

**FOP AIR MONITORING PLAN**  
for  
**Former Operating Plant Remediation Activities and  
Remediation Consolidation Area Operation and Closure Activities**  
at  
**Exide Technologies Frisco Recycling Center**  
**Frisco, Texas**

Prepared by  
**Golder Associates Inc.**

**May 2019**

# Table of Contents

|                                                                                                   |           |
|---------------------------------------------------------------------------------------------------|-----------|
| <b>1.0 INTRODUCTION.....</b>                                                                      | <b>2</b>  |
| <b>2.0 ORGANIZATION OF PLAN.....</b>                                                              | <b>2</b>  |
| <b>3.0 PARTICULATE Matter MONITORING .....</b>                                                    | <b>3</b>  |
| 3.1 Equipment.....                                                                                | 3         |
| 3.2 Monitoring Locations .....                                                                    | 3         |
| 3.3 Take Action and Stop Work Levels Using Particulates as a Surrogate for Lead and Cadmium ..... | 3         |
| 3.3.1 Establishing Particulate Take Action and Stop Action Levels for Lead .....                  | 3         |
| 3.3.2 Establishing Particulate Matter Take Action and Stop Work Levels for Cadmium .....          | 4         |
| 3.3.3 Take Action and Stop Work Levels for PM <sub>10</sub> as Surrogate.....                     | 5         |
| 3.4 Stop Work Level for Wind.....                                                                 | 5         |
| 3.5 Particulate Monitors and Wind Data Monitoring and Notifications .....                         | 5         |
| 3.5.1 Particulate Monitors .....                                                                  | 5         |
| 3.5.2 Wind Speed and Direction Data Monitoring.....                                               | 5         |
| 3.5.3 Notifications.....                                                                          | 6         |
| 3.5.4 Stop Work Criteria for Monitors.....                                                        | 6         |
| 3.6 Dust Suppression Measures .....                                                               | 6         |
| 3.6.1 Particulate Matter Take Action Levels .....                                                 | 6         |
| 3.6.2 Particulate Stop Work Levels.....                                                           | 6         |
| 3.6.3 Visible Dust .....                                                                          | 6         |
| <b>4.0 AIR SAMPLES COLLECTED FOR LABORATORY ANALYSES.....</b>                                     | <b>7</b>  |
| 4.1 Metals Analyses .....                                                                         | 7         |
| 4.2 Metals Concentrations Take Action Levels .....                                                | 8         |
| 4.3 Metals Concentrations Stop Work Levels .....                                                  | 8         |
| <b>5.0 REPORTS.....</b>                                                                           | <b>9</b>  |
| <b>6.0 QUALITY ASSURANCE / QUALITY CONTROL.....</b>                                               | <b>10</b> |
| 6.1 Particulate Monitors .....                                                                    | 11        |
| 6.1.1 Quality Control .....                                                                       | 11        |
| 6.2 Air Samplers .....                                                                            | 12        |
| 6.2.1 Quality Control .....                                                                       | 12        |
| 6.2.2 Quality Assurance .....                                                                     | 12        |
| 6.3 Met Station.....                                                                              | 13        |
| 6.4 Laboratory Validation.....                                                                    | 13        |
| 6.5 Dust Concentration, Wind Speed and Direction Report Validation .....                          | 13        |
| 6.6 Sample Information Management .....                                                           | 14        |
| <b>7.0 POINTS OF CONTACT .....</b>                                                                | <b>14</b> |

## ATTACHMENTS

1. Descriptive Literature on E-BAM Particulate Monitors
2. NIOSH Method 7303

## **1.0 INTRODUCTION**

The purpose of the air monitoring and dust control plans is to identify the measures that will be taken to monitor and minimize emissions associated with remediation activities at the Former Operating Plant portion of the Exide Technologies Frisco Recycling Center in Frisco, Collin County, Texas (FOP), including operation and closure activities at the Remediation Consolidation Area (RCA). Specifically, this Air Monitoring Plan specifies the requirements and methods for monitoring ambient air quality for particulate matter (dust), lead, and cadmium during excavation, consolidation, and closure activities. This plan works in conjunction with the FOP/RCA Dust Control Plan, which describes operational controls to reduce dust emissions during these activities.

Remediation and closure activities are described in detail in other components of the Final Closure Plan, to which this Perimeter Air Monitoring Plan and the FOP/RCA Dust Control Plan are appendices. Excavation of soils at the FOP and sediment/soils in Stewart Creek and placement of the soils/sediment in the RCA are considered dust-generating activities for the purposes of this plan as well as any additional demolition activities that may be needed prior to placement of waste within the RCA.

Air monitoring is not required during the following activities:

- Initial construction of perimeter berms constructed of clean soil
- Placement of the cap once contaminated soil or waste is covered
- Backfill of excavation areas with clean soil.

Air quality monitoring will consist of ambient air monitoring using NIOSH Method 7303 to evaluate lead and cadmium concentrations in dust. E-BAM mass monitors will be used to evaluate dust concentrations. Monitoring will be conducted to ensure that potential off-site impacts are mitigated. Air quality will be monitored by the contractor during the remediation activities.

The primary objectives of the air monitoring are to

- Monitor the relationship between particulate matter (i.e., dust) levels and concentrations of lead and cadmium so that the particulate matter measurements can be used as a surrogate;
- Determine if concentrations of lead and cadmium and particulate emissions are in excess of air Take Action or Stop Work Levels established for the FOP; and
- Ensure that engineering controls and work practices help minimize potential off-site impacts. The Plan will help ensure that the contractor reacts quickly and makes appropriate changes to dust control measures as needed.

Air quality will be measured and documented at air quality monitoring stations during FOP remediation, RCA operation, and RCA closure activities in accordance with this Plan.

## **2.0 ORGANIZATION OF PLAN**

This plan addresses the air monitoring to be performed during dust-generating activities involving excavation and consolidation of contaminated soil or sediment and placement of soil or sediment (or other approved waste as described in the Final Closure Plan) at the FOP and ancillary demolition needed prior to placement of wastes in the RCA (the "dust generating activities"). This Plan addresses continuous perimeter monitoring for particulate matter (PM<sub>10</sub>) during the times that such dust generating activities are performed; explains how the relationship between particulate matter, lead, and cadmium will be established and monitored; and describes how the Take Action and Stop Work Levels will be identified and implemented for particulate matter. In addition, it describes how samples will be collected to directly

measure lead and cadmium and how that data will be used. The dust control procedures to be used during excavation and placement of waste are described in the Dust Control Plan.

### **3.0 PARTICULATE MATTER MONITORING**

#### **3.1 Equipment**

Real-time particulate matter air monitors (e.g., E-BAM mass monitor or equivalent) equipped with omnidirectional air intake devices and PM<sub>10</sub> impactor heads will be used at the FOP to monitor dust levels at or near the Exide property boundaries during dust generating activities. Real-time data from the downwind particulate matter monitors will be evaluated in 30-minute and 60-minute averaged blocks to provide immediate comparison to Take Action and Stop Work Level criteria. If there is a calm wind condition (i.e. 1 mile per hour or less averaged over a 30-minute period) the upwind monitor will be treated as a downwind monitor. The data collection and reporting system which utilizes data generated by this equipment is described further in Section 3.5. Attachment 1 provides specific information regarding the E-BAM mass monitors that will be utilized at the FOP.

#### **3.2 Monitoring Locations**

One upwind and up to six downwind monitoring locations will be established each day dust-generating activities involving contaminated soil or sediment are to be performed (so that there are at least one upwind and three downwind monitoring locations for the RCA activities that may be occurring at the FOP). Monitors will be placed near the FOP boundary to ensure adequate downwind coverage of both the remediation areas and the RCA to minimize the potential for impacts to property beyond the FOP. If multiple activities are being conducted concurrently (i.e., multiple remediation areas, RCA activities, etc.) the downwind monitoring network will be used to monitor all activities to the extent practicable. One monitor may be used to monitor both RCA activities and remediation activities, depending on wind direction. If wind direction and remediation activity locations warrant it, additional monitors will be added to ensure adequate downwind coverage. If Take Action or Stop Work criteria are exceeded, dust mitigation procedures applicable to each activity will be implemented. The contractor will utilize National Weather Service forecasts and review current conditions and recent trends from an on-site meteorological station, located near the North Corrective Action Management Unit, to position the monitors each morning prior to start of work. The locations of the monitors will be determined by GPS and recorded. Wind speed and direction will be recorded and the data sent to on-site personnel as described in Section 3.5. If there is a 90-degree change in the prevailing wind direction averaged over a 30-minute period during the work day, the initial placement of the downwind monitors will be appropriately relocated and dust-generating activities involving waste will be suspended until the monitors resume operation.

#### **3.3 Take Action and Stop Work Levels Using Particulates as a Surrogate for Lead and Cadmium**

The 2008 National Ambient Air Quality Standards (NAAQS) standard for lead, and the Texas Effects Screening Level (ESL) for cadmium have been utilized to establish "Take Action" and "Stop Work" levels for real-time particulate monitoring that will minimize off-site migration of dust associated with the remediation and RCA closure activities. The lead and cadmium-based PM<sub>10</sub> surrogate levels will be calculated based upon correlations derived from project monitoring data and the more stringent of the two surrogate levels (i.e., lead or cadmium) will be used to establish the ongoing "Take Action" and "Stop Work" levels for PM<sub>10</sub>.

