.1.1, 4.1.2 and 4.1.3 are primarily attributed to the sample matrix. If an interference caused by a particular sample matrix is **known**, in many **cases** it **can** be circumvented. However, when the nature of the sample **is unknown**, tests as outlined in **Sects. 4.2.1** through 4.2.4 **can** be used to ensure the analyst that neither positive nor negative interference effects **are** operative on any of the analyte elements thereby distorting the **accuracy** of the reported values.
- 4.2.1 **Serial dilution** - If the analyte concentration is **sufficiently** high (minimally a factor of 10 the MDL after dilution), an analysis of a dilution should agree within 10% of the original determination or within an established acceptable control limit. If not, a chemical or physical interference effect should be suspected.
- 4.2.2 **Analyte addition** - A post digestion analyte addition added at a minimum level of **20X** the MDL (maximum **100X**) to the original determination should be recovered to within 90% to 110% or within an established control limit. If not, a matrix effect should be suspected. **The** use of a standard addition analysis procedure can usually compensate for this effect. **CAUTION:** The standard addition technique does not detect coincident **spectral** overlap. If **suspected**, use of computerized compensation, an alternative wavelength, or comparison with an alternative method is recommended (Sect. 4.2.3).
- 4.2.3 **Comparison with alternative method of analysis** - When investigating a sample **matrix**, comparison tests may be performed with other analytical techniques, such as atomic absorption **spectrometry**, ICP-mass **spectrometry**, or other approved methodology.

4.2.4 Wavelength scanning of **analyte** line region - If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

5. SAFETY

- 5.1 The **toxicity** or **carcinogenicity** of each **reagent** used in this method has not been fully established. Each **chemical** should be regarded as a potential health **hazard**, **and** exposure to these compounds should be **as** low as reasonably achievable. Each laboratory is responsible for **maintaining** a **current** file of OSHA regulations regarding the safe **handling** of chemicals specified in this method ^{6,9}. A reference **file** of material data handling sheets should also be made available to all personnel involved in the chemical analysis. **Specifically, concentrated** nitric and hydrochloric acids are moderately toxic and extremely **irritating** to skin and mucus **membranes**. Use these **reagents** in a hood whenever possible and if eye or **skin** contact occurs, flush with large **volumes** of water. Always wear safety glasses or a shield for eye protection when **working** with these **reagents**.
- 5.2 Analytical plasma sources emit **radiofrequency** radiation and intense **W** radiation. Suitable precautions should be taken to protect personnel from such hazards.
- 5.3 All personnel handling **environmental** samples known to contain or to have been in contact with human waste should be **immunized** against known disease causative **agents**.
- 5.4 Precautions should also be taken to minimize potential hazards. Basic good housekeeping and safety practices such as the use of rubber or plastic gloves and safety glasses during cleaning of **labware** are highly recommended.

6. APPARATUS AND EQUIPMENT

6.1 ANALYTICAL INSTRUMENTATION

6.1.1 The ICP instrument may be a simultaneous or sequential spectrometer system that uses ionized argon gas as the plasma. However, the system and processing of **background** corrected signals must be computer controlled. The instrument must be **capable** of meeting and complying with the requirements and description of the technique given in Sect. 2.1 of the method. In particular, **it** is the responsibility of the analyst to investigate the **spectral** interference (Sect. 4.1.1) operative about **each** analytical wavelength used and to verify and **periodically confirm** that the instrument configuration and operating conditions used satisfy the analytical **requirements**.

6.1.2 Argon gas supply - Liquid, high purity **grade** (99.99%).

6.1.3 A **variable** speed peristaltic pump is required to deliver both standard and sample solutions to the nebulizer.

6.1.4 Mass flow **controllers** to regulate the argon flow rates, especially the aerosol **transport** gas, are highly recommended. Their use will provide more exacting control of reproducible plasma conditions.

6.1.5 For routine analyses of **solutions** containing dissolved solids > 1 A, a high solids **nebulizer** and a torch injector tube having an **i.d.** > 1.0 mm are recommended. (Consult the instrument manufacturer for guidance.)

6.1.6 For sustained analyses of solutions containing alkali **concentrations** > 0.5 % , an alumina torch injector tube is recommended to prevent **devitrification** of the normally-used **quartz** injector tube.

NOTE: Regular periodic cleaning of the quartz torch assembly and injector tube by soaking in aqua **regia** (Sect. 7.1.9) reduces **background** signal noise, calibration drift and potential memory effects.

6.2 SAMPLE PROCESSING EQUIPMENT

6.2.1 Air Displacement Pipetter: Digital **pipet** capable of delivering volumes ranging from 0.1 to 2500 **uL** with **an** assortment of high quality disposable **pipet** tips.

6.2.2 Hot Plate: Ceramic top, graduated dial **90 °C** to **450 °C** (Coming PC100 or equivalent).

6.2.3 Single pan balance: **Balance** capable of weighing to the nearest 0.01 g.

6.1.4 Mass flow controllers to regulate the argon flow rates, especially the aerosol transport gas, **are** highly recommended. Their use will provide **more** exacting control of reproducible plasma conditions.

6.1.5 For routine analyses of solutions containing dissolved solids $> 1\%$; a high solids nebulizer and a torch injector **tube** having an **i.d.** > 1.0 mm are recommended. (Consult the instrument **manufacturer** for guidance.)

6.1.6 For sustained analyses of solutions containing alkali concentrations > 0.5 % , **an** alumina torch injector tube is recommended to prevent **devitrification** of the normally-used quartz **injector** tube.

NOTE: Regular periodic cleaning of the quartz torch assembly and injector tube by soaking in aqua regia (Sect. 7.1.9) reduces background signal noise, calibration drift and potential memory effects.

6.2 SAMPLE PROCESSING EQUIPMENT

- 6.2.1 **Air Displacement Pipetter:** Digital **pipet capable** of delivering volumes ranging from 0.1 to 2500 **uL** with **an** assortment of high quality **disposable pipet tips**.
- 6.2.2 Hot Plate: Ceramic top, graduated dial 90 °C to 450 °C (Corning PC100 or **equivalent**).
- 6.2.3 Single pan balance: **Balance** capable of weighing **to** the nearest 0.01 **g**.
- 6.2.4 Analytical balance: Balance capable of weighing to the nearest 0.0001 **g**.
- 6.2.5 Centrifuge: Steel **cabinet** with guard bowl, electric timer and brake. (**International** Centrifuge, Universal Model UY or equivalent.)
- 6.2.6 Drying oven: Gravity convection oven, with thermostatic control capable of maintaining 180 °C + 5 °C.
- 6.3 **LABWARE** - For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination **sources** include improperly cleaned laboratory **apparatus** and general contamination **within** the laboratory environment from dust, etc. A clean laboratory work area, designated for trace element sample handling must be used. Sample containers can introduce positive and negative errors **in** the **determination** of trace elements by (1) contributing contaminants through surface **desorption** or leaching, (2) depleting element concentrations through adsorption processes. **All reuseable labware** (glass, quartz, polyethylene, Teflon, etc.), including the sample container, should be cleaned prior to use. **Labware** should be soaked overnight and thoroughly washed with laboratory-grade detergent and water, rinsed with water, and soaked for 4 hours in a mixture of dilute nitric and hydrochloric acid (**1+2+9**), followed by *rinsing with water, ASTM type I water, and oven drying*.

NOTE Chromic acid must not be used for cleaning glassware.

- 6.3.1 Glassware - Volumetric flasks, graduated cylinders, funnels and centrifuge tubes (glass and/or metal-free plastic).
- 6.3.2 Assorted calibrated pipettes.

6.3.3 Conical Phillips beakers, **250-mL** with **50-mm** watch glasses. **Griffin** beakers, **250-mL** with **75-mm** watch glasses. Teflon and/or quartz beakers, **250-mL** with Teflon covers (optional).

6.3.4 Wash bottle - One piece stem, Teflon FEP bottle with Tefzel **ETFE** screw closure, **125-mL** capacity.

7. REAGENTS AND CONSUMABLE MATERIALS

7.1 Reagents may contain elemental **impurities** which might affect analytical **data**. **Only** high-purity reagent should **be** used whenever possible. All acids used for this method must be of ultra **highpurity grade**. Suitable acids are available from a **number** of manufacturers or may be prepared by sub-boiling distillation.

7.1.1 Nitric acid, concentrated (**sp.gr.** 1.41) (**CASRN** 7697-37-2).

7.1.2 Nitric acid (1+1) - Add 500 **mL conc.** nitric acid to 400 **mL** of ASTM type I water and dilute to 1 L.

7.1.3 Nitric acid (1+9) - Add 100 **mL conc.** nitric acid to **400 mL** of ASTM type I water and dilute to 1 L.

7.1.4 Hydrochloric acid, **concentrated** (**sp.gr.** 1.19) (**CASRN** 7647-01-o).

7.1.5 Hydrochloric acid (1 + 1) - Add 500 **mL conc.** hydrochloric acid to 400 **mL** of **ASTM type I water and dilute to 1 L.**

7.1.6 **Hydrochloric** acid (1+4) - Add 200 **mL conc.** hydrochloric acid to **400 mL** ASTM type I water and dilute to 1 L.

7.1.7 Ammonium hydroxide, concentrated (**sp. g-r.** 0.902) (**CASRN** 133621-6).

7.1.8 **Tartaric** acid, ACS reagent grade (**CASRN** 87-69-4).

7.1.9 Aqua **regia** - Add 100 **mL conc.** nitric acid to 300 **mL conc.** hydrochloric acid and **100 mL** ASTM type I water.

7.2 WATER - For all sample preparation and dilutions, **ASTM** type I water (ASTM **D1193**) 1 ° is required. Suitable water maybe prepared by passing **distilled** water through a **mixed** bed of anion and cation exchange rosins.

7.3 STANDARD STOCK SOLUTIONS - May be purchased from a reputable commercial source or prepared from **ultra** high-purity grade **chemicals** or metals (99.99 - 99.999% pure). **All salts** should be dried for one hour at 105 °C, unless

otherwise specified. (CAUTION: Many metal salts **are** extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling). Stock solutions should be stored in Teflon bottles.

The following procedures may be used for preparing standard stock solutions:

NOTE: Some **metals**, particularly those which form surface oxides **require** cleaning prior to being weighed. This may **be** achieved by pickling the surface of the metal in acid. An amount in excess of the **&sired** weight should be pickled repeatedly, rinsed with water, dried and weighed until the **desired** weight is achieved.

- 7.3.1 Aluminum solution, stock 1 **mL** = 1000-**ug** Al: Pickle aluminum metal in warm (1 + 1) hydrochloric acid to an exact weight of 0.100 g. Dissolve in 10 **mL conc.** hydrochloric acid and 2 **mL conc. nitric acid**, **heating** to effect solution. Continue heating until volume is reduced to 4 **mL**. **Cool** and add 4 **mL ASTM type I** water. Heat until volume is reduced to 2 **mL**. **rnol** and dilute to 100 **mL** with ASTM type I water.
- 7.3.2 Antimony solution, stock 1 **mL** = 500 **ug** Sb: Dissolve 0.100 **g** Sb powder in 2 **mL** (1 + 1) nitric acid and 1.0 **mL conc.** hydrochloric acid. Add 10 **mL** ASTM type I water and 0.15 **g tartaric** acid. Warm slightly to effect complete solution. Cool and **dilute** to 200 **mL** with ASTM type I water.
- 7.3.3 Arsenic solution, stock 1 **mL** = 1000 **ug** **As**: Dissolve 0.1320 **g** **As₂O₃** in a mixture of 50 **mL** ASTM type I water and 1 **mL conc.** ammonium hydroxide. **Heat** gently to dissolve. Cool and acidify the solution with 2 **mL conc.** nitric acid. **Dilute** to 100 **mL** with **ASTM** type I water.
- 7.3.4 Barium solution, stock 1 **mL** = 500 **ug** Ba: Dissolve 0.1437 **g** **BaCO₃** in a solution mixture of 10 **mL** ASTM type I water and 5 **mL conc.** nitric acid. Heat and stir to effect solution and **degassing**. Dilute to 200 **mL** with ASTM type I water.
- 7.3.5 Beryllium solution, stock 1 **mL** = 500 **ug** Be: Dissolve 1.965 **g** **BeSO₄·4H₂O** (DO NOT DRY) in 50 **mL** ASTM Type I water. Add 2 **mL conc. nitric acid**. Dilute to 200 **mL** with **ASTM type I water**.
- 7.3.6 Boron solution, stock 1 **mL** = 1000 **ug** B: **DO NOT DRY**. Dissolve 0.5716 **g** anhydrous **H₃BO₃** in 20 **mL** ASTM type I water. Dilute to 100 **mL** with ASTM type I water, mix and **immediately transfer** to a Teflon bottle for storage. Use a reagent meeting ACS **specifications**, keep the bottle tightly stoppered and store in a desiccator to prevent the entrance of atmospheric moisture.
- 7.3.7 Cadmium solution, stock 1 **mL** = 1000 **ug** Cd: Pickle **cadmium** metal in (1+9) nitric acid to an exact weight of 0.100 **g**. Dissolve in 5 **mL** (1 + 1) **nitric acid**, **heating** to effect solution, **Cool and** dilute to 100 **mL** with ASTM type I water.

- 7.3.8 Calcium solution, stock 1 mL = 1000 ug Ca: Suspend 0.2498 g CaCO₃ dried at 180°C for 1 hour before weighing, in 20 mL of ASTM type I water. Dissolve cautiously (reaction is vigorous) by adding-dropwise, 10 mL (1 + 1) hydrochloric acid. Dilute to 100 mL with ASTM type I water.
- 7.3.9 Chromium solution, stock 1 mL = 500 ug Cr: Dissolve 0.1923g CrO₃ in a solution mixture of 10 mL ASTM type I water and 2 mL conc. nitric acid. Dilute to 200 mL with ASTM type I water.
- 7.3.10 Cobalt solution, stock 1 mL = 1000 ug Co: Pickle cobalt metal in (1+9) nitric acid to an exact weight of 0.100 g.
- Dissolve in 5 mL (1 + 1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.
- 7.3.11 Copper solution, stock 1 mL = 1000 ug Cu: Pickle copper metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1 + 1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.
- 7.3.12 Iron solution, stock, 1 mL = 1000 ug Fe: Pickle iron metal in (1 +1) hydrochloric acid to an exact weight of 0.100 g. Dissolve in 10 mL (1 + 1) hydrochloric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.
- 7.3.13 Lead solution, stock 1 mL = 1000 ug Pb: Dissolve 0.1599 g PbNO₃ in 5 mL (1+1) nitric acid. Dilute to 100 mL with ASTM type I water.
- 7.3.14 Lithium solution, stock 1 mL = 500 ug Li: Dissolve 0.5324 g Li₂CO₃ in 20 mL ASTM type I -water. Add 2 mL conc. nitric acid and dilute to 200 mL with ASTM type I water.
- 7.3.15 Magnesium solution, stock 1 mL = 1000 ug Mg: Dissolve 0.100 g cleanly polished magnesium ribbon in 5 mL (1 + 1) hydrochloric acid. (Add acid slowly, reaction is vigorous) Add 2 mL (1 + 1) nitric acid and dilute to 100 mL with ASTM type I water.
- 7.3.16 Manganese solution, stock 1 mL = 1000 ug Mn: Pickle manganese flake in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1 + 1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.
- 7.3.17 Mercury solution, stock 1 mL = 500 ug Hg: DO NOT DRY, highly toxic, poison. Dissolve 0.1354 g HgCl₂ in 20 mL ASTM type I water. Add 10 mL conc. nitric acid and dilute to 200 mL with ASTM type I water.

- 7.3.18 Molybdenum solution, stock 1 mL = 1000 µg Mo: Dissolve 0.1500 g MoO₃ in a solution mixture of 10 mL ASTM type I water and 1 mL conc. ammonium hydroxide, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.
- 7.3.19 Nickel solution, stock 1 mL = 1000 µg Ni: Dissolve 0.100 g nickel powder in 5 mL conc. nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.
- 7.3.20 Phosphorus solution, stock 1 mL = 1000 µg P: Dissolve 0.3745 g NH₄H₂PO₄ in 20 mL ASTM type I water. Dilute to 100 mL with ASTM type I water.
- 7.3.21 Potassium solution, stock, 1 mL = 1000 µg K: Dissolve 0.1907 g KCl, previously dried at 110 °C for 3 hrs in 20 mL ASTM type I water. Add 2 mL (1+1) hydrochloric acid and dilute to 100 mL, with ASTM type I water.
- 7.3.22 Selenium solution, stock 1 mL = 500 µg Se: Dissolve 0.1405 g SeO₂ in 20 mL ASTM type I water. Dilute to 200 mL with ASTM type I water.
- 7.3.23 Silica solution, stock, 1 mL = 1000 µg SiO₂: Do not dry. Dissolve 0.2964 g NH₄SiF₆ in 20 mL solution mixture of ASTM type I water and 1 mL conc. hydrochloric acid, heating at 85°C for 5 min to effect solution. Cool, dilute to 100 mL with ASTM type I water, mix and immediately transfer to Teflon bottle for storage.
- 7.3.24 Silver solution, stock 1 mL = 250 µg Ag: Dissolve 0.125 g silver metal in 10 mL (1+ 1) nitric acid, heating to effect solution. Cool and dilute to 500 mL with ASTM type I water. Store in amber container.
- 7.3.25 Sodium solution, stock 1 mL = 1000 µg Na: Dissolve 0.2542 g NaCl in 20 mL ASTM type I water. Add 2 mL (1 + 1) nitric acid and dilute to 100 mL with ASTM type I water.
- 7.3.26 Strontium solution, stock 1 mL = 500 µg Sr: Suspend 0.1685 g SrCO₃ in 20 mL ASTM type I water. Dissolve continuously by adding dropwise 10 mL (1 + 1) hydrochloric acid. Dilute to 200 mL with ASTM type I water.
- 7.3.27 Thallium solution, stock 1 mL = 500 µg Tl: Dissolve 0.1303 g TlNO₃ in a solution mixture of 10 mL ASTM type I water and 2 mL conc. nitric acid. Dilute to 200 mL with ASTM type I water.
- 7.3.28 Tin solution, stock 1 mL = 1000 µg Sn: Dissolve 0.100 g Sn shot in 20 mL (1+1) hydrochloric acid, heating to effect solution. Cool and dilute to 100 mL with (1 + 1) hydrochloric acid.

7.3.29 Vanadium solution, stock 1 mL = 1000 µg V: Pickle vanadium metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1 + 1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.30 Yttrium solution, stock 1 mL = 1000 µg Y: Dissolve 0.1270 g Y₂O₃ in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 1000 mL with ASTM type I water.

7.3.31 Zinc solution, stock 1 mL = 500 µg Zn: Pickle zinc metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 10 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 200 mL with ASTM type I water.

7.4 **MIXED CALIBRATION STANDARD (CAL) SOLUTIONS--Prepare** mixed CAL solutions (Sects. 7.4.1 through 7.4.5) by combining appropriate volumes of the stock standard solutions in 500-mL volumetric flasks. First, add 20 mL of (1 + 1) nitric acid and 20 mL of (1 + 1) hydrochloric acid, then add the appropriate stock standard aliquots and dilute to 500 mL with ASTM type I water. prior to preparing the mixed CAL solutions, each stock solution should be analyzed separately to determine the presence of impurities. Transfer the freshly prepared mixed CAL solutions to an acid clean, not previously used FEP fluorocarbon or polyethylene bottles for storage. Fresh mixed CAL solutions should be prepared as needed with the realization that concentration can change on aging. The CAL solutions must be initially verified using a quality control sample and monitored weekly for stability (Sect. 7.12). Although not specifically required, the listed CAL solution combinations should be followed when using the specific wavelengths and recommended background correction locations listed in Table 1. If different combinations are used, the mixture should be verified for compatibility, stability and absence of spectral interference between analytes. This same requirement would apply if different wavelengths and/or background correction locations are utilized.

7.4.1 CAL Solution I (Volume = 500.0 mL)

<u>Analyte</u>	<u>Stock Solution</u>	<u>Aliquot Vol. mL</u>	<u>Analyte Conc. µg/mL</u>
Ag	7.3.24	1.0	0.5
As	7.3.3	5.0	10.0
B	7.3.6	1.0	2.0
Ba	7.3.4	1.0	1.0
ca	7.3.8	5.0	10.0
Cd	7.3.7	1.0	2.0
cu	7.3.11	1.0	2.0

Mn	7.3.16	1.0	2.0
Sb	7.3.2	5.0	5.0
Se	7.3.22	5.0	5.0

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of ASTM type I water and warm the flask until the solution clears. For the acid concentration used in the CAL solutions, the silver concentration should be limited to 0.5 mg/L. Higher concentrations of silver require additional hydrochloric acid.

7.4.2 CAL Solution II (Volume = 500.0 mL)

<u>Analyte</u>	<u>Stock Solution</u>	<u>Aliquot Vol. mL</u>	<u>Analyte Conc. µg/mL</u>
K	7.3.21	10.0	20.0
Li	7.3.14	5.0	5.0
MO	7.3.18	5.0	10.0
Na	7.3.25	5.0	10.0
Sr	7.3.26	1.0	1.0

7.4.3 CAL Solution III (volume = 500.0 mL)

<u>Analyte</u>	<u>Stock Solution</u>	<u>Aliquot Vol. mL</u>	<u>Analyte Conc. µg/mL</u>
co	7.3.10	1.0	2.0
V	7.3.29	1.0	2.0
P	7.3.20	5.0	10.0

7.4.4 CAL Solution IV (Volume = 500.0 mL)

<u>Analyte</u>	<u>Stock Solution</u>	<u>Aliquot Vol. mL</u>	<u>Analyte Conc. µg/mL</u>
Al	7.3.1	5.0	10.0
Cr	7.3.9	5.0	5.0
Hg	7.3.17	2.0	2.0
SiO	7.3.23	5.0	10.0
Sn	7.3.28	2.0	4.0
Zn	7.3.31	5.0	5.0

7.4.5 CAL Solution V (Volume = 500.0 mL)

<u>Analyte</u>	<u>Stock Solution</u>	<u>Aliquot Vol. mL</u>	<u>Analyte Conc. µg/mL</u>
Be	7.3.5	1.0	1.0
Fe	7.3.-12	5.0	10.0
Mg	7.3.15	5.0	10.0
Ni	7.3.19	1.0	2.0
Pb	7.3.13	5.0	10.0
Tl	7.3.27	5.0	5.0

7.5 **BLANKS** - Three types of blanks are **required** for this method. A calibration blank is used to establish the **analytical calibration curve**, a laboratory reagent **blank** is used to assess possible contamination from the sample preparation **procedure** and a **rinse** blank is used to flush the instrument uptake system and **nebulizer** between standards and samples to reduce memory interferences.

7.5.1 Calibration blank - **Prepare** by diluting a mixture of 20 mL of (1 + 1) **nitric** acid and 20 mL of (1 + 1) **hydrochloric** acid to 500 mL with **ASTM** type I water. Store in a Teflon bottle.

7.5.2 **Laboratory reagent** blank (**LRB**) - Contains all the reagents in the same volumes used in processing the samples. The **LRB** must be carried through the entire **preparation** procedure and analysis scheme. The **final** solution should **contain** the same acid concentrations as sample solutions for analysis.

7.5.3 **Rinse** blank - Prepare this acid wash solution in the same manner as the calibration blank and store in a convenient manner.

7.6 **PLASMA SOLUTION** - This solution is used for determining the optimum viewing height of the plasma above the work coil prior to using the method (Sect. 9.3.3). The solution is **prepared** by adding a 5 mL **aliquot** from each of the stock standard solutions of arsenic (Sect. 7.3.3) and **lead** Sect. **7.3.13**), and a 10 mL **aliquot** from each of the **stock** standard solutions of selenium (Sect. 7.3.22) and thallium (Sect. **7.3.27**), to a mixture of 20 mL (1 + 1) nitric acid and 20 mL (1 + 1) **hydrochloric** acid and diluting to 500 mL with ASTM type I water. Store in a Teflon bottle.

7.7 **TUNING SOLUTION** - This solution is used for adjusting the aerosol argon gas flow prior to calibration and analysis (Sect. 9.4). The solution is prepared by adding a 5 mL **aliquot** from each of the stock standard solutions of copper (Sect.

7.3.11) and lead (Sect. 7.3.13) to a mixture of 20 mL (1+ 1) nitric acid and 20 mL (1 + 1) hydrochloric acid and diluting to 500 mL with ASTM type I water. Store in a Teflon bottle.

7.8 LABORATORY PERFORMANCE CHECK (LPC) SOLUTION - This solution is prepared by adding the following listed aliquot volumes of the individual stock standards to the mixture of 20 mL (1+1) nitric acid and 20 mL (1+1) hydrochloric acid and diluting to 500 mL with ASTM type I water. Immediately transfer the freshly prepared LPC to an acid cleaned, not previously used, Teflon tattle.

<u>Analyte</u>	<u>Stock Solution</u>	<u>Aliquot Vol. mL</u>	<u>Analyte Conc. µg/mL</u>
Ag	7.3.24	1.0	0.5
Al	7.3.1	1.0	2.0
As	7.3.3	1.0	2.0
B	7.3.6	1.0	2.0
Ba	7.3.4	2.0	2.0
Be	7.3.5	2.0	2.0
Ca	7.3.8	1.0	2.0
Cd	7.3.7	1.0	2.0
co	7.3.10	1.0	2.0
Cr	7 3 9	2.0	2.0
cu	73 11	1.0	2.0
Fe	7 3.12	1.0	2.0
Hg	7 3.17	2.0	2.0
K	7.3.21	5.0	10.0
Li	7.3.14	2.0	2.0
Mg	7.3.15	1.0	2.0
Mn	7.3.16	1.0	2.0
MO	7.3.18	1.0	2.0
Na	7.3.25	1.0	2.0
Ni	7.3.19	1.0	2.0
P	7.3.20	5.0	10.0
Pb	7.3.13	1.0	2.0
Sb	7 3.2	2.0	2.0
Se	7 3.22	2.0	2.0
Si02	7.3.23	5.0	10.0
Sn	7.3.28	1.0	2.0
Sr	7 3.26	2.0	2.0
Tl	7 3.27	2.0	2.0
V	7.3.29	1.0	2.0
Zn	7.3.31	2.0	2.0

7.9 SPECTRAL INTERFERENCE CHECK (SIC) SOLUTIONS - Once the **interelement** spectral interference correction factors have been determined (Sect. 4.1.1) and the procedural routine for their use **has** been established, the operative **process** should be **periodically** tested and updated as needed. It is usually not **practical** to test and **update** the entire **corrective** process on a daily or weekly **basis**. The frequency of **confirming** and/or updating the entire corrective process is the **responsibility** of the analyst and should be dictated by instrument **stability**, **type** of samples **analyzed** and the **expected interference** encountered. The following **procedure** is recommended for testing the **interelement spectral** correction **process**. A general description of the procedure is given in Sect. 7.9.1. In Sect. 7.9.2 through 7.9.4 instructions are given for the preparation of SIC solutions that are specific to the wavelengths and background correction locations given in Table 1. The SIC solutions are designed to monitor and detect a 10% change in a partial **list** of the **interference** correction factors given in Table 3. The factors selected for monitoring were determined by dividing each of the listed **correction factors** by 10 and multiplying the quotient by the concentration of the interfering element in the **respective** SIC solution given below. If the resulting product was a number equal to or greater than two times the analyte MDL, the **correction** factor was included for monitoring.