##### **3.3.1 Establishing Particulate Take Action and Stop Action Levels for Lead**

The target level for lead on a one-hour basis (TPb), has been derived from the current (2008) NAAQS for Pb, 0.15 µg/m<sup>3</sup>, which is expressed as a three-month rolling average. The lead action level (AL<sup>Pb</sup>) derived from the NAAQS will be implemented on the basis of 30-minute and 60-minute block-averaged particulate

readings. The particulate Take Action Level notification will be based on a 30-minute downwind block average (TAL<sup>PM-30</sup>). The particulate Stop Work Level will be set on 30-minute (SWL<sup>PM-30</sup>) and 60-minute (SWL<sup>PM-60</sup>) downwind block averages.

According to Appendix D, "Averaging Period Concentration Estimates," of EPA-454/R-92-024 "Workbook of Screening Techniques for Assessing Impacts of Toxic Air Pollutants (Revised)" dated December 1992, the appropriate multiplying factor in converting one-hour averaged concentrations to three-month averages is 0.1. Therefore, to set an equivalent one-hour allowable concentration consistent with the three-month averaged Pb NAAQS, the NAAQS value of 0.15 µg/m<sup>3</sup> is divided by 0.1, yielding 1.5 µg/m<sup>3</sup> = 0.0015 mg/m<sup>3</sup> Pb = TPb. Until the AL<sup>Pb</sup> is established as described below, the default TAL<sup>PM-30</sup> will be 0.1 mg/m<sup>3</sup>, and the SWL<sup>PM-30</sup> will be 0.2 mg/m<sup>3</sup> (two times the default TAL<sup>PM-30</sup>). The default SWL<sup>PM-60</sup> will be 0.1 mg/m<sup>3</sup>. Work completed to date on other portions of the FOP have shown that this is an appropriate default level.

The AL<sup>Pb</sup> will be calculated by the following method:

The lead content fraction (FPb), taking into account downwind air sampling stations, will be determined from project-collected particulate matter and lead concentration data based upon the following relationship in the measured downwind particulate matter monitoring data. Any sample results for lead which are reported from the laboratory as being below the detection limits will be entered into this calculation as half of the reported detection limit rather than as zero. The calculation of FPb will be completed for the averaged data from each of the downwind particulate monitors and air sampler pairs.

$$\frac{Pb (mg/m^3)}{PM_{10} (mg/m^3)} = FPb (unitless)$$

The highest of the calculated values from the downwind particulate monitor and air sampler pairs will be the FPb. The AL<sup>Pb</sup> for the particulate matter monitors for the action levels described above will then be calculated as follows:

$$\frac{TPb (0.0015 mg/m^3)}{FPb (unitless)} = AL^{Pb} (mg/m^3 \text{ as particulates, } PM_{10})$$

The lowest correlated particulate matter Take Action Levels for lead calculated from the averaged data for each day from each of the downwind particulate matter monitor and air sampler pairs will be used to calculate a two-week rolling average that will be utilized for the dust monitors' AL<sup>Pb</sup> until the next correlation is performed.

### 3.3.2 Establishing Particulate Matter Take Action and Stop Work Levels for Cadmium

The Texas Commission on Environmental Quality (TCEQ) short-term Effects Screening Level (ESL) for cadmium is 0.0001 mg/m<sup>3</sup>. Until the AL<sup>Cd</sup> is established as described below, the default TAL<sup>PM-30</sup> will be 0.1 mg/m<sup>3</sup>, and the default SWL<sup>PM-30</sup> will be 0.2 mg/m<sup>3</sup> (two times the default TAL<sup>PM-30</sup>). The default SWL<sup>PM-60</sup> will be 0.1 mg/m<sup>3</sup>.

In order to derive a comparable PM<sub>10</sub> Take Action Level, the AL for cadmium based upon the content of cadmium in the measured dust (FCd) is determined from the downwind project-collected particulate matter and cadmium concentration data by the following equations. Any sample results for cadmium which are reported from the laboratory as being below the detection limits will be entered into this calculation as half of the reported detection limit rather than as zero. The calculation of FCd will be completed for the averaged data from each of the downwind particulate monitors and air sampler pairs.

$$\frac{Cd (mg/m^3)}{PM_{10} (mg/m^3)} = FCd (unitless)$$

The highest of the calculated values from the downwind particulate matter monitors and air sampler pairs will be the FCd. The AL<sup>Cd</sup> for the dust monitors for the action levels described above will then be calculated as follows:

$$\frac{ESL\ Cd\ (0.0001\ mg/m^3)}{FCd\ (unitless)} = AL^{Cd}\ (mg/m^3\ as\ particulates,\ PM_{10})$$

The lowest correlated particulate matter Take Action Levels for cadmium calculated from the averaged data for each day from each of the downwind particulate matter monitor and air sampler pairs will be used to calculate a two-week rolling average that will be utilized for the dust monitors' AL<sup>Cd</sup> until the next correlation is performed.

### **3.3.3 Take Action and Stop Work Levels for PM<sub>10</sub> as Surrogate**

The TAL<sup>PM-30</sup> (i.e., 30-minute block average Take Action Level) and SWL<sup>PM-60</sup> (i.e., 60-minute block average Stop Work Level) for PM<sub>10</sub> will be the LOWER of the calculated AL<sup>Pb</sup> and AL<sup>Cd</sup>. In no event will the TAL<sup>PM-30</sup> and the SWL<sup>PM-60</sup> be greater than 0.15 mg/m<sup>3</sup>. The SWL<sup>PM-30</sup> (i.e., 30-minute block average Stop Work Level) will be two times the TAL<sup>PM-30</sup>.

The lowest correlated particulate matter Take Action Levels for cadmium and lead calculated from the averaged data for each day from each of the downwind particulate matter monitor and air sampler pairs will be used to calculate a two-week rolling average that will be utilized for the dust monitors' AL<sup>PM</sup> until the next correlation is performed.

### **3.4 Stop Work Level for Wind**

A wind speed Stop Work Level notification will be set on a ten-minute block average using data from an on-Site meteorological station located near the North Corrective Action Management Unit. If the sustained wind speed (the wind speed obtained by averaging the measured values over a ten minute period) exceeds 20 miles per hour, all active soil excavation, stockpiling, and loading must cease until the sustained wind speed declines to 20 miles per hour or lower for at least 10 consecutive minutes. Non-dust producing activities (equipment maintenance, etc.) may still be conducted during these periods.

### **3.5 Particulate Monitors and Wind Data Monitoring and Notifications**

#### **3.5.1 Particulate Monitors**

The data obtained from the particulate monitors will be monitored at a remote location by Field Data Solutions (FDS). FDS hosts and manages a computer-based monitoring system which will provide Take Action and Stop Work Level notifications to both field and management personnel on a real time basis as well as provide real time access to values from each instrument. Each of the E-BAM monitors will be equipped with a wireless modem to transmit data. Cellular communication gateways will be installed at the FOP to act as central communication hubs.

#### **3.5.2 Wind Speed and Direction Data Monitoring**

Wind speed and direction will be monitored using data from an on-Site meteorological station located near the North Corrective Action Management Unit. The data will be transmitted to FDS directly via telemetry. This data will be integrated with the FDS monitoring system to provide Stop Work Level notifications to both field and management personnel on a real time basis as well as provide real time access to the current wind direction.

### 3.5.3 Notifications

Notifications of exceedances of the particulate or wind speed Take Action or Stop Work Levels at the downwind monitors will be sent via text message to field personnel. Notifications to the field office will be sent via email. The notifications will be sent to the contractor's on-site Project Manager and Air Monitoring Technician. The notifications will be sent as a Take Action Level notification or a Stop Work Level notification. The Air Monitoring Technician will be the primary individual responsible for monitoring the notifications and ordering implementation of dust mitigation procedures. However, both of these individuals will have the authority to order implementation of dust mitigation procedures, if needed.

### 3.5.4 Stop Work Criteria for Monitors

If the signal from either the downwind particulate monitors or the on-Site meteorological station located near the North Corrective Action Management Unit is lost for five minutes or more, all dust generating activities involving contaminated soil or waste will be suspended until the downwind particulate monitors and the meteorological station are operational and the signal to the FDS system is re-established.

## **3.6 Dust Suppression Measures**

### 3.6.1 Particulate Matter Take Action Levels

If the 30-minute average  $PM_{10}$  concentration at a downwind monitor exceeds or is equal to the Take Action Levels presented in Table 1 ( $TAL^{PM-30}$ ), the contractor will immediately implement increased dust suppression activities as described in the FOP/RCA Dust Control Plan.