7.9.1 **Prepare** an acid matrix solution of the **interfering** element at a high level of concentration (e.g., 50 **mg/L**). Complete 10 analyses of the solution and **determine** the standard deviation of the mean **concentration**. From the **data** **calculate** a concentration equal to 4.52 times the **standard deviation**. (This **calculated concentration estimates** the 95% **confidence** interval of the **interferent** mean concentration). Multiply the **calculated** concentration by the correction factor to **be** tested. Disregarding the **numerical** sign of the product, add a concentration value equivalent to 2.2X the **MDL** of the analyte that is being corrected. The sum of the two concentrations, when bisected by the calibration blank, describes an acceptable apparent analyte concentration **range**. If the apparent analyte concentration from the analysis of the **interferent** solution is within the acceptable **range**, the **correction** process is considered to be in control. If the apparent analyzed concentration is outside the range, **as** either a positive or negative concentration, a change in the correction process is indicated and an update of the process may be required.

NOTE: The interfering solution should be analyzed more than **once to confirm** a change **occurred** with **adequate** rinse time **between** solutions and **before** the subsequent analysis of the **calibration** blank

7.9.2 SIC solution I (50 **mg/L** Mo) - Add a 5 **mL aliquot** of the stock standard solution of molybdenum (Sect. 7.3.18) to a mixture of 4 **mL (1 + 1) nitric acid** and 4 **ml (1 + 1) hydrochloric acid** and dilute to 100 **mL** with **ASTM** type I **water**. **Store in** a Teflon bottle. This solution is used to evaluate the molybdenum **interelement spectral** correction factors on the **analytes**: Al, Sb, Se, **Sn**, and V. (See Table 3).

7.9.3 SIC solution II (10 mg/L Co; 20 mg/L Cr, Mn and V; and 40 mg/L Cu) - Add a 1 mL aliquot from the stock standard solution of cobalt (Sect. 7.3.10), a 2 mL aliquot from each of the stock standard solutions of manganese (Sect. 7.3.16) and vanadium (Sect. 7.3.29) and a 4 mL aliquot from the stock standard solutions of chromium (Sect. 7.3.9) and copper (7.3.11) to a mixture of 4 mL (1+1) nitric acid and 4 mL (1+1) hydrochloric acid and dilute to 100 mL with ASTM Type I water. Store in a Teflon bottle. This solution is used to evaluate the following list of interelement spectral correction factors (See Table 3).

<u>Analyte</u>	<u>Interferent</u>
Pb	Co
Sb	Cr
Mo	Mn
As	V
Be	V
Zn	cu

7.9.4 SIC Solution III (20 mg/L Ni, 30 mg/L Al and 150 mg/L Fe) Add a 2 mL aliquot from the stock standard solution of nickel (Sect 7.3.19), a 3 mL aliquot from the stock standard solution of aluminum (Sect. 7.3.1) and a 15 mL aliquot from the stock standard solution of iron (Sect. 7.3.12) to a mixture of 4 mL (1+1) nitric acid and 4 mL (1+1) hydrochloric acid and dilute to 100 mL with ASTM Type I water. Store in a Teflon bottle. This solution is used to evaluate the following list of interelement spectral correction factors (See Table 3).

<u>Analyte</u>	<u>Interferent</u>
Sb	Ni
Zn	Ni
As	Al
Ag	Fe
Cr	Fe
Mn	Fe

7.10 LABORATORY FORTIFYING STOCK SOLUTION - This solution is used in preparing the laboratory fortified blank and the laboratory fortified sample matrix. Prepare the solution in a 200-mL volumetric flask by adding the following listed aliquot volumes of the individual stock solutions to a mixture of 4 mL (1+1) nitric acid and 20 mL (1+1) hydrochloric acid. Dilute to the mark with ASTM type I water. Transfer the freshly prepared solution to a Teflon bottle for storage.

<u>Analyte</u>	<u>Stock Solution</u>	<u>Aliquot Vol. mL</u>	<u>Analyte Conc. $\mu\text{g/mL}$</u>
Ag	7.3.24	2.0	2.5
Al	7.3.1	5.0	25
As	7.3.3	S.O	25
B	7.3.6	5.0	25
Ba	7.3.4	10.0	25
Be	7.3.5	2.0	5
Cd	7.3.7	2.0	10
co	7.3.10	2.0	10
Cr	7.3.9	10.0	25
cu	7.3.11	5.0	25
Fe	7.3.12	5.0	25
Hg	7.3.17	2.0	5
Li	7.3.14	10.0	25
Mn	7.3.16	5.0	25
Mo	7.3.18	2.0	10
Ni	7.3.19	5.0	25
P	7.3.20	10.0	50
Pb	7.3.13	5.0	25
Sb	7.3.2	10.0	25
Se	7.3.22	10.0	25
SiO₂	7.3.23	5.0	25
Sn	7.3.28	2.0	10
Sr	7.3.26	10.0	25
Tl	7.3.27	10.0	25
V	7.3.29	2.0	10
Zn	7.3.31	10.0	25

NOTE: The **analytes** Ca, K, Mg, and Na are not **included** in the fortifying stock solution because their concentrations vary widely in environmental samples. The **analytes** B and SiO₂ should **be disregarded** if samples are processed and diluted in **borosilicate labware because** of the known **contamination** that occurs from **borosilicate** glass.

- 7.11 **LABORATORY FORTIFIED BUNK (LFB)** - To a **100 mL aliquot** of ASTM **type** water add **2 mL of (1 + 1)** nitric acid, **1.0 mL (1 + 1)** hydrochloric acid and **2 mL** of the **laboratory** fortifying **stock** solution (Sect. 7.10). The LFB must be **carried** through the entire sample **preparation procedure** and analysis scheme. The **final** solution should **be** diluted to **50 mL** as **are** the samples. Listed below is the expected concentration of each **analyte** based on the original **100 mL** of -water.

<u>Analyte</u>	<u>Conc. $\mu\text{g/mL}$</u>
Ag	0.05
Al	0.5
As	0.5
B	0.5
Ba	0
Be	0.1
Cd	0.2
co	0.2
Cr	0.5
cu	0.5
Fe	0.5
Hg	0.1
Li	0.5
Mn	0.5
Mo	0.2
Ni	0.5
P	1.0
Pb	0.5
Sb	0.5
Se	0.5
SiO ₂	0.5
Sn	0.2
Sr	0.5
Tl	0.5
V	0.2
Zn	0 . 5

7.12 **QUALITY CONTROL SAMPLE** - The quality control sample (Sect. 3.18) should be prepared in the same acid matrix as the **calibration** standards at a concentration near 1 **mg/L**, except silver, which must **be** limited to a concentration of 0.5 **mg/L**. Follow the instructions provided by the supplier and store the sample in a Teflon bottle. The **Quality Assurance** Research Division of **EMSL-Cincinnati** will either supply a quality control sample or provide information where one of equal **quality can be** procured.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Prior to collection of an aqueous sample, **consideration** should be given to the type of **data required**, (i.e., dissolved or total recoverable), so **that** appropriate preservation and **pretreatment** steps **can** be taken. **Filtration**, acid **preservation**, etc., should be performed at the time of **sample** collection or **as soon thereafter** as **practically** possible.

a.2 For determination of dissolved elements, the sample must be **filtered** through a **0.45- μm membrane filter**. (Glass or plastic filtering apparatus is recommended to avoid possible contamination. Only plastic apparatus should be used when determination of boron or **silica** is critical (Sect. 1.6). Use a portion of the **filtered** sample to **rinse** the filter flask, **discard this** portion and collect the required volume of **filtrate**. Acidify the **filtrate** with **(1 + 1)** nitric acid immediately **following filtration to a pH < 2**.

8.3 For the **determination** of total recoverable elements in aqueous **samples**, acidify with (1+1) nitric acid at the time of collection to a **pH < 2** (normally, 3 mL of (1 + 1) acid per liter of sample is sufficient for most ambient and **drinking** water samples). The sample should not **be** filtered prior to analysis (Sect. 1.6).

NOTE: Samples **that cannot** be acid preserved at the time of collection because of sampling limitations or transport restrictions should be **acidified** with nitric acid to a **pH < 2** -upon receipt in the laboratory. Following acidification, the sample should be held for 16 hours before withdrawing an **aliquot** for sample processing.

8.4 Solid samples us ally **require** no **preservation** prior to analysis other than storage at **40C**.

9. CALIBRATION AND STANDARDIZATION

9.1 Recommended wavelengths **and** background **correction** locations are listed in Table 1. Other wavelengths and background **correction** locations may be **substituted** if they **can** provide the **needed** sensitivity **and are** corrected for **spectral** interference. In Table 4 **specific** instrument **operating** conditions are recommended. However, **because** of the difference among various makes and models of spectrometers, the analyst should follow the instrument manufacturer's instructions, and if possible, approximate the recommended operating conditions.

9.2 Allow the **instrument** to become thermally stable before beginning. This usually requires at least 30 min of operation prior to plasma optimization, plasma tuning and/or **calibration**.

9.3 **PLASMA OPTIMIZATION - Prior to the use of this method optimize time plasma operating conditions using the following procedure.** The purpose of **plasma optimization is to provide a maximum signal to background ratio for the least sensitive element in the analytical array.** The use of a mass flow controller to **regulate** the **nebulizer gas flow rate** greatly **facilitates the procedure**.

9.3.1 Select an **appropriate** incident of power with minimum reflected power (see Table. 4 for recommendations) and **aspirate** the 1000 $\mu\text{g/mL}$ solution of yttrium (Sect. 7.3.30). Following the instrument manufacturer's instructions adjust the aerosol

carrier gas flow rate through the nebulizer SJ a **definitive** blue emission region of the plasma extends approximately from 5 to 20 mm above the top of the work coil.⁽¹¹⁾ Record the **nebulizer gas flow rate** or pressure setting for future reference.

- 9.3.2 **After** establishing the **nebulizer gas flow rate**, determine the **solution uptake rate** of the **nebulizer** in **mL/min** by aspirating a known volume acid blank for a period of **at least 3 min**. **Divide the spent volume by three and record the uptake rate**. Set the **peristaltic pump to deliver the uptake rate in a steady even flow**.
- 9.3.3 After horizontally aligning the plasma **and/or optically profiling** the **spectrometer**, use the selected instrument conditions from Sects. 9.3.1 and 9.3.2, and aspirate the plasma solution (Sect. 7.7), **containing 10 µg/mL each** of **As, Pb, Se and Tl**. Collect intensity data at the wavelength **peak** for each **analyte** at 1 mm intervals from 14 to 18 **mm** above the top of the work coil. (This region of the plasma is commonly referred to **as the analytical zone.**)¹² **Repeat** the process using the calibration blank. Determine the net signal to blank intensity ratio for each **analyte** for each viewing height setting. Choose the height for viewing the plasma that provides the largest **intensity** ratio for the **least** sensitive element of the four **analytes**. If more than one position provides the **same** ratio, select the position that **provides the best compromise** of **intensity** ratios of all four **analytes**.
- 9.3.4 The instrument **operating** condition finally selected as being optimum should **provide the lowest reliable IDLs and MDLs similar to those listed in Table 2**.
- 9.3.5 If either the instrument **operating conditions**, (such as incident power and/or **nebulizer gas flow rate**) **are changed, or a new torch injector tube having a different orifice i.d.** is installed, the plasma and plasma viewing height should be **reoptimized**.
- 9.3.6 Before daily calibration and after the instrument warm-up period (Sect. 9.2), the **nebulizer gas flow** must be reset to the determined **optimized** flow. If a mass flow controller is being used, it should **be** either reset to the recorded optimized flow rate or the optional plasma tuning **procedure** given in Sect. 9.4 should be followed to **reconfigure** the plasma. In order to provide and maintain valid **interelement spectral** correction factors the **nebulizer gas flow rate** must be well controlled. The **change** in signal **intensity** with a change in **nebulizer gas flow rate** for both "hard" (**Pb 220.353 nm**) and "soft" (**Cu 324.754 nm**) **lines is illustrated in Figure 1**.
- 9.4 **PLASMA TUNING (Optional)** - **This procedure can be used on a daily basis to collect the data necessary for fine tuning the plasma to a set Cu/Pb concentration ratio** that reflects the **optimized** Conditions **determined** in Sect. 9.3. **The analytical zone of the plasma can be altered by varying** the aerosol **carrier gas flow** entering the plasma. **This procedure requires the use of a mass flow controller for**

adjusting the **nebulizer gas flow rate** to reset the **Cu/Pb** concentration ratio. (This procedure can be used even when the front surface entrance optics degrade in a non-uniform manner over the visible and ultraviolet wavelength regions.)

- 9.4.1 Set the instrument to the **optimized** operating conditions (Sect. 9.3). After instrument warm-up, horizontal **alignment** of the plasma and/or optical profiling of the spectrometer, **aspirate** the **tuning solution** (Sea. 7.7) and collect 10 replicate **measurements** of the **Cu** (324.75 nm) and **Pb** (220.35 nm) intensity signals at **every 25 mL/min interval** over the flow rate **range** of 500 to **800 mL/min**. Repeat the **operation** using the **calibration** blank solution. Subtract the **respective** mean blank value and calculate the net mean intensity value for both metals at each flow rate. Plot the net mean intensity values versus flow rate **as** illustrated in Figure 1. From the plot determine the maximum signal intensity flow rate-for-each metal.
- 9.4.2 To **determine** the **Cu/Pb concentration** ratio, set the instrument to the optimized operating conditions. After warm-up and **optical** profiling, **calibrate** the instrument for both Cu (324.75 nm) and Pb (220.35 nm) at their **respective** maximum intensity flow rates (See Figure 1, Cu 750 mL/min, Pb 535 mL/min) with the **calibration** blank set at **the** optimum flow (e.g., 620 mL/min).
- 9.4.3 **Reset the nebulizer gas flow to the rate established in Sect. 9.3.1 (e.g., 620 mL/min)** and collect data from 10 **replicate** analyses of the tuning solution (Sect. 7.6). Ratio the **determined copper concentration** to the **determined** lead concentration on each analysis and **compute** the **standard** deviation and mean value of the 10 **ratios**. (Note: **Disregard** the fact **that** the **determined** concentrations do not equal the prepared **concentrations** of the tuning solution.) The **mean** value is used for **resetting** the ratio on a daily basis.
- 9.4.4 For tuning the plasma on a daily basis calibrate the instrument as described in Sect. 9.4.2. Reset the **nebulizer** gas flow rate to the optimum flow (e.g. 620 mL/min) and analyze the tuning solution. Calculate the **Cu/Pb** concentration ratio from the analysis. If the calculated ratio is not within two standard deviations of the mean value established in Sect. 9.4.3, adjust the **nebulizer** gas flow and **reanalyze** the tuning solution until the ratio is within **range**. Lowering the gas flow **rate** will **increase the lead concentration, decrease the copper concentration, and, therefore, lower the ratio. The opposite** is true when the gas flow is **increased. Day-to-day** variations in the **nebulizer** gas flow should **be < + 10 mL/min. Larger** changes should alert the analyst to possible instrumental problems.
- 9.4.5 **Once** an acceptable ratio is **achieved**, the instrument is **ready** for **analytical** calibration.

- 9.4.6 If either the selected instrument operating conditions are changed or instrument components replaced that require the **plasma to be reoptimized** (Sect. 9.3.5), the **Cu/Pb** concentration ratio must be reestablished.
- 9.5 CALIBRATION - **Calibrate** the instrument according to the instrument **manufacturer's** instructions using the **prepared calibration blank** (Sect. 7.5.1) and CAL solutions (Sect. 7.4). The following **operational** steps should be used for both CAL solutions and samples.
- 9.5.1 Using a peristaltic pump **introduce** the standard or **sample to nebulizer** at a uniform rate (e.g., 1.2 **mL/min**).
- 9.5.2 To allow equilibrium to be **reached** in the plasma, aspirate the standard or sample solution for 30 **sec** after **reaching** the plasma before **beginning** integration of the background corrected signal.
- 9.5.3 When possible use the average value of four 5 **sec** background corrected integration **periods** as the atomic emission signal to **be correlated to analyte concentration**.
- 9.5.4 Between each **standard** or **sample, flush the nebulize; and solution** uptake system with the **rinse** blank acid solution (Sect. 7.5.3) for 60 **sec** or for the required **period of time to ensure that analyte memory effects are not occurring**.
- 9.6 Analyze the **LPC** solution (Sect. 7.8) and **calibration** blank (Sect. 7.5.1) immediately following **calibration**, after every tenth sample and at the end of the **sample** run. The **analyzed** value of each **analyte** in the LPC solution should be within 95 % to 105 % of its expected value. If an **analyte value** is outside the interval, **reanalyze** the **LPC**. If the **analyte** is again outside the $\pm 5\%$ limit, the instrument should be **recalibrated** and **all** samples following the last acceptable **LPC** solution should be reanalyzed.
- 9.7 **Periodically** verify the validity of the interelement **spectral** interference **correction** process. The frequency of this testing is the **responsibility** of the analyst, however, **confirmation** prior to analysis of **solid sample extracts** is particularly useful. **See** sect. 7.9 for guidance and criteria.
- 9.8 If **methods** of standard addition **are required**, the following procedure is **recommended**.
- 9.8.1 **The standard** addition **technique**¹³ involves preparing new **standards** in the sample matrix by **adding known** amounts of standard to one or more **aliquots** of the processed sample solution. This **technique compensates** for a sample constituent that enhances or depresses the **analyte** signal thus **producing** a different slope from

that of the calibration standards. It will not **correct** for additive interference that causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows. Two identical aliquots (Volume V_x) of the sample solution, are taken. To the first [labeled A] is added a small volume V_s of a standard **analyte** solution of concentration **c_s** . To the second (labeled B) is **added** the **same** volume V_s of the solvent. The **analytical** signals of A and B are **measured** and corrected for **non-analyte** signals. The unknown sample concentration C_X is **calculated**:

$S_B - V_S C_S$

$C_X =$

$(S_A - S_B) V_X$ where S_A and S_B are the analytical signals (corrected for the **blank**) of solutions A and B, respectively. V_s and **c_s** should **be** chosen so that S is roughly twice B on the average. It is best if V_s is made much less than V_x , and thus **c_s** is much greater than C_X , to avoid excess **dilution** of the sample **matrix**. If a **separation** or concentration step is used, the additions are best made **first** and **carried** through the entire procedure. For results from this technique to be valid, the **following** limitations must be **taken** into consideration:

1. The **analytical** curve must be linear.
2. The **chemical form** of the **analyte** added must respond the same as **the analyte** in the sample.
3. The interference **effect** must **be** constant over the working **range** of concern.
4. The signal must be corrected for any additive interference.

10. QUALITY CONTROL

10.1 Each **laboratory** using this method is required to operate a **formal** quality control (QC) program. The minimum requirements of this **program** consist of an initial demonstration of laboratory capability and analysis of laboratory reagent blanks and **fortified** blanks and samples as a continuing check on performance. The laboratory is required to maintain **performance** records that **define** the quality of data generated.

10.2 **INITIAL DEMONSTRATION OF PERFORMANCE.**

10.2.1 The **initial demonstration** of **performance** is used to **characterize instrument performance** (**MDLs** and **linear** calibration ranges) and laboratory performance (analysis of quality control sample) for 'analyses **conducted** by this method.

10.2.2 **MDLs** should be established for all **analytes**, using reagent water (blank) fortified at a concentration of two to three times the estimated detection **limit**⁴. To determine MDL values, take seven replicate **aliquots** of the **fortified** reagent water and process through the **entire analytical** method. **Perform** all calculations defined in the method and report the **concentration** values in the appropriate units. **Calculate** the MDL as follows:

$$\text{MDL} = (t) \times (S)$$

where:

- t = Student's t value for a 99% **confidence** level and a **standard** deviation estimate with n-1 degrees of **freedom** [t = 3.14 for seven **replicates**].
S = **standard** deviation of the **replicate** analyses.

MDLs should be determined every 6 months or whenever there is a **significant** change in the background or instrument response.

10.2.3 **Linear calibration ranges** - The **upper** limit of the **linear calibration** range should be established for each **analyte** by ~~determining~~ the signal responses from a minimum of **three different concentration standards**, one of which is **close** to the **upper** limit of the **linear range**. The **linear calibration** range which may be used for the analysis of samples should be judged by the analyst **from** the resulting data. **Linear calibration** ranges should be **determined** whenever there is a **significant** change in **instrument response** and **every** 6 months for those **analytes** that **periodically** approach their **linear limit**.

10.2.4 **Quality Control Sample (QCS)** - When beginning the use of this method and on a quarterly basis, **verify** acceptable laboratory performance with the preparation and analyses of a quality control sample (Sect. 7.12). The QCS is carried through the **entire analytical operation** of the method. If the **determined** concentrations are not within + 5% of the stated values of 1 **mg/L**, laboratory performance is unacceptable. The source of the problem should be identified and corrected before continuing analyses.

10.3 ASSESSING LABORATORY PERFORMANCE - **REAGENT** AND FORTIFIED **BLANKS**

10.3.1 **Laboratory reagent blank (LRB)** - The laboratory must analyze at least one **LRB** (Sect. 7.5.2) with each set of sample. **LRB data** are used to assess **contamination from** the **laboratory environment**. If an **analyte value** in the reagent blank exceeds its **determined MDL**, then **laboratory** or reagent **contamination** should be suspected. Any determined source of **contamination** should be corrected and the samples reanalyzed.

10.3.2 Laboratory fortified **blank (LFB)** - The laboratory must analyze at least one **LFB** (Sect. 7.11) with each batch of samples. Calculate accuracy as percent recovery (Sect. 10.4.2). If the recovery of any analyte falls outside the control limits (Sect. 10.3.3), that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

10.3.3 Until **sufficient LFB data become** available (usually a minimum of 20 to 30 analyses), the **laboratory** should assess **laboratory performance** against recovery limits of 85-115%. When **sufficient** internal performance data becomes available, develop control limits from the percent mean recovery (\bar{x}) and the standard deviation (S) of the mean recovery. These **data** are used to establish upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = \bar{x} + 3s$$

$$\text{LOWER CONTROL LIMIT} = \bar{x} - 3s$$

After each **5** to 10 new recovery measurements, new control **limits** should be **calculated** using only the most recent 20 to 30 data **points**.

10.4 ASSESSING ANALYTE RECOVERY - LABORATORY FORTIFIED SAMPLE MATRIX

10.4.1 The laboratory -must add a known amount of each analyte to a minimum of 10% of the routine samples or one sample per sample set, whichever is greater. **Ideally** for water samples, the analyte concentration should be the same as that **used** in the LFB (Sect. 10.3.2). This is also recommended for solid samples, however, the concentration added should be expressed **as mg/kg** and calculated by multiplying the values given in Sect. 7.11 by the factor 100. Over time, samples from all routine sample sources should be **fortified**.

10.4.2 Calculate the percent recovery for each analyte, corrected for background concentrations measured in the **unfortified** sample, and compare these values to the control limits established in Sect. 10.3.3 for the analyses of **LFBs**. Recovery calculations **are** not required if the **concentration** added is less than 10% of the sample background concentration. Percent recovery may be calculated in units appropriate to the **matrix**, using the following **equation**:

$$R = \frac{C_s}{C} \times 100$$

where:

- R = percent recovery.
- cs = fortified sample concentration.
- C = sample background concentration.
- s = **concentration** equivalent of **analyte** added to sample.

10.4.3 If recovery of any **analyte** falls outside the **designated** range and **laboratory performance** for that **analyte** is shown to be in control (Sect. 10.3), the recovery problem encountered with the **fortified** sample is judged to be matrix related, not system related. The data **user** should be informed that the result for that **analyte** in the unfortified sample is suspect due to **matrix** effects and analysis by method of standard addition (Sect. 9.8) should be considered.

11. PROCEDURE

11.1 AQUEOUS SAMPLE PREPARATION - DISSOLVED ELEMENTS

11.1.1 For the determination of dissolved elements in ground and surface waters, take a 100 mL (+ 1 mL) aliquot of the filtered acid preserved sample, add 2 mL of (1+1) nitric acid and 1 mL (1+1) hydrochloric acid. The sample is now ready for analysis. Allowance for **sample** dilution should be made in the calculations.

NOTE: If a precipitate is formed during **acidification**, **transport** or **storage**, the **sample aliquot must be treated using the procedure in Sect. 11.2.1 prior to analysis.**

11.2 AQUEOUS SAMPLE PREPARATION - TOTAL RECOVERABLE ELEMENTS

11.2.1 For determination of total recoverable elements in water or waste water, other than marine and **estuarine** water, take a 100 mL (+ 1 mL) aliquot from a well mixed, acid preserved sample and transfer it to a 250-mL Griffin beaker. [For drinking water compliance monitoring certain **analytes** require 4X **preconcentration** prior to analysis (Sect. 1.7)]. Add 2 mL of (1+1) nitric acid and 1.0 mL of (1+1) hydrochloric acid. **Heat** the sample on a hot plate at 85°C until the volume has been reduced to approximately 20 mL, ensuring that the sample **does** not boil. (A **spare beaker containing 20 mL of water can be used as a gauge.**)

NOTE: For proper heating **adjust the temperature control** of the hot plate such that an uncovered **beaker** containing 50 mL of water **located** in the center of the hot plate **can be** maintained at a temperature no higher **than 85OC**. **Evaporation** time for 100 mL of sample at **85OC** is approximately 2 h with the rate of evaporation rapidly **increasing** as the sample volume approaches 20 mL.

Cover the **beaker** with a watch glass and reflux for 30 min. Slight boiling may occur but vigorous boiling should be avoided. Allow to **cool** and quantitatively transfer to either a **50-mL** volumetric or a **50-mL** class A stoppered graduated cylinder. Dilute to volume with **ASTM** type I water and mix. **Centrifuge** the sample or allow to stand **overnight** to **separate** insoluble material. The sample is now **ready** for analysis. **Because** the effects of various matrices on the stability of diluted samples **cannot be characterized**, samples should **be** analyzed as soon as possible after **preparation**.

11.2.2 For determination of total recoverable elements in marine and estuarine water, take a 100 **mL** aliquot from a well mixed, aci- preserved sample and transfer to a **250-mL** Griffin **beaker**. Add 2 **mL** of (1 + 1) nitric **acid**, and heat on a hot plate at 85°C until the volume has been reduced to approximately 25 **mL**, ensuring that the sample does not boil. (See NOTE in Sect. 11.2.1). Cover the beaker with a watch glass and **reflux** for 30 min. Slight boiling may occur but vigorous boiling should **be** avoided. Allow to cool and **dilute** to 100 **mL** with **ASTM** **type** I water. Centrifuge the sample or allow to stand overnight to separate insoluble material. The **sample** is now ready for analysis by the method of standard addition (Sect. 9.8). **Because** the effects of various matrices on the stability of diluted samples **cannot be characterized**, samples should be analyzed as **soon as** possible after **preparation**.

11.3 **SOLID** SAMPLE PREPARATION - **TOTAL** RECOVERABLE ELEMENTS

11.3.1 For determination of total recoverable elements in solid samples (sludge, soils, and *sediments*), mix the sample thoroughly to achieve homogeneity and weigh accurately a 1.0 + 0.01 g portion of the sample. Transfer to a **250-mL** Phillips beaker. Add 4 **mL** (1 + 1) nitric acid and 10 **mL** (1 + 4) hydrochloric acid. Cover with a watch glass. Heat the sample on a hot plate and gently **reflux** for 30 min. **Very** slight boiling may occur, however, vigorous boiling must be avoided to prevent the loss of the HCl-H₂O **azeotrope**.

NOTE: For proper heating adjust the temperature control of the hot plate such that an uncovered **Griffin beaker** containing 50 **mL** of water located in the center of the hot plate **can be** maintained at a **temperature** of approximately but no higher than 85 °C.

Allow the **sample** to cool and quantitatively transfer to **100mL** **volumetric** flask. Dilute to volume with **ASTM** type **I** water and mix. Centrifuge the sample or allow to stand overnight to **separate** insoluble **material**. The sample is now ready for analysis. **Because** the **effects** of various matrices on the stability of diluted **samples cannot be characterized**, samples should **be** analyzed as soon as possible after **preparation**.

NOTE: Determine the percent solids in the sample for calculating and reporting data on a dry weight basis. To determine the dry weight, transfer a **separate**, uniform 1 g **aliquot** to an evaporating dish and dry to a constant weight at 103-105 C.

11.4 SAMPLE ANALYSIS

- 11.4.1 Analyze the samples by the procedural routine described **in** Sects. 9.5, 9.6 and 9.7. If method of **standard** additions are required follow the instructions given in Sect. 9.8. Samples **having concentrations** higher than the **established linear dynamic range (LDR)** should **be** diluted into range and reanalyzed. The sample may **first** be analyzed for trace analytes providing the elements in high **concentration** do not **cause** a severe **matrix** effect and any **interelement** spectral interference or shift in background **intensity can be** properly corrected.
- 11.4.2 For **drinking** water compliance monitoring, if the **concentration** of a primary **contaminant** is **determined** to be 90% of its **MCL** or above and the combined **Mg and Ca concentration** equals **500 mg/L**, the sample should be **analyzed** by the standard **addition** technique (Sect. 9.8).

12. CALCULATIONS

- 12.1 Sample **data** should **be** reported in units of **mg/L** for aqueous samples **and mg/kg dry weight for solid** samples. **Do not report** element **concentrations** below the **determined MDL**.
- 12.2 For **aqueous** samples prepared by total recoverable procedure (Sect. 11.2.1), multiply solution **concentrations** by the dilution factor 0.5. Round the data to the thousandth place and **report** the data in **mg/L** up to **three** significant figures.
- 12.3 For **estuarine** and **marine** water samples prepared by total recoverable procedure (Sect. 11.2.2), **read** the **concentration** directly from the instrument **and** calculate the sample concentration by the procedure described **in** Sect. 9.8. Round the data **to** the **thousandth place** and report the data in **mg/L** up to **three significant** figures.
- 12.4 For **solid samples prepared** by **total** recoverable **procedure** (Sect. 11.3) round the solution **concentrations** ($\mu\text{g/mL}$ in the analysis solution) to the **thousandth** place and multiply by the **dilution** factor 100. Report the data to a 0.1 **mg/kg** up to **three significant** figures **taking** into account the **percent** solids as *noted in* Sect. 11.3 when the data are reported on a dry weight basis.
- 12.5 If additional dilutions were performed or if a **drinking** water **sample** was **preconcentrated** 4x for analysis, the appropriate factor must be applied to sample values.

- 12.4 The QC data obtained during sample analyses provide an indication of the quality of the sample data and should be provided with the sample results.

13. PRECISION AND ACCURACY

- 13.1 **Listed** in Table 2 are **MDLs** determined using the procedure described in Sect. 10.2.2. **The MDLs** were determined in the reagent blank matrix (best case situation) following sample **preparation** given in Sect. **11.2.1**. Teflon beakers were used to avoid boron and **silica** contamination from glassware with the **final** dilution to 50 **mL** completed in polypropylene centrifuged tubes.
- 13.2 Data obtained from single laboratory method testing are summarized in Table 5 for five types of water samples consisting of drinking water, surface water, ground water, and two wastewater effluents. Samples were prepared using the procedure described in Sec. 11.2.1.

For each matrix, five **replicate aliquots** were prepared, analyzed and the average of the five determinations used to **define** the sample background concentration levels. For each method analyte, the sample background concentration, mean percent **recovery**, **standard** deviation of the percent recovery, and relative **percent difference** between the **duplicate** fortified samples are listed in Table 5. The variance of the **five replicate** sample background **determinations** is included in the **calculated** standard deviation of the percent **recovery** when the analyte **concentration in the** sample was greater **than** the **MDL**. The tap and well waters were **processed** in Teflon and quartz **beakers** and d&t'd in polypropylene centrifuged tubes. The **nonuse** of **borosilicate** glassware is reflected in the precision and recovery data for boron and silica in those two sample types.

- 13.3 Data **obtained** from single laboratory method testing are **summarized** in Table 6 for three solid **samples** consisting of **EPA 884 Hazardous Soil**, **SRM 1645 River Sediment**, and **EPA 286 Electroplating Sludge**. Samples were prepared using the procedure described in Sect. 11.3. For each method analyte, the sample background concentration, mean **percent** recovery of the **fortified** additions, the standard deviation of the percent **recovery**, and relative percent difference between **duplicate** additions were determined **as** described in **Sect. 13.2**.
- 13.4 Data obtained from **single laboratory method testing** when using the procedure given in Sect. **11.2.1** but utilizing the 4X **preconcentration** step prior to analysis as required for the **determination** of certain drinking water contaminants are summarized in Table 7. Seven **replicate** aliquots of Cincinnati, Ohio, **tapwater** were prepared and analyzed to determine background concentrations. In addition, two more sets of seven **replicates** each were fortified at different levels of **concentration** with an attempt to **bracket** or match either current or proposed Maximum Contaminant Level concentrations. For each method analyte, the

sample background concentration, concentration added, mean percent recovery of the fortified addition, and relative standard deviation of the mean recovery are listed in Table 7. **All aliquots** were processed in Teflon beakers and diluted to volume in polypropylene centrifuged tubes. The sample **analyte** less than values **indicate 4X MDLs**. The **4X MDL** values for the **analytes: Al, B, Ba, Mn, Sr, and Zn are** 0.01, 0.002, 0.0003, 0.0002, **0.0002**, and 0.001 **mg/L**, respectively.

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14.5 **Appendix** to Method 200.7, Inductively Coupled Plasma Atomic Analysis of Drinking Water, Revision 1.3, March 1987, U.S. **Environmental Protection** Agency, **Office of Research** and Development, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio 45268.

TABLE 1. **RECOMMENDED WAVELENGTHS WITH LOCATIONS FOR BACKGROUND CORRECTION AND ESTIMATED INSTRUMENT DETECTION LIMITS (IDL)**

Analyte	Wavelength, nm ¹	Location for Bkgd. Correction	Estimated IDLs mg/L ⁽²⁾
Ag	328.068	+0.070 nm	0.005
Al	308.215	+0.070 nm	0.05
As	193.696	+0.070 nm	0.03
B	249.678x2	+0.035 nm	0.006
Ba	493.409	-0.064 nm	0.001
Be	313.042	-0.064 nm	0.0007
Ca			
Cd	315.887 226.502	+0.070 nm	0.02 0.002
Co	228.616	-0.064 nm	0.007
Cr	205.552x2	-0.032 nm	0.007
Cu	324.754	-0.064 nm	0.003
Fe	259.940	+0.070 nm	0.007
Hg	194.227x2	-0.032 nm	0.02
K	766.491	-0.064 nm	0.7
Li	670.784	+0.070 nm	0.005
Mg	279.079	-0.064 nm	0.03
Mn	257.610	+0.070 nm	0.0008
Mo	203.844	-0.064 nm	0.02
Na	588.995	+0.070 nm	0.03
Ni	231.604x2	+0.035 nm	0.009
P	214.914x2	+0.035 nm	0.09
Pb	220.353	-0.064 nm	0.03
Se	206.833	+0.070 nm	0.03
Si	251.611 196.090	+0.070 nm	0.08 0.02
O ₂			
Sn	189.980x2	-0.032 nm	0.02
Sr			
Tl	421.552 190.864	+0.070 nm	0.0006 0.03
V	292.402	+0.070 nm	0.009
Zn	213.856x2	+0.035 nm	0.002

(1) Wavelength x 2 indicates wavelength is read in second order.

(2) The IDLs were estimated from three times the standard deviation of 10 replicate measurements of the calibration blank. The calculated IDL was rounded upward and reported to a single digit.

Analyte	Aqueous, mg/L	Solids, mg/kg
Ag		
Al	0.002 0.02	0.3 3
AS	0.008	2
B	0.003	
Ba	0.001	0.2
Ca	0.0003	0.1
Cd	0.01 0.001	0.2 2
Co	0.002	0.4
Cr	0.004	0.8
Cu	0.003	0.5
Fe	0-03-	6
		2
Hg	0.007	60
Li	0.001	2
Mg	0.02	3
Mn	0.001	0.2
Mo	0.004	1
Na	0.03	20
Ni	0.005	1
P	0.06	12
Pb	0.01	2
		2
Sb	0.008	5
SiO ₂	0.02	
Sn	0.007	2
Sr	0.0003	0.1
Tl	0.02	3
V		1
Zn	0.003 0.002	0.3

(1) MDL Concentrations are computed for original matrix with allowance for 2x sample **preconcentration** during preparation. Samples were **processed** in Teflon and diluted in 50-mL plastic centrifuge tubes.

(2) Based on aqueous solution determination.

Boron not reported because of glassware contamination.
Silica not determined in solid samples.

- Elevated value due to fume hood contamination.

TABLE 3. LISTING OF POTENT IA1 INTERELEMENT SPECTRAL INTERFERENCE

<u>Analyte</u>	<u>Wavelength, nm</u>	<u>Interfering Element</u>	<u>Correction Factor</u>	<u>Analyte</u>	<u>Wavelength, nm</u>	<u>Interfering Element</u>	<u>Correction factor</u>				
Ag	328.068	Fe	-0.0002	Co	228.616	%a	0.0009				
		Mn	0.0001			Cr	0.0002				
		V	-0.0001			Mo	0.0001				
		Ni	0.0003								
Al	308.215	Co	-0.0020	Cr	205.552x2	Be	0.0014				
		Mo	0.0107			CU	-0.0004				
			0.0082			Fe	-0.0009				
		Mo	0.0009								
As	193.696	Al	0.0067	Cu	324.754	Mo	0.0005				
		Be	-0.0007			Fe	259.940	None	--		
		CO	0.0004					Hg	194.227x2	Mo	0.0004
		Fe	0.0003							V	0.0030
		Mo	-0.0012	K	766.491	None	--				
		Ni	0.0001	Li	670.784	None	--				
V	0.0120	Mg	279.079	Mn	-0.0030						
				Mo	-0.0029						
B	249.678x2	None	--	Mn	257.610	Fe	-0.0004				
%a	493.409	None	--			Mo	203.844	Al	-0.0002		
%e	313.042	V	0.0041	Fe	-0.0001						
Ca	315.887	Co	0.0016	Mn	-0.0041						
		Cr	-0.0002								
		Mo	0.0033								
Cd	226.502	Co	-0.0012								
		fe	-0.00004								
		Ni	0.0004								
		Sn	-0.0003								

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TABLE 3. (Continued)

<u>Analyte</u>	<u>Wavelength, nm</u>	<u>Interfering Element</u>	<u>Correction Factor</u>	<u>Analyte</u>	<u>Wavelength, nm</u>	<u>INTERFERING Element</u>	<u>CORRECTION FACTOR</u>
Na	588.995	None	--	Si	251.611	None	--
Ni	231.604x2	co Mo Tl	0.0011 -0.0016 0.0005	Sn	189.980x2	Fe Mn Mo Sb Si	0.0004 0.0004 -0.0114 -0.0009 0.0002
P	214.914x2	Al Ca CU Mo	-0.0019 -0.0014 0.0121 0.0060	Sr	421.552	None	--
Pb	220.353	Al co Cr cu Fe Ni V	0.0013 -0.0332 -0.0021 0.0005 -0.0002 -0.0012 -0.0016	Tl	190.864	Co Fe Mn Mo P V	0.0054 0.0008 0.0021 0.0057 0.00008 0.0038
Sb	206.833	co Cr Fe Mo Ni Sn	-0.0030 0.0114 0.00008 0.0082 -0.0092 0.0024	V	292.402	Cr Fe Mo	0.0006 0.0005 0.0026
Se	196.090	AS CO fe Mo V	-0.0025 -0.0047 0.0004 -0.0152 -0.0022	Zn	213.856x2	Cu Fe Ni	0.0011 0.0001 0.0034

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TABLE 4. INDUCTIVELY COUPLED PLASMA INSTRUMENT OPERATING CONDITIONS

Incident rf power	1100 watts
Reflected rf power	< 5 watts
Viewing height above work coil	15 mm
Injector tube orifice i.d.	1 mm
Argon supply	liquid argon
Argon pressure	40 psi
Coolant argon flow rate	19 L/min
Aerosol carrier argon flow rate	620 mL/min
Auxiliary (plasma) argon flow rate	300 mL/min
Sample uptake rate controlled to	1.2 mL/min

PB-CU ICP-AES EMISSION PROFILE

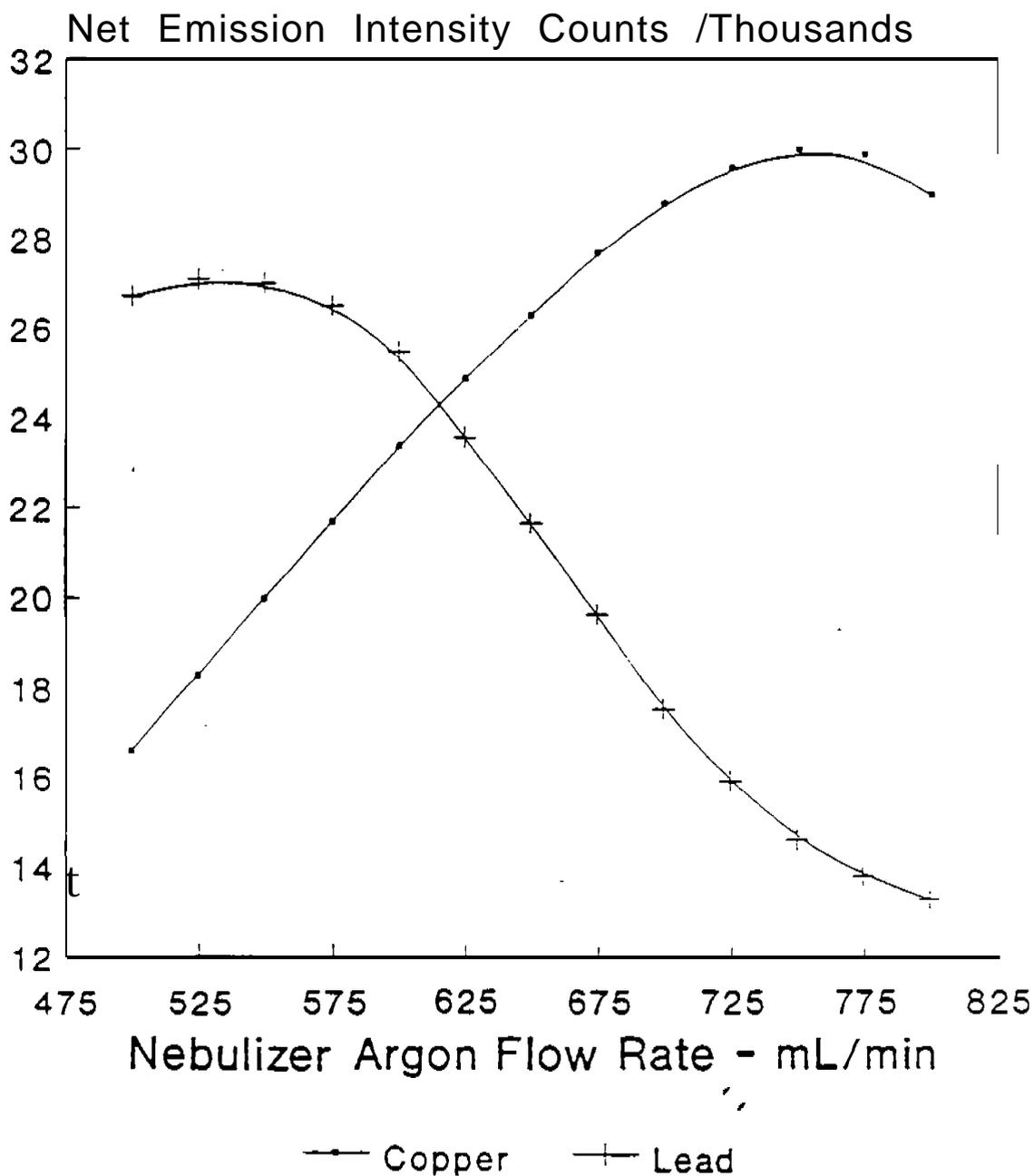


FIGURE 1

TABLE 5. PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES

TAP WATER

ANALYTE	SAMPLE	LOW	AVERAGE			HIGH	AVERAGE		
	CONC	SPIKE	RECOVERY	S(R)	RPD	SPIKE	RECOVERY	S(R)	RPD
	mg/L	mg/L	R(%)			mg/L	R(%)		
Ag	<0.002	0.05	95	0.7	2.1	0.2	96	0.0	0.0
Al	0.185	0.05	98	8.8	1.7	0.2	105	3.0	3.1
AS	<0.008	0.05	108	1.4	3.7	0.2	101	0.7	2.0
B	0.023	0.1	98	0.2	0.0	0.4	98	0.2	0.5
Ba	0.042	0.05	102	1.6	2.2	0.2	98	0.4	0.8
Be	<0.0003	0.01	100	0.0	0.0	0.1	99	0.0	0.0
Ca	35.2		101	8.8	1.7	20.0	103	2.0	0.9
Cd	<0.001	0.01	105	3.5	9.5	0.1	98	0.0	0.0
co	<0.002	0.02	100	0.0	0.0	0.2	99	0.5	1.5
Cr	<0.004	0.01	110	0.0	0.0	0.1	102	0.0	0.0
Cu	<0.003	0.02	103	1.8	4.9	0.2	101	1.2	3.5
Fe	0.008	0.1	106	1.0	1.8	0.4	105	0.3	0.5
Hg	<0.007	0.05	103	0.7	1.9	0.2	100	0.4	1.0
K	1.98	5.0	109	1.4	2.3	20.0	107	0.7	1.7
Li	0.006	0.02	103	6.9	3.8	0.2	110	1.9	4.4
Mg	8.08	5.0	104	2.2	1.5	20.0	100	0.7	1.1
Mn	<0.001	0.01	100	0.0	0.0	0.1	99	0.0	0.0
Mo	<0.004	0.02	95	3.5	10.5	0.2	108	0.5	1.4
Na	10.3	5.0	99	3.0	2.0	20.0	106	1.0	1.6
Ni	<0.005	0.02	106	1.8	4.7	0.2	104	1.1	2.9
P	0.045	0.1	102	13.1	9.4	0.4	104	3.2	1.3
Pb	to.01	0.05	95	0.7	2.1	0.2	100	0.2	0.5
Sb	<0.008	0.05	99	0.7	2.0	0.2	102	0.7	2.0
Se	<0.02	0.1	87	1.1	3.5	0.4	99	0.8	2.3
SiO ₂	6.5	5.0	104	3.3	3.4	20.0	96	1.1	2.3
Sn	<0.007	0.05	103	2.1	5.8	0.2	101	1.8	5.0
Sr	0.181	0.1	102	3.3	2.1	0.4	105	0.8	1.0
Tl	<0.02	0.1	101	3.9	10.9	0.4	101	0.1	0.3
V	<0.003	0.05	101	0.7	2.0	0.2	99	0.2	0.5
Zn	0.005	0.05	101	3.7	9.0	0.2	98	0.9	2.5

S(R) Standard deviation of percent recovery.

RPD Relative percent difference between duplicate spike determinations.

< Sample concentration below established method detection limit.

• Spike concentration <1% of sample background concentration.

TABLE 5. PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES (Cont'd.)

POND WATER

ANALYTE	SAMPLE CONC mg/L	LOW SPIKE mg/L	AVERAGE RECOVERY			HIGH AVERAGE SPIKE RECOVERY			RPD
			R(%)	S(R)	RPD	mg/L	R(%)	S(R)	
Ag	to.002	0.05	92	0.0	0.0	0.2	94	0.0	0.0
Al	0.819	0.2	88	10.0	5.0	0.8	100	2.9	3.7
As	<0.008	0.05	102	0.0	0.0	0.2	98	1.4	4.1
B	0.034	0.1	111	8.9	6.9	0.4	103	2.0	0.0
Ba	0.029	0.05	96	0.9	0.0	0.2	97	0.3	0.5
Be	<0.0003	0.01	95	0.4	1.1	0.1	95	0.0	0.0
Ca	53.9	5	*	*	0.7	20.0	100	2.0	1.5
Cd	to.001	0.01	107	0.0	0.0	0.1	97	0.0	0.0
co	co.002	0.02	100	2.7	7.5	0.2	97	0.7	2.1
Cr	<0.004	0.01	105	3.5	9.5	0.1	103	1.1	2.9
Cu	0.003'	0.02	98	2.1	4.4	0.2	100	0.5	1.5
Fe	0.875	0.2	95	8.9	2.8	0.8	97	3.2	3.6
Hg	<0.007	0.05	97	3.5	10.3	0.2	98	0.0	0.0
K	2.48		106	0.3	0.1	20.0	103	0.2	0.4
Li	<0.001	0.0:	110	0.0	0.0	0.2	106	0.2	0.5
Mg	10.8		102	0.5	0.0	20.0	96	0.7	1.3
Mn	0.632	0.0:	*	*	0.2	0.1	97	2.3	0.3
Mo	<0.004	0.02	105	3.5	9.5	0.2	103	0.4	1.0
Na	17.8	5	103	1.3	0.4	20.0	94	0.3	0.0
Ni	<0.005	0.02	96	5.6	9.1	0.2	100	0.7	1.5
P	0.196	0.1	91	14.7	0.3	0.4	108	3.9	1.3
Pb	<0.01	0.05	96	2.6	7.8	0.2	100	0.7	2.0
Sb	<0.008	0.05	102	2.8	7.8	0.2	104	0.4	1.0
Se	<0.02	0.1	104	2.1	5.8	0.4	103	1.6	4.4
SiO ₂	7.83	5	151	1.6	1.3	20.0	117	0.4	0.6
Sn	to. 007	0.05	98	0.0	0.0	0.2	99	1.1	3.0
Sr	0.129	0.1	105	0.4	0.0	0.4	99	0.1	0.2
Tl	<0.02	0.1	103	1.1	2.9	0.4	97	1.3	3.9
V	0.003	0.05	94	0.4	0.0	0.2	98	0.1	0.0
Zn	0.006	0.05	97	1.6	1.8	0.2	94	0.4	0.0

S(R) Standard deviation of percent recovery.
 RPD Relative percent difference between duplicate spike determinations.
 < Sample concentration below established method detection limit.
 * Spike concentration <10% of sample background concentration.

TABLE 5. PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES (Cont'd.)

WELLWATER

ANALYTE	SAMPLE	LOW	AVERAGE	S(R)	RPO	HIGH	AVERAGE	S(R)	RPD
	CONC	SPIKE	RECOVERY			SPIKE	RECOVERY		
	mg/L	mg/L	R(%)			mg/L	R(%)		
Ag	<0.002	0.05	97	0.7	2.1	0.2	96	0.2	0.5
Al	0.036	0.05	107	7.6	10.1	0.2	101	1.1	0.8
As	<0.008	0.05	107	0.7	1.9	0.2	104	0.4	1.0
B	0.063	0.1	97	0.6	0.7	0.4	98	0.8	2.1
Ba	0.102	0.05	102	3.0	0.0	0.2	99	0.9	1.0
Be	<0.0003	0.01	100	0.0	0.0	0.1	100	0.0	0.0
Ca	93.8	5.0	*	*	2.1	20.0	100	4.1	0.1
Cd	0.002	0.01	90	0.0	0.0	0.1	96	0.0	0.0
Co	co.002	0.02	94	0.4	1.1	0.2	94	0.4	1.1
Cr	to. 004	0.01	100	7.1	20.0	0.1	100	0.4	1.0
CU	0.005	0.02	100	1.1	0.4	0.2	96	0.5	1.5
Fe	0.042	0.1	99	2.3	1.4	0.4	97	1.4	3.3
Hg	<0.007	0.05	94	2.8	8.5	0.2	93	1.2	3.8
K	6.21	5.0	96	3.4	3.6	20.0	101	1.2	2.3
Li	0.001	0.02	100	7.6	9.5	0.2	104	1.0	1.9
Mg	24.5	5.0	95	5.6	0.3	20.0	93	1.6	1.2
Mn	2.76	0.01	*	*	0.4	0.1	*	*	0.7
MO	<0.004	0.02	108	1.8	4.7	0.2	101	0.2	0.5
Na	35.0	5.0	101	11.4	0.8	20.0	100	3.1	1.5
Ni	<0.005	0.02	112	1.8	4.4	0.2	96	0.2	0.5
P	0.197	0.1	95	12.7	1.9	0.4	98	3.4	0.9
Pb	co.01	0.05	87	4.9	16.1	0.2	95	0.2	0.5
Sb	<0.008	0.05	98	2.8	8.2	0.2	99	1.4	4.0
Se	to.02	0.1	102	0.4	1.0	0.4	94	1.1	3.4
SiO ₂	13.1	5.0	93	4.8	2.8	20.0	99	0.8	0.0
Sn	co.007	0.05	98	2.8	8.2	0.2	94	0.2	0.5
Sr	0.274	0.1	94	5.7	2.7	0.4	95	1.7	2.2
Tl	<0.02	0.1	92	0.4	1.1	0.4	95	1.1	3.2
V	<0.003	0.05	98	0.0	0.0	0.2	99	0.4	1.0
Zn	0.538	0.05	*	•	0.7	0.2	99	2.5	1.1

S(R) Standard deviation of percent recovery.
 RPD Relative percent difference between duplicate spike determinations.
 < Sample concentration below established method detection limit.
 * Spike concentration <10% of sample background concentration.