### 3.6.2 Particulate Stop Work Levels

If the one-hour (60-minute) average or thirty-minute (30-minute) average  $PM_{10}$  concentration at a downwind monitor exceeds or is equal to the applicable Stop Work Level ( $SWL^{PM-60}$  or  $SWL^{PM-30}$ ) presented in Table 1, the contractor will immediately stop all dust-generating activities involving contaminated soil or waste. During the work stoppage period (minimum 15 minutes), the contractor must make dust suppression adjustments to reduce airborne particulate matter concentrations below the Take Action Level concentration for particulate matter. The dust suppression adjustments are described in the FOP/RCA Dust Control Plan.

After dust suppression adjustments have been implemented (minimum 15-minute period), the work may resume. During the first 30 minutes after resumption of work activities, the air monitoring technician will continuously monitor the dust levels utilizing the real time data sent to the on-site computer to ensure the dust suppression adjustments are effective. Adjustments to dust suppression activities will be made if needed. If particulate matter Stop Work Levels are exceeded at a downwind particulate matter monitor twice in one work day, the contractor must immediately stop work for the remainder of that work day and design and implement a more effective dust control program prior to resuming work the following work day. During this period, equipment maintenance and other non-dust-producing activities may be performed.

### 3.6.3 Visible Dust

If visible dust is present in the active work zone, increased wetting of the area using water trucks or spray misters will be implemented. If visible dust is observed leaving the active work zone, work will stop until additional dust control measures are implemented as described in the FOP/RCA Dust Control Plan. This criteria also applies to excavation of sediment in Stewart Creek, although it is unlikely to occur because it is naturally wetted and therefore unlikely to generate dust. In addition, stockpiles of sediment will be covered when they are not actively being added to or loaded and trucks transporting sediment will be covered during transport.

## **4.0 AIR SAMPLES COLLECTED FOR LABORATORY ANALYSES**

### **4.1 Metals Analyses**

Air samples will be collected upwind and downwind of remediation activities and the RCA for laboratory analyses of both lead and cadmium during activities involving contaminated soil or waste using high volume (10 liters per minute) particulate matter air samplers. The samples will be collected approximately 2-3 feet away from the E-BAMs to mitigate any air-flow disturbances that may be caused by the E-BAM enclosure.

This analytical data will be correlated with the real-time particulate matter concentration data collected by the E-BAM monitors on a weekly basis, provided validated sampling results are received in a timely manner, and at a minimum every two weeks. Two weeks of analytical data will be correlated with the corresponding real-time particulate matter concentration data collected by the E-BAM monitors to establish a two-week rolling average.

The lowest correlated particulate matter Take Action Levels for cadmium and lead calculated from the averaged data for each day from each of the downwind particulate matter monitor and air sampler pairs will be used to calculate a two-week rolling average that will be utilized for the dust monitors' AL<sup>PM</sup> until the next correlation is performed.

Air samples for these metals analyses will be collected by the contractor on the first work day of every week and every other day through the week during remediation activities involving contaminated soil or waste. Samples will not be collected on days when remediation activities are not occurring.

Air samples for metals analyses will be collected over a full working shift (typically eight to ten hours) using sampling pumps capable of operating at 10 liters per minute. The intakes of the filter cassettes will be positioned adjacent to the inlet of the collocated E-BAM air inlet. The inlet port of the filter will be in a downward position. The air sampling interval may be less than eight hours in the event of inclement weather during the air sampling period (such as severe thunderstorms or rain which stop the work activities). Air samples will be collected by attaching laboratory-provided air sample filter cartridges (0.8-µm mixed cellulose ester membrane filter cartridges) to the pump and setting the air sample filter cartridges approximately five feet above ground level at the E-BAM monitor locations. When the downwind air samplers are relocated with the E-BAM monitors due to a 90-degree change in the prevailing wind direction, averaged over a 30-minute period, the air samplers will be shut off during the relocation and started in the new location without a filter change. The air sample pumps will be set at a flow rate of approximately ten liters per minute, thereby resulting in an air sample volume of approximately 4,800-6,000 liters per air sample.

Following air sample collection, the air sample cartridges will be securely capped, labeled, and delivered with chain of custody documentation to ALS Laboratory Group, in Salt Lake City, Utah, for analysis of lead and cadmium. ALS is accredited by the TCEQ for analysis of environmental samples and is accredited by the American Industrial Hygiene Association (AIHA) for analysis of air samples and lead in soil, dust, paint and air. Laboratory analyses on an expedited 72-hour turnaround will be requested. Metals will be analyzed using NIOSH Method 7303. Test method details are provided in Attachment 2. This method is specifically accredited by the AIHA.

Laboratory data will be validated by Exide's consultant (Golder Associates, Inc.) and provided to the TCEQ within two business days of receipt of validated analytical results, excluding the day that the results are received. If data is received that cannot be validated, an email notification will be provided to the TCEQ within two business days with a brief description of the issue(s). Upon receipt of the corrected data from the laboratory, Exide's consultant will validate and provide the data to TCEQ as described above.

## **4.2 Metals Concentrations Take Action Levels**

Following receipt of the lead and cadmium analytical laboratory reports, the analytical data from the downwind air samplers will be compared to the lead and cadmium Take Action Levels shown on Table 1. If either concentration in the downwind samples exceeds the relevant Take Action Level, the contractor will immediately implement increased dust suppression activities as described in the FOP/RCA Dust Control Plan.

## **4.3 Metals Concentrations Stop Work Levels**

Following receipt of the lead and cadmium analytical laboratory reports, the analytical data from the downwind air samplers will be compared to the Stop Work Levels shown on Table 1. The Stop Work Level for lead has been derived from the current (2008) NAAQS for lead, adjusted as appropriate to address the differences in averaging periods. According to Appendix D, "Averaging Period Concentration Estimates," in EPA-454/R-92-024 "Workbook of Screening Techniques for Assessing Impacts of Toxic Air Pollutants (Revised)" dated December 1992, the appropriate multiplying factor in converting eight-hour averaged concentrations to three-month averages is 0.14. Accordingly, the NAAQS value of 0.15  $\mu\text{g}/\text{m}^3$  is divided by 0.14, yielding 1.07  $\mu\text{g}/\text{m}^3$  average concentration as the lead Stop Work Level. For cadmium, the TCEQ short term ESL of 0.1  $\mu\text{g}/\text{m}^3$  average concentration is the Stop Work Level. The Take Action Levels for the lead and cadmium sample results are set at 75% of the Stop Work Levels.

If the lead or cadmium Stop Work Levels are exceeded, the contractor will immediately stop dust-generating activities involving contaminated soil or waste and design and implement a more effective dust control program prior to resuming work. The additional dust suppression activities are described in the FOP/RCA Dust Control Plan.

**Table 1** provides, in chart form, the default action levels and responses for particulate matter, lead, and cadmium. When sufficient site data has been collected following the start of the remediation activities, the action and stop work levels for particulate matter will be updated based upon the relationship between dust and lead concentrations utilizing the formulas in Section 3.3.1 and based upon the dust and cadmium concentrations utilizing the formulas in Section 3.3.2. Take Action and Stop Work levels will be updated weekly, provided timely sampling results are received, and at least every two weeks based upon the relationship between dust and measured metals concentrations. Work performed to date on other portions of the FOP have shown that the current concentrations used for stop work levels are an appropriate default.

| <b>TABLE 1</b>                            |                                                |                                |                                                                                                                                                                                           |                                                                                                                                                                                                                                      |
|-------------------------------------------|------------------------------------------------|--------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>Initial Action Levels and Response</b> |                                                |                                |                                                                                                                                                                                           |                                                                                                                                                                                                                                      |
| <b>Contaminant of Concern</b>             | <b>Monitoring Method</b>                       | <b>Frequency of Monitoring</b> | <b>Take Action Level to Increase Dust Suppression / Emission Controls</b>                                                                                                                 | <b>Stop Work Level</b>                                                                                                                                                                                                               |
| Particulate Matter                        | Visual                                         |                                | Visible dust within the active Work Zone – Implement additional dust control measures.                                                                                                    | Dust leaving the Placement Zone perimeter – Stop Work. Implement additional dust control measures.                                                                                                                                   |
|                                           | PM <sub>10</sub> Downwind Particulate Monitors | 30-minute block average        | PM <sub>10</sub> > or equal TAL <sup>PM-30</sup><br>Default TAL <sup>PM-30</sup> - 0.1 mg/m <sup>3</sup> average 30-minute concentration –<br>Implement additional dust control measures. | PM <sub>10</sub> > or equal SWL <sup>PM-30</sup><br>Default SWL <sup>PM-30</sup> (two times TAL <sup>PM-30</sup> ) - 0.2 mg/m <sup>3</sup> average 30-minute concentration<br>Stop Work. Implement additional dust control measures. |
|                                           | PM <sub>10</sub> Downwind Particulate Monitors | 60-minute block average        |                                                                                                                                                                                           | PM <sub>10</sub> > or equal SWL <sup>PM-60</sup><br>Default SWL <sup>PM-60</sup> - 0.1 mg/m <sup>3</sup> average hourly concentration<br>Stop Work. Implement additional dust control measures.                                      |
| Lead                                      | High Volume Particulate Samplers               | Three days per week            | 0.8 µg/m <sup>3</sup> – Implement additional dust control measures.                                                                                                                       | 1.07 µg/m <sup>3</sup> average concentration.                                                                                                                                                                                        |
| Cadmium                                   | High Volume Particulate Samplers               | Three days per week            | 0.075 µg/m <sup>3</sup> – Implement additional dust control measures.                                                                                                                     | 0.1 µg/m <sup>3</sup> average concentration (TCEQ short term Cd ESL).                                                                                                                                                                |

## **5.0 REPORTS**

Daily Dust Concentration (PM<sub>10</sub>) and Wind Speed and Direction summary reports will be prepared by FDS. These summary reports will include the average 30-minute net block average PM<sub>10</sub> results for each downwind E-BAM instrument and the 30-minute block average wind speed and direction data. Take Action or Stop Work Level exceedances and the dust suppression adjustment activities implemented in response will be documented in the summary reports.