TABLE 5. PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES (Cont'd.)

SEWAGE TREATMENT PRIMARY EFFLUENT

ANALYTE	SAMPLE CONC mg/L	LOU SPIKE mg/L	AVERAGE RECOVERY			HIGH SPIKE mg/L	AVERAGE RECOVERY		
			R(%)	S(R)	RPD		R(%)	S(R)	RPD
Ag	0.009	0.05	92	1.5	3.6	0.2	95	0.1	0.0
Al	1.19	0.05	•	•	0.9	0.2	113	12.4	2.1
As	<0.008	0.05	99	2.;	6.1	0.2	93	2.1	6.5
B	0.226	0.1	217	16.3	9.5	0.4	119	13.1	20.9
Ba	0.189	0.05	90	6.8	1.7	0.2	99	1.6	0.5
Be	<0.0003	0.01	94	0.4	1.1	0.1	100	0.4	1.0
Ca	87.9	5.0	•	•	0.6	20.0	101	3.7	0.0
Cd	0.009	0.01	89	2.6	2.3	0.1	97	0.4	1.0
co	0.016	0.02	95	3.1	0.0	0.2	93	0.4	0.5
Cr	0.128	0.01	•	•	1.5	0.1	97	2.4	2.7
c u	0.174	0.02	•	33.1	4.7	0.2	98	3.0	1.4
Fe	1.28	0.1	*	•	2.8	0.4	111	7.0	0.6
Hg	to.007	0.05	102	1.4	3.9	0.2	98	0.5	1.5
K	10.6	5.0	104	2.8	1.3	20.0	101	0.6	0.0
Li	0.011	0.02	103	8.5	3.2	0.2	105	0.8	0.5
Mg	22.7	5.0	100	4.4	0.0	20.0	92	1.1	0.2
Mn	0.199	0.01	*	•	2.0	0.1	104	1.9	0.3
Mo	0.125	0.02	110	21.;	6.8	0.2	102	1.3	0.9
Na	236	5.0	*	•	0.0	20.0	*	*	0.4
Ni	0.087	0.02	12.;	10.7	4.5	0.2	98	0.8	1.1
P	4.71	0.1	•	•	2.6	0.4	*	•	1.4
Pb	0.015	0.05	9.;	3.;	5.0	0.2	96	2.3	2.9
Sb	<0.008	0.05	97	0.7	2.1	0.2	103	1.1	2.9
Se	to.02	0.1	108	3.9	10.0	0.4	101	2.6	7.2
SiO ₂	16.7	5.0	124	4.0	0.9	20.0	108	1.1	0.8
Sn	0.016	0.05	90	3.8	0.0	0.2	95	1.0	0.0
Sr	0.515	0.1	103	6.4	0.5	0.4	96	1.6	0.2
Tl	<0.02	0.1	105	0.4	1.0	0.4	95	0.0	0.0
V	0.003	0.05	93	0.9	2.0	0.2	97	0.2	0.5
Zn	0.160	0.05	98	3.3	1.9	0.2	101	1.0	1.4

S(R) Standard deviation of percent recovery.
 RPD Relative percent difference between duplicate spike determinations.
 < Sample concentration below established method detection limit.
 * Spike concentration <10% of sample background concentration.

TABLE 5. PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES(Cont'd.)

INDUSTRIAL EFFLUENT

ANALYTE	SAMPLE CONC mg/L	LOW SPIKE mg/L	AVERAGE RECOVERY			HIGH AVERAGE SPIKE RECOVERY			
			R(%)	S(R)	RPD	mg/L	R(%)	S(R)	RPD
Ag	to.003	0.05	88	0.0	0.0	0.2	84	0.9	3.0
Al	0.054	0.05	88	11.7	12.2	0.2	90	3.9	8.1
As	to.02	0.05	82	2.8	9.8	0.2	88	0.5	1.7
B	0.17	0.1	162	17.6	13.9	0.4	92	4.7	9.3
Ba	0.083	0.05	86	8.2	1.6	0.2	85	2.3	2.4
Be	<0.0006	0.01	94	0.4	1.1	0.1	82	1.4	4.9
Ca	500	5.0			2.8	20.0	•	•	2.3
Cd	0.008	0.01	8;	4.;	6.1	0.1	82	1.4	4.4
co	<0.004	0.02	93	1.8	5.4	0.2	83	0.4	1.2
Cr	0.165	0.01	•	*	4.5	0.1	106	6.6	5.6
CU	0.095	0.02	93	23.3	0.9	0.2	95	2.7	2.8
Fe	0.315	0.1	88	16.4	1.0	0.4	99	6.5	8.0
Hg	<0.01	0.05	87	0.7	2.3	0.2	86	0.4	1.2
K	2.87	5.0	101	3.4	2.4	20.0	100	0.8	0.4
Li	0.069	0.02	103	24.7	5.6	0.2	104	2.5	2.2
Mg	6.84	5.0	a7	3.1	0.0	20.0	87	0.9	1.2
Mn	0.141	0.01	•	*	1.2	0.1	a9	6.6	4.8
Mo	1.27	0.02	*	*	0.0	0.2	100	15.0	2.7
Na	1500	5.0	*	•	2.7	20.0	•	•	2.0
Ni	0.014	0.02	•	4.4	3.0	0.2	8;	0.5	1.1
P	0.326	0.1	105	16.0	4.7	0.4	97	3.9	1.4
Pb	0.251	0.05	80	19.9	1.4	0.2	88	5.0	0.9
Sb	2.81	0.05	*	*	0.4	0.2	*	•	2.0
Se	0.021	0.1	106	2.6	3.2	0.4	105	1.9	4.6
SiO ₂	6.83	5.0	99	6.8	1.7	20.0	100	2.2	3.0
Sn	<0.01	0.05	a7	0.7	2.3	0.2	86	0.4	1.2
Sr	6.54	0.1	*	*	2.0	0.4	*	•	2.7
Tl	to.03	0.1	87	1.8	5.8	0.4	84	1.;	3.6
V	<0.005	0.05	90	1.4	4.4	0.2,	84	1.1	3.6
Zn	0.024	0.05	a9	6.0	4.4	0.2	91	3.5	8.9

S(R) Standard deviation of percent recovery.

RPD Relative percent difference between duplicate spike determinations.

< Sample concentration below established method detection limit.

* Spike concentration <10% of sample background concentration.

TABLE 6. PRECISION AND RECOVERY DATA IN SOLID MATRICES

EPA HAZARDOUS SOIL #884

ANALYTE	SAMPLE CONC mg/kg	LOW ⁺ SPIKE mg/kg	AVERAGE RECOVERY			HIGH+ SPIKE mg/kg	AVERAGE RECOVERY		
			R(%)	S(R)	RPD		R(%)	S(R)	RPD
Ag	1.1	20	98	0.7	1.0	100	96	0.2'	0.6
Al	5080	20	*		7.2	100	*	*	5.4
AS	5.7	20	95	5.;	10.6	100	96	1.4	3.6
B	20.4	100	93	2.7	5.3	400	100	2.1	5.5
Ba	111	20	98	71.4	22.2	100	97	10.0	1.0
Be	0.66	20	97	0.7	2.0	100	99	0.1	0.2
Ca	85200			-	-				
Cd		20	93	0.7	1.0	100	94	0.2	0.4
co	5.;	20	96	3.5	7.7	100	93	0.8	2.1
Cr	79.7	20	87	28.8	16.5	100	104	1.3	1.1
CU	113,	20	110	16.2	4.4	100	104	4.0	4.2
Fe	16500								
Hg	<1.4	10	92	2.;	7.;	40	98	0.0	0.0
K	621	500	121	1.3	-	2000	107	0.9	1.8
Li	6.7	10	113	3.5	4.4	40	106	0.6	0.6
Mg	24400	500	•	*	8.4	2000		*	10.1
Mn	343	20	*	•		100	9;	11.0	1.6
MO	5.3	20	88	5.3	8.5	100	91	1.4	4.1
Na	195	500	102	2.2	13.2	2000	100	1.5	3.7
Ni	15.6	20	100	1.8	0.0	100	94	1.5	3.6
P	595	500	106	13.4	8.0	2000	103	3.2	2.7
Pb	145	20	88	3.9	17.9	100	108	15.6	17.4
Sb	6.1	20	83	14.7	52.4 7.5	100	81	1.9	5.9
Se	<5	20	79			100	99	0.7	2.1
Sn	16.6	20	91	34.6	5.8	80	112	8.7	2.8
Sr	102	100	84	9.6		400	94	2.5	4.6
Tl	<4	20	92	4.8	10.8 14.6	100	91	1.5	4.6
V	16.7	20	104		5.4	100	99	0.8	1.7
Zn	131	20	103	3.;	7.3	100	104	7.2	6.4

S(R) Standard deviation of percent recovery.

RPD Relative percent difference between duplicate spike determinations.

< Sample concentration below established method detection limit.

• Spike concentration <10% of sample background concentration.

Not spiked.

+ Equivalent

TABLE 6. PRECISION AND RECOVERY DATA IN SOLID MATRICES (Cont.)

EPA ELECTROPLATING SLUDGE #286

ANALYTE	SAMPLE CONC mg/kg	LOW ⁺ SPIKE mg/kg	AVERAGE RECOVERY			HIGH+ AVERAGE RECOVERY			
			R(%)	S(R)	RPD	SPIKE mg/kg	R(%)	S R)	RPD
Ag	6	20	96	0.2	0.4	100	93.2	0.1	0.4
Al	4980	20	•	*	4.4	100	*	*	5.6
AS	32	20	1.3	0.8	100	97	0.7	1.6	
B	210	100	113	2.0	1.6	400	98	1.9	3.5
Ba	39.8	20	0	6.8	0.3	100	0	1.6	5.7
Be	0.32	20	96	0.2	0.5	100	100.68	0.7	2.0
Ca	48500	-	-	-	-	-	-	-	-
Cd	108	20	98	2.5	0.8	100	96	0.5	0.5
co	5.9	20	93	2.9	5.7	100	93	0.6	1.5
Cr	7580	20	•	•	0.7	100	*	•	1.3
cu	806	20	☒	*	1.5	100	94	8.3	0.7
Fe	31100	-	-	-	-	-	-	-	-
Hg	6.1	10	90	4.0	4.0	40	97	-	4.3
K	2390	500	75	4.0	4.0	2000	94	1.7	3.8
Li	9.1	10	101	2.8	0.5	40	106	1.6	3.1
Mg	1950	500	110	2.0	0.8	2000	108	2.3	3.2
Mn	262	20	*	*	1.8	100	91	1.2	0.9
Mo	13.2	20	9;	2.1	2.9	100	92	0.3	0.0
Na	73400	500	*	•	1.7	2000	*	*	1.4
Ni	456	20	*	•	0.4	100	88	2.7	0.9
P	9610	500	4	☒	2.9	2000	114	7.4	3.4
Pb	1420	20	*	*	2.1	100	*	*	1.3
Sb	<2	20	76	0.9	3.3	100	7;	2.8	10.7
Se	6.3	20	86	16.6	16.6	100	103	1.6	2.7
Sn	24.0	20	87	4.0	2.7	100	92	0.7	0.0
Sr	145	100	90	8.1	8.1	400	93	2.4	4.6
Tl	16	20	89	4.6	5.3	100	92	0.8	0.9
V	21.7	20	95	1.2	1.0	100	96	0.4	0.9
Zn	12500	20	*	*	0.8	100	•	*	0.8

S(R) - Standard deviation of percent recovery.
 RPD Relative percent difference between duplicate spike determinations.
 < Sample concentration below established method detection limit.
 • Spike concentration <10% of sample background concentration.
 Not spiked.
 + Equivalent

TABLE 6. PRECISION AND RECOVERY DATA IN SOLID MATRICES (Cont.)

NBS 1645 RIVER SEDIMENT

ANALYTE	SAMPLE CONC mg/kg	LOW ⁺ SPIKE mg/kg	AVERAGE RECOVERY			HIGH ⁺ AVERAGE SPIKE RECOVERY			RPD
			R(%)	S(R)	RPD	mg/kg	R(%)	S(R)	
Ag	1.6	20	92	*	1.0	100	96	0.3	0.9
Al	5160	20	*	14.4	8.4	100	*	•	2.4
AS	62.8	20	89			100	97	2.9	5.0
B	31.9	100	116	7.1	9.7	400	100	0.6	1.5
Ba	54.8	20	95	6.1	12.8	100	100	1.2	1.3
Be	0.72	20	101	0.4	1.0	100	103	1.4	3.9
Ca	28000						-	-	
Cd	9.7	20		1.1		100	101	0.7	1.8
CO	9.4	20	100 98	3.8	0.0	100	98	0.9	1.8
Cr	28500	20	•	•	0.4	100	*	•	0.7
CU	109	20	115	8.5	0.0	100	102	1.8	1.0
Fe	84800	10					-	-	
Hg	3.1	500	95	4.3	7.;	40	96	0.7	1.0
K	452		98		2.0	2000	106	1.4	2.3
Li	3.7	10	101	2.0	0.7	40	108	1.3	3.0
Mg	6360	500	•	•	1.8	2000	93	2.7	1.0
Mn	728	20	*	*	3.5	100	98	12.4	2.2
Mo	17.9	20	97	12.5	18.5	100	97	0.6	0.0
Na	1020	100	100	2.6	0.0	2000	100	1.1	1.7
Ni	36.2	20	94	5.9	4.0	100	100	1.1	1.5
P	553	500	102	1.4	0.9	2000	100	0.8	1.6
Pb	707	20		•	0.8	100	103	5.9	0.4
Sb	22.8		86	1.0	27.1 0.0	100	88		0.9
Se	6.7	20	103			100	98	0.3 1	7.6
Sn	309	20	•	*	1.0	100	101	7.9	2.7
Sr	782	100	91	12.3	3.0	400	96	3.3	2.6
Tl	<4		90	0.0	0.0	100	95	1.3	4.0
V	20.1	20	89	5.4	5.8	100	98	0.7	0.0
Zn	1640	20	•	*	1.8	100	*	*	1.1

S(R) Standard deviation of percent recovery.
 RPD Relative percent difference between duplicate spike determinations.
 < Sample concentration below established method detection limit.
 * Spike concentration <10% of sample background concentration.
 Not spiked.
 + Equivalent

TABLE 7. DRINKING WATER 4X PRECONCENTRATION PRECISION AND RECOVERY DATA (1)

ANALYTE	SAMPLE CONC mg/L	LOW SPIKE mg/L	AVERAGE RECOVERY R(%)	RSD(%)	HIGH SPIKE mg/L	AVERAGE RECOVERY R(%)	RSD(%)
Ag	to. 001	0.025	95	0.5	0.12	95	4.6
Al	0.102	0.05	95	1.6	0.2	104	5.2
AS	<0.004	0.02	101	10.9	0.08	98	3.6
B	0.022	0.02	100	1.2	0.08	96	5.1
Ba	0.037	0.5	101	0.7	2.0	98	4.0
Be	<0.0002	0.001	100	0.0	0.004	100	5.0
Ca	32.6			1.9			
Cd	~0.0006	0.005	100	2.4	0.02	95	3.8
Cr	to. 002	0.05	99	1.0	0.2	96	3.9
cu	to. 001	0.5	99	0.7	2.0	96	3.3
Fe	to. 02	0.1	114	5.4	0.4	102	5.0
Hg	<0.003	0.01	84	7.1	0.04	94	6.6
K	2.09			2.2			
Mg	7.49			2.0		-	
Mn	0.002	0.005	100	1.4	0.02	110	4.8
mo	co. 003	0.01	103	5.3	0.04	102	4.6
Na	8.21			1.9			
Ni	<0.002	0.01	112	1.9	0.04	103	5.6
Pb	<0.005	0.01	105	11.4	0.04	108	4.4
Sb	to. 004	0.01	106	7.5	0.04	99	9.1
Se	to. 01	0.05	107	8.8	0.2	96	5-6
Sr	0.160	0.1	94	0.3	0.4	102	4.7
Tl	<0.008	0.02	98	8.6	0.08	100	4.4
V	to. 002	0.01	100	3.1	0.04	100	5.7
Zn	0.003	0.02	101	1.8	0.08	95	5.0

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ANNEX E-13

i-13-1

ARSENIC

Method 206.2 (Atomic Absorption, **furnace** technique)

STORET NO. Total 01002
Dissolved 01000
suspended 01001

Optimum Concentration Range: 5-100 **ug/l**

Detection Limit: 1 **ug/l**

Preparation of Standard Solution

1. Stock solution: Dissolve 1.320 g of arsenic trioxide, **As₂O₃** (analytical reagent grade) in 100 ml of deionized distilled water containing 4 g **NaOH**. Acidify the solution with 20 ml **conc. HNO₃** and dilute to 1 liter. 1 ml = 1 mg **As** (1000 **mg/l**).
2. Nickel **Nitrate** Solution, 5%: Dissolve 24.780 g of ACS reagent grade **Ni(NO₃)₂·6H₂O** in deionized **distilled** water and make up to 100 ml.
3. Nickel Nitrate Solution, 1%: Dilute 20 ml of the 5% nickel nitrate to 100 ml with deionized distilled water.
4. Working Arsenic Solution: Prepare **dilutions** of the stock solution to be used as calibration standards at the time of analysis. Withdraw **appropriate aliquots** of the stock solution, add 1 ml of **conc. HNO₃**, 2 ml of 30% **H₂O₂** and 2 ml of the 5% nickel **nitrate** solution. Dilute to 100 ml with deionized **distilled** water.

Sample **Preservation**

1. For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.

Sample **Preparation**

1. Transfer 100 **ml** of well-mixed sample to a 250 ml **Griffin** beaker, add 2 ml of 30% **H₂O₂** and sufficient **conc. HNO₃** to result in an acid concentration of 1% (v/v). Heat for 1 hour at 95 **°C** or until the volume is slightly less than 50 ml.
2. Cool and bring back to 50 ml with deionized distilled water.
3. **Pipet 5 ml of this digested solution into a 10-ml volumetric flask, add 1 ml of the 1% nickel nitrate solution and dilute to 10 ml with deionized distilled water.** The sample is now ready for injection into the furnace.

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NOTE: If **solubilization** or digestion is not required, adjust the **HNO₃** concentration of the sample to 1% (v/v) and add 2 ml of 30 % **H₂O₂** and 2 ml of 5 % nickel **nitrate** to each 100 ml of sample. The volume of the calibration standard should be adjusted with deionized distilled water to match the volume change of the sample.

Instrument **Parameters (General)**

1. **Drying Time** and **Temp**: 30 **sec**-125 °C.
2. **Ashing Time** and **Temp**: 30 **sec**-1100 °C.
3. **Atomizing Time** and **Temp**: 10 **sec**-2700 °C.
4. **Purge Gas Atmosphere**: Argon
5. **Wavelength**: 193.7 **nm**
6. Other operating parameters should be set **as** specified by the particular instrument manufacturer.

Analysis **Procedure**

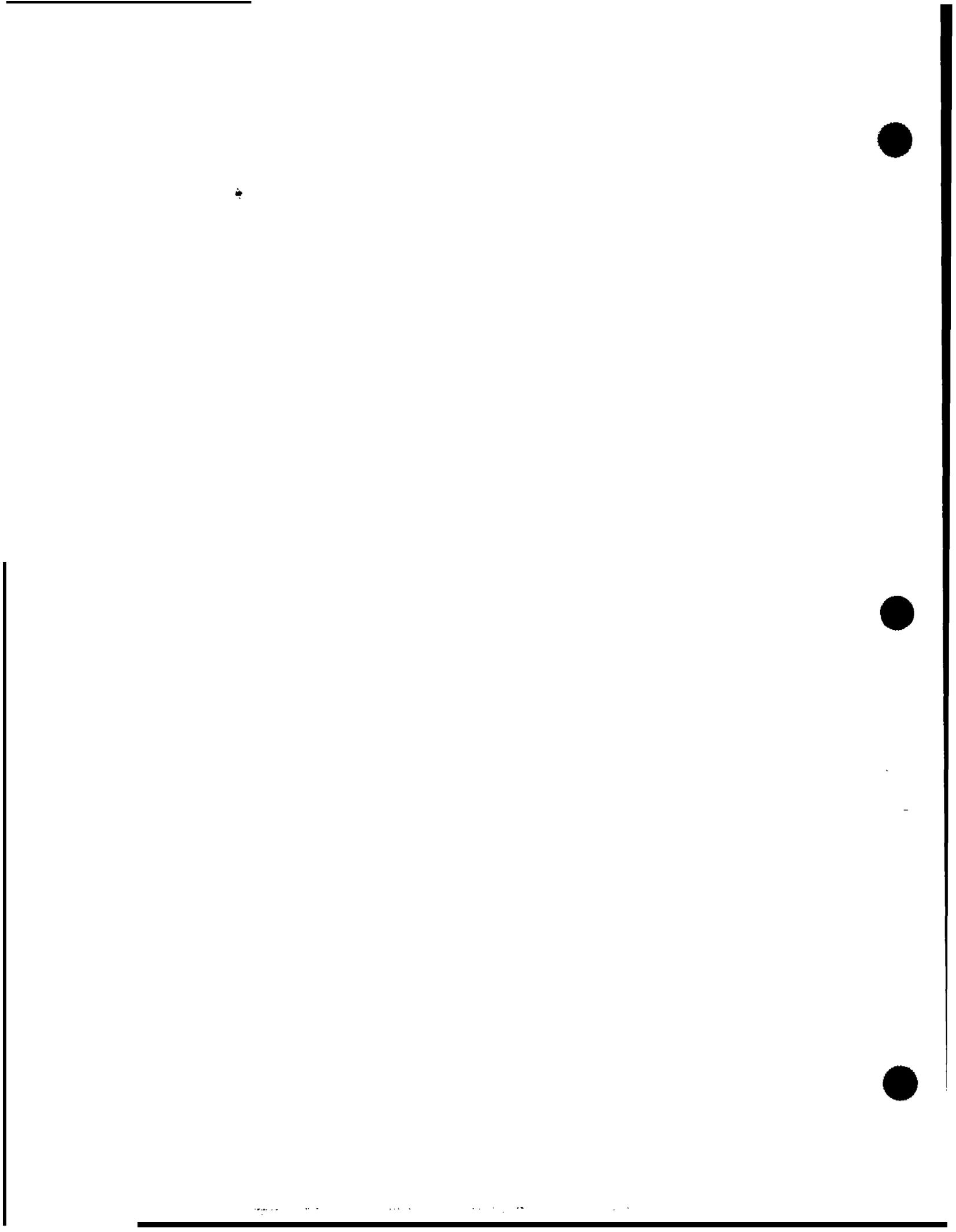
1. For the analysis procedure and the calculation, **see** "Furnace Procedure" part 9.3 of the Atomic Absorption Methods section of this manual.

Notes

1. The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, **based** on the use of a 20 **ul** injection, purge gas interrupt and non-pyrolytic graphite. **Smaller** size **furnace** devices or those employing faster rates of atomization **can** be **operated** using lower **atomization** temperatures for shorter time periods than the above recommended **settings**.
2. **The** use of background correction is recommended.
3. For every sample matrix analyzed, **verification** is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
4. If method of standard addition is required, follow the procedure given earlier in part 8.5 of the Atomic Absorption Methods section of this manual.
5. For quality control requirements and optional recommendations for use in drinking water analyses, see part 10 of the Atomic Absorption **Methods** section of this manual.
6. Data to be entered into STORET must **be** reported as **ug/l**.

Precision and **Accuracy**

1. In a single laboratory (**EMSL**), using a mixed **industrial-domestic** waste effluent containing 15 **ug/l** and spiked with **concentrations** of 2, 10 **and** 25 **ug/l**, recoveries of **85 %**, **90 %** and 88 % were obtained, **respectively**. The relative standard deviation at these **concentration** levels were **±8.8 %**, **±8.2 %**, **±5.4 %**, **and** **±8.7 %**, respectively.
2. In a single laboratory (**EMSL**), using Cincinnati, Ohio tap water spiked at concentrations of 20, 50 and 100 **ug As/l**, the standard deviations were **f0.7**, **± 1.1**, **and** **f1.6**, respectively. Recoveries at these levels were **105 %**, **106 %**, and **101 %**, respectively.



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ANNEX E-14

E-14-1

CADMIUM

Method 213.2 (Atomic Absorption, furnace technique)

STORET NO. 01027

Dissolved 0 1025

Suspended 01026

Optimum Concentration Range: **0.5-10 ug/l**

Detection Limit: **0.1 ug/l**

Preparation of Standard Solution

1. Stock solution: Prepare as described under "**direct** aspiration method."
2. Ammonium Phosphate solution (**40%**): Dissolve **40 grams** of ammonium phosphate, **(NH₄)₂HPO₄** (**analytical** reagent **grade**) in & ionized distilled water and dilute to 100 ml.
3. Prepare dilutions of the stock cadmium solution to be used as calibration standards at the time of **analysis**. To each 100 ml of standard and **sample** alike add 2.0 ml of the ammonium phosphate solution. The calibration standards should **be** prepared to contain 0.5% (v/v) **HNO₃**.

Sample **Preservation**

1. For sample handling and **preservation**, see part 4.1 of the Atomic Absorption Methods section of this manual.

Sample Preparation

1. Prepare as **described** under "**direct** aspiration method." Sample solutions for analysis should contain 0.5% (v/v) **HNO₃**.

Instrument **Parameters** (General)

1. Drying **Time** and Temp: 30 **sec**-125 °C.
2. **Ashing** Time and Temp: 30 **sec**-500 °C.
3. Atomizing **Time** and Temp: 10 **sec**-1900 °C.
4. Purge Gas Atmosphere: Argon
5. Wavelength: 228.8 nm
6. Other operating **parameters** should **be** set **as specified** by the particular instrument manufacturer. .