Summary reports must be completed within two business days of the receipt of analytical data for the monitoring day being reported. The data will be validated by Golder Associates, Inc. Summary reports of the validated data will be provided to the TCEQ within two business days of receipt of verifiable results, excluding the day that the results are received. If data are received that are not able to be validated, an email notification will be provided to the TCEQ with a brief description of the issue(s). The summary report with the corrected data will be resubmitted to Golder Associates, Inc., followed by validation. The summary report with validated data will then be submitted to TCEQ as described above.

## **6.0 QUALITY ASSURANCE / QUALITY CONTROL**

Quality assurance (QA) refers to the planned and systematic actions necessary to provide adequate confidence that a product or service will satisfy a given requirement for quality. QA is applied to location and equipment selection, equipment acquisition and installation, routine site operation, and data processing and reporting.

Quality control (QC) refers to the operational techniques and activities that are used to fulfill requirements for quality. QC procedures applied at each step provide checks for acceptable conditions with corrective procedures specified when necessary.

The purpose of QC procedures is to assess and document data quality and to define remedial corrective actions when operating conditions exceed pre-established limits. Routine QC procedures are designed to focus on areas most likely to have problems, based on experience and guideline documents. Table 2 shows the frequency of audits and routine QC measures for the air quality study. The following subsections describe the QC, calibration, and auditing procedures to be used during this project.

| <b>Table 2</b>                                                      |                                                                                                                                                              |                                                                                                                                                                                                                                                                    |
|---------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>Schedule of Audits, Calibrations, and Quality Control Checks</b> |                                                                                                                                                              |                                                                                                                                                                                                                                                                    |
| <b>Frequency</b>                                                    | <b>Activity</b>                                                                                                                                              | <b>Acceptable Limits</b>                                                                                                                                                                                                                                           |
| Prior to delivery, prior to start of the project                    | Calibration of E-BAM monitors                                                                                                                                |                                                                                                                                                                                                                                                                    |
| Prior to the start of work each week                                | Routine checks of E-BAM Monitors (tape checks, zero checks, leak check; clean size selective inlets; verify clock settings; housekeeping) and air samplers   | Leak check >1.0 L/min requires nozzle and vane cleaning<br><br>Leak check >1.5 L/min invalidates data to previous leak check                                                                                                                                       |
| Every three weeks                                                   | Flow rate calibration (perform barometric pressure sensor audit, temperature sensor audit prior to flow test), membrane test and pump test of E-BAM monitors | Flow rate $\pm 0.1$ L/min of traceable reference standard audit device<br><br>Barometric pressure audit – calibrate E-Bam<br><br>Temperature audit – calibrate E-Bam<br><br>Membrane test – pass/fail<br><br>Pump test – pass/fail<br><br>Membrane check pass/fail |
| Every tape change and at least monthly                              | Cleaning nozzle and vane of E-BAM monitors (leak check is required anytime detector tape is removed or a new tape is installed)                              | Leak check >1.0 L/min requires nozzle and vane cleaning<br><br>Leak check >1.5 L/min invalidates data to previous leak check                                                                                                                                       |
| Weekly                                                              | Field blanks collected for air samplers                                                                                                                      | See 6.4 below                                                                                                                                                                                                                                                      |
| Monthly                                                             | Trip blanks collected for air samplers                                                                                                                       | See 6.4 below                                                                                                                                                                                                                                                      |
| Yearly                                                              | Calibration for on-site meteorological station, including zero calibration for wind speed                                                                    | See 6.3 below                                                                                                                                                                                                                                                      |

## **6.1 Particulate Monitors**

### **6.1.1 Quality Control**

The E-BAM beta detectors are calibrated at the factory. The beta detector calibrations remain fixed for the life of the unit, and no user adjustments are required. Each unit has test membranes that are placed in the beta particle pathway to verify performance of the detector. The test membranes are thin sheets of material that absorb a fraction of beta particles equivalent to a known mass of particulate matter. Each

instrument has an individually matched membrane, and the factory-provided equivalent mass reading is stored in the instrument. The reference membrane tests are manually performed prior to the start of the project and at least every three weeks (the manufacturer recommends a frequency of one or two times per year for the E-BAM). The units are also equipped with zero-check inserts that are used in the same manner as the reference membranes. The zero check insert test will be performed prior to the start of the project, and prior to the start of work each week.

QC flow checks will be performed by the contractor every three weeks to ensure that the correct sample flow rate is being maintained to provide proper particle size separation. The flow rate calibration is performed using a traceable reference standard flow audit device (BGI deltaCal® or equivalent). The barometric pressure and ambient temperature must be audited and calibrated, if necessary, prior to the flow check. The ambient temperature and barometric pressure indicated on the traceable reference standard flow audit device is compared to the ambient temperature and barometric pressure indicated on the E-BAM. If necessary, the ambient temperature and barometric pressure indicated on the traceable standard flow audit device is entered into the E-BAM to correct the E-BAM internal ambient temperature and/or barometric pressure sensor reading. The flow rate calibration can then be performed. The E-BAM internal flow rate is audited based upon the flow rate indicated by the traceable reference standard flow audit device. If necessary, the E-BAM flow rate indicated on the traceable standard flow audit device is entered into the E-BAM to correct the E-BAM internal flow sensor reading. A pump test will also be performed every three weeks.

The E-BAM particle size selective inlets are designed to function at a flow rate of 16.7 L/min to maintain proper particle separation. Cleaning of the size selective inlets on the particulate monitors will be conducted prior to the start of each work week. The larger particles that are removed from the air flow are captured inside the PM<sub>10</sub> inlet heads. To maintain proper operation of the inlets, the particle deposits must be cleaned periodically. A leak check will be performed weekly and when the tape is removed or a new tape is installed. The nozzle and vane beneath the filter tape will be cleaned each time the tape is changed but at a minimum of once per month.

## **6.2 Air Samplers**

### **6.2.1 Quality Control**

Field and trip blank quality control samples will be collected. Field blank samples assess the possible contamination introduced by field sampling procedures, sampling media, sampling equipment, or shipment of the samples. Trip blanks verify the cleanliness of the sampling media.

The field blank will be shipped to the field, prepared, and handled as the other samples, and returned to the laboratory, without drawing air through the air sampler, for analysis. One field blank will be collected each week for metals analysis. The trip blank will be shipped to the field, left sealed in its packaging, and then returned to the laboratory for analysis. One trip blank will be analyzed per month.

### **6.2.2 Quality Assurance**

Precision and accuracy checks are both elements of QA. Precision checks are a measure of agreement among individual measurements of the same parameter, usually under prescribed similar conditions. Accuracy is the degree of agreement between an accepted reference measurement and the field measurement. Accuracy may be expressed as a total difference, or as a percentage of the reference value, or as a ratio. Precision checks are performed as collocated measurements.

Accuracy of ambient air sampling equipment is measured in terms of the accuracy of the flow rate measurement. Accurate determination of the air volume drawn through the air sampler is essential to the concentration calculation. Flow rates of the air samplers will be determined pre- and post-sampling using calibrated equipment appropriate to the sampling device.

Preventive maintenance will be part of the air samplers' QA program. Preventive maintenance is a combination of preventive and remedial actions taken to prevent or correct failure of the monitoring systems. Preventive maintenance for the air samplers includes inspection and cleaning of the inlets.

### **6.3 Meteorological Station**

The on-Site meteorological station will be zero checked once per year (or more often if values do not appear reasonable based on experience or comparison to a local weather source). The check for wind speed will include placing a small container over the sensor to zero check the wind measurement. The check for other meteorological data will be done by comparing readings to a local weather source. Once every two years the meteorological station will be calibrated by the manufacturer (the unit will be removed from service and shipped off-Site).

### **6.4 Laboratory Validation**

Data validation is used to interpret the quality of the analytical data received from the laboratory. The quality of the data is determined through evaluation of both the field and laboratory quality control samples. Data validation procedures determine whether individual project data are useable, useable with qualification, or unusable. Data will be reviewed in accordance with guidelines presented in the EPA's *National Functional Guidelines for Inorganic Superfund Data Review* (2010).