Analysis Procedure

1. For the analysis procedure and the calculation, see "**Furnace** Procedure" part 9.3 of the Atomic Absorption-Methods section of this manual.

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Notes

1. The above concentration values and instrument conditions are for a **Perkin-Elmer HGA2100**, based on the use of a **20** ul injection, continuous flow purge gas and non-pyrolytic graphite. **Smaller** size furnace devices or those employing faster rates of atomization can be **operated** using lower atomization temperatures for shorter time periods **than** the above recommended settings.
2. The **use** of background **correction** is recommend&.
3. Contamination **from** the work **area** is **critical** in cadmium analysis. Use of **pipet** tips which are free of cadmium is of particular importance. (See part 5.2.9 of the Atomic Absorption Methods section of this manual.)
4. For every sample **matrix** analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
5. If method of standard addition is required, follow the procedure given earlier in **part** 8.5 of the Atomic Absorption Methods section of this manual.
6. For quality control requirements and optional recommendations for use in drinking water analyses, see part 10 of the Atomic Absorption Methods section of this manual.
7. Data to be entered into **STORET** must **be** reported as **ug/l**.

Precision and Accuracy

1. In a single laboratory (**EMSL**), using Cincinnati, Ohio **tap** water spiked at concentrations of 2.5, 5.0 and 10.0 ug Cd/l, the standard deviations were 0.10, 0.16, and **±0.33, respectively**. Recoveries at these levels were **96%, 99%, and 98%, respectively**.



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ANNEX E-15

E-15-1

MERCURY

Method 245.1 (Manual Cold Vapor Technique)

STORET NO. Total 71900

Dissolved 71890

Suspended 71895

1. scope and Application

1.1 This method is applicable to drinking, surface, and saline waters, domestic and **industrial** wastes.

1.2 In addition to inorganic forms of mercury, organic **mercurials** may also be present. These **organo-mercury** compounds will not respond to the cold vapor atomic absorption technique unless they are **first** broken down and converted to mercuric ions. Potassium **permanganate** oxidizes many of these compounds, but recent studies have shown that a **number** of organic **mercurials, including phenyl** mercuric acetate **and methyl** mercuric chloride, are only **partially** oxidized by this reagent. Potassium **persulfate** has **been** found to give approximately 100% recovery when used **as** the oxidant with **these** compounds. Therefore, a **persulfate** oxidation step following the addition of the **permanganate** has been included to **insure** that **organo-mercury** compounds, **if** present, will be oxidized to the mercuric ion **before** measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a **natural** system. For distilled water the **heat** step is not necessary.

1.3 The range of the method may be varied through instrument and/or recorder **expansion**. Using a 100 ml sample, a **detection** limit of 0.2 ug **Hg/l** can be achieved; concentrations below this level should be reported as **< 0.2** (see Appendix 11 . 2).

2. Summary of Method

2.1 The flameless **AA** procedure is a physical method based on the absorption of radiation at 253.7 **nm** by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a **closed** system. The mercury vapor passes through a cell positioned **in** the light path of an atomic absorption **spectrophotometer**. Absorbance (**peak** height) is measured as a function of mercury concentration and recorded in the usual manner.

3. Sample Handling and Preservation

3.1 Until more conclusive data **are** obtained, samples should be preserved by acidification with nitric acid to a **pH** of 2 or lower immediately at the time of collection. If only dissolved mercury is to be determined, the **sample** should be filtered through an all glass **apparatus** before the acid is added. For total mercury the **filtration** is omitted.

4. Interference

- 4.1 Possible interference from sulfide is eliminated by the addition of potassium **permanganate**. Concentrations as high as 20 **mg/l** of sulfide as sodium sulfide do not interfere with the recovery of **added** inorganic mercury from distilled water.
- 4.2 **Copper** has also been reported to interfere; however, **copper** concentrations as high as 10 **mg/l** had no effect on recovery of mercury **from** spiked samples.
- 4.3 Sea waters, brines and **industrial emuents** high in chlorides require additional **permanganate** (as much as 25 ml). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation of 253 **nm**. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using **an** excess of hydroxylamine sulfate reagent (25 ml). In addition, the **dead** air space in the BOD bottle must be purged before the addition of **stannous** sulfate. Both inorganic and organic mercury spikes have **been** quantitatively recovered **from sea** water using this technique.
- 4.4 Interference **from certain volatile organic materials** which will absorb at this wavelength is also possible. A **preliminary** run without reagents should determine if this type of **interference** is present (see Appendix 11.1).

5. Apparatus

- 5.1 Atomic Absorption **Spectrophotometer**: (See Note 1) Any atomic absorption **unit** having an open sample presentation **area** in which to mount the absorption cell is suitable. Instrument settings recommended by the **panicular** manufacturer should be followed.
Note 1: Instruments designed **specifically** for the **measurement** of mercury using the cold **vapor** technique are commercially available **and** may **be** substituted for the atomic absorption **spectrophotometer**.
- 5.2 Mercury Hollow Cathode **Lamp**: Westinghouse WL-22847, argon **filled**, or equivalent.
- 5.3 Recorder: Any multi-range variable **speed** recorder that is compatible with the *W* detection system is suitable.
- 5.4 Absorption Cell: Standard spectrophotometer cells 10 cm long, having quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 1" O.D. X 4-1/2". The ends **are** ground perpendicular to the longitudinal axis and **quartz** windows (1" *diameter* X 1/16" thickness) are cemented in place. The cell is **strapped** to a burner for support and aligned in the light **beam** by use of two 2" by 2" **cards**. One inch diameter holes are cut in the middle of each **card**; the **cards** are then placed over each end of the cell. The cell **is then** positioned and adjusted vertically and horizontally to give the maximum **transmittance**.
- 5.5 **Air Pump**: Any **peristaltic** pump capable of delivering 1 liter of air per minute may **be** used. A **Masterflex** pump with electronic **speed** control has **been** found to be **satisfactory**.

- 5.6 Flowmeter: Capable of measuring an airflow of 1 liter per minute.
- 5.7 Aeration Tubing: A straight glass frit having a coarse porosity. Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return.
- 5.8 Drying Tube: 6" X 3/4" diameter tube containing 20 g of **magnesium perchlorate** (see Note 2). **The apparatus is assembled as shown in Figure 1.**
NOTE 2: In place of the magnesium **perchlorate** drying tube, a small **reading** lamp with 60W bulb may **be used to prevent condensation** of moisture inside the cell. The lamp is positioned to shine on the absorption cell **maintaining** the air temperature in the cell about 10 °C above ambient.
6. Reagents
- 6.1 Sulfuric Acid, **Conc.:** Reagent grade.
6.1.1 Sulfuric acid, 0.5 N: Dilute 14.0 ml of **conc.** sulfuric acid to 1 .0 liter.
- 6.2 **Nitric** Acid, **Conc:** Reagent grade of low mercury content (See Note 3).
NOTE3: If a high, reagent blank is obtained, it may be necessary to distill the nitric acid.
- 6.3 **Stannous** Sulfate: Add 25 g **stannous** sulfate to 250 ml of 0.5 N sulfuric acid. This mixture is a suspension and should **be** stirred continuously during use. (**Stannous chloride may be used in place of stannous sulfate.**)
- 6.4 Sodium Chloride-Hydroxylamine Sulfate Solution: Dissolve 12 g of sodium chloride and 12 g of **hydroxylamine** sulfate **in** distilled water and dilute to 100 ml. (**Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.**)
- 6.5 Potassium **Permanganate:** 5 A solution, **w/v.** Dissolve **5 g of** potassium **permanganate in** 100 ml of **distilled water.**
- 6.6 Potassium **Persulfate:** **5 %** solution, **w/v.** Dissolve **5 g** of potassium persulfate in 100 ml of distilled water.
- 6.7 Stock Mercury Solution: Dissolve 0.1354 g of mercuric chloride in 75 ml of **distilled** water. Add 10 ml of **conc.** nitric acid and adjust the volume to 100.0 ml.
1 ml = 1 mg Hg.
- 6.8 Working Mercury Solution: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 ug per ml. **This working standard and the dilutions of the stock mercury solution should be prepared fresh daily.** Acidity of the working **standard** should be maintained at 0.15 % nitric acid. This acid should be added to the flask as needed before the addition of the **aliquot.**

6. Reagents

6.1 Sulfuric Acid, **Conc.:** Reagent grade.

6.1.1 Sulfuric acid, 0.5 N: Dilute 14.0 ml of **conc.** sulfuric acid to 1 .0 liter.

6.2 Nitric Acid, **Conc: Reagent** grade of low mercury content (See Note 3).

NOTE 3: If a **high reagent** blank is **obtained**, it may be **necessary** to distill the nitric acid.

6.3 **Stannous Sulfate:** Add 25 g **stannous sulfate** to 250 ml of 0.5 N sulfuric acid- This mixture is a suspension and should be stirred **continuously** during **use.** (**Stannous chloride** may be used in **place** of **stannous sulfate.**)

6.4 **Sodium Chloride-Hydroxylamine Sulfate Solution:** Dissolve 12 g of sodium chloride and 12 g of **hydroxylamine** sulfate in distilled water and dilute to 100 ml. (Hydroxylamine hydrochloride may be **used** in place of **hydroxylamine** sulfate.)

6.5 Potassium **Permanganate:** 5% solution. w/v. Dissolve 5 g of potassium **permanganate** in 100 ml of distilled water.

6.6 Potassium **Persulfate:** 5% solution, w/v. Dissolve 5 g of potassium **persulfate** in 100 ml of **distilled water.**

6.7 Stock Mercury Solution: **Dissolve** 0.1354 g of mercuric chloride in 75 ml of distilled water. Add 10 ml of **conc.** nitric acid and adjust the volume to 100.0 ml. 1 ml = 1 mg Hg.

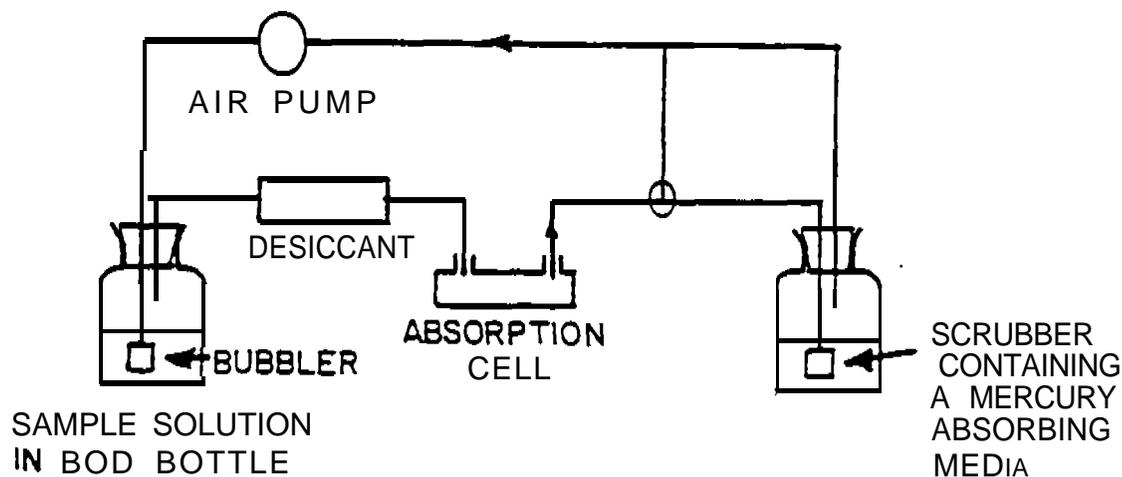


FIGURE 1. APPARATUS FOR FLAMELESS MERCURY DETERMINATION

7. Calibration

7.1 Transfer 0, 0.5, 1.0, 2.0, 5.0 and 10.0 ml **aliquots** of the working mercury solution containing 0 to 1 **.0 ug** of mercury to a series of 300 ml BOD bottles. Add enough distilled water to each bottle to make a total volume of 100 ml. Mix thoroughly and add 5 ml of **conc.** sulfuric acid (6.1) and 2.5 ml of **conc.** nitric acid (6.2) to each bottle. Add 15 ml of **KMnO₄** (6.5) solution to each **bottle and** allow to **stand** at least 15 minutes. Add **8** ml of potassium **persulfate** (6.6) to **each** bottle and **heat** for 2 hours in a water bath maintained at 95 °C. **Cool** and add 6 ml of **sodium chloride-hydroxylamine** sulfate solution (6.4) to reduce the excess **permanganate**. When the solution has been **decolorized** wait 30 seconds, add 5 ml of the **stannous** sulfate solution (6.3) and immediately attach the bottle to the aeration apparatus forming a closed system. At this point the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously **been** adjusted to a rate of 1 liter per minute, is allowed to **run** continuously (See Note 4). The absorbance will **increase** and reach maximum within 30 seconds. **As soon** as the recorder pen levels off, approximately 1 minute, open the bypass valve **and** continue the **aeration** until the absorbance **returns** to its minimum value (see Note 5). Close the bypass valve, remove the stopper and frit from the BOD **bottle** and **continue** the aeration. Proceed with the standards and construct a standard **curve** by plotting **peak** height versus micrograms of mercury.

NOTE 4: An **open** system where the mercury vapor is passed through the absorption cell only once may be used instead of the closed system.

NOTE 5: **Because** of the toxic nature of **mercury** vapor **precaution** must be taken to avoid its **inhalation**. Therefore, a bypass has been **included** in the system to either vent the mercury **vapor** into an exhaust hood or pass the vapor through some absorbing media, such as:

- a) equal volumes of 0.1 M **KMnO₄** and 10% **H₂SO₄**
- b) 0.25% iodine in a 3% **KI** solution

A specially **treated** charcoal that will adsorb mercury vapor is **also** available from Bamebey and Cheney, E. 8th Ave. and N. Cassidy St., Columbus, Ohio 43219, Cat. #580-13 or #580-22.

8. Procedure

8.1 Transfer 100 ml, or an **aliquot** diluted to 100 ml, containing not more than 1.0 **ug** of mercury, to a 300 ml BOD bottle. Add 5 ml of sulfuric acid (6.1) and 2.5 ml of **conc.** nitric acid (6.2) mixing after **each** addition. Add 15 ml of potassium **permanganate** solution (6.5) to each sample bottle. For sewage **samples** additional **permanganate** may **be** required. Shake and add additional portions of potassium **permanganate** solution, if necessary, until the **purple** color persists for at least 15 minutes. Add 15 ml of **potassium persulfate** (6.6) to each bottle and heat for 2 hours in a water bath at 95 °C. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate (6.4) to reduce the **excess permanganate**. After a delay of at least 30 seconds add 5 ml of stannous sulfate (6.3) and immediately attach the bottle to the aeration **apparatus**. Continue as described under Calibration.

9. Calculation

9.1 Determine the **peak** height of the u&own from the char& and read the mercury value from the standard curve.

9.2 Calculate the mercury concentration in the sample by the formula:

$$\text{ug Hg/l} = (\text{aliquot}) (\text{volume of aliquot in ml})$$

9.3 **Report** mercury **concentrations** as follows: Below 0.2 **ug/l**, **<0.2**; **between** 1 and 10 **ug/l**, **one** decimal; above 10 **ug/l**, whole numbers.

10. Precision and Accuracy

10.1 In a single **laboratory (EMSL)**, using an Ohio River composite sample with a background mercury concentration of 0.35 **ug/l**, spiked with concentrations of 1.0, 3.0 and 4.0 **ug/l**, the standard deviations were f0.14, ± 0.10 and ± 0.08 , respectively. Standard deviation at the 0.35 level was ± 0.16 . Percent recoveries at the three levels were 89, 87, **and 87%**, **respectively**.

10.2 In a joint **EPA/ASTM interlaboratory** study of the cold vapor technique for total **mercury** in water, increments of organic and inorganic mercury were added to natural **waters**. Reconvenes were determined by **difference**. A statistical summary of **this** study follows:

ml of sodium chloride-hydroxylamine **sulfate** (6.4) to reduce the **excess permanganate**. After a delay of at least 30 **seconds** add S ml of **stannous sulfate** (6.3) and immediately attach **the bottle** to the aeration apparatus. Continue **as described** under Calibration.

9. calculation

9.1 **Determine** the **peak height of the unknown from the chart** and **read the mercury value from the standard curve**.

9.2 Calculate the mercury concentration in **the sample** by **the formula**:

$$\text{ug Hg/l} = \left(\frac{\text{ug Hg in aliquot}}{\text{volume of aliquot in ml}} \right) \left(\frac{1.000}{\text{volume of aliquot in ml}} \right)$$

9.3 Report mercury concentrations as follows: Below 0.2 **ug/l**, **<0.2**; between 1 and 10 **ug/l**, one **decimal**; above 10 **ug/l**, whole numbers.

10. Precision and Accuracy

10.1 In a single laboratory (EMSL), using **an Ohio River composite sample** with a background mercury **concentration** of 0.35 **ug/l**, **spiked** with concentrations of 1.0, 3.0 and 4.0 **ug/l**, the standard deviations were **±0.14, ±0.10 and ±0.08, respectively**. Standard **deviation** at **the 0.35 level** was 0.16. **Percent recoveries** at the three levels were **89, 87, and 87%, respectively**.

10.2 In a joint EPA/ASTM interlaboratory study of the cold vapor technique for total mercury in water, increments of organic and inorganic mercury were added to natural **waters**. **Recoveries** were determined by **difference**. A statistical summary of this study follows:

<u>Number of Labs</u>	<u>True Values ug/liter</u>	<u>Mean value ug/liter</u>	<u>Standard Deviation ug/liter</u>	<u>Accuracy as % Bias</u>
76	0.21	0.349	0.276	66
80	0.27	0.414	0.179	53
82	0.51	0.674	0.541	32
77	0.60	0.709	0.390	18
82	3.4	3.41	1.49	0.34
79	4.1	3.81	1.12	-7.1
79	8.8	8.77	3.69	-0.4
78	9.6	9.10	3.57	-5.2

11. Appendix

11.1 **While** the possibility of absorption from certain organic **substances** actually being **present** in the sample **does** exist, **EMSL** has not **encountered such samples**. This is **mentioned** only to caution the analyst of the possibility. A simple correction that may be used is as follows: If an interference has been found to be present (4.4), the sample should be analyzed both by 'using the regular procedure and again under oxidizing conditions **only**,

11. Appendix

- 11.1 While the possibility of absorption from certain organic substances actually being present in the sample does exist, **EMSL** has not encountered such samples. This is mentioned only to caution the analyst of the possibility. A simple correction that may be **used** is as follows: **If** an interference has **been** found to be present **(4.4)**, the sample should be analyzed **both** by using the regular procedure and again under oxidizing conditions only, that **is** without the reducing reagents. The true mercury value **can** then **be obtained** by **subtracting** the two values.
- 11.2 If additional sensitivity is **required**, a 200 ml sample with recorder expansion may be used provided the instrument **does** not produce undue noise. Using a Coleman **MAS-50** with a drying tube of magnesium **perchlorate** and a variable recorder, 2 mv was set to read full scale. With 'these conditions, and distilled water solutions of mercuric chloride at concentrations of 0.15, 0.10, 0.05 and 0.025 **ug/l** the standard deviations were **±0.027**, **±0.006**, **±0.01** and **±0.004**. Percent recoveries at these levels were 107, 83, 84 and **96%**, respectively.
- 11.3 Directions for the disposal of **mercury-containing** wastes are given in **ASTM Standards**, Part 31, "Water," p 349, Method D3223 (1976).

Bibliography

1. Kopp, J.F., **Longbottom, M.C.** and **Lobring, L.B.**, "Cold Vapor Method for **Determining** Mercury," AWWA, vol **64**, p 20, **January** 1972.
2. Annual Book of **ASTM Standards**, **Part 31**, "Water," Standard **D3223-73**, p 343 (1976).
3. **Standard Methods** for the **Examination** of Water and Wastewater, 14th Edition, p 156 (1975).



Final Rpt, Kuwait Oil **Fire HRA** No. 39-26-L192-91, 5 May - 3 Dec 91

ANNEX E-16

E-16-1

LEAD

Method 239.2 (Atomic Absorption, furnace technique)

STORET NO. Total 01051
Dissolved 01049
suspended 01050

Optimum Concentration Range: S-100 ug/l
Detection Limit: 1 ug/l

Preparation of Standard Solution

1. Stock solution: Prepare as described under "direct aspiration method."
2. **Lanthanum Nitrate** Solution: Dissolve 58.64 g of ACS reagent grade La_2O_3 in 100 ml **conc. HNO_3** and dilute to 1000 ml with deionized distilled water. 1 ml = 50 mg La.
3. Working **Lead** Solution: Prepare dilutions of the stock lead solution to be used as calibration standards at the time of analysis. Each calibration standard should contain 0.5% (v/v) **HNO_3** . To each 100 ml of **diluted standard** add 10 ml of the **lanthanum nitrate solution**.

Sample Preservation

1. For **sample** handling and preservation, **see** part 4.1 of the Atomic Absorption Methods section of **this** manual.

Sample Preparation

1. **Prepare** as **described** under "direct aspiration method." Sample solutions for analysis should contain 0.5% (v/v) **HNO_3** .
2. To each 100 ml of prepared sample solution add 10 ml of the lanthanum nitrate solution.

Instrument Parameters (General)

1. Drying Time and Temp: 30 **sec-125 °C**.
2. **Ashing Time** and Temp: 30 **sec-500 °C**.
3. Atomizing Time and Temp: 10 **sec-2700 °C**.
4. Purge Gas Atmosphere: **Argon**
5. Wavelength: 283.3 **nm**
6. **Other operating** parameters should **be** set as specified by the particular instrument **manufacturer**.

Analysis Procedure

1. For the analysis procedure in the calculation see "Furnace Procedure," **part** 9.3 of the Atomic Absorption Methods section of this manual.

Notes

1. The above concentration values and instrument conditions are for a **Perkin-Elmer HGA-2100**, based on the use of a **20 ul** injection, continuous flow purge gas **and** non-pyrolytic graphite. Smaller size furnace devices or those employing faster rates of atomization **can** be operated using lower atomization temperatures for shorter time **periods** than the above recommended settings.
2. The use of background correction is recommended.
3. **Greater** sensitivity **can** be achieved using the **217.0 nm line**, but the optimum concentration range is reduced. The use of a **lead** electrodeless discharge lamp at this lower wavelength has been found to **be** advantageous. Also a lower atomization temperature (**2400 °C**) may be **preferred**.
4. To suppress sulfate interference (up to 1500 ppm) lanthanum is added as the nitrate to both samples and calibration standards. (Atomic Absorption Newsletter Vol. 15, No. 3, p 71, May-June 1976.)
5. Since glassware **contamination** is a severe problem in lead analysis, all glassware should be **cleaned** immediately prior to use, and once cleaned, should not be open to the atmosphere except when necessary.
6. For every sample matrix analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
7. For quality control requirements and optional recommendations for use **in** drinking water analyses, see part 10 of the Atomic Absorption **Methods** section of this manual.
8. If method of standard addition is required, follow the procedure given earlier in part 8.5 of the Atomic Absorption Methods section of this manual.
9. Data **to be** entered into **STORET** must be reported **as ug/l**.

Precision and Accuracy

1. In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at concentrations of 25, 50, and **100 ug Pb/l**, the standard deviations were ± 1.3 , ± 1.6 , and ± 3.7 , respectively. Recoveries at these levels were **88%**, **92%**, and **95%** respectively.



Final Rpt, Kuwait Oil Fire HRA No. 39-26-L192-91, 5 May - 3 Dec 91

ANNEX E-17

E-17-1

Procedure - **RJ Lee** Group **Hexavalent** Chromium Kit

Following these instructions and the contents of the kit prior to use to become familiar with the process will help eliminate sampling delays in the field

Step I Collection of Sand Samples

1. **Use the plastic spoon and the wooden stick to collect sand for Steps II and V.**
2. **Scoop** up sand and level the surface with the edge of the stick (**see** Figure 1).

Step II Extracting the Sand

1. Close the stopcock (see Figure 2) at **the** bottom of Syringe 1 and place it in the gray wooden rack with Tube A under it.
2. Pour approximately 20 ml of &ionized water from **Tube B** into this syringe. Use the markings on the tube to estimate this volume.
3. Place the funnel into Syringe 1 as shown in figure 2. Pour one **spoonful** of sand from Step I through the **funnel** into the solution. Allow it to stand for approximately 30 **minutes**.
4. After the 30 minute wait, remove the funnel, **open** the stopcock, and **allow** the solution to **drain into Tube A until the flow stops**.
5. **Pour approximately** 15 ml more of the solution from Tube **B into Syringe I, again** using the markings on the tube to estimate the volume, and allow it to drain until the flow stops. (It should take between 1 and 10 minutes for the solution to drain through the system.)
6. **Repeat** step 5 once more with the remaining solution flow from Tube B.
7. After no more solution flows from Syringe 1, attach the **Extra** Plunger found in the kit and squeeze out additional water. The expected total volume for this step is between 40 and **50** ml.

Note: If the solution **does** not drain from the sand, it may be **drawn** out using the syringe. To do this, attach Syringe 2 to the **bottom** of Syringe 1 via the **female/female** luer **connection**. Add any additional solution obtained in this **manner to Tube A**.

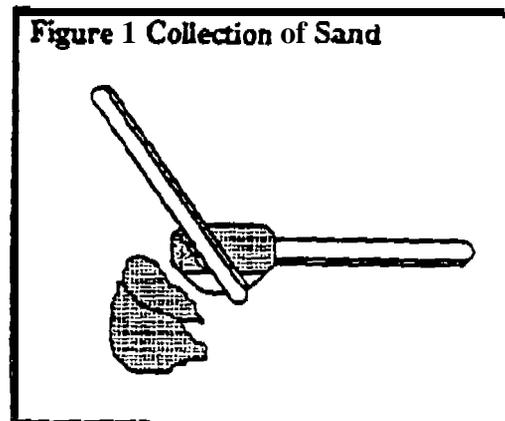
Procedure - RJ LeeGroup Hexavalent Chromium Kit

11/18/93

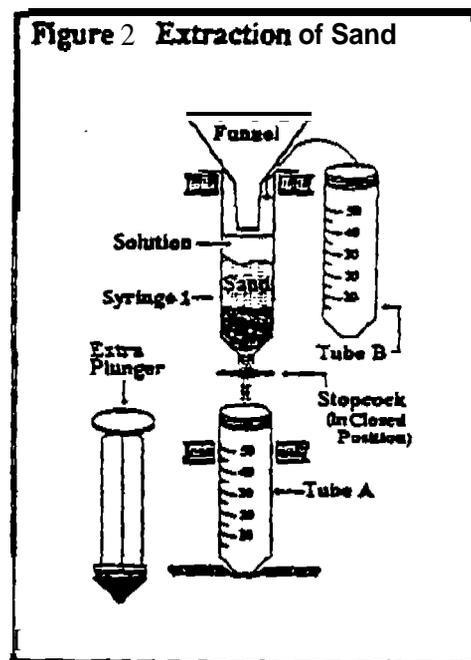
Examining these instructions and the contents of the kit prior to use to become familiar with the process will help eliminate sampling delays in the field.