The Laboratory will submit the analytical data and supporting quality assurance/quality control data to Exide's consultant, Golder Associates, Inc., for validation. The validation review will consist of a Level II review which includes the following:

- Blank samples (i.e., trip, method, equipment, field, etc.) are reviewed for detections which may indicate whether field or laboratory handling may have cross-contaminated samples causing false positive or high-biased data.
- Spike recovery samples (i.e., laboratory control sample, surrogate, or matrix spike) are reviewed to evaluate accuracy in the laboratory's ability to recover known concentrations that were intentionally spiked into the quality control samples.
- Duplicate samples (field and/or laboratory-prepared) are evaluated to determine precision, which is the level of agreement among individual measurements.

In addition to the above quality control samples, verification of appropriate analytical methods, reporting limits, sample preservation, and holding times are also reviewed to determine data usability.

Any potential bias (high or low) or cross-contamination observed as a result of the data review is usually addressed by addition of data qualifiers. These typically include one of the following: a non-detect (U) flag for blank detections resulting in potential cross-contamination; an estimated (J) flag for results that could be high or low biased due to accuracy or precision issues; rejection of data (R) due to results grossly outside their respective control limits or questionable data.

### **6.5 Dust Concentration, Wind Speed and Direction Report Validation**

The Daily Dust Concentration and Wind Speed and Direction summary reports will be prepared by FDS. The summary reports will be reviewed by Exide's consultant, Golder Associates, Inc., for validation. The review will include review of error reports, previous instrument flow and leak check information, and review of the data received to insure the data being reported is from the instruments being used at the site.

## **6.6 Sample Information Management**

The sample information management system for the study will be based on a uniform sample identification system. Each sample will receive a unique ID that is based on the unique combination of project, sampling date, sampling location and the Serial Number of the E-BAM Monitor with which the sample is associated.

The sample ID will be structured as follows:

FOPR-YYMMDD-LOC-XXX[-QQ], where

FOPR = Project (FOP Remediation)

YYMMDD = Sampling date (e.g., 11/01/2012 = 121101)

LOC = Sample Location (e.g. DW = Downwind)

XXX = E-BAM Monitor Sample Association – Last 3 digits of Serial Number,

QQ = Optional QA sample flag (TB = trip blank, FB = field blank, SC = duplicate)

For example, a sample collected at a downwind station on November 1, 2017, would be identified as FOPR 171101 DW 123.

## **7.0 POINTS OF CONTACT**

Concerns regarding activities conducted at the Exide Technologies Frisco Recycling Center should be addressed to the following points of contact:

Exide:

Eduardo Salazar

P.O. Box 250

Frisco, Texas 75034

Ph: 972-335-2121

Cell: 972-786-5404

eduardo.salazar@exide.com

Texas Commission on Environmental Quality:

Margaret Ligarde

Office of Legal Services

MC-173

P.O. Box 13087

Austin, Texas 78711

Ph: 512-239-3426

Fax: 512-239-0330

margaret.ligarde@tceq.texas.gov

City of Frisco:

Mack Borchardt

City of Frisco

6101 Frisco Square Blvd.

Frisco, Texas 75034

Ph: 972-292-5127

Fax: 972-292-6319

mborchardt@friscotexas.gov

## **ATTACHMENTS**

**ATTACHMENT 1**

**E-Bam Particulate Monitors**

# E-BAM

## E-BAM is a complete measurement system it comes with the following standard components:

- 8 Channel Datalogger
- Internal DC Vacuum Pump Standard
- Real-Time Concentration
- PM10 Inlet
- Aluminum Tripod
- Ambient Temperature Sensor
- Volumetric Flow Control
- Weatherproof Enclosure
- Filter Temperature Sensor
- Filter RH Sensor
- Filter Pressure Sensor
- Calibration Membrane

## Specifications

|                       |                                                                     |
|-----------------------|---------------------------------------------------------------------|
| Range                 | 0 - 65 mg per cubic meter                                           |
| Accuracy              | 2.5 µg or 10% in 24 hour period                                     |
| Measurement Cycle     | Hourly measurements with 1, 5, 10, 15, or 30 min real-time averages |
| Beta Source           | C14, less than 75 microcurie, Half life of 5730 years               |
| Detector:             | Scintillation probe                                                 |
| Analog Output         | 0-1V, 0-2.5v, 0-5V, selectable hourly or real-time output           |
| Filter Tape           | Continuous glass fiber filter                                       |
| Inlet                 | Compatible with EPA PM10 and PM2.5 inlets                           |
| Flow Rate:            | 16.7 liters per minute, adjustable                                  |
| Flow accuracy         | +/- 2% of reading, volumetric flow controlled                       |
| Sample Pump           | Dual diaphragm type, DC powered, 4000 hr rating                     |
| Alarm Signals         | Filter, flow, power and operation failure                           |
| Input Power           | 12 Volts DC @ 48 Watts max                                          |
| Alarm Contact Closure | 2 Amp @ 240 VAC max                                                 |
| Operating Temperature | -30 Deg C to 50 Deg C                                               |
| Enclosure             | 41 cm x 36 cm x 20 cm, 13kg                                         |

## Options and Accessories

- BX-302 Zero Calibration Kit
- BX-305 Leak check valve
- BX-307 Flow Calibrator
- BX-308 PM2.5 Sharp-Cut Cyclone
- BX-803 TSP Inlet
- EX-034 Wind speed and direction sensor
- EX-121 AC Power supply, 100-240 VAC, 12 VDC output
- EX-593 Ambient RH Sensor
- EX-996 Phone modem kit
- EX-911 Cell modem kit
- 460130 Filter tape, roll
- 9425 Wall mount bracket
- Airsis Satellite modem kit
- External AC Vacuum Pump
- MMP MicroMet Plus Software
- Solar Panel Array



The Met One E-BAM is a portable, real-time beta gauge which is comparable to U.S. EPA methods for PM<sub>2.5</sub> and PM<sub>10</sub> particulate measurements.

The Met One E-BAM has been built to satisfy users, regulators and those from the health community by providing truly accurate, precise, real time measurement of fine particulate matter automatically. In addition, it is rugged, portable, battery operated, and deployable in 15 minutes.

### The E-BAM offers the following advanced features:

1. Accuracy and precision consistent with U.S. EPA requirements for Class III PM<sub>2.5</sub> and PM<sub>10</sub> measurement.
2. Real-time, accurate results without correction factors, regardless of season or geographic location.
3. True ambient sampling provides accurate measurement of semi-volatile nitrates and organic compounds.
4. Lightweight, rugged construction is easily mounted on a tripod in minutes.
5. All-weather construction allows for true ambient sampling.
6. Operates on AC or DC power. Battery and Solar options available upon request.



**Met One Instruments, Inc.**

Corporate Sales & Service: 1600 Washington Blvd., Grants Pass, Oregon 97526 • Tel (541) 471-7111 • Fax (541) 471-7116  
Regional Sales & Service: 3206 Main Street, Suite 106, Rowlett, Texas 75088 • Tel (972) 412-4747 • Fax (972) 412-4716  
<http://www.metone.com> • [metone@metone.com](mailto:metone@metone.com)



**Met One Instruments, Inc.**

## Continuous Monitoring

The E-BAM automates particulate measurement by continuously sampling and reporting concentration data. Data records are updated every minute. E-BAM eliminates the old process of filter collection and manual filter weighing, and eliminates the need for more expensive, high maintenance instruments. Today, with the adaptation of Beta Attenuation to ambient monitoring this process became simple, streamlined, and inexpensive.

### About Accuracy

Real-time accurate, reliable, and repeatable measurement of ambient fine particulate matter has been the elusive goal of environmental regulators and health professionals for many years. Met One Instruments has developed advanced particulate monitoring instrumentation which is reliable, and is easy to operate. It will also automatically report results in near real time, eliminating the need for high levels of human intervention.

Because sampling occurs under true ambient conditions semi-volatile organic compounds and nitrates are easily detected thereby avoiding under measurement.

## Continuous Sampling

E-BAM is a lightweight portable instrument that operates directly in hostile environments without an exterior enclosure. E-BAM is a very robust portable sampler system that is easily installed in less than 15 minutes. No other sampler matches the portability and flexibility of the E-BAM.

### Set up

Quick setup of the E-BAM is assured with a series of prompts instructing the installer on the sequence to follow. Then the E-BAM performs a series of self test diagnostics and alerts the installer of any corrective action. Upon completion, the E-BAM automatically places itself in normal operate mode.

### Particulate size selection

Size selective concentration measurements are made using a variety of sampling inlets. The E-BAM may be supplied with TSP (Total Suspended Particulate), PM-10, PM 2.5 or PM 1 inlets. Flow dependent cut points in the size selective inlets are maintained using integral flow meter, pressure sensor and ambient temperature sensor.

The PM-10 inlet removes particles larger than 10 microns, the inlet is not affected by wind speed and wind direction. For PM 2.5 or PM 1 secondary size selection is made using a second downstream inlet.

## Construction etc.

The standard configuration of the E-BAM is a self-contained environmentally sealed aluminum enclosure placed on a rugged tripod. This system can be permanently placed on rooftops, near roads, at industrial sites, or rapidly deployed to monitor emergency situations.

'E-' represents Environment Proof instrument, E-BAM has been specifically designed to work in hostile environments without additional protection.

### Direct Field Reporting

Collecting real time or historical particulate data from a field site has never been easier. Advanced communication options include cellular phone, Line of Sight Radio, and for very remote sites, satellite communications are now available. E-BAM also supports the full line of standard MET ONE options, such as phone modem, and direct communications to a portable computer.

E-BAM data is recorded internally and may be retrieved using one of the communication options, or data may be forwarded to third party data acquisition system.

MicroMet Plus Software supports the E-BAM and provides a complete communication, data base and reporting modules with charting. Comet data retrieved software is included.

### Digital, Analog and Alarm Outputs

The E-BAM provides both continuous digital and analog outputs. Analog output is selectable to several full-scale voltages. Digital output is supplied as RS-232.

### Reporting modes

The internal data logger can store up over 182 days of concentration data at one hour sample times, and collect data from eight other measurements at the same time! Both digital and analog outputs are included to enable users to connect to other data recording systems.

### Easy to Operate

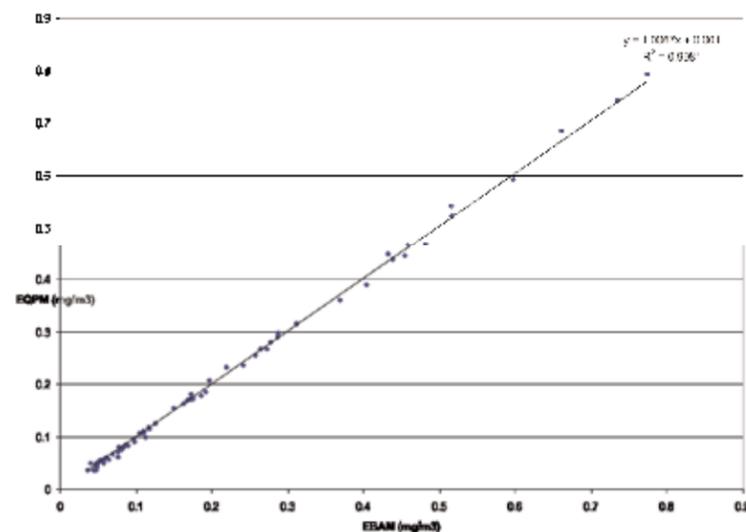
E-BAM has been programmed to operate at all times, except during calibration verification. Current data, historical data, and status information are available at all times without interrupting normal E-BAM operation.

### Data Validation

The operator may select various criteria for data validation, including deviation from rolling average, high value excursions, power failure and others. If an error occurs it is entered into the error log with date, time and type of error.



EPA Designated Method EQPM-0798-122 VS EBAM



**ATTACHMENT 2**  
**NIOSH Method 7303**

**ELEMENTS by ICP  
(Hot Block/HCl/HNO<sub>3</sub> Digestion)**

**7303**

MW: Table 1

CAS: Table 2

RTECS: Table 2

**METHOD: 7303, Issue 1**

**EVALUATION: PARTIAL**

**Issue 1: 15 March 2003**

**OSHA:** Table 2

**PROPERTIES:** Table 1

**NIOSH:** Table 2

**ACGIH:** Table 2

|                           |          |            |            |           |      |
|---------------------------|----------|------------|------------|-----------|------|
| <b>ELEMENTS:</b> aluminum | cadmium  | indium     | nickel     | strontium | zinc |
| antimony*                 | calcium  | iron       | palladium  | tellurium |      |
| arsenic                   | chromium | lead*      | phosphorus | thallium  |      |
| barium                    | cobalt   | magnesium  | platinum   | tin*      |      |
| beryllium                 | copper   | manganese  | potassium  | titanium  |      |
| bismuth*                  | gallium  | molybdenum | selenium   | vanadium  |      |
| boron                     | gold     | neodymium  | sodium     | yttrium   |      |

\* With certain restrictions (see Table 3)

| SAMPLING                  |                                                    | MEASUREMENT                   |                                                                      |
|---------------------------|----------------------------------------------------|-------------------------------|----------------------------------------------------------------------|
| <b>SAMPLER:</b>           | FILTER<br>(0.8- $\mu$ m, cellulose ester membrane) | <b>TECHNIQUE:</b>             | INDUCTIVELY COUPLED ARGON<br>PLASMA, ATOMIC EMISSION<br>SPECTROSCOPY |
| <b>FLOW RATE:</b>         | 1 to 4 L/min                                       | <b>ANALYTE:</b>               | See element list above                                               |
| <b>VOL-MIN:</b>           | Table 1                                            | <b>REAGENTS:</b>              | Conc. HCl, 1.25 mL; and conc. HNO <sub>3</sub> ,<br>1.25 mL          |
| <b>-MAX:</b>              | Table 1                                            | <b>FINAL SOLUTION:</b>        | 5% HCl and 5% HNO <sub>3</sub> , 25 mL                               |
| <b>SHIPMENT:</b>          | Routine                                            | <b>WAVELENGTH:</b>            | Element and instrument specific                                      |
| <b>SAMPLE STABILITY:</b>  | Stable                                             | <b>BACKGROUND CORRECTION:</b> | Spectral wavelength shift                                            |
| <b>BLANKS:</b>            | 2 to 10 field blanks per set                       | <b>CALIBRATION:</b>           | Elements in 5% HCl, 5% HNO <sub>3</sub>                              |
| <b>ACCURACY</b>           |                                                    | <b>RANGE:</b>                 | LOQ to 50,000 $\mu$ g/sample [1]                                     |
| <b>RANGE STUDIED:</b>     | 5,000 to 50,000 $\mu$ g/sample                     | <b>ESTIMATED LOD:</b>         | Varies with element; Table 1                                         |
| <b>BIAS:</b>              | Not determined                                     | <b>PRECISION (\$):</b>        | Not evaluated                                                        |
| <b>OVERALL PRECISION:</b> | Not determined                                     |                               |                                                                      |
| <b>ACCURACY:</b>          | Not determined                                     |                               |                                                                      |

**APPLICABILITY:** The working range of this method is up to 100 mg/m<sup>3</sup> for each element in a 500-L sample (the minimum range depends on the LOD for each sample; see Table 1). The analysis is not compound specific. Certain elemental compounds are known to be acceptable or unacceptable by this method (see Table 3). For unverified compounds, a test run should be conducted using a known amount of the compound in question to determine acceptability.

**INTERFERENCES:** Interferences are spectral in nature and are accounted for by choosing appropriate wavelengths, applying interelement correction factors, and background correction.

**OTHER METHODS:** Alternative, more sensitive methods exist for some elements by graphite furnace atomic absorption spectroscopy. This method is similar to NIOSH Method 7301, differing only in the use of the hot block for digestion of the sampler.

**REAGENTS:**

1. Hydrochloric acid,\* conc., ultra pure.
2. Nitric acid,\* conc., ultra pure.
3. Calibration stock solutions, 50-1000 µg/mL. Commercially available single element solutions or multielement solutions prepared as instructed by the instrument manufacturer.
4. Argon, prepurified.
5. Distilled, deionized, Type II water.
6. Diluting solution: 5% HCl : 5% HNO<sub>3</sub>. To about 600 mL of deionized water in a 1-L volumetric flask, slowly add 50 mL conc. HCl and 50 mL conc. HNO<sub>3</sub>. Dilute to the mark with deionized water.

\* See SPECIAL PRECAUTIONS

**EQUIPMENT:**

1. Sampler: cellulose ester membrane filter, 0.8-µm pore size, 37-mm diameter; in cassette filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Inductively coupled argon plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest.
4. Hot block apparatus at 95 °C.
5. Digestion vessels and caps, 50-mL.
6. Watchglasses.
7. Pipettes, electronic and mechanical.
8. Regulator, two-stage, for argon.
9. Forceps.

---

**SPECIAL PRECAUTIONS:** Concentrated acids are powerful oxidizers, toxic, and corrosive liquids. Wear protective clothing and work in a fume hood.

---

**SAMPLING:**

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of 200 to 2000 L for TWA measurements. Do not exceed a filter loading of approximately 2 mg total dust.

**SAMPLE PREPARATION:**

3. Open the cassette filter holder and with forceps remove the sample filter. Fold the filter into quarters taking care not to lose any sample, and transfer to a clean, 50-mL hot block digestion tube.
4. Add 1.25 mL HCl. Cover with a plastic watchglass. Place in the hot block and heat at an internal temperature of 95 °C for 15 minutes.  
NOTE: The internal temperature may vary from the digital readout. Calibrate the hot block prior to digestion.
5. Remove the sample from the hot block and cool for 5 minutes. Remove watchglass and add 1.25 mL HNO<sub>3</sub>. Replace watchglass and return to hot block at 95 °C for 15 minutes.
6. Remove the sample from the hot block and cool for at least 5 minutes. Rinse watchglass into the sample container and discard watchglass.
7. Dilute to 25-mL final volume with distilled, deionized Type II water.