Step I Collection of Sand Samples

1. Use the plastic spoon and the wooden stick to collect sand for Steps II and V.
2. Scoop up sand and level the surface with the edge of the tick (see Figure 1).

**Step II Extracting the Sand**

1. Close the stopcock (see Figure 2) at the bottom of Syringe 1 and place it in the gray wooden rack with Tube A under it.
2. Pour approximately 20 ml of deionized water from Tube B into this syringe. Use the markings on the tube to estimate this volume.
3. Place the funnel into Syringe 1 as shown in Figure 2. Pour one spoonful of sand from Step I through the funnel into the solution. Allow it to stand for approximately 30 minutes.
4. After the 30 minute wait, remove the funnel, open the stopcock, and allow the solution to drain into Tube A until the flow stops.
5. Pour approximately 15 ml more of the solution from Tube B into Syringe 1, again using the markings on the tube to estimate the volume, and allow it to drain until the flow stops. (It should take between 1 and 10 minutes for the solution to drain through the system.)
6. Repeat step 5 once more with the remaining solution from Tube B.
7. After no more solution flows from Syringe 1, attach the Extra Plunger found in the kit and squeeze out additional water. The expected total volume for this step is between 40 and 50 ml.



Note: If the solution does not drain from the sand, it may be drawn out using the syringe. To do this, attach Syringe 2 to the bottom of Syringe 1 via the female/female luer connection. Add any additional solution obtained in this manner to Tube A.

Step III Filtering **the** Soil Extract

1. Place Syringe 2 in the rack and remove the plunger. Connect the **filter** (disk shaped unit) to the bottom as shown in Figure 3 by twisting approximately **1/3** turns (Do Not Over tighten).
2. Pour the solution **from** Tube A into Syringe 2.
3. Replace the plunger and filter **the** solution into **Tube B** by applying pressure.

Step IV Trapping the Chromium-VI

1. Remove and discard the filter unit from Syringe 2. **Remove** the plunger and place the syringe in the rack.
2. Uncap the top and bottom of the SPE Cartridge and place the caps in one of the zipper lock bags for **later reuse**.
3. Attach the **cartridge** to the syringe and pour the **filtered** solution from Tube B into the syringe (see Figure 4).
4. Hold the **cartridge** over Tube **B** and push the solution and a small amount of air through the SPE Cartridge. You will need to press hard to accomplish this. This could take Several minutes to accomplish.
5. **Read the volume in Tube B and record this on the label on the small Zipper Lock bag.**
6. Remove the cartridge, then the plunger from the syringe, then replace the cartridge.
7. Pour the wash solution from **Tube C** into the syringe and push in through the **cartridge**.
8. Remove the cartridge from the syringe, draw up 50 ml of air, then replace the cartridge and push the air through it.
9. Cap the top and bottom of the **cartridge, return** it to the bag in which it was received, and place this into the large zipper lock bag.

Step III Filtering the Soil Extract

1. Place Syringe 2 in the rack and remove the plunger. Connect the filter (disk shaped unit) to the bottom as shown in Figure 3 by twisting approximately 1/3 turns (Do Not Over Tighten).
2. Pour the solution from Tube A into Syringe 2.
3. Replace the plunger and filter the solution into Tube B by applying pressure.

Step IV Trapping the Chromium-VI

1. Remove and discard the filter unit from Syringe 2. Remove the plunger and place the syringe in the rack.
2. Uncap the top and bottom of the SPE Cartridge and place the caps in one of the zipper lock bags for later reuse.
3. Attach the cartridge to the syringe and pour the filtered solution from Tube B into the syringe (see Figure 4).
4. Hold the cartridge over Tube B and push the solution and a small amount of air through the SPE Cartridge. You will need to press hard to accomplish this. This could take several minutes to accomplish.
5. Read the volume in Tube B and record this on the label on the small Zipper Lock bag.
6. Remove the cartridge, then the plunger from the syringe, then replace the cartridge.
7. Pour the wash solution from Tube C into the syringe and push it through the cartridge.
8. Remove the cartridge from the syringe, draw up 50 ml of air, then replace the cartridge and push the air through it.
9. Cap the top and bottom of the cartridge, return it to the bag in which it was received, and place this into the large zipper lock bag.

Figure 3 Filtering Soil Extract

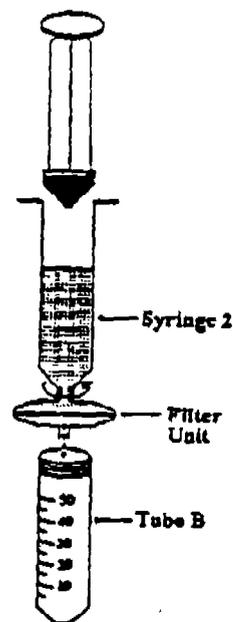
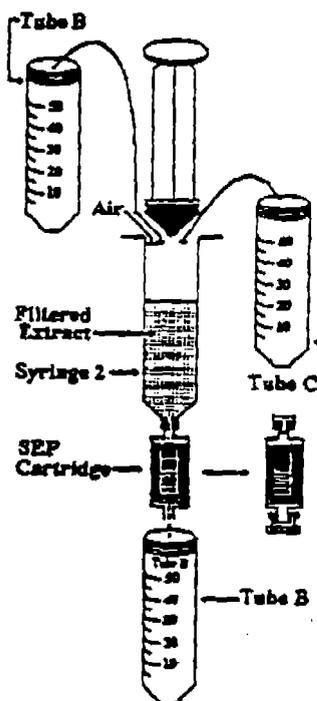


Figure 4 Trapping Chromium-VI



Step V Collection of Sand Sample

1. Collect at least four or five spoonfuls of sand in the small Zipper Lock bag provided (see Figure 5a).
2. Roll the small **Zipper** Lock bag, processing out as much air as possible, and zip the bag closed (see Figure 5b).
3. Place the small Zipper Lock **bag** containing the sand **into** the large Zipper Lock bag with the SPE **Cartridge** (see Figure 6).
4. Roll the large Zipper Lock bag to press out as much air as possible and zip it closed.

Note: Kits are identified with Bar Codes.

Do Not Mix **Components** from **Different Kits!**

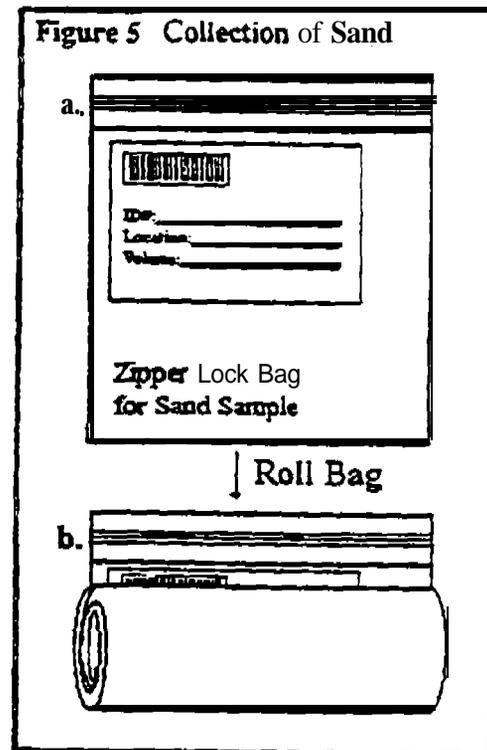
When the process is completed, only keep the small Zipper Lock Bag (containing the sand sample) and the bagged, capped SEP Cartridge. These should be enclosed in the Large Zipper Lock Bag (see Figure 6).

Other **supplies from** the kit may be **discarded**.

Step V Collection of Sand Sample

1. Collect at least four or five spoonfuls of sand in the small Zipper Lock bag provided (see Figure 5a).
2. Roll the small Zipper Lock bag, pressing out as much air as possible, and zip the bag closed (see Figure 5b).
3. Place the small Zipper Lock bag containing the sand into the large Zipper Lock bag with the SPE Cartridge (see Figure 6).
4. Roll the large Zipper Lock bag to press out as much air as possible and zip it closed.

Figure 5 Collection of Sand

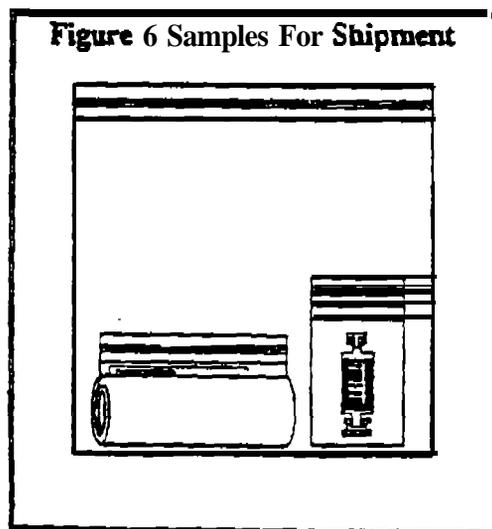


Note: Kits are identified with Bar Codes.
Do Not Mix Components from Different Kits!

When the process is completed, only keep the small Zipper Lock Bag (containing the sand sample) and the wedge - These should be enclosed in the Large Zipper Lock Bag (see Figure 6).

Other supplies from the kit may be discarded

Figure 6 Samples For Shipment



Kit Components

1. spoon
2. Stick
3. Small **Labeled** Zipper **Lock** Bag
4. syringe I (**Column**)
5. **Extra** Plunger
6. **Funnel**
7. **Tube A**
8. **Tube B**
9. Tube c
10. Syringe 2
11. Bagged Filter
- i2. Bagged, **Capped** SPE Cartridge

Final Rpt, Kuwait Oil Fire HRA No. 39-26-L192-91, 5 May - 3 Dec 91

ANNEX K-18

E-18-1

REPORT OF ANALYSIS

U.S. ARMY ENVIRONMENTAL HYGIENE AGENCY
Aberdeen Proving Ground, MD 21010

27 NOV 93

Organic Environmental Chemistry Division
Special Analysis Branch

Installation: Kuwait HRA
Project Number: 39-22-L192-94
Project Officer: Jack Heller
Analysis Requested: TO1 Method
Matrix: Air (custom Tenax tubes)

LCSD #: R3724 - R3800
Number of Samples: 77
Dates Sampled: 01 - 09 Nov 93
Date Received: 12 Nov 93
Dates Analyzed: 12 - 21 Nov 93

AQAD #	FIELD #	DATA FILE #
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R3726	TCTASF0600	>JA19A
R3727	TCTASF0605	>JA20A
R3728	TCTASF0602	>JA21A
R3729	TCTASF0606	>JA22A
R3730	TCTFB0603	>JA23A
R3731	T3ASF0809	>JA24A
R3732	T3ASF0803	>JA25A
R3733	T3ASF0808	>JA26A
R3734	T3ASF0802	>JA29A
R3735	TCTASF0501	>JA30A
R3737	TCTMSF0901	>JA32A
R3738	TCTASF0509	>JA33A
R3739	TCTASF0505	>JA34A
R3740	TCTASF0503	>JA35A
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R3742	T3MSF0902	>JA37A
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R3746	T3ASF0801	>JA44A
R3747	T3ASF0806	>JA45A
R3748	T3FB0800	>JA46A

Analyst: William J. Fenter
Reviewer: Robert V. J. [Signature]
Released by: Robert V. J. [Signature]
Date Released: 1 DEC 93

REPORT OF ANALYSIS

U.S. ARMY ENVIRONMENTAL HYGIENE AGENCY
Aberdeen Proving Ground, MD 21010

Organic Environmental Chemistry Division
Special Analysis Branch

Installation: Kuwait **HRA**
Project Number: **39-22-L192-94**
Project Officer: **Jack Heller**
Analysis Requested: **TO1 Method**
Matrix: Air (custom **Tenax** tubes)

LCSD #: **R3724 - R3800**
Number of Samples: **77**
Dates Sampled: **01 - 09 Nov 93**
Date Received: **12 Nov 93**
Dates Analyzed: **12 - 21 Nov 93**

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R3798	TCTTB0906	>KA08A
R3799	T - 0 6 3 5	>KA11A
R3800	T3TB0905	>KA12A

Analyst: *[Signature]*

Reviewer: *[Signature]*

Released by: *[Signature]*

Date Released: 1 DEC 93

REPORT OF ANALYSIS

U.S. ARMY ENVIRONMENTAL HYGIENE AGENCY
Aberdeen Proving Ground, MD 21010

Organic Environmental Chemistry Division
Special Analysis Branch

Installation: Kuwait HRA
Project Number: 39-22-L192-94
Project Officer: Jack Heller
Analysis Requested: TO1 Method
Matrix: Air (custom Tenax tubes)

LCSD #: R3724 - R3800
Number of Samples: 77
Dates Sampled: 01 - 09 Nov 93
Date Received: 12 Nov 93
Dates Analyzed: 12 - 21 Nov 93

AQAD #	FIELD #	DATA FILE #
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R3775	TCTASF0906	>JC79A
R3776	TCTMSF1703	>JC80A
R3777	TCTMSB1201	>JC81A
R3778	TCTASF1106	>JC82A
R3779	TCTMSF1701	>JC83A
R3780	TCTASF0909	>JC84A
R3781	TCTASB1209	>JA85A
R3782	TCTASF0908	>JA86A
R3783	TCTASF0905	>JB89A
R3784	T3ASF1507	>JB90A
R3785	T3ASB1600	>JB91A
R3786	T3ASF1509	>JB92A
R3787	T3MSF1702	>JB93A
R3788	T3ASF1303	>JB94A
R3789	T3ASB1605	>JA95A
R3790	T3ASF1302	>JA96A
R3791	T3FB1501	>KA01A
R3792	T3ASF1301	>KA02A
R3793	T3ASB1602	>KA03A
R3794	T3ASF1505	>KA04A
R3795	T3ASF1504	>KA05A
R3796	T3ASB1409	>KA06A
R3797	TCTFB1209	>KA07A
R3798	TCTTB0906	>KA08A
R3799	TCTTB0635	>KA11A
R3800	T3TB0905	>KA12A

Analyst: [Signature]

Reviewer: [Signature]

Released by: [Signature]

Date Released: DEC 93

23 November 1993

RESULTS OF T01 ANALYSIS
39-22-L192-94

Results Summary:

One hundred and sixteen T01 tubes were received by the USAEHA/OECD/SAB laboratory on 12 November 1993. These tubes were sorted by collection date and tube type (primary or backup). Seventy-seven of these tubes (all of the primary tubes, all of the blanks, and 15 of the backup tubes) were subsequently analyzed on 12 - 21 November 1993 using EPA Method T01: a thermal desorption / gas chromatography / mass spectrometric method (TD/GC/MS). All samples were analyzed within the recommended 14-day holding time.

This package contains the analytical results for these 77 samples.

All individual sample reports (Form 1's) contain a total volatile organic compound summation (which was determined by summing all the peaks in the chromatogram, subtracting out the ISTD and SURR areas, and then quantitating the area left relative to toluene-d8) and individual T01 analyte quantitative results. A summary sheet containing the total volatile organic results for all samples is also included.

Sample results were reported-in:

Total nanograms (NG) per tube for all samples and micrograms per meter cubed (uG/M3) for all samples with air volume data.

Tentatively Identified Compounds:

Two non-target compounds were detected at very high levels in the Camp Thunder Rock samples. Methyl methacrylate was detected in all CT primary samples at concentrations well above the target analyte range (approximately 1000 - 5000 ng relative to the ISTD). High concentrations of trichloroethylene were also detected in CT primary samples that were collected from 11/06/93 until the end of the survey. The Khobar Towers samples did not show the same high concentrations of these non-target compounds, although they were present at lower levels in some samples (<100 ng). All field and travel blanks showed no detections of these two compounds.

Quality Control

The T01 compounds included in the analytical standards and the internal quality control were:

- Compound
- =====
- Benzene
- n-Heptane
- Toluene
- Ethylbenzene
- meta/para Xylene
- ortho Xylene
- n-propyl Benzene

The limit of quantitation for all the T01 compounds listed above was 10 ng per T01 tube.

A five point calibration curve was run prior to sample analysis using the following standard concentration levels: 10 ng, 25 ng, 100 ng, 250 ng, and 500 ng. A 50 ng check standard was run daily to assure that analyte responses had not changed. A laboratory blank (a T01 tube that was filled with glass wool - not Tenax) was analyzed daily after the check standard to assure that the TD/GC/MS system was free of analyte contamination.

Toluene-d8 (50 ng) was spiked on all tubes immediately prior to analysis to correct for any changes in instrument response. Benzene-d6 and Ethylbenzene-d10 were spiked on all primary T01 tubes prior to sampling to demonstrate the retentive properties of the sorbenc.

Internal standard and surrogate recoveries are included in this data package (Forms 2 & 8). All internal standard and surrogate recoveries were within normal QC limits for this data package, except one internal standard recovery (R3770) and two surrogate recoveries (R3735 and R3758 - BZE-d6). Surrogate recoveries are given separately for the backup tubes. These tubes were not spiked with the surrogate mixture prior to sampling, so any recovery of either of the two surrogate compounds indicates analyte breakthrough to the backup tube.

All matrix spikes included in this package were spiked prior to sampling with an independent volatile standard mixture. Percentage matrix spike recoveries (relative to actual samples) are included in this package.

~~Sample Field Number Codes~~

~~ONS 26000000 15000000 00000000 00000000 00000000 00000000 00000000 00000000~~
Field Number = T####

Where:

T - Tenax air sample

- Site: 3 -Khobar Towers, CT-Camp Thunder Rock

- Type of sample
AS - Actual sample
FB - Field blank
MS - Matrix spike
TB - Travel blank

- F or B: Front (primary) or backup tube

- Tenax tube number, denotes conditioning batch number
(12 tubes per batch).

Analysis Data Sheet Qualifier Sheet Codes

=====

E - indicates reported value exceeds the upper limit of the
quantitation curve.

J - indicates reported value is an estimate; it is less than
the lower limit of the quantitation curve.

U - indicates compound was analyzed for, but not detected.

SUMMARY OF RESULTS
TOTAL VOLATILE ORGANICS IN AIR
KUWAIT HRA

LCSD NUMBER	FIELD NUMBER	VOLUME COLL'D (L)	DATE (TIME) COLLECTED	UNITS OF CONCENTRATION	
				NG/SAMPLE	UG/M3
R3724	TCTASF0506	19.1L	11/01/93 (1535)	2649.	138.7
R3725	TCTASF0507	19.1L	11/01/93 (1535)	2369.	124.0
R2726	TCTASF0600	20.0L	11/02/93 (0027)	2208.	110.4
X3727	TCTASF0605	17.6L	11/02/93 (0050)	2999.	170.4
R3728	TCTASF0602	17.2L	11/02/93 (1227)	3966.	230.6
R3729	TCTASF0606	18.2L	11/02/93 (1256)	1750.	96.2
R3730	TCTFB0603	NOVOL	11/02/93 (1500)	171.	N/A
R3731	T3ASF0809	13.3L	11/01/93 (1700)	718.	54.0
R3732	T3ASF0803	13.7L	11/01/93 (1700)	800.	58.4
R3733	T3ASF0808	14.3L	11/02/93 (0500)	1343.	93.9
R3734	T3ASF0802	14.4L	11/02/93 (0500)	1647.	114.4
R3735	TCTASF0501	17.1L	11/03/93 (0145)	2606.	152.4
R3737	TCTMSF0901	17.4L	11/03/93 (0145)	4146.	238.3
R3738	TCTASF0509	18.9L	11/04/93 (0200)	1325.	70.1
R3739	TCTASF0505	15.2L	11/04/93 (0200)	1688.	111.1
R3740	TCTASF0503	17.3L	11/04/93 (1400)	1906.	110.2
R3741	TCTASF0508	18.8L	11/04/93 (1400)	1148.	61.1
R3742	T3MSF0902	13.8L	11/03/93 (0500)	3026.	219.3
R3743	T3MSB0400	13.8L	11/03/93 (0500)	109.	7.9
R3744	T3ASF0807	14.3L	11/03/93 (0500)	441.	30.8
R3745	T3ASF0804	14.6L	11/03/93 (0500)	743.	50.9
R3736	T3ASB0405	14.6L	11/03/93 (0500)	79.	5.4
R3746	T3ASF0801	14.7L	11/03/93 (1700)	599.	40.7
R3747	T3ASF0806	14.7L	11/03/93 (1700)	612.	41.6
R13748	T3FB0800	NOVOL	11/03/93 (1700)	202.	N/A
R13749	T3ASF0705	13.7L	11/04/93 (0500)	628.	45.8
R3750	T3ASF0805	12.4L	11/04/93 (0500)	498.	40.2
R3751	T3ASB0306	12.4L	11/04/93 (0500)	147.	11.8
R3752	T3FB0707	NOVOL	11/04/93 (1100)	88.	N/A
R3753	TCTASF0608	14.1L	11/05/93 (1430)	1732.	172.4
R3754	TCTASF0607	16.3L	11/06/93 (0039)	1441.	88.4
R3755	TCTASB0207	16.3L	11/06/93 (0039)	152.	9.3
R3756	TCTASF0604	14.4L	11/06/93 (0039)	1746.	121.2
R3757	TCTMSF0904	16.6L	11/06/93 (1305)	14186.	854.6
R3758	TCTASF0601	16.7L	11/06/93 (1305)	2054.	123.
R3759	TCTASB1202	16.7L	11/06/93 (1305)	218.	13.0
R3760	T3ASF0703	14.8L	11/05/93 (0500)	421.	29.5
R3761	T3MSF0903	15.3L	11/05/93 (0500)	3396.	222.1
R3762	T3ASF0704	15.3L	11/05/93 (0500)	569.	37.1
R3763	T3ASB0304	15.3L	11/05/93 (0500)	13s.	9.8
R3764	T3ASF0702	6.99L	11/05/93 (1700)	415.	63.3
R3765	T3ASF0701	14.1L	11/05/93 (1700)	608.	43.1

SUMMARY OF RESULTS
TOTAL VOLATILE ORGANICS IN AIR FOR
KUWAIT HRA

LCSD NUMBER	FIELD NUMBER	VOLUME COLL'D (L)	DATE (TIME) COLLECTED	UNITS OF CONCENTRATION	
				NG/SAMPLE	UG/M3
R3766	TSASB0307	14.1L	11/05/93 (1700)	60.	4.3
R3767	T3ASF1508	14.9L	11/06/93 (0500)	802.	53.8
R3768	T3ASF1500	14.3L	11/06/93 (0500)	786.	54.9
R3769	T3ASF1506	14.3L	11/07/93 (0500)	644.	45.0
R3770	T3ASB1608	14.31;	11/07/93 (0500)	40.	2.8
R3771	T3ASF1502	15.2L	11/07/93 (1700)	864.	56.4
R3772	T3ASF1503	9.18L	11/07/93 (1700)	772.	84.1
R3773	TCTASF0907	16.7L	11/07/93 (0005)	4604.	275.7
R3774	TCTASB1206	18.6L	11/07/93 (0005)	98.	5.3
R3775	TCTASF0906	18.6L	11/07/93 (0005)	3095.	166.4
R3776	TCTMSF1703	16.3L	11/07/93 (1204)	5981.	366.9
R3777	TCTMSB1201	16.3L	11/07/93 (1204)	135.	a.3
R3778	TCTASF1106	16.41;	11/07/93 (12041)	1174.	71.6
R3779	TCTMSF1701	17.8L	11/07/93 (2326)	7114.	399.6
R3780	TCTASF0909	16.6L	11/07/93 (2326)	2997.	180.6
R3781	TCTASB1209	16.6L	11/07/93 (2326)	404.	24.3
R3782	TCTASF0908	15.4L	11/08/93 (1105)	2721.	176.7
R3783	TCTASF0905	16.1L	11/08/93 (1105)	3568.	221.6
R3784	T3ASF1507	13.3L	11/08/93 (0500)	669.	50.3
R3785	T3ASB1600	13.3L	11/08/93 (0500)	21.	1.6
R3786	T3ASF1509	14.3L	11/08/93 (0500)	792.	55.4
R3787	T3MSF1702	14.4L	11/09/93 (0655)	6026.	418.4
R3788	T3ASF1303	14.6L	11/09/93 (0655)	823.	56.4
R3789	T3ASB1605	14.6L	11/09/93 (0655)	62.	4.2
R3790	T3ASF1302	14.8L	11/09/93 (0655)	893.	60.3
R3791	T3FB1501	NOVOL	11/09/93 (0655)	193.	N/A
R3792	T3ASF1301	10.2L	11/09/93 (1845)	1635.	160.3
R3793	T3ASB1602	10.2L	11/09/93 (1845)	98.	9.6
R3794	T3ASF1505	5.47L	11/09/93 (1845)	898.	164.1
R3795	T3ASF1504	14.3L	11/07/93 (0500)	945.	66.1
R3796	T3ASB1409	14.3L	11/07/93 (0500)	94.	6.6
R3797	TCTFB1209	NOVOL	11/08/93 (????)	12.	N/A
R3798	TCTTB0906	NOVOL	TRVBLK1STSHIP	22.	N/A
R3799	TCTTB0635	NOVOL	TRVBLK2NDSHIP	12.	N/A
R3800	T3TB0905	NOVOL	TRVBLK1STSHIP	41.	N/A
SYSTEMBL	50 NG ISTD			24.	N/A
LAB BLAN	50 NG ISTD			11.	N/A
LABBLANK	50 NG ISTD			47.	N/A
LAB BLAN	50 NG ISTD			91.	N/A
LABBLANK	50 NG ISTD			87.	N/A
LAB BLAN	50 NG ISTD			131.	N/A
LAB BLAN	50 NG ISTD			8.	N/A
LAB BLAN	50 NG ISTD			19.	N/A
LABBLANK	50 NG ISTD			43.	N/A

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

SYSTEMBL

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: SO NG ISTD

Air Volume (L): NOVOL

Lab File ID: >JA16A

Date Received: 11/12/93

Date Analyzed: 11/12/93

Date Collected: N/A

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	24. NG	N/A uG/M3
-------------------------	--------	-----------

2: INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	D D D D D D D
n-Heptane	10.	N/A	
Toluene	10.	N/A	
Ethylbenzene	10.	N/A	
meta/para Xylene	10.	N/A	
ortho Xylene	10.	N/A	
n-propyl Benzene	10.	N/A	

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

LAB BLAN

Lab Name: USAEHA-OECD-SAB

Matrix: Air - Custom Tenax Tube

Air Volume (L): NOVOL

Date Received: 11/12/93

Date Collected: N/A

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	11. NG	N/A uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	U
n-Heptane	10.	N/A	U
Toluene	10.	N/A	U
Ethylbenzene	10.	N/A	U
meta/para Xylene	10.	N/A	U
ortho Xylene	10.	N/A	U
n-propyl Benzene	10.	N/A	U

COMMENTS:

FORM 1
 VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

LAB BLAN

4

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA .

Matrix: Air - Custom Tenax Tube

Field ID: 50 NG ISTD

Air Volume (L) : NOVOL

Lab File ID: >JAS3A

Date Received: 11/12/93

Date Analyzed: 11/16/93

Date Collected: N/A

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	91. NG	N/A uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:-

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	U
n-Heptane	10.	N/A	U
Toluene	10.	N/A	U
Ethylbenzene	10.	N/A	U
meta/para Xylene	10.	N/A	U
ortho Xylene	10.	N/A	U
n-propyl Benzene	10.	N/A	U

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

ROAD #

LABBLANK

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: 50 NG ISTD

Air Volume (L) : NOVOL

Lab File ID: >JA64A

Date Received: 11/12/93

Date Analyzed: 11/17/93

Date Collected: N/A

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	87. NG	N/A uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	I D D D D D D
n-Heptane	10.	N/A	
Toluene	10.	N/A	
Ethylbenzene	10.	N/A	
meta/para Xylene	10.	N/A	
ortho Xylene	10.	N/A	
n-propyl Benzene	10.	N/A	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

ROAD #

LAB BLAN

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom **Tenax** Tube

Field ID: 50 NG ISTD

Air Volume (L): NOVOL

Lab File ID: >JA76A

Date Received: 11/12/93

Date Analyzed: 11/18/93

Date Collected: N/A

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

=====			
TOTAL VOLATILE ORGANICS	131. NG	N/A	uG/M3
=====			

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	U
n-Heptane	10.	N/A	U
Toluene	10.	N/A	U
Ethylbenzene	10.	N/A	U
meta/para Xylene	10.	N/A	U
ortho Xylene	10.	N/A	U
n-propyl Benzene	10.	N/A	U
=====			

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

LAB BLAN

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: SO NG ISTD

Air Volume (L): NOVOL

Lab File ID: >JA88A

Date Received: 11/12/93

Date Analyzed: 11/19/93

Date Collected: N/A

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	8. NG	N/A uG/M3
-------------------------	-------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		
	NG/SAMPLE	uG/M3	Q
Benzene	10.	N/A	U
n-Heptane	10.	N/A	U
Toluene	10.	N/A	U
Ethylbenzene	10.	N/A	U
meta/para Xylene	10.	N/A	U
ortho Xylene	10.	N/A	U
n-propyl Benzene	10.	N/A	U

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

LABBLANK

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: 50 NG ISTD

Air Volume (L): NOVOL

Lab File ID: >KA10A

Date Received: 11/12/93

Date Analyzed: 11/21/93

Date Collected: N/A

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	43. NG	N/A uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	U
n-Heptane	10.	N/A	U
Toluene	10.	N/A	U
Ethylbenzene	10.	N/A	U
meta/para Xylene	10.	N/A	U
ortho Xylene	10.	N/A	U
n-propyl Benzene	10.	N/A	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

ROAD #

LAB BLAN

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: SO NG ISTD

Air Volume (L): NOVOL

Lab File ID: >JA98A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: N/A

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	12. NG	N/A uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	U
n-Heptane	10.	N/A	U
Toluene	10.	N/A	U
Ethylbenzene	10.	N/A	U
meta/para Xylene	10.	N/A	U
ortho Xylene	10.	N/A	U
n-propyl Benzene	10.	N/A	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

ROAD #

R3724

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0506

Air Volume (L) : 19.1

Lab File ID: >JA17A

Date Received: 11/12/93

Date Collected: 11/01/93 (1535)

I. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	2649. NG	138.7 uG/M3
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2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION NG/SAMPLE	UNITS: uG/M3	Q
Benzene	37.	2.0	
n-Heptane	25.	1.3	
Toluene	126.	6.6	
Ethylbenzene	22.	1.2	
meta/para Xylene	72.	3.8	
ortho Xylene	25.	1.4	
n-propyl Benzene	5.	.24	J

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

R3725

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0507

Air Volume (L): 19.1

Lab File ID: >JA18A

Date Received: 11/12/93

Date Analyzed: 11/12/93

Date Collected: 11/01/93(1535)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	2369. NG	124.0 uG/M3
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2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	33.	1.7	
n-Heptane	20.	1.0	
Toluene	109.	5.7	
Ethylbenzene	19.	.97	
meta/para Xylene	64.	3.4	
ortho Xylene	24.	1.3	
n-propyl Benzene	4.	.22	J

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3726

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0600

Air Volume (L) : 20.0

Lab File ID: >JA19A

Date Received: 11/12/93

Date Analyzed: 11/12/93

Date Collected: 11/02/93(0027)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	2208. NG	110.4 uG/M3
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2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	27.	1.4	
n-Heptane	22.	1.1	
Toluene	77.	3.9	
Ethylbenzene	12.	.62	
meta/para Xylene	40.	2.0	
ortho Xylene	15.	.77	
n-propyl Benzene	3.	.15	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3727

Lab Name: USAEHA-OECD-SAB Installation: KUWAIT HRA
 Matrix: Air - Custom Tenax Tube Field ID: TCTASF0605
 Air Volume (L) : 17.6 Lab File ID: >JA20A
 Date Received: 11/12/93 Date Analyzed: 11/12/93
 Date Collected: 11/02/93(0050)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	2999. NG	170.4 uG/M3
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2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		%
	NG/SAMPLE	uG/M3	
Benzene	28.	1.6	
n-Heptane	28.	1.6	
Toluene	90.	5.1	
Ethylbenzene	12.	.71	
meta/para Xylene	39.	2.2	
ortho Xylene	16.	.91	
n-propyl Benzene	3.	.18	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

R3728

Lab Name: USAEHA-OECD-SAB
 Matrix: Air - Custom Tenax Tube
 Air Volume (L): 17.2
 Date Received: 11/12/93
 Date Collected: 11/02/93 (1227)

Installation: KUWAIT HRA
 Field ID: TCTASF0602
 Lab File ID: >JA21A
 Date Analyzed: 11/12/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	3966. NG	230.6 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	29.	1.7	
n-Heptane	12.	.73	
Toluene	60.	3.5	
Ethylbenzene	7.	.42	J
meta/para Xylene	22.	1.3	
ortho Xylene	9.	.50	J
n-propyl Benzene	10.	.58	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3729

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0606

Air Volume (L) : 18.2

Lab File ID: >JA22A

Date Received: 11/12/93

Date Analyzed: 11/13/93

Date Collected: 11/02/93 (1256)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1750. NG	96.2 uG/M3
-------------------------	----------	------------

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	20.	1.1	
n-Heptane	14.	.77	
Toluene	52.	2.9	
Ethylbenrene	8.	.44	J
meta/para Xylene	27.	1.5	
ortho Xylene	10.	.57	
n-propyl Benzene	2.	.11	3

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AOAD #

R3730

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT ERA

Matrix: Air - Custom **Tenax** Tube

Field ID: TCTFB0603

Air Volume (L): NOVOL

Lab File ID: >JA23A

Date Received: 11/12/93

Date Analyzed: 11/13/93

Date Collected: 11/02/93(1500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	171. NG	N/A uG/M3
-------------------------	---------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	U
n-Heptane	10.	N/A	U
Toluene	2.	N/A	J
Ethylbenzene	10.	N/A	U
meta/para Xylene	10.	N/A	U
ortho Xylene	10.	N/A	U
n-propyl Benzene	10.	N/A	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3731

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0809

Air Volume (L): 13.3

Lab File ID: >JA24A

Date Received: 11/12/93

Date Analyzed: 11/13/93

Date Collected: 11/01/93 (1700)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	718. NG	54.0 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	35.	2.6	
n-Heptane	23.	1.7	
Toluene	102.	7.7	
Ethylbenzene	22.	1.7	
meta/para Xylene	60.	4.5	
ortho Xylene	24.	1.8	
n-propyl Benzene	5.	.39	J

COMMENTS:

E-18-26

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3732

Lab Name: USAEHA-OECD-SAB
 Matrix: Air - Custom Tenax Tube
 Air Volume (L) : 13.7
 Date Received: 11/12/93
 Date Collected: 11/01/93 (1700)

Installation: KUWAIT HRA
 Field ID: T3ASF0803
 Lab File ID: >JA25A
 Date Analyzed: 11/13/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	800. NG	58.4 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	40.	2.9	
n-Heptane	2s.	1.8	
Toluene	115.	8.4	I
Ethylbenzene	25.	1.8	
meta/para Xylene	65.	4.8	
ortho Xylene	24.	1.8	
n-propyl Benzene	6.	.46	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3733

Lab Name: **USAEHA-OECD-SAB**
 Matrix: Air - Custom **Tenax** Tube
 Air Volume (L) : 14.3
 Date Received: 11/12/93
 Date Collected: 11/02/93 (0500)

Installation: KUWAIT HRA
 Field ID: **T3ASF0808**
 Lab File ID: >**JA26A**
 Date Analyzed: 11/13/93

1. TOTAL VOLATILE ORGANIC **COMPOUND** SUMMATION:

TOTAL VOLATILE ORGANICS	1343. NG	93.9 uG/M3
-------------------------	----------	------------

2. INDIVIDUAL **T01** COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	83.	5.8	
n-Heptane	54.	3.8	
Toluene	255.	18.	
Ethylbenzene	52.	3.6	
meta/para Xylene	132.	9.3	
ortho Xylene	55.	3.9	
n-propyl Benzene	11.	.80	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

R3734

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0802

Air Volume (L) : 14.4

Lab File ID: >JA29A

Date Received: 11/12/93

Date Analyzed: 11/13/93

Date Collected: 11/02/93 (0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1647. NG	114.4 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL TO 1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	UG/M3	
Benzene	87.	6.0	
n-Heptane	55.	3.8	
Toluene	252.	18.	
Ethylbent-ene	50.	3.4	
meta/para Xylene	135.	9.4	
ortho Xylene	55.	3.8	
n-propyl Benzene	12.	.80	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3735

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0501

Air Volume (L): 17.1

Lab File ID: >JA30A

Date Received: 11/12/93

Date Analyzed: 11/13/93

Date Collected: 11/03/93 (0145)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	2606. NG	152.4 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	26.	1.5	
n-Heptane	30.	1.8	
Toluene	62.	3.6	
Ethylbenzene	8.	1.34	J
meta/para Xylene	23.		
ortho Xylene	10.	.58	J
n-propyl Benzene	10.	.58	U

COMMENTS:

This sample had a 0% recovery of BZE-d6, most probably due to a spiking error.

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3737

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTMSF0901

Air Volume (L): 17.4

Lab File ID: >JA32A

Date Received: 11/12/93

Date Analyzed: 11/13/93

Date Collected: 11/03/93(0145)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	4146. NG	238.3 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	123.	7.1	
n-Heptane	28.	1.6	
Toluene	151.	8.7	
Ethylbenzene	103.	5.9	
meta/para Xylene	222.	13.	
ortho Xylene	116.	5.7	
n-propyl Benzene	109.	5.3	

COMMENTS :

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD

R3738

Lab Name: USAEHA-OECD-SAB Installation: KUWAIT HRA
 Matrix: Air - Custom Tenax Tube Field ID: TCTASF0509
 Air Volume (L) : 18.9 Lab File ID: >JA33A
 Date Received: 11/12/93 Date Analyzed: 11/13/93
 Sate Collected: 11/04/93(0200)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1325. NG	70.1 uG/M3
-------------------------	----------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	15.	.77	
n-Heptane	15.	.77	
Toluene	22.	1.1	
Ethylbenzene	3.	.17	J
meta/para Xylene	9.	.48	J
ortho Xylene	4.	.21	J
n-propyl Benzene	10.	.53	U

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET
 QAD #

R3739

Lab Name: USAEHA-OECD-SAB
 Matrix: Air - Custom Tenax Tube
 Air Volume (L) : 15.2
 Date Received: 11/12/93
 Date Collected: 11/04/93 (0200)

Installation: KUWAIT HRA
 Field ID: TCTASF0505
 Lab File ID: >JA34A
 Date Analyzed: 11/13/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1688. NG	111.1 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	11.	.75	
n-Heptane	11.	.70	
Toluene	23.	1.5	
Ethylbenzene	3.	.17	J
meta/para Xylene	7.	.48	J
ortho Xylene	3.	.23	J
n-propyl Benzene	10.	.66	U

COMMENTS :

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3740

Lab Name: USAEHA-OECD-SAB
 Matrix: Air - Custom Tenax Tube
 Air Volume (L) : 17.3
 Date Received: 11/12/93
 Date Collected: 11/04/93 (1400)

Installation: KUWAIT HRA
 Field ID: TCTASF0503
 Lab File ID: >JA35A
 Date Analyzed: 11/13/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1906. NG	110.2 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	13.	.75	
n-Heptane	10.	.58	U
Toluene	27.	1.5	
Ethylbenzene	3.	.18	
meta/para Xylene	8.	.49	
ortho Xylene	4.	.24	
n-propyl Benzene	10.	.58	

COMMENTS:

E-18-34

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3741

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0508

Air Volume (L) : 18.8

Lab File ID: >JA36A

Date Received: 11/12/93

Date Analyzed: 11/13/93

Date Collected: 11/04/93(1400)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1148. NG	61.1 uG/M3
-------------------------	----------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	14.	.75	
n-Whptane	13.	.57	
Toluene	23.	1.2	
Ethylbenzene	3.	.17	U
meta/para Xylene	8.	.44	U
ortho Xylene	4.	.21	U
n-prcpyl Benzene	10.	.53	U

COMMENTS :

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3742

Lab Name: USAEHA-OECD-SAB
 Matrix: Air - Custom Tenax Tube
 Air Volume (L) : 13.8
 Date Received: 11/12/93
 Date Collected: 11/03/93(0500)

Installation: KUWAIT HRA
 Field ID: T3MSF0902
 Lab File ID: >JA37A
 Date Analyzed: 11/13/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:-

TOTAL VOLATILE ORGANICS	3026. NG	219.3 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	121.	8.8	
n-Heptane	11.	.83	
Toluene	153.	11.	
Ethylbenzene	113.	8.2	
meta/para Xylene	228.	17.	
ortho Xylene	125.	9.1	
n-propyl Benzene	111.	8.0	

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

R3743

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3MSB0400

Air Volume (L): 13.8

Lab File ID: >JA38A

Date Received: 11/12/93

Date Analyzed: 11/14/93

Date Collected: 11/03/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	109. NG	7.9 uG/M3
-------------------------	---------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	7.	.54	J
n-Heptane	10.	.72	J
Toluene	2.	.15	J
Ethylbenzene	10.	.72	J
meta/para Xylene	10.	.72	J
ortho Xylene	10.	.72	J
n-propyl Benzene	10.	.72	J

COMMENTS :

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

ROAD #

R3744

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0807

Air Volume (L): 14.3

Lab File ID: >JA39A

Date Received: 11/12/93

Date Analyzed: 11/14/93

Date Collected: 11/03/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	441. NG	30.8 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	20.	1.4	
n-Heptane	12.	.86	
Toluene	53.	3.7	
Ethylbenzene	10.	.69	J
meta/para Xylene	26.	1.8	
ortho Xylene	11.	.78	
n-propyl Benzene	3.	.18	J

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3745

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0804

Air Volume (L) : 14.6

Lab File ID: >JA42A

Date Received: 11/12/93

Date Analyzed: 11/14/93

Date Collected: 11/03/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	805. NG	55.1 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	21.	1.	
n-Heptane	11.	.7	
Toluene	58.	4.	
Ethylbenzene	10.	.7	
meta/para Xylene	26.	2.	
ortho Xylene	11.	.8	
n-propyl Benzene	2.	.2	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3736

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASB0405

Air Volume (L): 14.6

Lab File ID: >JA43A

Date Received: 11/12/93

Date Analyzed: 11/14/93

Date Collected: 11/03/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	79. NG	5.4 uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.7	DDDDDD
n-Heptane	10.	.7	DDDDDD
Toluene	10.	.7	DDDDDD
Ethylbenzene	10.	.7	DDDDDD
meta/para Xylene	10.	.7	DDDDDD
ortho Xylene	10.	.7	DDDDDD
n-propyl Benzene	10.	.7	DDDDDD

COMMENTS:

E-18-40

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3746

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0801

Air Volume (L) : 14.7

Lab File ID: >JA44A

Date Received: 11/12/93

Date Analyzed: 11/14/93

Date Collected: 11/03/93(1700)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	599. NG	40.7 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	41.	2.8	
n-Heptane	28.	1.9	
Toluene	99.	5.7	
Ethylbenzene	28.	1.9	
meta/para Xylene	78.	5.3	
ortho Xylene	29.	2.0	
n-propyl Benzene	6.	.41	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3747

Lab Name: USAEHA-OECD-SAB
Matrix: Air - Custom Tenax Tube
Air Volume (L) : 14.7
Date Received: 11/12/93
Date Collected: 11/03/93 (1700)

Installation: KUWAIT HRA
Field ID: T3ASF0806
Lab File ID: >JA4 SA
Date Analyzed: 11/14/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	612. NG	41.6 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	42.	2.9	
n-Heptane	27.	1.9	
Toluene	97.	6.6	
Ethylbenzene	28.	1.9	
meta/para Xylene	77.	5.2	
ortho Xylene	29.	2.0	
n-propyl Benzene	6.	.42	J

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3748

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3FB0800

Air Volume (L) : NOVOL

Lab File ID: >JA46A

Date Received: 11/12/93

Date Analyzed: 11/14/93

Date Collected: 11/03/93 (1700)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	202. NG	N/A	uG/M3
-------------------------	---------	-----	-------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	4.	N/A	P D D D D D D
n-Heptane	10.	N/A	
Toluene	4.	N/A	
Ethylbenzene	10.	N/A	
meta/para Xylene	2.	N/A	
ortho Xylene	10.	N/A	
n-propyl Benzene	10.	N/A	

COMMENTS :

E-18-43

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FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3749

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0705

Air Volume (L): 13.7

Lab File ID: >JA47A

Date Received: 11/12/93

Date Analyzed: 11/14/93

Date Collected: 11/04/93 (0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	628. NG	45.8 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	30.	2.2	
n-Heptane	31.	2.2	
Toluene	70.	5.1	
Ethylbenzene	22.	1.6	
meta/para Xylene	53.	3.8	
ortho Xylene	21.	1.5	
n-propyl Benzene	5.	.33	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3750

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0805

Air Volume (L) : 12.4

• Lab File ID: >JA48A

Date Received: 11/12/93

Date Analyzed: 11/14/93

Date Collected: 11/04/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	498. NG	40.2 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	23.	1.8	
n-Heptane	20.	1.5	
Toluene	54.	4.3	
Ethylbenzene	17.	1.4	
meta/para Xylene	42.	3.4	
ortho Xylene	15.	1.3	
n-propyl Benzene	10.	.81	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3751

Lab Name: USAEHA-OECD-SAB
Matrix: Air - Custom Tenax Tube
Air Volume (L) : 12.4
Date Received: 11/12/93
Date Collected: 11/04/93 (0500)

Installation: KUWAIT HRA
Field ID: T3ASB0306
Lab File ID: >JA49A
Date Analyzed: 11/14/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	147. NG	11.8 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION		Q
	NG/SAMPLE	UNITS: uG/M3	
Benzene	5.	.37	55555555
n-Heptane	3.	.27	
Toluene	7.	.31	
Ethylbenzene	10.	.31	
meta/para Xylene	3.	.27	
ortho Xylene	10.	.31	
n-propyl Benzene	10.	.31	

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3752

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3FB0707

Air Volume (L) : NOVOL

Lab File ID: >JA50A

Date Received: 11/12/93

Date Analyzed: 11/14/93

Date Collected: 11/04/93 (1100)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	88. NG	N/A uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	U
n-Heptane	10.	N/A	U
Toluene	5.	N/A	U
Ethylbenzene	10.	N/A	U
meta/para Xylene	10.	N/A	U
ortho Xylene	10.	N/A	U
n-propyl Benzene	10.	N/A	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

ROAD #

R3753

Lab Name: USAEHA-OECD-SAB
 Matrix: Air - Custom Tenax Tube
 Air Volume (L): 14.1
 Date Received: 11/12/93
 Date Collected: 11/05/93(1430)

Installation: KUWAIT HRA
 Field ID: TCTASF0608
 Lab File ID: >JA51A
 Date Analyzed: 11/14/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

=====

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	UG/M3	
Benzene	11.	.75	
n-Heptane	10.	.71	U
Toluene	20.	1.4	
Ethylbenzene	2.	.15	J
meta/para Xylene	4.	.29	J
ortho Xylene	2.	.17	J
n-propyl Benzene	10.	.71	U

=====

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3754

Lab Name: USAEHA-OECD-SAE

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0607

Air Volume (L): 16.3

Lab File ID: >JA54A

Date Received: 11/12/93

Date Analyzed: 11/16/93

Date Collected: 11/06/93(0039)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1441. NG	88.4 uG/M3
-------------------------	----------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	18.	1.1	
n-Heptane	12.	.71	
Toluene	24.	1.5	
Ethylbenzene	4.	.25	J
meta/para Xylene	8.	.51	J
ortho Xylene	5.	.29	J
n-propyl Benzene	10.	.61	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3755

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASB0207

Air Volume (L) : 1.63

Lab File ID: >JA55A

Date Received: 11/12/93

Date Analyzed: 11/16/93

Date Collected: 11/06/93(0039)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	152. NG	9.3 uG/M3
-------------------------	---------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.51	U
n-Heptane	10.	.61	U
Toluene	10.	.61	U
Ethylbenzene	10.	.61	U
meta/para Xylene	10.	.61	U
ortho Xylene	10.	.61	U
n-propyl Benzene	10.	.61	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3756

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0604

Air Volume (L): 14.4

Lab File ID: >JA56A

Date Received: 11/12/93

Date Analyzed: 11/16/93

Date Collected: 11/06/93(0039)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1746. NG	121.2 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	32.	2.2	
n-Heptane	22.	1.5	
Toluene	46.	3.2	
Ethylbenzene	9.	.62	J
meta/para Xylene	18.	1.2	
ortho Xylene	10.	.68	J
n-propyl Benzene	10.	.69	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3757

Lab Name: USAEHA-OECD-SAB
 Matrix: Air - Custom Tenax Tube
 Air Volume (L): 16.6
 Date Received: 11/12/93
 Date Collected: 11/06/93(1305)

Installation: KUWAIT HRA
 Field ID: TCTMSF0904
 Lab File ID: >JA57A
 Date Analyzed: 11/16/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	14186. NG	854.6 uG/M3
-------------------------	-----------	-------------

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	119.	7.1	U
n-Heptane	10.	.60	
Toluene	134.	8.1	
Ethylbenzene	109.	6.5	
meta/para Xylene	219.	13.	
ortho Xylene	119.	7.2	
n-propyl Benzene	113.	6.0	

COMMENTS :

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3758

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0601

Air Volume (L) : 16.7

Lab File ID: >JA58A

Date Received: 11/12/93

Date Analyzed: 11/16/93

Date Collected: 11/06/93(1305)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	2054. NG	123.0 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	24.	1.4	
n-Heptane	10.	.60	U
Toluene	75.	4.5	
Ethylbenzene	4.	.23	J
meta/para Xylene	10.	.54	J
ortho Xylene	10.	.27	J
n-propyl Benzene	10.	.60	U

COMMENTS:

This sample had a low recovery of BZE-d6 (66%).

Handwritten notes in cursive script, mostly illegible due to fading and bleed-through.