**CALIBRATION AND QUALITY CONTROL:**

8. Calibrate the spectrometer according to the manufacturer's recommendations. Use standards consisting of the same 5% HCl : 5% HNO<sub>3</sub> matrix as the samples.
9. Analyze a standard every 10 samples.
10. Analyze a media blank every 20 samples, and a reagent blank every 10 samples.
11. Analyze a set of two laboratory control samples every 40 samples of a given matrix for a given analyte.
12. Check recoveries with at least two spiked media blanks per ten samples.  
NOTE: In the determination of lead, there may be a measurement interference (for example, samples with high aluminum levels). More recent instruments have a correction for this.

**MEASUREMENT:**

13. Set spectrometer to conditions specified by manufacturer.
14. Analyze standards, samples and quality control checks.

NOTE: If the elemental value for a sample is above the linear range of the element(s) in question, dilute the sample solution with 5% HCl: 5% HNO<sub>3</sub> diluting solution, reanalyze and apply the appropriate dilution factor in the calculations.

**CALCULATIONS:**

15. Obtain the solution concentrations for the sample, C<sub>s</sub> (µg/mL), and the average media blank, C<sub>b</sub> (µg/mL), from the instrument.
16. Using the solution volumes of sample, V<sub>s</sub> (mL), and media blank, V<sub>b</sub> (mL), calculate the concentration, C (mg/m<sup>3</sup>), of each element in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \text{mg} / \text{m}^3$$

NOTE: µg/L = mg/m<sup>3</sup>

**EVALUATION OF METHOD:**

The method was evaluated for all elements and compounds listed in Table 1 and Table 2 between 1999 and 2001 using known amounts of bulk material [4]. Evaluation is ongoing for additional elements and compounds. The limits of detection and quantitation were also determined for each element. Two ICP instruments were used in the evaluation, a Thermal Jarrell Ash Model 61E [5] and a TJA IRIS [6], operated according to the manufacturer's instructions.

**REFERENCES:**

- [1] WOHL [2001]. Metals validation using hot block digestion, Unpublished data. Wisconsin Occupational Health Laboratory, Madison, WI.
- [2] NIOSH [1994]. Method 7300: Elements by ICP, NIOSH Manual of Analytical Methods, Fourth Edition, Issue 2, Aug. 15, 1994.
- [3] WOHL [2001]. Metals Manual 2001, WOHL Internal Document, Updated Apr. 1, 2001. Wisconsin Occupational Health Laboratory, Madison, WI.
- [4] WOHL [2001]. WOHL General Operations Procedures Manual, WOHL Internal Document, Updated 2001. Wisconsin Occupational Health Laboratory, Madison, WI.
- [5] Thermal Jarrell Ash [1991]. ICAP 61E Plasma Spectrometer Operator's Manual, Thermal Jarrell Ash Corp., Part No. 128832-01, Feb., 1991.
- [6] Thermal Jarrell Ash [1997]. IRIS Plasma Spectrometer User's Guide, Thermal Jarrell Ash Corp., Part No. 135811-0, Feb. 4, 1997.

**METHOD WRITTEN BY:**

Jason Loughrin, Lyle Reichmann, Doug Smieja, Shakker Amer, Curtis Hedman  
Wisconsin Occupational Health Laboratory (WOHL).

TABLE 1: ANALYTE INFORMATION FOR VALID ELEMENTS AND COMPOUNDS

| Analyte                                   | Properties |         | LOD<br>(µg/mL) | LOQ<br>(µg/mL) | Estimated<br>LOQ<br>(µg/sample)* | Minimum**<br>air vol. (L) | Maximum***<br>air vol. (L) |
|-------------------------------------------|------------|---------|----------------|----------------|----------------------------------|---------------------------|----------------------------|
|                                           | MW         | MP (°C) |                |                |                                  |                           |                            |
| Al                                        | 26.98      | 660     | 0.111          | 0.37           | 9.25                             | 2                         | 10,000                     |
| As                                        | 74.92      | 817     | 0.009          | 0.03           | 0.075                            | 8                         | 5,000,000                  |
| Au                                        | 196.97     | 10.63   | 0.015          | 0.05           | 1.25                             | 1                         | 3,300                      |
| B                                         | 10.81      | 2177    | 0.0094         | 0.0283         | 0.71                             | 1                         | 3,300                      |
| Ba                                        | 137.34     | 3.51    | 0.0018         | 0.006          | 0.15                             | 1                         | 100,000                    |
| Be                                        | 9.01       | 2178    | 0.00075        | 0.0025         | 0.062                            | 35                        | 25,000,00                  |
| Bi                                        | 208.98     | 271     | 0.025          | 0.085          | 2.12                             | 1                         | 10,000                     |
| Ca                                        | 40.08      | 842     | 0.099          | 0.33           | 8.25                             | 2                         | 10,000                     |
| CaO                                       | 56.08      | 2927    | 0.139          | 0.462          | 11.6                             | 3                         | 10,000                     |
| Cd                                        | 112.4      | 321     | 0.0037         | 0.012          | 0.30                             | 3                         | 500,000                    |
| Co                                        | 58.93      | 1495    | 0.003          | 0.011          | 0.27                             | 3                         | 500,000                    |
| Cr                                        | 52.00      | 1890    | 0.009          | 0.03           | 0.75                             | 8                         | 500,000                    |
| Cu                                        | 63.54      | 1083    | 0.020          | 0.060          | 1.50                             | 15                        | 500,000                    |
| Fe                                        | 55.85      | 1535    | 0.070          | 0.20           | 5.00                             | 1                         | 5,000                      |
| Fe <sub>2</sub> O <sub>3</sub><br>(as Fe) | 159.69     | 1462    | 0.070          | 0.20           | 5.00                             | 1                         | 5,000                      |
| Ga                                        | 69.72      | 29.75   | 0.03           | 0.09           | 2.25                             | 1                         | 3,300                      |
| In                                        | 114.82     | 156.3   | 0.015          | 0.05           | 1.25                             | 15                        | 500,000                    |
| Mg                                        | 24.31      | 651     | 0.047          | 0.14           | 3.50                             | 1                         | 10,000                     |
| MgO                                       | 40.32      | 2825    | 0.078          | 0.23           | 5.75                             | 5                         | 33,000                     |
| Mn                                        | 54.94      | 1244    | 0.0012         | 0.004          | 0.10                             | 0.05                      | 10,000                     |
| Mo                                        | 95.94      | 651     | 0.0072         | 0.024          | 0.60                             | 0.5                       | 10,000                     |
| Nd                                        | 92.906     | 2477    | 0.01           | 0.03           | 0.75                             | 0.1                       | 3,300                      |
| Ni                                        | 58.71      | 1453    | 0.012          | 0.039          | 0.98                             | 1                         | 50,000                     |
| P                                         | 30.97      | 44      | 0.3            | 1.0            | 25                               | 250                       | 500,000                    |
| Pb                                        | 207.19     | 328     | 0.023          | 0.07           | 1.75                             | 35                        | 100,000                    |
| Pd                                        | 106.4      | 1550    | 0.009          | 0.03           | 0.75                             | 0.1                       | 3,300                      |
| Pt                                        | 195.09     | 1769    | 0.0045         | 0.015          | 0.38                             | 200                       | 25,000,000                 |
| Sb                                        | 121.75     | 630.5   | 0.018          | 0.06           | 1.50                             | 3                         | 100,000                    |
| Se                                        | 78.96      | 217     | 0.021          | 0.064          | 1.60                             | 8                         | 250,000                    |
| Sn                                        | 118.69     | 232     | 0.015          | 0.05           | 1.25                             | 1                         | 25,000                     |
| Sr                                        | 87.62      | 769     | 0.002          | 0.006          | 0.15                             | 300                       | 100,000,000                |
| Te                                        | 127.60     | 450     | 0.15           | 0.5            | 12.5                             | 125                       | 500,000                    |
| Ti                                        | 47.90      | 1675    | 0.005          | 0.016          | 0.40                             | 0.1                       | 10,000                     |
| Tl                                        | 204.37     | 304     | 0.044          | 0.133          | 3.32                             | 35                        | 500,000                    |
| V                                         | 50.94      | 1890    | 0.003          | 0.01           | 0.25                             | 2.5                       | 500,000                    |
| Y                                         | 88.91      | 1495    | 0.001          | 0.003          | 0.075                            | 0.1                       | 50,000                     |
| Zn                                        | 65.37      | 419     | 0.022          | 0.066          | 1.65                             | 0.5                       | 10,000                     |
| ZnO                                       | 81.37      | 1970    | 0.027          | 0.082          | 2.05                             | 0.5                       | 10,000                     |

\* Value based on a 25-mL sample volume.

\*\* The minimum sampling volume needed to obtain the OSHA PEL at the LOQ for the element/compound at a sample digestion volume of 25 mL.

\*\*\* The maximum sampling volume for a given sample, calculated by taking 50,000 µg as the limit for the element/compound per sample.

NOTE: The LOD and LOQ values are dependent on the particular analytical instrument used. Also, LOD and LOQ values may vary for a particular element due to certain interelement interferences.

TABLE 2. EXPOSURE LIMITS, CAS #, RTECS

| Element<br>(Symbol) | CAS #      | RTECS     | Exposure Limits, mg/m <sup>3</sup> (Ca = carcinogen) |                                                             |                                                     |
|---------------------|------------|-----------|------------------------------------------------------|-------------------------------------------------------------|-----------------------------------------------------|
|                     |            |           | OSHA                                                 | NIOSH                                                       | ACGIH                                               |
| Silver (Ag)         | 7440-22-4  | VW3500000 | 0.01 (dust, fume, metal)                             | 0.01 (metal, soluble)                                       | 0.1 (metal)<br>0.01 (soluble)                       |
| Aluminum (Al)       | 7429-90-5  | BD0330000 | 15 (total dust)<br>5 (respirable)                    | 10 (total dust)<br>5 (respirable fume)<br>2 (salts, alkyls) | 10 (dust)<br>5 (powders, fume)<br>2 (salts, alkyls) |
| Arsenic (As)        | 7440-38-2  | CG0525000 | varies                                               | C 0.002, Ca                                                 | 0.01, Ca                                            |
| Barium (Ba)         | 7440-39-3  | CQ8370000 | 0.5                                                  | 0.5                                                         | 0.5                                                 |
| Beryllium (Be)      | 7440-41-7  | DS1750000 | 0.002, C 0.005                                       | 0.0005, Ca                                                  | 0.002, Ca                                           |
| Calcium (Ca)        | 7440-70-2  | --        | varies                                               | varies                                                      | varies                                              |
| Cadmium (Cd)        | 7440-43-9  | EU9800000 | 0.005                                                | lowest feasible, Ca                                         | 0.01 (total), Ca<br>0.002 (respir.), Ca             |
| Cobalt (Co)         | 7440-48-4  | GF8750000 | 0.1                                                  | 0.05 (dust, fume)                                           | 0.02 (dust, fume)                                   |
| Chromium (Cr)       | 7440-47-3  | GB4200000 | 0.5                                                  | 0.5                                                         | 0.5                                                 |
| Copper (Cu)         | 7440-50-8  | GL5325000 | 1 (dust, mists)<br>0.1 (fume)                        | 1 (dust)<br>0.1 (fume)                                      | 1 (dust, mists)<br>0.2 (fume)                       |
| Iron (Fe)           | 7439-89-6  | NO4565500 | 10 (dust, fume)                                      | 5 (dust, fume)                                              | 5 (fume)                                            |
| Potassium (K)       | 7440-09-7  | TS6460000 | --                                                   | --                                                          | --                                                  |
| Lanthanum           | 7439-91-0  | --        | --                                                   | --                                                          | --                                                  |
| Lithium (Li)        | 7439-93-2  | --        | --                                                   | --                                                          | --                                                  |
| Magnesium (Mg)      | 7439-95-4  | OM2100000 | 15 (dust) as oxide<br>5 (respirable)                 | 10 (fume) as oxide                                          | 10 (fume) as oxide                                  |
| Manganese (Mn)      | 7439-96-5  | OO9275000 | C 5                                                  | 1; STEL 3                                                   | 5 (dust)<br>1; STEL 3 (fume)                        |
| Molybdenum (Mo)     | 7439-98-7  | QA4680000 | 5 (soluble)<br>15 (total insoluble)                  | 5 (soluble)<br>10 (insoluble)                               | 5 (soluble)<br>10 (insoluble)                       |
| Nickel (Ni)         | 7440-02-0  | QR5950000 | 1                                                    | 0.015, Ca                                                   | 0.1 (soluble)<br>1 (insoluble, metal)               |
| Phosphorus (P)      | 7723-14-0  | TH3500000 | 0.1                                                  | 0.1                                                         | 0.1                                                 |
| Lead (Pb)           | 7439-92-1  | OF7525000 | 0.05                                                 | 0.05                                                        | 0.05                                                |
| Antimony (Sb)       | 7440-36-0  | CC4025000 | 0.5                                                  | 0.5                                                         | 0.5                                                 |
| Selenium (Se)       | 7782-49-2  | VS7700000 | 0.2                                                  | 0.2                                                         | 0.2                                                 |
| Tin (Sn)            | 7440-31-5  | XP7320000 | 2                                                    | 2                                                           | 2                                                   |
| Strontium (Sr)      | 7440-24-6  | --        | --                                                   | --                                                          | --                                                  |
| Tellurium (Te)      | 13494-80-9 | WY2625000 | 0.1                                                  | 0.1                                                         | 0.1                                                 |
| Titanium (Ti)       | 7440-32-6  | XR1700000 | --                                                   | --                                                          | --                                                  |
| Thallium (Tl)       | 7440-28-0  | XG3425000 | 0.1 (skin) (soluble)                                 | 0.1 (skin) (soluble)                                        | 0.1 (skin)                                          |
| Vanadium (V)        | 7440-62-2  | YW2400000 | --                                                   | C 0.05                                                      | --                                                  |
| Tungsten            | 7440-33-7  | --        | 5                                                    | 5<br>10 (STEL)                                              | 5<br>10 (STEL)                                      |
| Yttrium (Y)         | 7440-65-5  | ZG2980000 | 1                                                    | N/A                                                         | 1                                                   |
| Zinc (Zn)           | 7440-66-6  | ZG8600000 | --                                                   | --                                                          | --                                                  |
| Zirconium (Zr)      | 7440-67-7  | ZH7070000 | 5                                                    | 5, STEL 10                                                  | 5, STEL 10                                          |

TABLE 3: VALIDATION SUMMARY

| Analyte                        | Status <sup>1</sup>          | Analyte                        | Status                       | Analyte                        | Status                       |
|--------------------------------|------------------------------|--------------------------------|------------------------------|--------------------------------|------------------------------|
| Ag                             | Not Valid                    | CuO                            | Valid                        | S                              | Not Valid                    |
| Al                             | Valid                        | Fe                             | Valid                        | Sb                             | Partially Valid <sup>4</sup> |
| Al <sub>2</sub> O <sub>3</sub> | Not Valid                    | Fe <sub>2</sub> O <sub>3</sub> | Valid                        | Sb <sub>2</sub> O <sub>3</sub> | Partially Valid <sup>5</sup> |
| As                             | Valid                        | Ga                             | Valid                        | Se                             | Valid                        |
| Au                             | Valid                        | In                             | Valid                        | Si                             | Not Valid                    |
| B                              | Valid                        | KCl                            | Pending                      | Sn                             | Partially Valid <sup>6</sup> |
| Ba                             | Pending                      | Mg                             | Valid                        | SnO                            | Pending                      |
| BaO                            | Pending                      | MgO                            | Valid                        | SnO <sub>2</sub>               | Pending                      |
| BaO <sub>2</sub>               | Pending                      | Mn                             | Valid                        | Sr                             | Valid                        |
| BaCl <sub>2</sub>              | Valid                        | MnO                            | Valid                        | SrCrO <sub>4</sub>             | Valid (by Cr)                |
| BaSO <sub>4</sub>              | Pending                      | Mo                             | Valid                        | Te                             | Valid                        |
| Be                             | Valid                        | NaCl                           | Pending                      | Ti                             | Valid                        |
| Bi                             | Partially Valid <sup>2</sup> | Nd                             | Valid                        | Tl                             | Valid                        |
| Ca                             | Valid                        | Ni                             | Valid                        | V                              | Valid                        |
| CaCO <sub>3</sub>              | Valid                        | P                              | Valid                        | V <sub>2</sub> O <sub>5</sub>  | Valid                        |
| CaO                            | Valid                        | Pb                             | Partially Valid <sup>3</sup> | Y                              | Valid                        |
| Cd                             | Valid                        | PbCrO <sub>4</sub>             | Valid (by Cr)                | Zn                             | Valid                        |
| Co                             | Valid                        | PbO                            | Valid                        | ZnO                            | Valid                        |
| Cr                             | Valid                        | Pd                             | Valid                        | Zr                             | Not Valid                    |
| Cu                             | Valid                        | Pt                             | Valid                        | ZrO                            | Not Valid                    |

<sup>1</sup> Status definitions

Valid: The method is suitable for samples up to at least 0.0500 g bulk material with recoveries of between 90 and 110 percent. This weight exceeds most expected levels encountered in work environments.

Partially Valid: The method is suitable with bulk-material recoveries of between 90 and 110 percent under certain conditions (as footnoted above).

Not Valid: The method procedure is not suitable for samples at any weight with recoveries of between 90 and 110 percent. An alternative method should be used.

<sup>2</sup> Valid up to 10,000 µg/sample and within 7 days of sample digestion.

<sup>3</sup> Valid up to 50,000 µg/sample and at least 24 hours after sample digestion; Valid up to 15,000 µg/sample within 24 hours of sample digestion.

<sup>4</sup> Valid up to 25,000 µg/sample and within 7 days of sample digestion.

<sup>5</sup> Valid up to 25,000 µg/sample and within 7 days of sample digestion.

<sup>6</sup> Valid up to 30,000 µg/sample and within 7 days of sample digestion.

NOTE: The upper limits of the method can be extended by serial dilution of the samples at the time of analyses.