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3759

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASB1202

Air Volume (L) : 16.7

Lab File ID: >JA59A

Date Received: 11/12/93

Date Analyzed: 11/16/93

Date Collected: 11/06/93 (1305)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	218. NG	13.0 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.60	U
n-Heptane	10.	.60	U
Toluene	10.	.60	U
Ethylbenzene	10.	.60	U
meta/para Xylene	10.	.60	U
ortho Xylene	10.	.60	U
n-propyl Benzene	10.	.60	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

R3760

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0703

Air Volume (L): 14-8

Lab File ID: >JA60A

Date Received: 11/12/93

Date Analyzed: 11/16/93

Date Collected: 11/05/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	421. NG	28.5 uG/M3
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a

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	27.	1.9	
n-Heptane	16.	1.1	
Toluene	55.	3.7	
Ethylbenzene	15.	.99	
meta/para Xylene	35.	2.4	
ortho Xylene	15.	1.0	
n-propyl Benzene	3.	.22	J

COMMENTS

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

ACAD #

R3761

Lab Name: USAEHA-OECD-SAB
 Matrix: Air - Custom Tenax Tube
 Air Volume (L) : 15.3
 Date Received: 11/12/93
 Date Collected: 11/05/93 (0500)

Installation: KUWAIT HRA
 Field ID: T3MSF0903
 Lab File ID: >JA61A
 Date Analyzed: 11/16/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	3396. NG	222.0 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	134.	a.7	
n-Heptane	16.	1.0	
Toluene	160.	10.	
Ethylbenzene	122.	8.0	
meta/para Xylene	250.	16.	
ortho Xylene	134.	a.0	
n-propyl Benzene	117.	7.7	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

R3762

Lab Name: USAEHA-OECD-SAB
 Matrix: Air - Custom Tenax Tube
 Air Volume (L) : 15.3
 Date Received: 11/12/93
 Date Collected: 11/05/93 (0500)

Installation: KUWAIT HRA
 Field ID: T3ASF0704
 Lab File ID: >JA62A
 Date Analyzed: 11/16/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	569. NG	37.2 uG/M3
-------------------------	---------	------------

2 : INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CON-TION NG/SAMPLE	UNITS : uG/M3	Q
Benzene	31.	2.0	
n-Heptane	16.	1.0	
Toluene	57.		
Ethylbenzene	14.	37.93	
meta/para Xylene	37.	2.4	
ortho Xylene	15.	1.0	
n-propyl Benzene	4.	.25	J

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3763

Lab Name: **USAEHA-OECD-SAB**
 Matrix: Air - Custom **Tenax** Tube
 Air Volume (L) : 15.3
 Date Received: 11/12/93
 Date Collected: 11/05/93(0500)

Installation: **KUWAIT HRA**
 Field ID: **T3ASB0304**
 Lab File ID: >**JA65A**
 Date Analyzed: 11/17/93

1. **TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:**

TOTAL VOLATILE ORGANICS	135. NG	8.8 uG/M3
-------------------------	---------	-----------

2. **INDIVIDUAL TO1 COMPOUND DETERMINATION:**

ANALYTE	CONCENTRATION UNITS:		
	NG/SAMPLE	uG/M3	I Q
Benzene	10.	.65	U
n-Heptane	10.	.65	U
Toluene	8.	.55	U
Ethylbenzene	10.	.65	U
meta/para Xylene	10.	.65	U
ortho Xylene	10.	.65	U
n-propyl Benzene	10.	.65	U

COMMENTS :

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3764

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0702

Air Volume (L) : 6.9

Lab File ID: >JA66A

Date Received: 11/12/93

Date Analyzed: 11/17/93

Date Collected: 11/05/93 (1700)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	416. NG	60.3 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	26.	3.7	
n-Heptane	13.	1.8	
Toluene	61.	8.8	
Ethylbenzene	16.	2.3	
meta/para Xylene	41.	6.0	
ortho Xylene	19.	2.7	
n-propyl Benzene	4.	.56	J

COMMENTS:

FORM 1
 VOLATILE ORGANICS ANALYSIS DATA SHEET

 AQAD #
 R3765

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF0706

Air Volume (L): 14.1

Lab File ID: >JA67A

Date Received: 11/12/93

Date Analyzed: 11/17/93

Date Collected: 11/05/93(1700)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

 =====

TOTAL VOLATILE ORGANICS	608. NG	43.1 uG/M3
-------------------------	---------	------------

 =====

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	40.	2.8	
n-Heptane	21.	1.5	
Toluene	84.	6.0	
Ethylbenzene	26.	1.8	
meta/para Xylene	64.	4.5	
ortho Xylene	28.	2.0	
n-propyl Benzene	6.	.43	J

 =====

COMMENTS:

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3766

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT EZRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASB0307

Air Volume (L): 14.1

Lab File ID: >JA68A

Date Received: 11/12/93

Date Analyzed: 11/17/93

Date Collected: 11/05/93 (1700)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	60. NG	4.3 uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.71	U
n-Heptane	10.	.71	U
Toluene	10.	.71	U
Ethylbenzene	10.	.71	U
meta/para Xylene	10.	.71	U
ortho Xylene	10.	.71	U
n-propyl Benzene	10.	.71	U

COMMENTS:

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3767

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF1508

Air Volume (L): 14.9

Lab File ID: >JA69A

Date Received: 11/12/93

Date Analyzed: 11/17/93

Date Collected: (1/06/93 (0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	802. NG	53.8 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	50.	3.4	
n-Heptane	31.	2.1	
Toluene	107.	7.2	
Ethylbenzene	30.	2.0	
meta/para Xylene	7s.	5.1	
ortho Xylene	33.	2.2	
n-propyl Benzene	8.	.51	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #
R3768

Lab Name: USAEHA-OECD-SAB Installation: KUWAIT HRA
 Matrix: Air - Custom Tenax Tube Field ID: T3ASF1500
 Air Volume (L) : 14.3 Lab File ID: >JA70A
 Date Received: 11/12/93 Date Analyzed: 11/17/93
 Date Collected: 11/06/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	786. NG	54.9 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	49.	3.5	
n-Heptane	30.	2.1	
Toluene	100.	7.3	
Ethylbenzene	29.	2.0	
meta/para Xylene	75.	5.3	
ortho Xylene	32.	2.2	
n-propyl Benzene	a.	.53	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

8 1105

AQAD #

R3769

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF1506

Air Volume (L): 14.3

Lab File ID: >JA71A

Date Received: 11/12/93

Date Analyzed: 11/17/93

Date Collected: 11/07/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	644. NG	45.0 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	44.	3.1	
n-Heptane	20.	1.4	
Toluene	107.	7.5	
Ethylbenzene	30.	2.1	
meta/para Xylene	72.	5.0	
ortho Xylene	32.	2.3	
n-propyl Benzene	a.	.53	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3770

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASB1608

Air Volume (L) : 14.3

Lab File ID: >JA72A

Date Received: 11/12/93

Date Analyzed: 11/17/93

Date Collected: 11/07/93 (0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	40. NG	2.0 uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.70	U
n-Heptane	10.	.70	U
Toluene	10.	.70	U
Ethylbenzene	10.	.70	U
meta/para Xylene	10.	.70	U
ortho Xylene	10.	.70	U
n-propyl Benzene	10.	.70	U

COMMENTS:

This sample had a low internal standard recovery.

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3771

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF1502

Air Volume (L): 15.2

Lab File ID: >JA73A

Date Received: 11/12/93

Date Analyzed: 11/17/93

Date Collected: 11/07/93 (1700)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	864. NG	56.8 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	52.	3.4	
n-Heptane	33.	2.2	
Toluene	154.	10.	
Ethylbenzene	46.	3.0	
meta/para Xylene	115.	7.6	
ortho Xylene	50.	3.3	
n-propyl Benzene	11.	.71	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3772

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF1503

Air Volume (L) : 9.2

Lab File ID: >JA74A

Date Received: 11/12/93

Date Analyzed: 11/17/93

Date Collected: 11/07/93(1700)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	772. NG	84.1 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	52.	5.6	
n-Heptane	30.	3.3	
Toluene	140.	15.	
Ethylbenzene	37.	4.1	
meta/para Xylene	98.	11.	
ortho Xylene	43.	4.7	
n-propyl Benzene	9.	1.0	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AOAD #

R3773

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0907

Air Volume (L) : 16.7

Lab File ID: >JA77A

Date Received: 11/12/93

Date Analyzed: 11/18/93

Date Collected: 11/07/93(0005)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	4604. NG	275.7 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL TOL COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	16.	.9s	
n-Heptane	10.	.60	U
Toluene	35.	2.1	
Ethylbenzene	s.	.32	J
meta/para Xylene	14.	.82	
ortho Xylene	7.	.42	J
n-propyl Benzene	10.	.60	U

COMMENTS:

..... This sample contained ~ 5500 ng TRichloroethane.....
.....
.....

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3775

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0906

Air Volume (L): 18.6

Lab File ID: >JC78A

Date Received: 11/12/93

Date Analyzed: 11/18/93

Date Collected: 11/07/93(0005)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	3095. NG	166.4 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		o
	NG/SAMPLE	uG/M3	
Benzene	21.	1.1	
n-Heptane	16.	.88	
Toluene	42.	2.2	
Ethylbenzene	7.	.40	J
meta/para Xylene	16.	.85	
ortho Xylene	8.	.43	J
n-propyl Benzene	10.	.54	U

COMMENTS:

E-18-69

FORM 1

VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3774

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASB1206

Air Volume (L) : 18.6

Lab File ID: >JC79A

Date Received: 11/12/93

Date Analyzed: 11/18/93

Date Collected: 11/07/93(0005)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

=====		
TOTAL VOLATILE ORGANICS	98. NG	5.3 uG/M3
=====		

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.5	U
n-Heptane	10.	.5	U
Toluene	10.	.5	U
Ethylbenzene	10.	.5	U
meta/para Xylene	10.	.5	U
ortho Xylene	10.	.5	U
n-propyl Benzene	10.	.5	U
=====			

COMMENTS:

E-18-70

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3776

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTMSF1703

Air Volume (L): 16.3

Lab File ID: >JC80A

Date Received: 11/12/93

Date Analyzed: 11/18/93

Date Collected: 11/07/93 (1204)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

=====			
TOTAL VOLATILE ORGANICS	5981. NG	366 . 9	uG/M3
=====			

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS		o
	NG/SAMPLE	uG/M3	
Benzene	204.	12.	
n-Heptane	10.	.6	U
Toluene	200.	12.	
Ethybenzene	200.	12.	
meta/para Xylene	394.	24.	
ortho Xylene	213.	13.	
n-propyl Benzene	189.	12.	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3777

Lab Name: USAEHA-OECD-SAB Installation: KUWAIT HRA
 Matrix: Air - Custom Tenax Tube Field ID: TCTMSB1201
 Air Volume (L) : 16.3 Lab File ID: >JC81A
 Date Received: 11/12/93 Date Analyzed: 11/18/93
 Date Collected: 11/07/93 (1204)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	135. NG	8.3 uG/M3
-------------------------	---------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.60	J
n-Heptane	10.	.61	U
Toluene	10.	.60	J
Ethylbenzene	10.	.61	U
meta/para Xylene	10.	.61	U
ortho Xylene	10.	.61	U
n-propyl Benzene	10.	.61	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3778

Lab Name: USAEHA-OECD-SAB
Matrix: Air - Custom Tenax Tube
Air Volume (L) : 16.4
Date Received: 11/12/93
Date Collected: 11/07/93 (1204)

Installation: KUWAIT HRA
Field ID: TCTASF1106
Lab File ID: >JC82A
Date Analyzed: 11/18/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1174. NG	71.6 uG/M3
-------------------------	----------	------------

2. INDIVIDUAL TO 1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	9.	.55	J
n-Heptane	10.	.61	U
Toluene	10.	.63	
Ethylbenzene	2.	.13	J
meta/para Xylene	5.	.29	J
ortho Xylene	3.	.16	J
n-propyl Benzene-	10.	.61	U

COMMENTS:

The sample contained ~ 700 ng of benzene.

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3779

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTMSF1701

Air Volume (L) : 17.8

Lab File ID: >JC83A

Date Received: 11/12/93

Date Analyzed: 11/18/93

Date Collected: 11/07/93 (2326)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	7114. NG	399.6 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	201.	11.	I
n-Heptane	10.	.6	U
Toluene	210.	12.	
Ethylbenzene	198.	11.	
meta/para Xylene	397.	22.	
ortho Xylene	221.	12.	
n-propyl Benzene	207.	12.	

COMMENTS:

This sample was analyzed on 11/18/93

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3780

Lab Name: USAEHA-OECD-SAB
Matrix: Air - Custom Tenax Tube
Air Volume (L): 16.6
Date Received: 11/12/93
Date Collected: 11/07/93 (2326)

Instailacion: KUWAIT ERA
Field ID: TCTASF0909
Lab File ID: >JC84A
Date Analyzed: 11/18/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	2997. NG	180.6 uG/M3
-------------------------	----------	-------------

2: INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION NG/SAMPLE	UNITS: uG/M3	Q
Benzene	18.	1.1	
n-Heptane	10.	.60	U
Toluene	28.	1.7	
Ethylbenzene	5.	.29	J
meta/para Xylene	10.	.61	
ortho Xylene	6.	.36	J
n-propyl Benzene	10.	.60	J

COMMENTS:

This sample contained no detectable

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3781

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASB1209

Air Volume (L) : 16.6

Lab File ID: >JA85A

Date Received: 11/12/93

Date Analyzed: 11/19/93

Date Collected: 11/07/93 (2326)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	404. NG	24.3 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		
	NG/SAMPLE	uG/M3	Q
Benzene	6.	.35	J
n-Heptane	10.	.60	U
Toluene	45.	2.7	
Ethylbenzene	10.	.60	U
meta/para Xylene	10.	.60	U
ortho Xylene	10.	.60	U
n-propyl Benzene	10.	.60	U

COMMENTS:

E-18-76

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3782

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0908

Air Volume (L) : 15.4

Lab File ID: >JA86A

Date Received: 11/12/93

Date Analyzed: 11/19/93

Date Collected: 11/08/93(1105)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	2721. NG	176.7 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	11.	.74	
n-Heptane	10	.65	U
Toluene	21.	1.4	
Ethylbenzene	3.	.20	J
meta/para Xylene	7.	.45	J
ortho Xylene	3.	.21	J
n-propyl Benzene	10.	.65	U

COMMENTS:

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.....
.....

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

43703

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTASF0905

Air Volume (L) : 16.1

Lab File ID: >JB89A

Date Received: 11/12/93

Date Analyzed: 11/19/93

Date Collected: 11/08/93(1105)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

-----d-----		
TOTAL VOLATILE ORGANICS	3568. NG	221.6 uG/M3
=====		

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	11.	.71	
n-Heptane	10.	.62	U
Toluene	13.	1.2	
Ethylbenzene	3.	.29	J
meta/para Xylene	5.	.33	J
ortho Xylene	3.	.21	J
n-propyl Benzene	10.	.62	U

COMMENTS:

 THIS SAMPLE CONTAINED 24% ...

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3784

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF1507

Air Volume (L) : 13.3

Lab File ID: >JB90A

Date Received: 11/12/93

Date Analyzed: 11/19/93

Date Collected: 11/08/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	669. NG	50.3 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		
	NG/SAMPLE	uG/M3	Q
Benzene	41.	4.1	I
n-Heptane	2s.	1.8	I
Toluene	108.	8.1	
Ethylbenzene	27.	2.3	
meta/para Xylene	70.	5.3	
ortho Xylene	29.	2.2	
n-propyl Benzene	5.	.41	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3785

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASB1600

Air Volume (L) : 13.3

Lab File ID: >JB91A

Date Received: 11/12/93

Date Analyzed: 11/19/93

Date Collected: 11/08/93(0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

=====		
TOTAL VOLATILE ORGANICS	21. NG	1.6 uG/M3
=====		

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.75	U
n-Heptane	10.	.75	U
Toluene	10.	.75	U
Ethylbenzene	10.	.75	U
meta/para Xylene	10.	.75	U
ortho Xylene	10.	.75	U
n-propyl Benzene	10.	.75	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3786

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF1509

Air Volume (L) : 14.3

Lab File ID: >JB92A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: 11/08/93 (0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	792. NG	55-4 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	40.	2.8	I
n-Heptane	26.	1.8	
Toluene	134.	9.4	
Ethylbenzene	29.	2.0	
meta/para Xylene	73.	5.1	
ortho Xylene	31.	2.1	
n-propyl Benzene	6.	.41	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3787

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3MSF1702

Air Volume (L): 14.4

Lab File ID: >JB93A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: 11/09/93(0655)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

=====		
TOTAL VOLATILE ORGANICS	6026. NG	418.4 uG/M3
=====		

2. INDIVIDUAL TO1 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	248.	17.	
n-Heptane	41.	3.	
Toluene	317.	22.	
Ethylbenzene	229.	16.	
meta/para Xylene	472.	33.	
ortho Xylene	250.	17.	
n-propyl Benzene	203.	14.	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3788

Lab Name: **USAEHA-OECD-SAB**

Installation: KUWAIT HRA

Matrix: Air - Custom **Tenax** Tube

Field ID: **T3ASF1303**

Air Volume (L) : 14.6

Lab File ID: >**JB94A**

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: 11/09/93 (0655)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	823. NG	56.4 µG/M3
-------------------------	---------	------------

2. INDIVIDUAL **T01** COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION		Q
	NG/SAMPLE	UNITS: µG/M3	
Benzene	56.	3.8	
n-Heptane	39.	2.7	
Toluene	133.	9.1	
Ethylbenzene	32.	2.2	
meta/para Xylene	85.	5.9	
ortho Xylene	38.	2.5	
n-propyl Benzene		.53	J

COMMENTS :

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3789

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASB1605

Air Volume (L): 14.6

Lab File ID: >JA95A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: 11/09/93(0655)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	62. NG	4.2 uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	2.	.11	J
n-Heptane	10.	.68	U
Toluene	10.	.68	U
Ethylbenzene	10.	.68	U
meta/para Xylene	10.	.68	U
ortho Xylene	10.	.68	U
n-propyl Benzene	10.	.68	U

COMMENT:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3790

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF1302

Air Volume (L) : 14.8

Lab File ID: >JA96A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: 11/09/93(0655)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	893. NG	60.3 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	59.	4.0	
n-Heptane	38.	2.6	
Toluene	134.	9.1	
Ethylbenzene	33.	2.2	
meta/para Xylene	81.	5.5	
ortho Xylene	36.	2.5	
n-propyl Benzene	8.	.55	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3791

Lab Name: **USAEHA-OECD-SAB**
 Matrix: Air - Custom **Tenax** Tube
 Air Volume (L): NOVOL
 Date Received: 11/12/93
 Date Collected: 11/09/93(0655)

Installation: KUWAIT HRA
 Field ID: **T3FB1501**
 Lab File ID: >KA01A
 Date Analyzed: 11/20/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	193. NG	N/A uG/M3
-------------------------	---------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	4.	N/A	J
n-Heptane	10.	N/A	J
Toluene	9.	N/A	J
Ethylbenzene	10.	N/A	J
meta/para Xylene	3.	N/A	J
ortho Xylene	10.	N/A	3
n-propyl Benzene	10.	N/A	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3792

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF1301

Air Volume (L): 10.2

Lab File ID: >KA02A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: 11/09/93(1845)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	1635. NG	160.3 uG/M3
-------------------------	----------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	308.	12.	
n-Heptane		7.	
Toluene		30.	
Ethylbenzene	88.	0	
meta/para Xylene	236.	27.	
ortho Xylene	98.	10.	
n-propyl Benzene	21.	2.	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3793

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASB1602

Air Volume (L): 10.2

Lab File ID: >KA03A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: 11/09/93(1845)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	98. NG	9.6 uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.98	U
n-Heptane	10.	.98	U
Toluene	4.	.43	J
Ethylbenzene	10.	.98	U
meta/para Xylene	10.	.98	U
ortho Xylene	10.	.98	U
n-propyl Benzene	10.	.98	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3794

Lab Name: **USAEHA-OECD-SAB**

Installation: KUWAIT HRA

Matrix: Air - Custom **Tenax** Tube

Field ID: **T3ASF1505**

Air Volume (L) : 5.5

Lab File ID: >KA04A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: 11/09/93(1845)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	898. NG	164.1 uG/M3
-------------------------	---------	-------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	68.	12.	
n-Heptane	35.	6.	
Toluene	175.	32.	
Ethylbenzene	50.	9.	
meta/para Xylene	134.	24.	
ortho Xylene	57.	10.	
n-propyl Benzene	12.	2.	

COMMENTS

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3795

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: T3ASF1504

Air Volume (L) : 14.3

Lab File ID: >KA05A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: 11/07/93 (0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	945. NG	66.1 uG/M3
-------------------------	---------	------------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	46	3.2	
n-Heptane	35	2.5	
Toluene	35	2.5	
Ethylbenzene	35	2.5	
meta/para Xylene	35	2.5	
ortho Xylene	35	2.5	
n-propyl Benzene		.53	J

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3796

Lab Name: **USAEHA-OECD-SAB** Installation: KUWAIT HRA
 Matrix: Air - Custom **Tenax** Tube Field ID: **T3ASB1409**
 Air Volume (L) : 14.3 Lab File ID: >KA06A
 Date Received: 11/12/93 Date Analyzed: 11/20/93
 Date Collected: 11/07/93 (0500)

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	94. NG	6.6 uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	.70	U
n-Heptane	10.	.70	U
Toluene	7.	.46	J
Ethylbenzene	10.	.70	U
meta/para Xylene	10.	.70	3
ortho Xylene	10.	.70	U
n-propyl Benzene	10.	.70	U

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3797

Lab Name: **USAEHA-OECD-SAB**
 Matrix: **Air** - Custom **Tenax** Tube
 Air Volume (L): **NOVOL**
 Date Received: **11/12/93**
 Date Collected: **11/08/93(????)**

Installation: **KUWAIT HRA**
 Field ID: **TCTFB1209**
 Lab File ID: **>KA07A**
 Date Analyzed: **11/20/93**

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	I D D D D D D D
n-Heptane	10.	N/A	
Toluene	6.	N/A	
Ethylbenzene	10.	N/A	
meta/para Xylene	10.	N/A	
ortho Xylene	10.	N/A	
n-propyl Benzene	10.	N/A	

COMMENTS:

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3798

Lab Name: USAEHA-OECD-SAB

Installation: KUWAIT HRA

Matrix: Air - Custom Tenax Tube

Field ID: TCTTB0906

Air Volume (L) : NOVOL

Lab File ID: >KA08A

Date Received: 11/12/93

Date Analyzed: 11/20/93

Date Collected: TRVBLK1STSHIPM

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	22. NG	N/A uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	DDDD
n-Heptane	10.	N/A	DDDD
Toluene	5.	N/A	DDDD
Ethylbenzene	10.	N/A	DDDD
meta/para Xylene	10.	N/A	DDDD
ortho Xylene	10.	N/A	DDDD
n-propyl Benzene	10.	N/A	DDDD

COMMENTS

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3799

Lab Name: **USAEHA-OECD-SAB**
Matrix: Air - Custom **Tenax** Tube
Air Volume (L): NOVOL
Date Received: 11/12/93
Date Collected: TRVBLK2NDSHIP

Installation: KUWAIT HRA
Field ID: **TCTTB0635**
Lab File ID: >**KAl1A**
Date Analyzed: 11/21/93

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

TOTAL VOLATILE ORGANICS	12. NG	N/A uG/M3
-------------------------	--------	-----------

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS.:		°
	NG/SAMPLE	uG/M3	
Benzene	10.	N/A	U D D D D D D
n-Heptane	10.	N / A	
Toluene	10.	N / A	
Ethylbenzene	10.	N / A	
meta/para Xylene	10.	N / A	
ortho Xylene	10.	N / A	
n-propyl Benzene	10.	N / A	

COMMENTS

FORM 1
VOLATILE ORGANICS ANALYSIS DATA SHEET

AQAD #

R3800

Lab Name: **USAEHA-OECD-SAB**

Installation: KUWAIT HRA

Matrix: Air - Custom **Tenax** Tube

Field ID: **T3TB0905**

Air Volume (L): NOVOL

Lab File ID: **>KA12A**

Date Received: 11/12/93

Date Analyzed: 11/21/93

Date Collected: TRVBLK1STSHIP

1. TOTAL VOLATILE ORGANIC COMPOUND SUMMATION:

=====		
TOTAL VOLATILE ORGANICS	41. NG	N/A uG/M3
=====		

2. INDIVIDUAL T01 COMPOUND DETERMINATION:

ANALYTE	CONCENTRATION UNITS:		Q
	NG/SAMPLE	uG/M3	
Benzene	4.	N/A	J
n-Heptane	10.	N/A	U
Toluene	10.	N/A	U
Ethylbenzene	10.	N/A	U
meta/para Xylene	10.	N/A	U
ortho Xylene	10.	N/A	U
n-propyl Benzene	10.	N/A	U

COMMENTS:

FORM 5

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION - BROMOFLUOROBENZENE (BFB)

Lab Name: USAEHA-OECD-CAB

Lab File ID: >BFK12

BFB Injection Date: 11/12/93

Instrument ID: CHEMSTATION

BFB Injection Time: 17:12

Matrix: AIR

Column: (pack/cap) Cap

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% OF MASS 95	22.6
75	30.0 - 60.0% OF MASS 95	48.6
95	Base peak, 100% relative abundance	100
96	5.0 - 9.0% of mass 95	5.7
173	Less than 2.0% of mass 174	0.0 (0.0)1
174	Greater than 50.0% of mass 95	74.8
175	5.0 - 9.0% of mass 174	6.0 (8.1)1
176	Greater than 95.0%, but less than 101.0% of mass 174	73.1 (97.7)1
177	5.0 - 9.0% of mass 176	5.0 (6.9)2

1-Value is % mass 174

2-Value is % mass 176

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS, AND STANDARDS:

	AQAD SAMPLE NO.	FIELD SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01	50 NG CHECK	50 NG ISTD/SU	>JA15A	11/12/93	17:57
02	SYSTEMBLANK	50 NG ISTD	>JA16A	11/12/93	18:53
03	R3724	TCTASF0506	>JA17A	11/12/93	20:13
04	R3725	TCTASF0507	>JA18A	11/12/93	21:07
05	R2726	TCTASF0600	>JA19A	11/12/93	22:01
06	R3727	TCTASF0605	>JA20A	11/12/93	22:55
07	R3728	TCTASF0602	>JA21A	11/12/93	23:49
08	R3729	TCTASF0606	>JA22A	11/13/93	0:43
09	R3730	TCTFB0603	>JA23A	11/13/93	1:37
10	R3731	T3ASF0809	>JA24A	11/13/93	2:31
11	R3732	T3ASF0803	>JA25A	11/13/93	3:25
12	R3733	T3ASF0806	>JA26A	11/13/93	4:19
13					
14					
15					

Final Rpt, Kuwait Oil Fire HRA No. 39-26-L192-91, 5 May - 3 Dec 91

ANNEX E-19

E-19-1

ANALYSIS REPORT

U.S ARMY ENVIRONMENTAL HYGIENE AGENCY
Aberdeen Proving Ground, MD 21010
Organic Environmental Chemistry Division
Special Analysis Branch

Installation: KUWAIT/ SAUDIA ARABIA

Project Number: 39-22-L192

Project Officer: J. Heller

Number of Samples: 23

Matrix: XAD-2

Analyzed for: PAHs

Method: TO13

LCSD No.	FIELD NO.	FILE NO.
R3701	PS/CT/AS/S/XAD-07C	>SSB07
R3702	PS/CT/AS/S/XAD-08C	>SSB08
R3703	PS/CT/AS/S/XAD-06C	>SSC10
R3704	PS/CT/AS/S/XAD-05C	>SSC11
R3705	PS/CT/AS/S/XAD-04C	>SSC05
R3706	PS/CT/AS/S/6B/S/XAD-03C	>SSC12
R3707	PS/CT/TB/XAD-02TB	>SSC13
R3708	PS/CT/FB/XAD-02B	>SSC14
R3709	PS/3/AS/XAD/07	>SSC15
R3710	PS/3/AS/XAD/06	>SSC16
R3711	PS/3/AS/XAD/09	>SSC17
R3712	PS/3/AS/XAD/10	>SSC18
R3713	PS/3/AS/XAD/08	>SSC19
R3714	PS/3/AS/XAD/11	>SSC02
R3715	PS/3/AS/XAD/05	>SSC01
R3716	PS/CT/AS/S/6B/XAD-01C	>SSC09
R3717	PS/CT/AS/S/6B/XAD-02C	>SSC08
R3718	PS/CT/FB/S/XAD-03FB	>SSC07
R3719	PS/CT/BL/N/XAD-03B	>SSC06
R3720	PS/3/AS/XAD/01	>SSC04
R3721	PS/3/AS/XAD/02	>SSC03
R3722	PS/3/FB/XAD/03	>SSC02
R3723	PS/3/TB/XAD/04	>SSC01

Analyst: Michael J

Date: 13 Dec 93

Reviewed by: Robert J Vales

Date: 14 Dec 93

Reviewed by: Wesley Smith

Date: 14 Dec 93